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## INTRODUCTION

These proceedings of a Workshop held in Braga (Portugal) on 5-7 November 2008 are the second public record of the scientific activity developed within COST Action IE0601 “Wood Science for Conservation of Cultural Heritage (WoodCultHer)”.

COST Action IE0601 started formally with a kick-off Management Committee (MC) meeting of held in Brussels on 17-18 April 2007, devoted to administrative and organizational issues. It was closely followed by a second MC meeting of the Action in Tervuren (Belgium) on 8-9 June 2007, together with a Workshop which allowed for an initial approach to the many themes potentially covered by the Action. Every following MC meeting of COST Action IE0601 has been organised together with an international conference:

- 3<sup>rd</sup> MC meeting and 1<sup>st</sup> conference in Florence (Italy), 8-10 November 2007
- 4<sup>th</sup> MC meeting and 2<sup>nd</sup> conference in Braga (Portugal) on 5-7 November 2008
- 5<sup>th</sup> MC meeting and 3<sup>rd</sup> conference in Hamburg (Germany) on 5-7 November 2009
- 6<sup>th</sup> MC meeting and 4<sup>th</sup> conference in Izmir (Turkey) on 20-23 October 2010

A final MC meeting and conference is planned around the end of the Action, that was granted a 6-months extension until xxx.

In addition to these annual conferences, a number of “focused meetings” (FM) or “training schools” (TS) have been organised by MC delegates, and many participants have already benefited from “short term scientific missions” (STSM). Following the example of Tervuren workshop, for all these and other types of formal and informal contacts, information is made available on the web site of COST Action IE0601 ([www.woodculther.org](http://www.woodculther.org)), including, reports, minutes of meetings, draft and final papers by the authors or oral presentations and posters.

COST Actions aim at promoting a field of research through the stimulation of networking activities. They are mostly managed by volunteering scientists and have a limited life span, usually 4 to 4.5 years. The continuation of the related activities after the completion of the period relies of the will of the scientists involved. This motivated in 2001, the creation of the European Society for Wood Mechanics (ESWM) by Pierre Morlier, who led two successive COST Action on wood mechanics between 1990 ad 2000. The aim of ESWM was to ensure the continuation of networking activities in the field of wood mechanics, especially the regular organisation of European conferences addressing the topics of interest to this community. After three ESWM conferences were organized in Lausanne (CH) 19-21/4/2001, Stockholm (SE) 25-28/5/2003 and Vila Real (PT) 5-8/9/2004 it was decided that the coming events would be, whenever possible, held jointly with meetings of running COST Actions, in order to benefit from the financial support of the COST system while creating new opportunities of scientific interaction, as well as avoid the risk of dispersion arising from the increasing number of meeting opportunities in the field. This principle had already been tried in Villa Real, with the organisation of a working group (WG) meeting of COST Action E35 “Fracture mechanics and micromechanics of wood and wood composites with regard to wood machining” in the continuity of the ESWM meeting. Going one step further, the International conference on integrated approach to wood structure, behaviour and application, Florence (IT) 15-17/5/2006 was explicitly a joint meeting of ESWM and COST Action E35. This was made easy by the fact that two WGs of E35 clearly addressed ESWM issues.

This meeting in Braga was the occasion to try a new idea, the use of a joint meeting to stimulate the interest of wood mechanicians to a field of application which had never been explicitly addressed during preceding ESWM meetings. Regarding conservation issues, wood mechanics is a part of wood science that has much to contribute. From the three aspects mentionned in the call for porposal (dimensional response to environment and ageing; strength of aged load-bearing structures; biodeterioration), the first two addressed explicitly wood mechanics issues. As a matter of fact, the challenge of attracting newcomers was not that clearly fulfilled within the meeting itself, however the meeting format was certainly useful to highlight and advertize important research needs within ESWM community.

### A summary report from the Workshop

The Braga conference was organized locally by Prof. Manuel F. Costa, member of the MC of COST Action IE0601, Professor of Physics at the nearby University of Minho. He was supported by the SC of the Action, especially his Chair, Luca Uzielli. It took place in Museu D. Diogo de Sousa, Braga (PT), at a walking distance from the hotels where the participants were lodged. All meals were taken in the cafeteria of the Museum, so that all the available time could be used by participants also for individual discussions, visits to the posters, etc. and no time was wasted for transfers to/from the meeting place.

In total 83 Participants attended, coming from 29 Countries (AT, BE, CA, CH, CZ, DE, DZ, EE, ES, FI, FR, GR, HU, IR, IT, JP, LV, MK, MT, NL, NO, NZ, PL, PT, RO, SE, SI, TR, UK).

Several of the Participants (two invited Speakers, others offering Oral or poster presentations) came from Countries being not COST members (Algeria, Canada, Iran, Japan, New Zealand). Some of them were from Institutions who had applied for participation to the Action as non-COST Countries. Of the Participants, 36 were MC Members, or substituting for MC Members. In total 61 scientists benefited from reimbursement of travel expenses by COST.

Annex 1 includes a detailed Program, and the schedule of the presentations (Orals and Posters).

The Scientific Committee of the conference was composed by the steering committee of the COST Action who met together with the local organiser in Paris, 1-2 September 2008, to select and allocate the papers on the basis of extended abstracts submitted, and prepare the program. The submitted papers were allocated to 5 groups in relation with the 3 themes announced in the call for papers:

- (1) Dimensional Response to environment and ageing (theme 1)
- (2) Strength of Aged Load Bearing Structures (theme 2)
- (3) Biodeterioration (theme 3)
- (4) Other subject in the scope of IE0601
- (5) Out of the scope of IE0601 but of general wood science interest

The conference was organized into plenary Keynotes, 3 parallel sessions corresponding to each of the 3 themes, a poster session, parallel WG meetings followed by a final discussion. Regular orals were presented and discussed during the parallel sessions, as well as related poster briefly presented as short orals. The allowed time slot, including discussion, was 45' for a Keynote, 30' for an Oral and 5' for a Short Oral. Papers from groups (4) and (5) were presented as hanging poster only.

In total:

- 6 Keynote lectures were presented (2 each Conference day)
- 27 Orals were planned and presented (except one cancelled at last moment), i.e. 9 Orals x 3 Sessions
- 21 Posters were planned and 18 were presented as OPS, i.e. 6 posters x 3 Sessions
- 6 Posters assigned to group (4) and 3 Posters assigned to group (5) were hanged and presented during the Poster session.

Posters were on display during the whole meeting time, in the coffee-break room and in the nearby corridor, so that Participants could look at them and discuss them with the Authors during any free moment.

A Management Committee (MC) meeting of COST Action IE0601 took place, divided in two parts: on the 5th (We) from 17:45 to 18:30 and on the 7th from 14:30 to 16:00.

During and at the end of the Conference, Participants had the occasion for visiting the Museum, which is mainly dedicated to the Roman archaeology of the *Bracara Augusta* area, and is of high cultural and scientific interest (<http://mdds.imc-ip.pt>). At the end of the Conference, after Part 2 of the MC Meeting (Friday, November 7, 2008, 16:30-19:00) a visit had been organized to the Mosteiro S. Martinho de Tibães ([www.mosteirodetibaes.org](http://www.mosteirodetibaes.org)), a splendid Monastery founded in 11<sup>th</sup> century, that in 16<sup>th</sup> century became the mother house of the Portuguese monastic complexes, as well as an excellence place of Portuguese thought and art, and was presently under restoration. Among other extraordinary architectural, artistic and historical features, visitors were allowed to access the rear part and the

structure of the huge baroque wooden altar, an aesthetic and scientific occasion not easy to be forgotten.

#### Main objectives and results

The main objectives of the Conference can be summarized as follows.

- a) To present a “state of the art” about selected subjects dealt with by the Action:
- b) To improve mutual knowledge and acquaintance between MC Members, and WG members.
- c) To present current ongoing research, and inform specialists of different fields, all working towards conservation of WCHOs (Wooden Objects belonging to the Cultural Heritage), about the work of their Colleagues and the scientific work being conducted in different Institutions.
- d) To provide an occasion of discussing interdisciplinary subjects, which are very important for study and conservation of WCHOs.
- e) To identify the main subjects to be carried out in the following activities.

All such objectives have been accomplished at quite satisfactory level, by means of (i) the already mentioned Parallel sessions, (ii) the Keynote Lectures, (iii) the formal and informal discussions carried out during sessions and during free time, (iv) the discussions during WG meetings, and (v) the general discussions session.

Especially meaningful was considered the presence of:

- Researchers from RISH (Research Institute for Sustainable Humanosphere) Kyoto University (Japan), and from Scion (New Zealand Forest Research Institute), Institutions from non-COST Countries, since our MC has voted in favor of their application to participate in our Action.
- Prof. Vasco Fassina, Chairman of CEN Technical Committee 346 “Conservation of Cultural Property”, Invited Speaker giving a Keynote Lecture on the activities of such Committee after a four year period, representing the interest of our Action to cooperate in the field of technical Standardization in the field of Cultural Heritage.

#### Allocating and refereeing the Papers

For this Workshop, the Steering Committee (see Annex 3) fulfilled the role of Scientific Committee, preparing and managing its scientific aspects, and organising the refereeing process of the submitted papers, all of which have been peer-reviewed for this publication.

Emphasis should be given to the fact that all accepted paper were deemed of high scientific calibre and deserved the same “dignity”, no matter whether they were allocated as Oral presentations or Posters, such allocation being purely dictated by unescapable time constraints.

Unfortunately some Authors were not able to submit their paper for publication, or did not return a corrected version as suggested by the referees; the titles and the abstracts of their papers – when available – have been reported, for the sake of completeness. Others, not being of English mother tongue, submitted papers with some language inaccuracies; as long as such inaccuracies did not affect the comprehensibility of the text, the SC decided not to ask for a very strict language editing.

Two authors were not able to attend the conference at the last minute. Their papers, however, were reviewed like the others and included in these proceedings.

For the purpose of clarity, the classification of the papers was made according to the content of the papers and does not follow strictly the organisation of the conference. They were grouped into 6 parts:

- (A) Material properties
- (B) Biodeterioration
- (C) Characterization and measurement techniques
- (D) Conservation
- (E) Structures
- (F) Others

Within each part, Keynote papers were placed first, followed by other papers in alphabetical order of the 1<sup>st</sup> Author.

Acknowledgements

As the Editor of these Proceedings, I wish to deeply and sincerely thank:

- each and all the members of Steering Committee, who accepted to undertake their heavy task – and especially Luca Uzielli, Chairman of the Action, who did as usual most of the administrative work
- the anonymous referees who revised the papers, and provided effective and beneficial comments and suggestions
- the Authors of the papers, who shared their knowledge and their work, aimed at improving conservation (in Europe and worldwide) of Wooden Cultural Heritage Objects.
- the friends and colleagues who were instrumental in making possible and organizing the Workshop, including the COST Office personnel, Piotr Swiatek (at that time Science Officer, later replaced by Caroline Whelan) and Milena Stoyanova (Administrative Officer), The Action's Rapporteur Zsolt Kajcsos, the members of the Organizing Committee cited above.

*Joseph GRIL*

*Vice-chair of COST Action IE0601 and president of the European Society for Wood Mechanics*

## (A) MATERIAL PROPERTIES



# SORPTION OF MOISTURE AND DIMENSIONAL CHANGE OF WOOD SPECIES USED IN HISTORIC OBJECTS

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## Abstract

'General' moisture sorption and swelling/shrinkage patterns which would apply as a first approximation to any wood species constituting cultural objects were proposed. They were obtained from the experimental data measured for 21 historically important wood species used in the past for panel paintings and woodcarving. Information on further wood species of interest to the wood conservation community can be added to the database and used to constantly improve the general relationships.

## 1. Introduction

*Uncontrolled* variations of ambient temperature and relative humidity have been the principal agents of deterioration for historic and artistic wooden objects. The variations in the environment induce physical damage to the wood due to its hygroscopic nature and dimensional response to the moisture sorption or desorption.

Over the centuries, a great number of wood species have been used for constructional, utilitarian and decorative purposes with each geographic region, historic period and application field favouring specific woods for reasons of availability, ease of processing or fashion. Therefore museum collections or interior furnishings of historic buildings are mixture of various wood species having different responses to climatic variations. Wood variability within species and due to aging adds an additional dimension to the complexity of the problem. As a result, acquiring information on moisture sorption and related dimensional response by wood species present in a particular collection or building can be rarely possible due to high cost and long time necessary for the laboratory work, as well as a difficulty in withdrawal of samples from cultural objects. By necessity, the assessment of wood damage risk related to the fluctuating climate should be rather based on some general climate – wood relationships which for most practical purposes could be applied to mixed wood collections or furnishings.

Therefore, the main aim of this study was to evaluate water vapour sorption and dimensional response for 21 historically important wood species used in the past for panel paintings and woodcarving and to propose a 'general' moisture sorption and swelling/shrinkage patterns which would apply as a first approximation to any wood species constituting cultural objects, as well as ranges of moisture uptake and dimensional response behaviour which can be expected.

## 2. Materials and methods

Wood species selected for this study are listed in Table 1. The selection was based on the list of types of woods used in the past for panel paintings and woodcarving published by Grosser and Geier [1] for softwoods, and by Grosser and Grässle [2] for hardwoods. The specimens were obtained from defect-free wooden boards seasoned for at least 3 years at room conditions at the restoration workshop of the National Museum in Krakow, Poland.

Moisture adsorption and desorption isotherms were determined at 20°C and for a full range of water vapour relative pressures. The measurements were done gravimetrically with the use of a Sartorius vacuum microbalance. Typically, a 0.05 g piece of wood was weighed and outgassed prior to a measurement under a vacuum of a residual pressure less than 10<sup>-3</sup> mbar. The aim was to move air out of the wood and to eliminate most of the species physisorbed during storage of the sample, especially water. Vacuum was maintained until a constant weight was obtained, then subsequent portions of

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water vapour were introduced, and the respective mass increases due to the sorption and the respective equilibrium pressures were recorded. Samples were considered to have reached equilibrium when the weight changes were less than 0.05% in 40 minutes. The equilibrium moisture contents (EMC) were calculated on the basis of the initial weight of the out-gassed sample.

The dimensional change of wood accompanying water vapour sorption was measured in the radial and tangential directions using two inductive transducers of accuracy 2  $\mu\text{m}$ . The dimensions of wood specimens coincided with the principal anatomical directions in wood – radial 2 cm x tangential 2 cm x longitudinal 0.5 cm. The measurements were taken in a specially built specimen holder placed in a vacuum vessel. The vessel was connected to the same outgassing and water vapour dosing system which was used to determine water vapour sorption isotherms; in fact the two measurements were taken simultaneously. Both adsorption and desorption branches of the sorption and dimensional change isotherms were recorded. The process was fully automated and rapid - measuring of 10 adsorption and 10 desorption points took on average 15 hours.

After the measurements of dimensional changes induced by water vapour sorption and desorption, each wood specimen was immersed in distilled water until full saturation. At the saturation state, its dimensions were measured with a micrometer.

### 3. Sorption isotherms

The sorption of water on wood can be described by sigmoid shape of the type II isotherm in the IUPAC 1985 classification [3]. It indicates the monolayer-multilayer physisorption in which an adsorbed surface layer progressively thickens as the vapour pressure is increased up to the saturation pressure, close to which the adsorbed layer becomes a bulk liquid. The three-parameter Guggenheim-Andersen-de Boer sorption equation [4] is used to interpret the sorption data for wood by expressing the equilibrium moisture content as a function of water vapour activity:

$$v(a_0) = \frac{v_m c k a_0}{(1 - k a_0)(1 + (c - 1)k a_0)} \quad (1)$$

where  $a_0$  is water vapour activity taken equal to  $p/p_0$  where  $p$  is the experimental partial water vapour pressure and  $p_0$  its value at saturation (thus if RH is the relative humidity expressed as a percentage  $a_0 = \text{RH}/100$ ),  $v$  the amount water vapour sorbed by a gram of wood at water vapour activity  $a_0$ ,  $v_m$  the monolayer capacity in the same units as  $v$ ,  $c$  - energy constant related to the difference of free enthalpy of water molecules in the liquid state and in the monolayer, and  $k$  - the third parameter, characterizing the state of the sorbed molecules beyond the first layer. The usefulness of the GAB model in the wood science has been demonstrated [5].

In the present study, the GAB constants were determined by a least-squares regression of the GAB relation for  $a_0 < 0.85$ , identified as the applicability interval. The regression was performed separately for the adsorption and desorption branches. By way of example, the experimental data for water vapour sorption by the lime wood are compared in Fig. 1 with curves calculated using the GAB equation. RH scale is used on the ordinate axis of the plot. The GAB constants for adsorption and desorption for the lime wood, as well as 20 other wood species investigated, are given in Table 1.

As one can see in Fig. 1, the applicability range for the GAB equation for the adsorption branch is  $0.05 < a_0 < 0.85$  and an upswing of the adsorption points above the upper limit of this range is observed. The shape of the isotherm has been interpreted in terms of 3 sorption stages occurring as the water activity increases: the direct adsorption of water molecules on the sorbent within a monolayer, the formation of the multilayer film of water molecules beyond the first layer, and the formation of the liquid-like water interacting insignificantly with the solid due to the increased distance.

For the desorption branch, the hysteresis effect is observed i.e. higher moisture content after desorption when compared to that after adsorption at any given RH value. The applicability range for the GAB equation is wider and covers the highest RH range close to 100%. It can be seen that the monolayer value obtained for desorption is higher than the adsorption value (7 compared to 5 grams of water per 100 g of wood). Further research must elucidate if the effect can be explained by a real increase of the internal surfaces with more adsorption sites made available in the material or a delayed removal of water entrapped in the smallest capillaries and channels in wood on desorption.



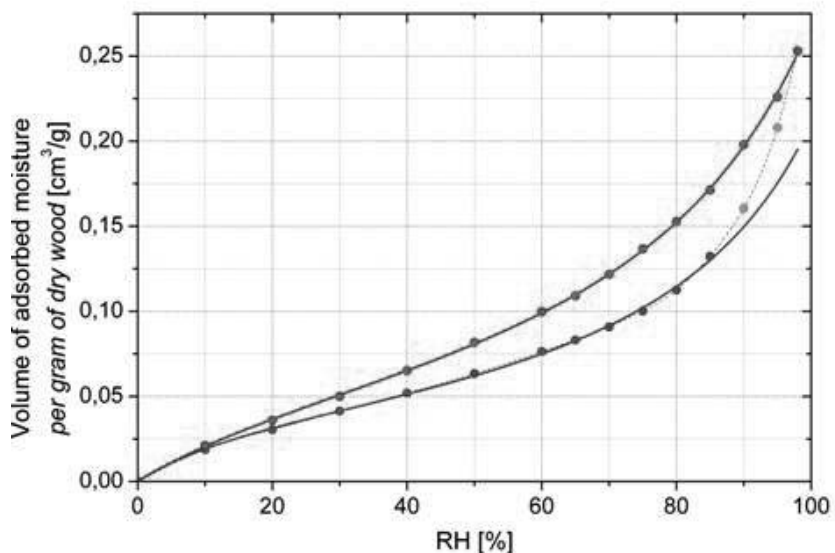


Fig. 1 – Adsorption and desorption isotherms of water vapour for lime wood at 20°C: solid line shows prediction of the GAB equation for adsorption (green, down) and desorption (red, up) branches.

The ‘general’ or ‘average’ water vapour sorption isotherm was calculated by fitting the GAB equation to the adsorption and desorption data for all 21 wood species and is shown in Fig. 2. The data for the least sorbing specimen (dark fossil oak wood) and the most sorbing one (mahogany) are given for comparison. The average values of the GAB constants calculated from the adsorption data were:  $v_m = 6.83$  g H<sub>2</sub>O per 100 g of wood (range: 5.65 – 9.13),  $c = 8.61$  (range: 3.98 – 14.16),  $k = 0.66$  (range: 0.45 – 0.74).

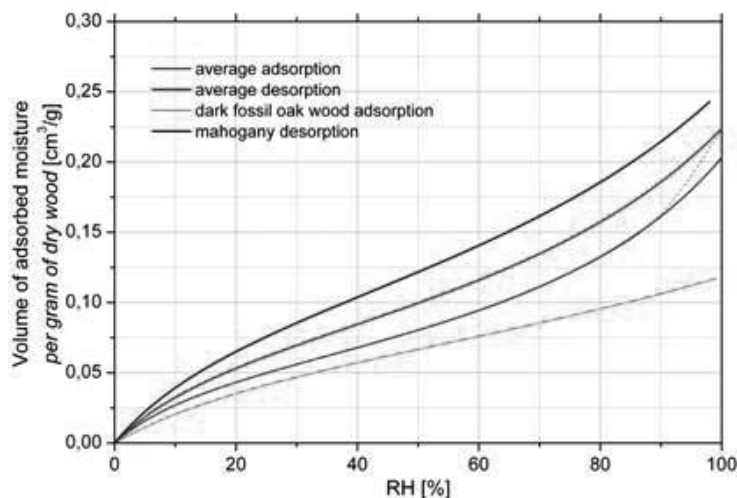


Fig. 2 – The average adsorption and desorption isotherms of water vapour at 20°C: the adsorption data for the least sorbing (dark fossil oak wood, lowest curve) and the desorption data for the most sorbing (mahogany, upper curve) wood species are shown for comparison.

Table 1: GAB constants for water vapour adsorption and desorption isotherms for 21 species of wood as well as their coefficients of tangential and radial dimensional change

Species	Adsorption/desorption			Dimensional change coefficients	
	$v_m \cdot 10^{-2}$	c	k	tangential	radial
Softwoods					
pine ( <i>Pinus</i> spp.)	7.25/10.07	13.5/8.21	0.49/0.44	0.38	0.13
Japanese cypress ( <i>Chamaecyparis obtusa</i> )	7.58/10.19	4.38/7.41	0.63/0.53	0.23	0.12
spruce ( <i>Picea</i> spp.)	6.82/7.96	10.04/12.49	0.74/0.70	0.34	0.14
larch ( <i>Larix decidua</i> )	7.07/7.33	9.89/9.05	0.74/0.72	0.45	0.13
fir ( <i>Abies</i> spp.)	7.40/11.35	8.90/7.55	0.66/0.50	0.32	0.12
Hardwoods					
sycamore ( <i>Acer pseudoplatanus</i> )	6.67/12.49	9.01/7.01	0.58/0.30	0.38	0.19
oak ( <i>Quercus</i> spp.)	6.19/11.1	7.36/7.26	0.72/0.51	0.33	0.19
dark fossil oak wood	7.57/11.13	7.36/12.50	0.45/0.24	0.62	0.39
rosewood ( <i>Dalbergia</i> spp.)	6.11/9.84	9.31/7.38	0.66/0.53		0.33
walnut ( <i>Juglans</i> spp.)	5.88/10.41	7.35/4.36	0.69/0.56	0.21	0.14
cherry ( <i>Prunus avium</i> )	5.91/8.64	9.89/7.01	0.72/0.66	0.25	0.14
lime ( <i>Tilia</i> spp.)	4.51/7.69	7.42/4.70	0.79/0.66	0.30	0.19
sour cherry ( <i>Prunus cerasus</i> )	6.80/8.15	8.74/9.83	0.69/0.71	0.29	0.16
elm ( <i>Ulmus procera</i> )	5.65/9.07	14.16/7.03	0.74/0.66	0.31	0.17
poplar ( <i>Populus</i> spp.)	5.95/8.13	9.58/7.88	0.73/0.68	0.27	0.12
mahogany ( <i>Swietenia mahagoni</i> )	7.80/11.18	10.00/7.86	0.65/0.59	0.28	0.11
beech ( <i>Fagus sylvatica</i> )	6.98/11.92	7.11/4.88	0.70/0.53	0.36	0.17
birch ( <i>Betula pendula</i> )	6.67/10.60	7.85/5.64	0.71/0.57	0.30	0.21
willow ( <i>Salix</i> spp.)	6.59/9.07	8.03/7.03	0.73/0.66	0.20	0.07
apple ( <i>Malus domestica</i> )	6.83/14.95	7.70/5.15	0.73/0.64	0.35	0.20
ash ( <i>Fraxinus excelsior</i> L)	6.63/6.54	8.92/8.32	0.68/0.20	0.29	0.21
Average	6.83/9.36	8.61/7.59	0.66/0.57		

#### 4. Dimensional change isotherms

The amount wood specimens swell or shrink was measured as relative dimensional change versus RH. The relationships were described by fitting the polynomial of degree 3 to the adsorption (swelling) and desorption (shrinkage) data. As for the sorption isotherms, the ‘average’ swelling and shrinking isotherms were calculated by fitting the data for all 21 wood species and they are shown for the tangential and radial directions in Fig. 3. The constant coefficients of the polynomial  $a_0$  to  $a_3$ , for the swelling and shrinkage, are listed in Table 2.

Table 2: The constant coefficients of the polynomial predicting the average swelling and shrinkage isotherms.

	$a_0$	$a_1$	$a_2$	$a_3$
swelling tangential	-0.0088	4.8412	-5.1062	6.0800
swelling radial	-0.0031	3.0628	-3.1238	3.2034
shrinkage tangential	0.0131	5.2690	0.6517	-0.4018
shrinkage radial	0.0051	3.5639	-0.6023	-0.0461

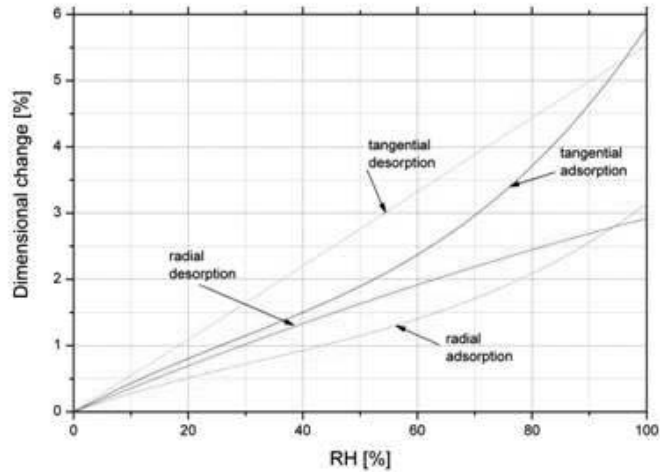


Fig. 3 – The average dimensional change isotherms for the adsorption and desorption of water vapour at 20°C.

A convenient way of expressing swelling and shrinkage behaviour of individual wood species is to plot dimensional change as a function of EMC. By way of example, such relationship is shown in Fig.4 for the tangential and radial directions in the lime wood.

All the experimental points for the EMC values between 0 and 25% were obtained by adsorption and desorption of water vapour at varying RH whereas the last point corresponds to a wooden specimen fully saturated under distilled water. Generally, the measurements did not show any differences between the dimensional change values at the same EMC but obtained after adsorption or desorption. In this way, the so-called secondary effects of moisture sorption on dimensional changes [6] proved insignificant at least for any practical purposes.

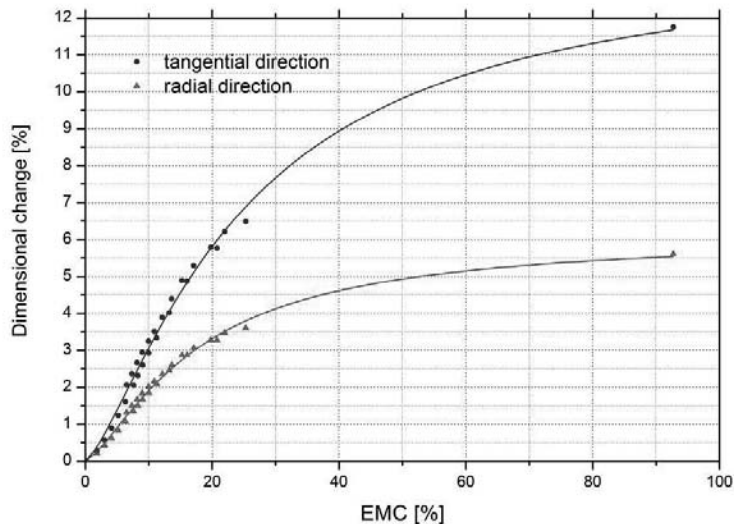


Fig. 4 – Dimensional change of lime wood in the radial and tangential directions plotted as a function of the equilibrium moisture content.

The three parameter sigmoid function was used to interpret the individual swelling and shrinkage data:

$$d(EMC) = d_{\max} \frac{EMC^n}{k^n + EMC^n} \quad (2)$$

where EMC is the equilibrium amount of water vapour sorbed,  $d$  the relative dimensional change at a given EMC and  $d_{\max}$  reflects the maximum dimensional change for wood saturated with water.

The function shows an early exponential growth at low EMC, then slows to a linear growth for the broad medium part between approximately 5-15%, and then approaches a constant value as the capillary system of wood is saturated with water. The dimensional change coefficients were determined by the linear fit across the linear range of the plots and are given in Table 1.

## 5. Conclusions

In addition to individual sorptive characteristics and dimensional responses of wood species, constituting cultural heritage objects, ‘general’ moisture sorption and swelling/shrinkage patterns which would apply as a first approximation to any wooden furnishings and collections were proposed. They were obtained from the experimental data measured for 21 historically important wood species used in the past for panel paintings and woodcarving. Information on further wood species of interest to the wood conservation community can be added to the database and used to constantly improve the general relationships.

## Acknowledgements

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# VIBRATIONAL PROPERTIES OF TROPICAL WOODS WITH HISTORICAL USES IN MUSICAL INSTRUMENTS

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## Abstract

This paper presents a collection of wood species with important uses in musical instruments, in reference to historical and geographical cultural specificities, with ranges of viscoelastic vibrational properties by species. Data combine our experimental characterizations and extensive literature review, gathered in a specific relational database. An overview of vibrational properties' distribution on c.400 species is introduced. Two case studies of wood choices for a given function in different epochs or regions are presented: woods for European historical bows, and woods for idiophone bodies in different continents. Trends are contrasted: very different properties associated to historical changes in the first case; some common features over different regions in the second one.

## 1. Introduction

Present-day musical instruments are frequently associated to a few « emblematic » wood species for given functions, and these chosen materials participate to the specificities of instruments. Amongst the now archetypal species for Western musical instruments, several are tropical hardwoods, which uses were adopted at different epochs in the last centuries. Well-known examples are Pernambuco (*Caesalpinia echinata*) which started to be used at the end of the XVIIIth century and then became the first choice for violin bows; or Rosewoods (*Dalbergia nigra*, then *D. latifolia*) which became standards for guitar bodies starting from the 19<sup>th</sup> century. Comparison with other wood species reveals some specific combinations of physical-mechanical properties, especially concerning vibrational properties: the above species are distinguished [10] [14] by atypical values of damping coefficient, a property which is recognized as a key factor for several parts of instruments. Thus, the adoption in the past of “new” wood resource participated to the development of designs and techniques, and to the cultural identity of the resulting instruments. As such, documentation and mechanical characterization of woods employed at different times are important both for the critical study, and for the conservation, restoration or re-construction of these objects of musical heritage.

Of course, the introduction throughout history of “new” or exotic woods in the instrument making culture goes far beyond the two examples introduced above, and many particular species were introduced in this way at different times and for different instruments. A good example of it is the co-evolution in shape/structure and in wood choice for bows, from convex to concave profiles, with different sections, and different chosen species or groups of species.

Another branch of cultural heritage concerns the collections of extra-European instruments, though there is usually quite little historical documentation in this case. Wood choice and its more or less strong permanence represent also in this case an important aspect of the specificity of a given heritage (considering both tangible and intangible heritages).

This paper aims at exploring the diversity of wood vibrational properties, with special reference to tropical woods, in relation with historical or geographical specificities in instruments.

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## 2. Methodology

### 2.1. Experimental characterizations

#### Material

Two main samplings of woods were studied. On one hand, woods provided by skilled instrument makers, preferentially off-cuts from pieces from which instruments had been effectively built, and when possible with appreciations. In this paper we will separate the sampling of woods used for bows of early-music quartet and fiddles. This work was run in collaboration with the specialized bow maker Nelly Poidevin (based in Bretagne, France). On the other hand, a wider sampling of many different species, with about 70% tropical ones, was collected, mainly from the well-identified wood stocks from CIRAD (in Montpellier, France). The “bows” sampling covered a total of 250 test specimens belonging to 10 species: 5 temperate ones believed to have been used until Renaissance era and 5 tropical woods used from Baroque –including Snakewood *Brosimum guianense*- to modern eras – including Pernambuco *Caesalpinia echinata*. The “general” sampling contained a total of c.1500 test specimens prepared from 77 species (including 70% tropical hardwoods, 15% temperate hardwoods and 15% softwoods). All studied woods had been stored for several years in ambient conditions before testing.

#### Methods

Specimens (12×2×150mm, R×T×L) were first dried (in order to reach equilibrium in adsorption) for 48h at 60°C. All measurements were performed after at least 3 weeks stabilization in controlled conditions of 20±1°C and 65±2%RH. Specific gravity and equilibrium moisture content were recorded. Vibrational measurements were made by non-contact forced vibrations of free-free bars (e.g. [12]). Specimens were made to vibrate through a tiny iron piece (weight 15-20 mg) glued on one end, facing an electric magnet. Their displacement was measured using a laser triangulation displacement sensor. Vibration emission and detection were computer-driven using a National Instruments card and a new semi-automated interface that we specifically developed [2] using Labview® Software.  $E'/\rho$  was deduced from the first resonant frequency according to the Euler-Bernoulli equation. Damping or loss coefficient –expressed as  $\tan\delta$ - was measured both through the ‘quality factor’ Q (bandwidth at half-power; frequency domain) and through logarithmic decrement  $\lambda$  of amplitude after stopping the excitation (time domain). These two methods of determination shall be equivalent. Measurement frequencies were in the range of 200-600 Hz. 3 repetitions were made for each probe and mean error on properties was  $\leq 5\%$ .

### 2.2. Literature review and relational database

Data obtained through our above-cited experimental characterizations were much extended by an extensive literature review on wood viscoelastic (i.e. data including damping coefficients) vibrational properties. Data were collected from 30 sources, including some hard-to-obtain ones. Great care has been taken about checking the compatibility of all collected values, especially considering the hygrothermic and frequency conditions of measurements. The data compiled were obtained on woods stabilized in controlled conditions of 20-25°C, at c.65% RH (55-70% RH were accepted but specified) and in the frequency range of 200-1500Hz (data at higher frequencies are listed separately). Basic set of properties includes specific gravity  $\rho$ , Young’s modulus E, specific dynamic Young’s modulus  $E'/\rho$  and damping coefficient  $\tan\delta$ , along the grain (some information on anisotropy ratios were also collected). This “wood vibrational properties” collection contains data for 395 woody species (corresponding to the results of nearly 6000 tests), including 224 tropical hardwoods, 102 temperate hardwoods, 61 softwoods, and 8 monocotyledons.

This collection constitutes the “vibrational properties” datatable in a relational database we created on the specific subjects of “Woods and Instruments Diversity” [3]. The other main datatables include: “uses of woods in instruments” and “woody species taxonomy and information”, with additional modules about species conservation and nomenclature (botanical synonyms and common names). All tables are dynamically related together and the whole database includes nearly 700 species, and some information on wood uses in about 160 instruments or the World, also collected from numerous references from several disciplines and languages.

### 3. General results: an overview of the diversity of wood vibrational properties

The general distributions in specific gravity, Young's modulus and vibrational properties  $E'/\rho$  and  $\tan\delta$  over the 395 documented wood species are presented in Fig. 1, separating broad categories: tropical or temperate hardwoods, softwoods, and monocotyledons. The different ranges in specific gravity is a well known fact and confirms that very dense woods ( $\rho$  higher than 1) can barely be found outside of tropical hardwoods. This, combined with a higher proportion of  $E'/\rho$ , leads to the fact that highest values of Young's modulus can only be found in tropical hardwoods. This point could have been determinant in the case of the application to bows. Finally, the distributions in damping coefficient show that softwoods are centered around the mean values on all woods, while temperate hardwoods are mostly associated to higher than average damping values. Tropical hardwoods have a broad distribution of  $\tan\delta$ , but a high number of species have lower than average damping coefficient, and the lowest values can barely be found for other wood categories. This confirms and extends the statistical validity of some previous statements [9] about the difficulty of finding temperate species with low enough damping to be envisaged as substitutes to tropical species used in some instruments.

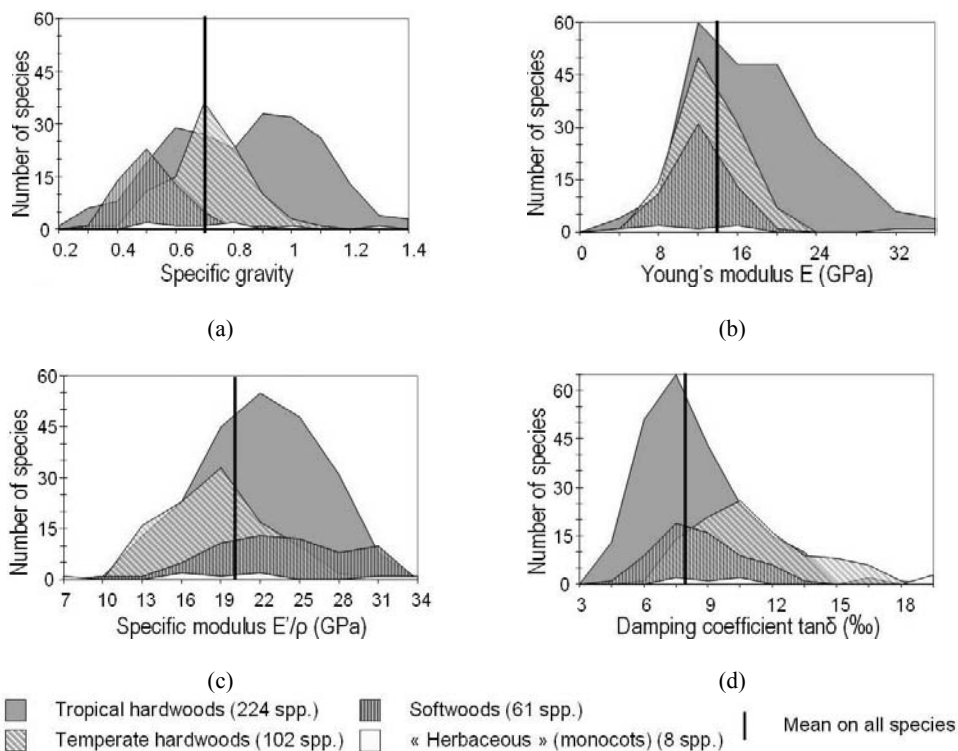


Fig. 1. General distribution of specific gravity (a), Young's modulus (b) and vibrational properties  $E'/\rho$  (c) and  $\tan\delta$  (d) over 395 species.

This specificity of very low  $\tan\delta$  for some tropical hardwoods may also be considered from a more fundamental point of view. Previous works have shown that there was a high correlation between  $\tan\delta$  and  $E'/\rho$  over different species [13], which resulted from the common effects of microfibril angle [12] and/or natural grain angle [4]. This could define a "standard" relation. If we observe (not shown in Fig. 1) the distribution of the deviations of  $\tan\delta$  to this "standard", the vast majority of woods with "abnormally low  $\tan\delta$ " are tropical hardwoods, which may be related to their chemical composition, and especially to high extractives contents as it had been demonstrated for some species [2] [11] [14].

## 4. Two case studies on wood choice at different epochs or regions

### 4.1. Woods for historical bows in Europe

Violin bows are now firmly associated to Pernambuco (*Caesalpinia echinata*) wood (although its recent inscription on annex II of CITES might renew the interest for alternative species). This wood started to be tried for bows in the second half of the 18th century, and later became the exclusive standard for highly prized “classical-modern” models of bows. Characterizations of this wood showed an often high Young’s modulus (although seldom in the extreme range) and an exceptionally low damping [10]. Woods for bows “should” have adequate specific gravity (for balance and playability), Young’s modulus (for stability and holding the tension of hair) and internal damping (in regard to energy dissipation, playability and dynamics). Though, if the “adequate” ranges are associated to those of Pernambuco for classic-modern bows, this may not be so for earlier models (Baroque and Renaissance, and before for Medieval fiddles) which structures are different, and used for different playing modes and musical tastes. Fig. 2 presents the ranges in specific gravity, Young’s modulus and vibrational properties ( $E'/\rho$  and  $\tan\delta$ ) for several species of woods that have been used at different epochs. Historical comments below owe a lot to information provided by N. Poidevin.

The first two observations may be that: i) on the whole studied set of species used for different bows, very wide ranges of properties are recorded; ii) within an ensemble related to different models, ranges are much more focused, and the different ensembles barely overlap. Rather clear wood-structure (and presumably musical trends) associations can be separated.

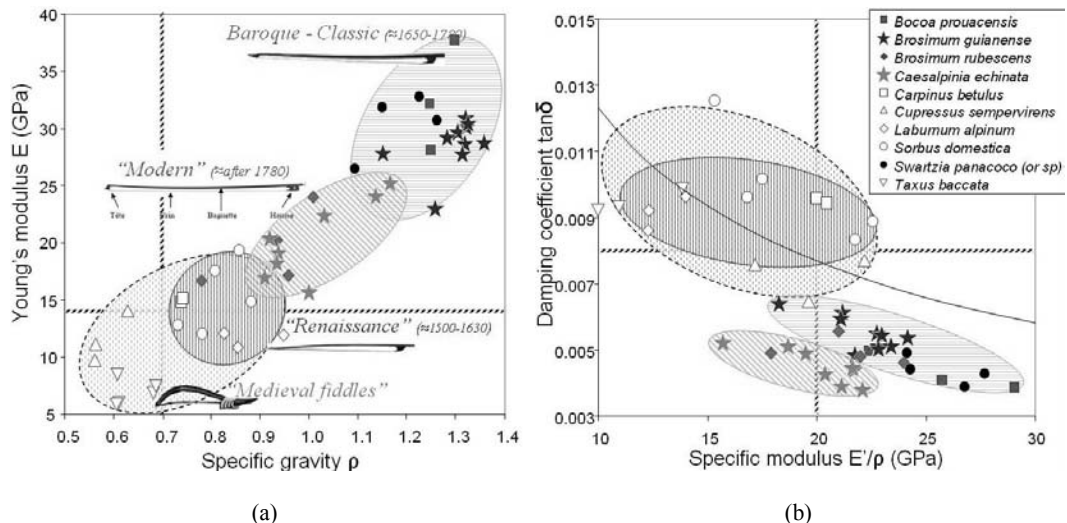


Fig. 2. Specific gravity and Young’s modulus (a) and vibrational properties  $E'/\rho$  and  $\tan\delta$  (b) for woods used in models of bows from different epochs. Authors’ experimental data. One mark = mean value of 2-30 samples for one tree and/or “quality”. Dashed lines indicate mean values on 395 woods, and plain curve in (b) represents the “normal” relationship between  $\tan\delta$  and  $E'/\rho$  [13].

Most hardwoods used from Renaissance are heavier than average and up to extreme values, and have a Young’s modulus between average (temperate hardwoods in Renaissance) and exceptionally high (tropical woods in Baroque bows). The South-American woods (including the most often seen Snakewood) adopted in Baroque era had extremely high density and modulus, usually higher than Pernambuco (it shall be noted that Pernambuco was apparently not used in that epoch, although its trade towards Europe was important since 16th century). The introduction of these woods probably allowed the development of the long and thin bow sticks of Baroque era, associated with new trends in playing techniques and places of performances. Slenderness was counterparted by much reducing the curve to maintain stability. These woods used in Baroque bows also have much lower than average damping, which may have provided more playability and dynamics. For the earlier models of bows, damping may not be a critical parameter, or, alternatively, low damping may not be desirable and



medium-high values best suited to musical styles and performing conditions; this may also have been simply the result of the difficulty in finding low-damping temperate hardwoods.

From the second half of XVIIIth century, “transitional” bows were made, using both “Baroque” woods, and some lighter species such as *Brosimum rubescens* and Pernambuco, which thereafter became the standard for the classical models. These later models have a concave curve, higher head, and were associated to increasingly virtuoso playing technique, and wider performing rooms. The lower (as compared to Baroque woods) density and exceptionally low damping of Pernambuco may have facilitated very dynamic playing techniques and more powerful emission. The often lower Young’s modulus of this wood was dealt with reverse curve in order to keep stiffness and stability.

Finally, it shall be noted that the woods used at different epochs, albeit having very different properties, are adequate to the model there are used for (structure, musical specificity) and material choice seems to involve adaptation rather than maximization. Of course, it is difficult to explore in-depth all facets of this subject in a short article, and a deeper understanding of this subject will ask for mechanical analyses combining structural and material parameters, together with craftsmanship and musicological approaches. Yet, from the “wood” point of view, these results provide an insight into the contribution of chosen material resource (local or imported wood species) to some technical and esthetical developments.

#### 4.2. Woods for vibrating bodies of idiophones through different continents

In the case of “idiophones” (i.e. the primary vibrating part is the main structure of the instrument), studies about “Western” instruments (concert marimbas and xylophones) pointed out that the main woods’ physical parameter related to quality appreciation was a very low damping coefficient [1] [8]. Other parameters were a high density, high impact hardness, medium-high specific modulus [9]. It shall be remembered that this type of instruments is somehow of recent introduction in “Western” cultures: about the end of 19<sup>th</sup> century. Before that, the historical pathways of xylophone-like instruments in tropical regions (Africa, Indonesia, Mesoamerica) have been the subject of several hypotheses, although it seems that the theory of African origin would now prevail. Whatever the historical pathways, we might wonder about the properties of woods traditionally selected for “xylophones” in their several areas of repartition. Another interesting type of wooden idiophone instruments are the slit-drums, which have strong cultural importance in the Pacific regions, and served for long-distance transmission of “spoken” messages in Africa [5] [6]. Fig. 3 presents the ranges in properties of several species used for these idiophones in different continents.

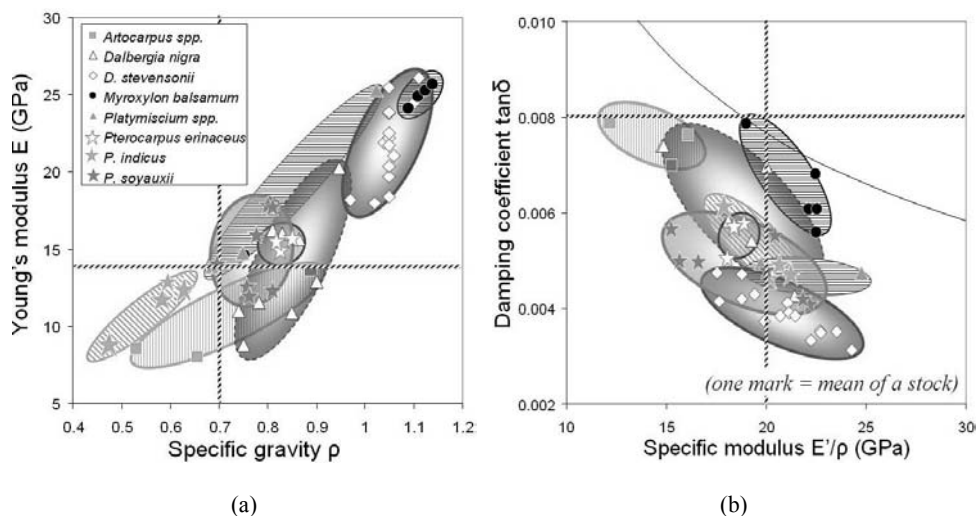


Fig. 3. Basic properties of woods used for vibrating bodies of xylophones and slit-drums through different continents. Authors’ data + literature. Dashed lines and plain curve in (b): see Fig. 2.

The most preferred wood for “Western” concert xylophones is Honduras Rosewood (*Dalbergia stevensonii*), and its damping coefficient is indeed amongst the very lowest not only of the selection above, but on all woods from our collection. There are indications of an early use of Rio Rosewood (*D. nigra*), but apparently its “sound” was considered less bright than Honduran, which is consistent with their respective  $\tan\delta$  values. Interestingly, in Central-America (the bio-geographic range for Honduran Rosewood and several others *Dalbergia spp.*), the preferred woods for the very important traditional marimbas are from an other genus: *Platymiscium spp.* Woods from this genus would deserve more mechanical characterization, but, according to available data, they appear to have also a very low damping, yet sometimes lower density. These species are stated to be “the preferred woods”, while “the most durable and resistant wood is *Myroxylon balsamum*”, but the later has much higher  $\tan\delta$ , so it seems that the traditional choice was firstly based on “acoustic” aspects.

The diversity and cultural importance of “xylophones” and slit-drums in Africa are well-known facts. It has been shown [6] [7] that, the more prominent the purely “acoustic” function of xylophones or slit-drums, the higher the proportion of use of *Pterocarpus spp.* (often *P. soyauxii* in Central Africa), this choice becoming nearly exclusive for the slit-drums for message transmission. While the specific gravity of this species is not very high, its  $\tan\delta$  is amongst the lowest values. In other regions (Mali to Burkina), another species of this genus is the preferred material (*Pterocarpus erinaceus*). Broader characterizations of this wood would be useful, yet it appears that its ranges in properties overlap those of *P. soyauxii*.

*Artocarpus spp.* are frequently used from SE Asia to Melanesia (Papua-New Guinea to Vanuatu), either for xylophone-like instruments (SE Asia) or slit-drums (the monumental slit-drums of Vanuatu and especially the anthropomorphic slit-drums of Ambrym islands are good examples). It seems that in Vanuatu, *Pterocarpus indicus* is considered as giving a “brighter” sound [personal communication, D. Cardon], which would be consistent with its lower value of  $\tan\delta$ .

This short synthesis indicates that: i) relatively high specific gravity is indeed a frequent feature, but far from being systematic; ii) specific Young’s modulus (related to resonance frequencies for given geometries) is not a key factor: changes in geometry can efficiently deal with actual frequencies; iii) all important woods selected for “xylophones” and/or slit drums are characterized by damping coefficients that are not only lower than the mean values for all kind of woods, but also lower than mean values for woods at equivalent specific modulus  $E'/\rho$  [13], whatever the region of the world and local or imported flora. This finding not only supports, but mostly extends the statements of previous authors considering only Western musical instruments [1] [8] [9]. It also suggests that, in the case of these idiophone instruments, the “acoustical” function of the material predominates and leads to rather comparable choices in different continents and cultures.

Another interesting fact is that, apart from the *Artocarpus spp.* (Moraceae), most of the important species presented here belong not only to the same family (Leguminosae-Papilionoideae), but even to the same tribe (Dalbergioideae). Given that, for some *Dalbergia spp.* [14] and *Pterocarpus spp.* [2] [4], extractives were found to be responsible of their exceptionally low damping, we might suspect that there may be some kind of connection between chemotaxonomy, and the empirical selection of these materials for “acoustic” functions throughout different regions of the world.

## 5. Conclusion

An attempt has been made into clarifying some relations between diversity of wood vibrational properties, and cultural specificities of wood uses in musical instruments. The distribution of properties on 395 species confirmed that very high specific gravity and Young’s modulus can only be found in tropical hardwoods, and gave some statistical evidence that very low damping is also associated to this category of woods. Two cases studies of wood choice for a same function but different historical or geographical cultures were introduced. Woods used for historical models of European bows are rather homogeneous in properties for a given model, and very different between different models. The introduction of imported South-American woods allowed structural –and probably musical- developments. On the contrary, woods traditionally selected for xylophones and slit drums in different continents share some common features, mainly a very low damping coefficient. Several civilizations carefully selected appropriate species from a very diverse local flora. Of course,

further interdisciplinary (mechanics, acoustics, wood sciences, musicology, organology, ethnobotany) analyses would be necessary to fully grasp this wide subject. Yet, collected data and documentation may be useful both in the context of documentation for conservation-restoration, and for an approach of the history and ethnology of techniques in the specific domain of wooden musical instruments.

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# CREEP PROPERTIES OF HEAT TREATED WOOD IN RADIAL DIRECTION

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## **Abstract.**

This paper discuss about ageing-like process of heating wood in completely dry conditions and the effects on creep properties for poplar wood (*Populus alba*). Creep tests have been performed in the radial direction, for heat treated and untreated samples over 10 hours. Beforehand some samples were heat treated at 150°C during a few days. Differences in creep phenomenon between new and aged samples are discussed with the help of graphical methods such as the approximated complex plane.

Key words: *heat treatment, poplar, creep tests, radial direction.*

## **1. Introduction**

Recently, the author and coworkers have been interested in the mechanical behavior of wood panel paintings [1,2]. Collaborations were established to perform and analyze the monitoring of dimensional changes on 3 to 6 centuries old wooden boards. A large amount of data on long term period were obtained, which raised also many questions on the properties of ancient wood. Because of the real difficulty of analyzing the material of panel paintings, we are now facing the lack of data to understand the behavior of ancient wood.

It has already been shown that ancient wood exhibits different mechanical properties than fresh wood [3]. Partly due to the variation in chemical composition, several properties like stiffness, axial strength, hardness and dimensional stability slightly improve within a few centuries, while color is getting darker. In order to reproduce partly the effects of time, heat treatment was used in many studies as an ‘accelerated ageing’ and showed so far good results on different softwood with respect to the darker color, fragile behavior and the improved stability against humidity changes [4,10]. In the cultural heritage context, this technique was used for instance in the restoration of wooden statues [5,6].

Further applications in cultural heritage, possibly in structures, would imply to get interested more about the mechanical behavior of heat treated wood. It has already been shown that heat treatment in dry conditions tends to reduce the elastic modulus in longitudinal direction [7], while the ancient wood get naturally stronger, probably due to the crystallization of cellulose in the earlier centuries [8]. But so far very few attempts have been made to show the long term behavior or water-related mechanical behavior of artificially heat treated wood.

This study was conducted on hardwood for several reasons. So far most of the studies were conducted on softwood. Moreover European poplar is one of the most common specie for panel paintings and thus was chosen for our experiments. The creep experiments were all conducted in radial direction; so far, most of the literature on the subject was done in the longitudinal direction.

## **2. Material and method**

This study was conducted on poplar wood (*Populus alba*) from Italy. Creep tests were all performed on radial samples, with dimensions 10mm (T), 1.6mm (L) and 60mm (R); for practical reasons, adsorption isotherms were checked by weighting the same size of samples. All samples were first air-dried for 24 hours at 60°C, and then vacuum-dried over P<sub>2</sub>O<sub>5</sub> at the same temperature and duration to reach the oven dry state.

Heat treatment was performed on dried samples at 150°C. Two different durations (24 hours and 48 hours) were tested on two different groups of samples. Durations were decided in accordance with

previous results on Hinoki (*Chamaecyparis obtusa*) [4], in order to obtain significant decrease in hygroscopicity.

Static bending tests were performed until rupture on 5 samples at least for each group (non-treated, treated for 24h and 48h) at 65% relative humidity

In order to obtain both isotherm curves and creep at different moisture contents, samples were conditioned in 9 different relative humidity (RH% 11,22,43,53,62,75,84,92,97 at 24°C), obtained with saturated solutions, at 25°C for nearly 11 days. Adsorption isotherms were measured at each humidity conditions while creep tests were only performed at 22, 62, 84, 92 and 97% relative humidity, for both treated and non-treated samples.

Constant humidity during creep tests was obtained by wrapping samples in polyethylene bags. Room temperature regulation was considered to be satisfactory for this study. Moisture stability of the wrapped samples was checked by weighting before covering and after experiments, with no significant deviation.

Creep tests were performed on a three points bending apparatus. Constant load was applied, and maximum strain applied to the material was estimated to be 0.20%, most of the samples being deformed at around 0.15%.

Each sample was at least loaded for 10 hours, and data points were collected at a 0.1 interval, given in natural logarithm of the time in second. To go through the variability, all data were interpreted in terms of compliance.

### 3. Results and discussion

As expected, hygroscopicity of poplar wood was reduced by heat treatment. Static bending test showed that elastic modulus at 65% is also decreasing with the treatment duration. Hygroscopicity of poplar treated during 24 and 48 hours were almost the same. If sufficient time was dedicated to this study, it would have been interesting to investigate regularly spaced durations on the  $\log(\text{time})$  axes. Fig 1a and Fig 1b show the adsorption isotherms with experimental data and fitted equation as used by Hailwood and Horrobin [11]:

$$A + B * rh + C * rh^2 = \frac{rh}{mc}$$

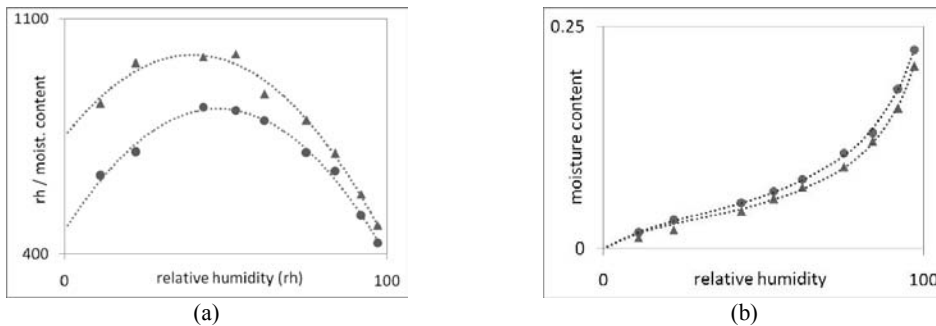


Fig. 1: adsorption isotherms plotted in (a) relative humidity divided by moisture content versus relative humidity, and (b) relative humidity versus moisture content. ● non-treated samples ▲ treated samples.

In this paper, two different approaches are proposed to analyze creep data: first the material properties are described as functions of  $\ln(\text{time}_{\text{sec}})$  and moisture content, and the second approach will be to plot data in the approximated complex plane to find a suitable rheological model. All results are presented in terms of creep compliance in order to avoid dispersions in the samples properties.

#### 3.1. Influence of moisture content on elastic properties

Fig. 2 shows the relative variations of elastic modulus, estimated during creep tests at  $t=5\text{sec}$ , depending on the moisture content. For treated and non-treated samples, all data are divided by

modulus at 64%. Radial modulus ( $E_R$ ) at 64% ( $E_{R64\%}$ ) in static bending was around 1.1GPa for non-treated wood and 0.97GPa for treated wood. Values found in creep tests were close to these values. Non-treated wood shows clear linear dependency between  $E_R$  and mc%, and strong reduction with increasing humidity.

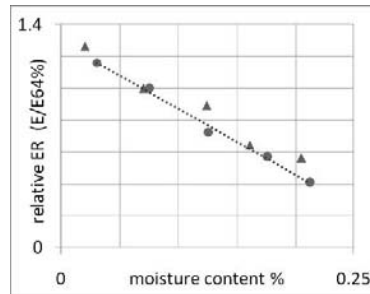


Fig. 2: relative elastic modulus depending on moisture content (modulus at any moisture content divided by modulus at 64% relative humidity), ● non-treated samples ▲ treated samples.

In former studies [4], static bending tests have suggested that heat treatment increases the elastic compliance at standard ambient conditions. For practical applications, it would be useful to know if the same effect can be expected for creep compliance, as well as for higher levels of relative humidity.

### 3.2. Analysis in terms of compliance

The compliance at  $t=10$ seconds and at  $t=9$ hours are both plotted on Fig. a, as a function of moisture content. Instantaneous compliance ( $J_{10sec}$ ) shows a regular increasing with moisture content (see dotted line on Fig. 3a) while the curves have particular shape depending on treatment. The difference between the two curves ( $J_{9hours} - J_{10sec}$ ) i.e. the contribution of delayed behavior of wood, is called creep compliance and is plotted in Fig. 3b. This curve points out the differences in creep phenomenon between treated and non-treated samples.

After heat treatment, if the dependency between creep compliance and moisture content is the same, the two curves of Fig. 3b might be superimposed. In other words, if heat treatment results only in a reduction of moisture content, experimental points representing heat treated wood should be on the untreated samples curve, with only a shift due to the reduced moisture content.

However it is not the case: the delayed deflection of heat treated samples is sometimes higher, sometimes lower, depending on moisture content. This observation supposes that the influence of heat treatment should not be considered systematic, that the effect might depend on moisture content during loading.

The difference in shape of the two curves also implies to explain what happen at 6% moisture content in the new wood, where a clear minimum is shown in creep compliance (see Fig. 3). The same effect does not occur in heat treated wood.

The differences in creep behavior can also be shown by plotting the time spectrum of treated and untreated samples, as shown on Fig. 4. Time spectrum consists in plotting characteristic time  $\tau$  versus moisture content, where  $\tau$  is the time to reach a given creep rate in the compliance curve. The faster the deformation increases, the lower the characteristic time  $\tau$ . Thus the curves in Fig. 4 show that modification of delayed behavior of heat treated wood depends on moisture content.

The previous observations suppose that the chemical composition of wood changed, and that the components that were modified play an important role around 6% moisture content (this is shown by the reduction of minimum pick after treatment).

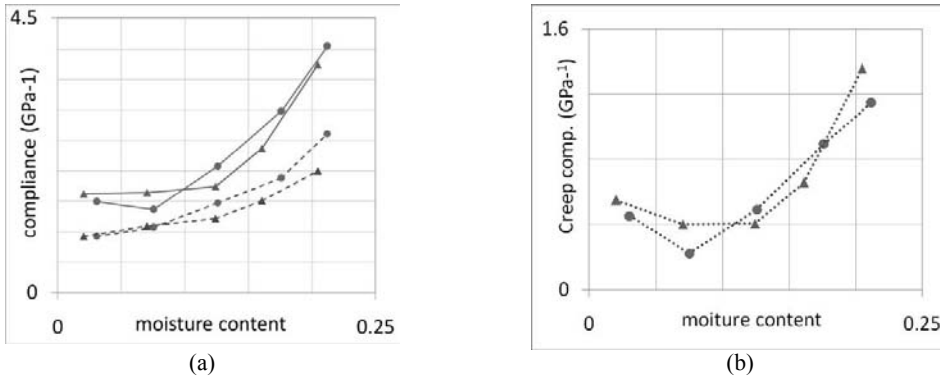


Fig. 3: (a) compliance versus moisture content, (plain line) after 9hours, (dotted line) instantaneous, (b) creep compliance after 9 hours. ● non-treated samples ▲ treated samples.

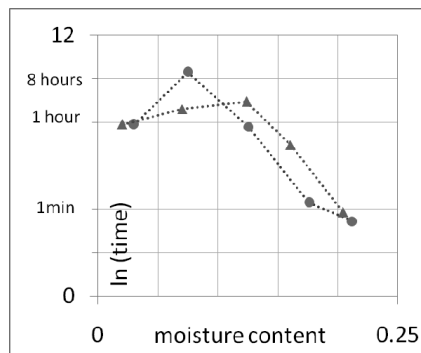


Fig. 4: time response spectrum,  $\ln(\tau)$  in seconds) versus moisture content:  $\tau$  is the time for which the rate of creep compliance is equal to a given value ( $dJ/d\ln t = 10^{-4}$ ,  $J$  given in MPa and  $t$  in seconds).

### 3.3. Analysis by approximate complex plane (ACP)

Based on an approximation by Alfrey (1948) of the complex compliance usually considered for dynamical studies, we can interpret our data in a different way. With this method, on X axis we plot the creep compliance as calculated from the data, and Y axis is proportional to the compliance rate:

$$X = J(\ln t); Y = \frac{\pi}{2} * \frac{dJ}{d \ln t}$$

On the graphic method proposed by Huet [9] we identify a rheological model that can fit to our data. First step is to determine the type of plot in the approximate complex plane. On the basis of pattern in Fig. 6, we assume that the phenomenon is described by a linear curve in the ACP, this for all moisture content and both treated and untreated wood. A slope  $\ll 1$ , like observed here, means that the suitable model would have a large spectrum of Kelvin models.

Then we analyze and discuss the effect of moisture content on the parameters of the linear regression used as model. Fig. 7 gives the slope of the linear regression and the intersection with X axis, respectively the spreading of Kelvin spectrum and the elastic compliance given by the model fitting. Experimental measurements of instantaneous compliance are also re-plotted in dotted line to show the difference between fitted model and initial data.

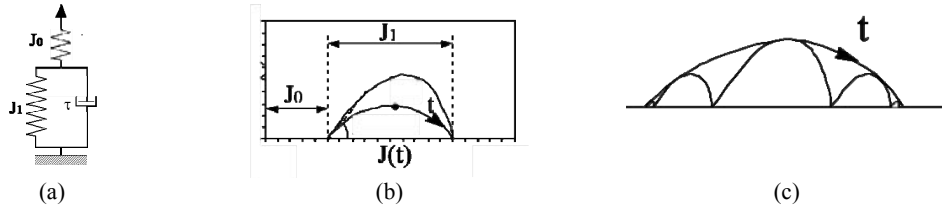


Fig. 5: (a) Kelvin body, (b) representation of a single Kelvin body in the ACP, (c) spectrum of several Kelvin bodies in the ACP.

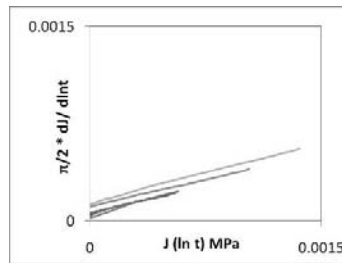


Fig. 6: an example of creep curves in complex plane for non-treated wood.

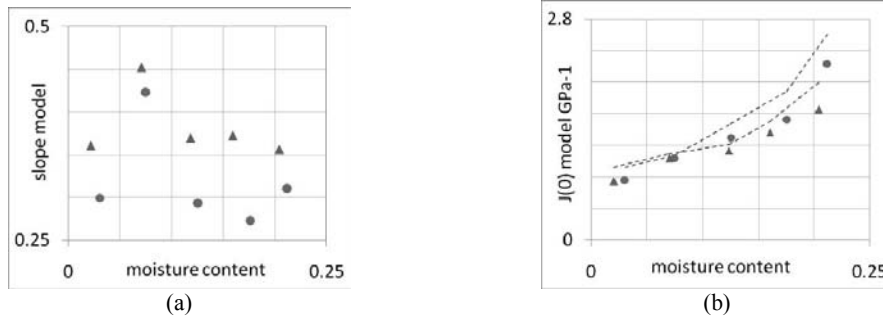


Fig. 7: parameters given by model in complex plane: (a) slope of model (b) instantaneous compliance as represented by model; dotted line correspond to measured compliance at 10 seconds, as shown in Fig. 3.

#### 4. Conclusion

New data have been obtained on a hardwood (poplar) and in radial direction; while studies conducted so far and available in the literature concern usually softwood in longitudinal direction. Mechanical behavior in transverse plane (R,T) is of great interest, from the panel paintings to the structural applications.

As expected, hygroscopicity of poplar wood was reduced by heat treatment at 150°C for 1 day. Results suggest that the effect of heat treatment on creep deflection is depending on moisture content under service conditions. The discussion has to be detailed on several points: the shape of creep curve on natural wood, with appearance of a minimum around six percent moisture content, and the modification of this shape due to heat treatment.

With further experiments, it would be interesting to compare these results with other directions (longitudinal and tangential). It would be also interesting to draw experiments with a large range of treatment times and temperatures.

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# MEASUREMENT OF THE ELASTIC PROPERTIES OF MINUTE SAMPLES OF WOOD ALONG THE THREE MATERIAL DIRECTIONS

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## Abstract

Reaction wood is an important trait of secondary growth in trees, which allows the mechanical structure to be adapted to its environment. It is well-known that reaction wood is different from normal wood, in terms of anatomical and ultrastructural features. Among the important differences between reaction and normal wood, we can list: thicker cell walls, chemical composition, microfibril orientation... A complete characterisation of the mechanical properties at the tissue level remains however seldom, especially in the three material directions. This work proposed a complete characterisation of the stiffness of poplar, at the tissue level, for normal wood, reaction wood, opposite wood. The material comes from a poplar tree artificially bent for several years to oblige the stem to produce reaction wood. This procedure allows large zones of tension, normal and opposite wood to be easily obtained for sample preparation. Two different micro-testing machines were designed and built in our laboratory: A tensile testing device with determination of the strain field using an optical microscope and image correlation (Radial and Tangential directions), with specimens about:  $20 \times 4.5 \times 1.8 \text{ mm}^3$ ; a 4-point bending device to determine the Longitudinal Young modulus on very small samples with determination of the deflection using a laser micrometer, with specimens about  $30 \times 2 \times 0.7 \text{ mm}^3$ . As most significant results, one may note the low transverse stiffness of tension wood in spite of its high density and the very high longitudinal stiffness of tension wood. Indeed, the specific longitudinal Young Modulus of tension wood reaches **70% that of crystalline cellulose**.

**Keywords:** reaction wood, normal wood, tensile, optical microscope, 4-point bending, poplar.

## 1. Introduction

Wood is an anisotropic material whose physical and mechanical properties depend dramatically on the material direction and on its structural features [2] (anatomy, cell wall macromolecular arrangement). Thus, the development of experimental devices able to measure local properties of on well-identified minute samples of wood is of great interest. In this context, original methodologies were developed to measure mechanical properties of wood on micro-samples (a few millimetres) in longitudinal and transverse (radial or tangential) direction. The measurements presented in this paper were performed on samples cut from a poplar tree artificially bent for several years, which forced it to produce tension wood. In addition to custom experimental devices, the whole approach required microsampling methodologies to be developed, that enable accurate mechanical measurement on specific types of wood and along the three material directions.

## 2. Method

### 2.1. Mechanical property measurement according to the longitudinal direction

In these tests, a device was conceived and built to perform a rigorous 4-point bending test on micro-samples. The wood sample, which can be considered as a beam, is placed on two fixed cylinders, free to rotate, and this wood sample defines the measurement length (L). The force is evenly applied on two moving cylinders of same diameter separated by the length (l) (Fig. 1).

The force is applied stepwise to the sample. For each load level, the beam curvature is measured without contact using a laser micrometer. Due to the small sample size and to avoid creep effects, the forces applied to samples ranged from 0 to 1.5 N, by steps of 0.1 N. Each step lasted 5 seconds. The

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comparison of loading and unloading curves permitted us to validate that measurements took place in the linear elasticity domain. The infradensity of each sample after drying at 103°C was also determined.

From the experimental displacement / force curves, the Young's modulus was calculated using the following equation:

$$E = \frac{l^2 (L - l)}{32 E I . \alpha} \quad (\text{where } \alpha \text{ is the slope of the displacement/ force curve})$$

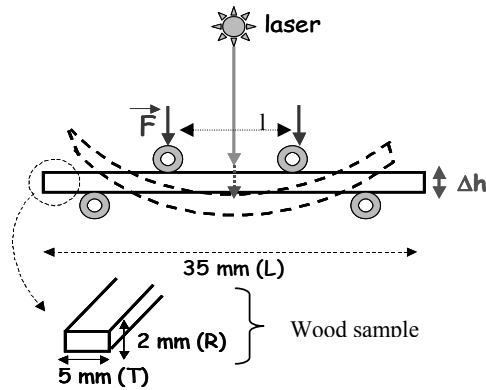


Fig. 1 Experimental configuration for the 4-point bending tests.

## 2.2. Tensile tests along the radial and tangential directions

### Experimental device

Tensile tests were performed with a manual tensile system, whose micrometer screw allows precise increments. In these tests, the stress value,  $\sigma$ , is measured with a load cell, assuming the section to be uniform and the strain value,  $\varepsilon$ , is obtained directly on the sample without contact using image correlation. For this purpose, the optical microscope is set in reflexion mode and the sample surface has to be carefully polished [7]. In the specific case of the tangential direction, because of their small size, the samples have been glued to plastic jaws to facilitate their attachment to the bending device. Particular attention was paid to the alignment of the jaws and the wood sample before they were placed on the experimental apparatus. In the case of radial measurement, the sample size was sufficient for direct fixing to the bending device. During the test, the apparatus itself was attached to the stage of an optical microscope with two screws. This allows the position of the image to be carefully chosen and this zone of interest to be tracked during the test (Fig. 2).

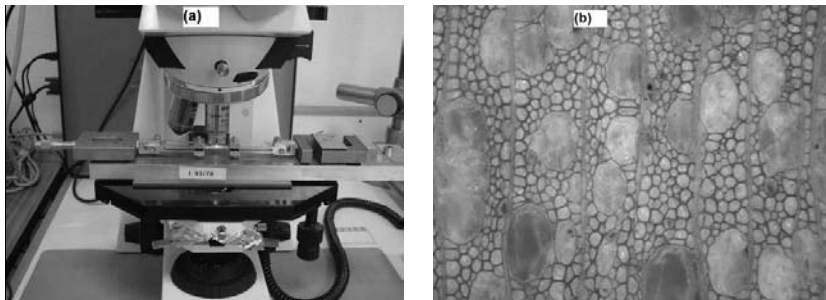


Fig. 2: a) Our custom tensile device placed on the stage of the optical microscope and b) example of image obtained in reflexion mode.

At each imposed displacement increment, the indication of the load cell is noted down and an image of the sample is grabbed. These images were subsequently treated by an image analysis software to determine the strain tensor in the focal plane of the microscope [5]. The Young's modulus of the sample was finally calculated from the linear part of the experimental force/ deformation curve.

#### Measuring the strain tensor by image correlation using the software MeshPore

The software *MeshPore*, developed in LERMAB [8] was used to determine the cell deformation of wood sample at each displacement increment. An initial approximation of the strain tensor was obtained by contour integral of a chain of points plotted on the initial image and on the deformed image (Fig. 3). Depending on the image quality, three types of anatomical features have been used to define the points of this chain [5]:

- the triple point which corresponds to the intersection of three cells;
- the mid-segment: the point situated halfway between two triple points. This was the solution adopted when the image was of poor quality.
- the lumen centre.

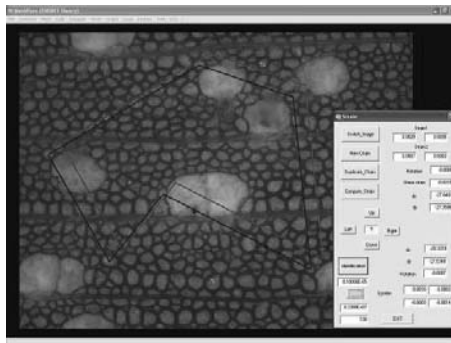


Fig. 3 Correlation image by MeshPore

The approximate value of the strain field was subsequently refined using an inverse method. This second step is the real image correlation procedure, whereas then first step allows us to avoid local minima.

### 3. Material

The study was performed on a clone of poplar (*Populus*) that was ten years old and that came from an in vitro culture carried out by the Forestry Physiological and Genetical Improvement Unit (INRA, Orléans). Planted in 1996, it was artificially bent from 1998 in order to let it produce a highly located area of tension wood in the cross section. Considering the small tree diameter and its age, samples are all in juvenile wood.

Three kinds of wood were collected in this tree: reaction (tension); opposite ( $180^\circ$  from the tension wood zone) and normal wood ( $90^\circ$  from the tension wood zone). From an anatomical point of view, vessels of tension wood are fewer and smaller in diameter than the opposite and normal zones. Tension wood exhibits a more important fibre ratio and thicker cell walls with very rounded contours that result to smaller cell diameter than opposite and normal ones. Microscopic observations of tension wood fibres revealed the presence of a gelatinous layer, the so-called G-layer.

#### 3.1. Sampling for tests in longitudinal direction

Sampling was prepared in two successive stages. First, wood was sawed in the radial and the longitudinal directions by a circular micro-saw (Fig. 4). The rotation speed and the sample advance rate were 900 rounds/min and 250  $\mu\text{m}$ /sec respectively. After, the previously machined block was cut by a razor blade in the tangential direction.

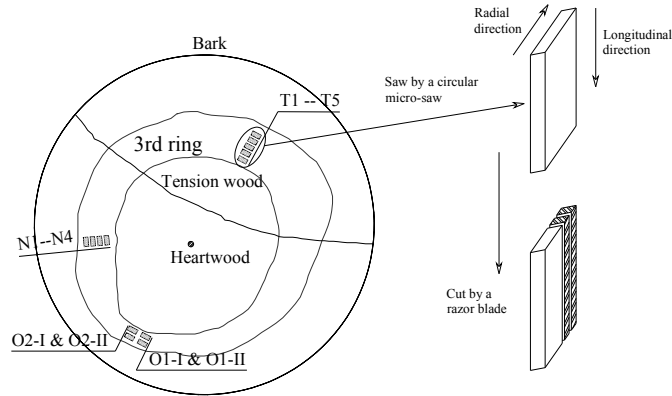


Fig. 4 Sampling for tests in longitudinal direction

In a second stage, the previously sample was polished in their longitudinal direction (direction of their length). A rigid sample holder was designed to obtain a constant thickness along the entire length (Fig. 5).

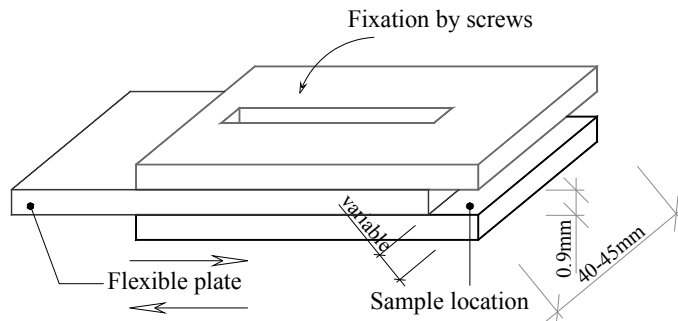


Fig. 5: Sample holder used for to polish the machined blocks at a constant section.

The size of wood samples was  $35 \times 1.8 \times 0.9 \text{ mm}^3$  according to longitudinal, tangential and radial direction respectively.

### 3.2. Sampling for tests in transverse directions

Samples were prepared with a diamond wire saw which produces parallel faces. For each type of wood, two blocks were taken: one in the tangential direction and the other in the radial direction (Fig. 6). From these blocks, thin bars (longitudinal thickness) were cut at constant thickness.

One of the wood cross section was then polished to allow microscopic observations during tensile tests. This operation was done with the same sample holder to ensure a uniform thickness over the entire sample (Fig. 5).

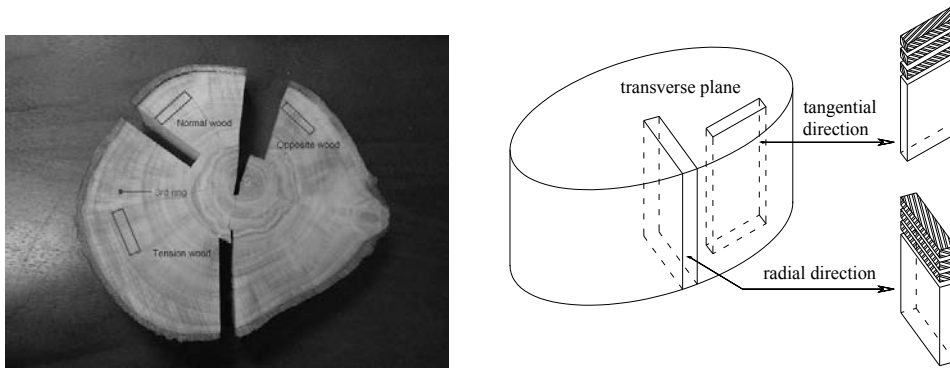


Fig. 6 : Sampling in radial and tangential directions.

4. Results

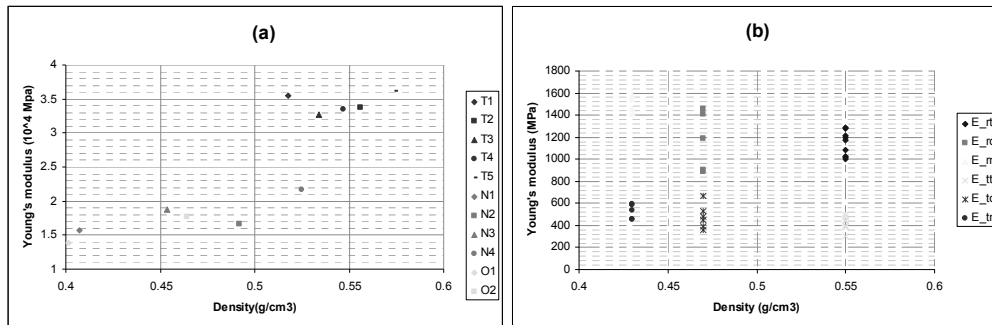


Fig. 8: Young's modulus of tension (T), normal (N) and opposite (O) wood in the longitudinal (a), radial and tangential (b) directions in relation with their density.

Table 1 : Specific modulus of tension (T), opposite (O) and normal (N) poplar wood in radial (R), tangential (T) and longitudinal (L) directions.

	$E_R^{(c)}$	$E_T$	$E_L$	Density	$E_R^{(a)}$	$E_T$	$E_L$
T	1 143	416	34 300	0.55	2 078	756	62 364
SD <sup>(b)</sup>	111	60	1 400	0.02			
O	1 170	478	15 500	0.47	2 489	1 017	32 979
SD	269	109	2 000	0.05			
N	1 484	573	18 200	0.43	3 451	1 333	42 326
SD	75	65	2 600	0.04			

<sup>(a)</sup>E': Specific modulus (Mpa.kg<sup>-1</sup>.m<sup>3</sup>)

<sup>(b)</sup>SD: Standard deviation

<sup>(c)</sup>E: Young's modulus (MPa)  
Density (g/cm<sup>3</sup>)

In the longitudinal direction, the variability of Young's modulus in the differences samples of each type of wood (tension, normal and opposite wood) is low compared to the transversal direction. Particularly, in the case of opposite wood, the standard deviation of the Young's modulus is rather high; the Young's modulus is variable despite the proximity of all samples to the same annual ring. This could be due to the fact that, as ring widths are very narrow in opposite wood, the sample can easily involve adjacent annual rings. For normal wood and tension wood, quite good values of standard deviation have to be noticed.

In the longitudinal direction, the difference between tension wood and the group of normal and opposite wood is huge, E=35000MPa for tension wood and E=17000MPa for opposite and normal wood. On the contrary, the results in the transversal direction present low differences of Young's

modulus, from  $E=400$  to  $500$  MPa in the tangential direction (corresponding to the result of L.Brancheriau 2002 [6] when the density varies from  $0.43 \text{ g/cm}^3$  to  $0.55 \text{ g/cm}^3$ ,  $E_{\text{tangential}} \sim 500 \text{ MPa}$ ) and from  $E=1200$  to  $1500$  MPa in the radial direction [9]. Otherwise, we can see that normal wood was more rigid than tension wood in the transversal direction ( $E_n/E_t$  about  $500 \text{ MPa}/400 \text{ MPa}$  in tangential direction and about  $1500 \text{ MPa}/1200 \text{ MPa}$  in radial direction). This could be explained by the lack of cohesion of the *G*-layer with the rest of the wall [1]. In this case, the real part of tension wood participating in the mechanical strength would be thinner than in the normal wood ( $S1 + S2$  instead of  $S1 + S2 + S3$ ).

The wood anisotropy is even higher in tension wood ( $E_{\text{longitudinal}}/E_{\text{tangential}} = 87$  and  $E_{\text{longitudinal}}/E_{\text{radial}} = 30$ ) than in normal and opposite woods (45 and 10 respectively). This is due to the presence of the *G*-layer [1] and its crystalline cellulose mainly oriented along the cell axis. Besides, the effect of weak microfibril angle of the *S2* and *G*-layer is also a possible reason for the high mechanical behaviour of tension wood [3], [6].

## 5. Conclusions and prospects

Custom devices and the corresponding procedure (sampling and testing protocols) have been resented here for the characterisation of mechanical properties of minute wood samples in their 3 directions of anisotropy. Several improvements of this domain of experimental investigation are currently in progress in our laboratory: compression tests with large deformations, characterisation of green samples...

By its ability to produce very localized measurements, this methodology can be applied to many questions of wood science: different type of wood, different tissues, but also sample with homogeneous treatment (extraction, chemical attacks, enzymatic attacks, locally modified wood...)

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# PREDICTION OF LINEAR DIMENSIONAL CHANGE OF UNRESTRICTED WOOD AT DIFFERENT LEVELS OF EQUILIBRIUM MOISTURE CONTENT

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## Abstract

A computer routine to predict variations in linear dimensions and warping of wood was designed and tested. This routine was applied on both experimental data obtained from laboratory-prepared wood samples as well as other data found in the literature. The two outcomes were compared and contrasted. Predictions resulting from the computer procedure resulted to be reliable only for wood which is straight grained and free from physical constraints and natural defects

## 1. Aim

The aim of the computer analysis exercise was to predict linear dimensional changes as well as any possible warping (cupping) of unrestricted wood.

## 2. Method

A computer routine was devised by using two computer software packages, namely Microsoft Excel and AutoCAD 2000. Data found in the relevant literature was used in order to carry out the necessary workings. The four stages of the computer analysis routine consist of:

- (i) the end grain photography and line drawing on AutoCAD;
- (ii) the merging of data into a Microsoft Excel spreadsheet;
- (ii) the interpretation of data and the relevant calculations; and
- (iv) the re-construction of the panel end grain, considering the predicted contraction/expansion and warping using AutoCAD.

### 2.1. End grain photography and line drawing by AutoCAD

The first step involved capturing an image of the end view of the panel. In this study, both direct scanning and digital photography were applied. One has to consider that photography has an element of inaccuracy due to perspective illusion.

The picture was transferred into AutoCAD and the outline traced. The drawing was then divided into a number of quadrilateral sections, as in the case of ABCD shown in Fig. 1. In areas where the radius of curvature of the annual rings was quite small or complex, smaller quadrilateral sections were constructed. The diagonals in each section were then marked (as AD and BC in Fig. 1) and the orientation of the grain was marked (as EF in Fig. 1). All dimensions (of lines AB, CD, AC, BD, AD and BC) and angles of orientation (0° to 90°) were measured using AutoCAD.

All the following data was then fed into a Microsoft Excel spreadsheet:

- initial temperature (T);
- initial relative humidity (RH);
- final T;
- final RH;
- literature value for longitudinal shrinkage (left out if length of panel was not being considered) and when data was not available, a value of 0.1% was assumed; [1]
- literature value for tangential shrinkage of the given type of wood (left out if the width and the thickness of the panel were not being considered);



- literature value for radial shrinkage of the wood under test (left out if the width and the thickness of the panel were not being considered);
- literature value for the fibre saturation point (FSP), and when not available, a value of 30% was assumed.

AutoCAD line drawing

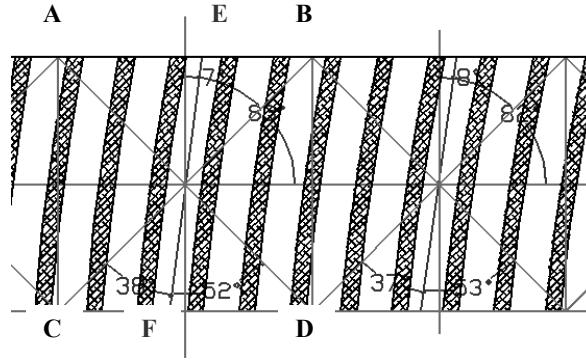


Fig. 1. Measuring sides, diagonals some of the angles to grain orientation EF

## 2.2. Interpretation of Data and Calculations

### Equilibrium Moisture Content (EMC)

Calculation of the initial and final EMC values using a Microsoft Excel spreadsheet was carried out by using formula (1) [2]:

$$EMC = \frac{1800}{W} \left[ \frac{K.RH}{1 - K.RH} + \frac{K_1.K.RH + 2K_1K_2K^2.RH^2}{1 + K_1.K.RH + K_1K^2.RH^2} \right] \quad (1)$$

where:  $EMC$  = equilibrium moisture content (in %)  
 $RH$  = relative humidity (in %)

For temperature  $T$  in °C:

$$W = 349 + 1.29T + 0.0135T^2$$

$$K = 0.805 - 0.000736T - 0.00000273 T^2$$

$$K_1 = 6.27 - 0.00938T - 0.000303 T^2$$

$$K_2 = 1.91 + 0.0407T - 0.000293T^2$$

### Theoretical Dimensional Change

An estimate of the value for shrinkage/expansion was obtained by using formula (2) giving the theoretical dimensional change [3]:

$$\Delta D = \frac{D_i (MC_i - MC_f)}{\frac{FSP}{S} - FSP + MC_i} \quad (2)^\diamond$$

where:  $\Delta D$  = dimensional change  
 $D_i$  = initial dimension  
 $MC_i$  = initial moisture content (%)  
 $MC_f$  = final moisture content (%)  
 $FSP$  = fibre saturation point (%)

<sup>◊</sup> Note from the Editor: although Equation (2) is correctly cited from Hoadley, the term  $-FSP + MC_i$ , which anyway adds a negligible contribution to the denominator, does not bear any physical meaning and should be omitted

$S$  = published value for shrinkage (%)

### Linear Dimensional Change

Considering that all dimensions in every orientation would undergo radial contraction/expansion, the extent of tangential contraction/expansion needs to be found. Formula (3) giving the linear dimensional change [4] was used to arrive at the final increase/decrease for every dimension recorded.

$$D_f = \frac{D_i}{100} \times \left[ 100 - \left( \left( \frac{V_t - V_r}{90} \times O^0 \right) + V_r \right) \right] \quad (3)$$

where:

- $D_f$  = final dimension
- $D_i$  = initial dimension
- $V_t$  = calculated tangential shrinkage (%)
- $V_r$  = calculated radial shrinkage (%)
- $O^0$  = orientation (in degrees)

### **2.3. Reconstruction of the Panel End Grain presenting Contraction/Expansion and Warping on AutoCAD**

The results obtained as outlined above, by using a Microsoft Excel spreadsheet, were then transferred back to AutoCAD. The resulting outline (based on the new set of data) led to the indications of dimensional change as well as any cupping the panel would undergo as a result of changes in the T and the RH. Longitudinal contraction/shrinkage is deemed as insignificant, especially over short lengths, but it can still be predicted by using the same computer routine.

### **3. Limitations of the computer analysis**

The computer analysis only leads to approximate results. These results are only reliable under given conditions while certain other conditions affect the behaviour of wood. Such instances outlined hereafter.

The wood panels should be free from natural defects such as: cross grains, knots, compression/tension wood, juvenile wood, etc.

The wood should not be restricted by crossbars, frames and/or mounting hardware.

Preparation and painting layers (as in panel paintings) lower the accuracy of the results obtained from this computer analysis, especially when cupping is evaluated.

Published literature data for the coefficient of shrinkage (tangential, radial and longitudinal) as well as the FSP are all average values, which might result to be quite different in reality. Dimensional response of aged wood may result to be slightly lower due to decrease in hygroscopicity and/or the mechanical effects of repeated shrinkage/swelling cycles. [5] In the case of panel paintings, unpredictable cupping may take place even on radially cut boards due to rapid moisture adsorption by the untreated back. [6]

### **4. Possibility of errors**

As the grain orientation may change, adjacent sides of sections may have slightly different dimensions. Such discrepancies are negligible and are not visible to the naked eye. They may be eliminated by narrowing the sections and, consequently, any changes in grain orientation result to be more gradual.

### **5. Testing the computer procedure**

#### **5.1. Aim**

This test was carried out in order to simulate situations of contraction/expansion as well as warping. Literature data for Shellbark Hickory (*Carya laciniosa*) were chosen for this test, due to the high tangential and radial shrinkage values, i.e. 12.6% and 7.6% respectively. [7] The test was also carried out on actual pine samples.

## 5.2. Test on two shellbark hickory (*Carya laciniosa*) boards

Two ends of two imaginary boards, having dimensions 400mm by 25mm, were drawn. Four tests were carried out on these virtual Hickory panels. Both the initial T and the final T were taken as 21°C. The three shrinkage coefficients were taken as follows: tangential shrinkage = 12.6%, radial shrinkage = 7.6%, longitudinal shrinkage = 0.1%. The value of the FSP was taken as 30%. Table 1 shows the initial and final RH values for the four tests.

Table 1: Data used for computer tests 1 to 4

Test number	Initial RH (%)	Final RH (%)
1	90	20
2	20	90
3	78	0
4	78	100

The boards were virtually subjected to theoretical, extreme climatic conditions to simulate expansion, contraction and fluctuation of the cupping from convex to concave warping. The outcome of Tests 3 and 4 are illustrated in Fig. 3.

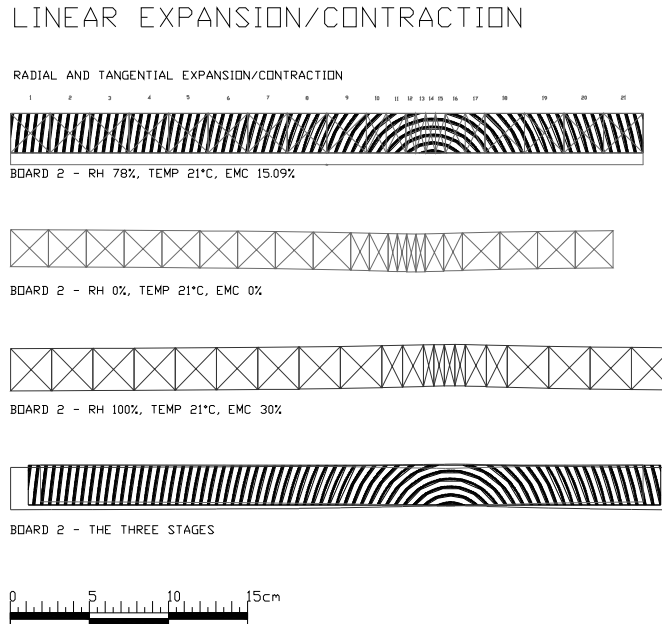


Fig. 9 - Computer prediction – Test 3: expansion and contraction

## 5.3. Test on Actual Pine (*Pinus sp.*) Samples

### 5.3.1. Method

Four pine samples were selected for this test. One of the samples had a tangential grain orientation across the width to ensure that cupping would be evaluated apart from linear changes.

The samples were dried in an oven (MEMMERT GmbH, UE/ULE/SLE 500) at a temperature of 103°C to reach an EMC of 0%, and then placed on a shelf in a sealed container with deionised water to attain a constant RH of 100%, until they reached a FSP value of 31.9%. The longitudinal face and the end grain were scanned just after the sample was oven dried and cooled back to room temperature, and when it reached 31.9% MC.

Line drawings using AutoCAD were made when the samples were at 0% MC and at room temperature. Drawings were drawn using a scale of 10:1, for higher accuracy. All dimensions and grain orientations were measured and introduced into the Microsoft Excel spreadsheet.

The following two predictions were done:

- (i) Case 1: calculations based on literature data of *Pinus eliotta* (Slash pine). [8]
- (ii) Case 2: calculations based on experimental results.

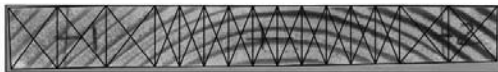
Table 2 shows the data used for these tests.

Table 2: Data used for pine computer program

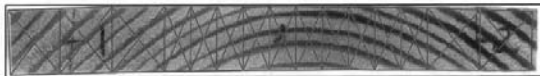
DATA USED FOR PINE SAMPLE								
TEST	INITIAL TEMP.°C	INITIAL RH %	FINAL TEMP.°C	FINAL RH%	PUBLISHED DATA FOR			FSP%
					TAN%	RAD%	LONG%	
CASE 1	17.0	0.0	15.1	100.0	7.6	5.4	0.1	30.0
CASE 2					7.91	3.73	0.31	

Fig. 3 shows the final drawings, where the resulting diagrams for both Case 1 and Case 2 were superimposed onto the true scanned images of the pine sample.

**ORIGINAL**



**CASE I - PINE SAMPLE using data collected for Slash Pine**



DATA USED FOR THE PREDICTIONS OF THE PINE SAMPLE							
Ti	Tf	RHi%	RHf%	Tan%	Rad%	Long%	FSP%
17.00°	15.10°	00.00	100.00	7.60	5.40	0.10	30.00

**CASE II - PINE SAMPLE using data collected from Physical Tests**



DATA USED FOR THE PREDICTIONS OF THE PINE SAMPLE							
Ti	Tf	RHi%	RHf%	Tan%	Rad%	Long%	FSP%
17.00°	15.10°	00.00	100.00	7.91	3.73	0.31	27.01



Fig. 3 – Results of computer analysis carried out on the pine sample

**5.3.2. Test Limitations**

The samples were too small and therefore the margin of error in measurement results to be larger. There must have been greater error with the thickness measurements (c.11mm) when compared with those for the length (c.78mm) and the width (c.89mm).

**5.4. Presentation and analysis of the computer procedure**

As can be seen from Fig. 3, the computer procedure is quite accurate. Although slightly different from published values, experimental results were not very significant considering the sample was quite small, and subjected to extreme conditions (from 0 to 31% MC). As expected, due to the difference between experimental and literature data (7.91-7.60% tangential value higher by 0.31%) the

predictions made using literature values resulted shorter by about 0.75%; experimental results gave slightly higher cupping than actually resulted.

In general, results seemed promising although the computer procedure should be tested on more wood species. Samples containing preparation and paint layers should also be tested to simulate more closely the situation in panel paintings.

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6. Ibid.:20.
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8. Ibid.

# AGEING OF WOOD – DESCRIBED BY THE ANALYSES OF OLD BEAMS

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## Abstract

Wood underlies ageing processes which means that the properties of old wood have been changed. Wood density shows very close relationships to the amount of latewood (for softwoods only). There are no changes of the wood anatomy due to ageing; except due to destruction by insects or fungi. Interior beams of two buildings (felling of the trees 1720 AD and 1854 AD) were sampled. To understand the effect of ageing on wood density, x-ray densitometric analyses of 10 beams from pith to bark were done. Plotting radial profiles of percentage of latewood and wood density sometimes showed an increasing divergence of these trends with increasing cambial age.

## 1. Introduction

Many buildings are preserved as important cultural heritage. A lot of these buildings are unique architectural and artistic objects [1]. Usually a lot of wooden structures can be found within these buildings.

Wood remains durable when it is protected from weathering and biological attacks (insects and fungi) but still underlies ageing processes. That means that the properties of old wood are altered [2, 3, 4].

Obataya [2] reported that wood loses strength, but stiffer with increasing time. Schniewind [7] found evidence, that wood density of used, old wood can be lower as well as higher compared to recent material. Wood quality, i.e. wood density, varies strongly – within the cell wall, within tree-rings, within trees and across sites [5]. Wood density should always be compared with respect to the cambial age (that is the number of tree-rings outside the pith) of the sample [6]. Typically wood density of softwoods (as well as mechanical strength) increases with increasing cambial age (number of rings). This is due to juvenile wood effects and due to age-related trends in wood formation [8]. Fig. 1 shows the general trends of ring-width, latewood percentage and wood density with increasing tree-age.

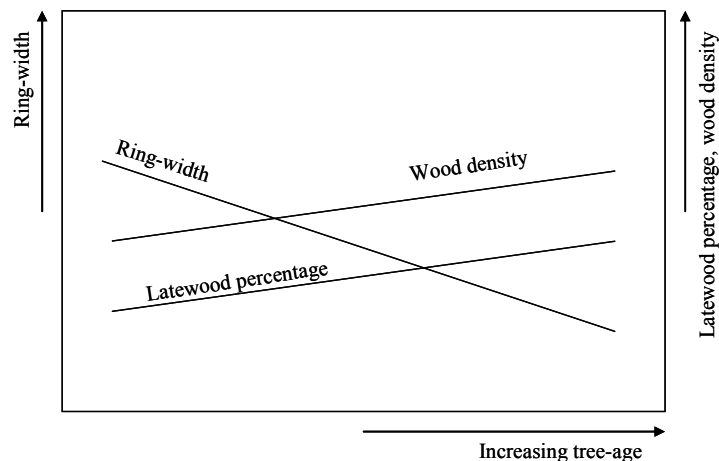


Fig. 1: General trends of ring-width, latewood percentage and wood density with increasing tree-age (for softwood only).

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All these statements make it complicated to compare aged samples with recent material. Generally, due to the high variability of all wood quality parameters along the stem, the growing sites and so on, it is hard to compare old material, from which the provenience is often not known, with recent grown wood.

In Literature, changes of the chemical composition of wood due to ageing effects are often described [1, 3, 4]. These changes can also influence the wood density. There are no changes in the anatomical structure due to ageing; except due to destruction by insects or fungi. Wood density shows very close relationships to the amount of latewood (for softwoods only). Studying the latewood percentage should help to understand changes in wood density, which could be attributed to ageing effects.

## 2. Material and methods

Ten interior beams out of two buildings from Eastern Austria (felling of the trees 1720 AD and 1854 AD) were sampled. The beams were made out of Norway spruce (*Picea abies* (L.)Karst.) and Silver fir (*Abies alba* Mill.) trees, which were between 65 and 147 years old (number of tree-rings). Wood density at standard climate (DIN 52184) and bending strength (DIN 52186) were studied across the whole radius (from pith to bark). All beams did not present obvious attacks of insects or fungi.

All tests were performed using 15x15 mm wide samples (the length was given by the standards). It was possible to prepare up to five samples across the radius – starting at the outermost part up to the pith.

To study the influence of ageing on the wood density, x-ray densitometric analyses [9] were performed using the whole radius from pith to bark (a part of such a profile is shown in Fig. 2). This analysis allows to extract data of ring-width, earlywood - and latewood width as well as mean ring density, mean earlywood - and latewood density at standard climate. The measurements of density and ring width are done at 35µm resolution. The analysis of density follows standard procedures [9]. The calibration is done using a eight step cellulose-acetate wedge.



Fig. 2: x-ray densitometric image showing some “decayed” parts.

## 3. Results

The mean values of the bending strength, showed the expected trend (from pith to bark): increasing strength with increasing distance from the pith (see Fig. 3). But, at the outermost position (close to the bark) the trend was inverted, and the results were decreasing.

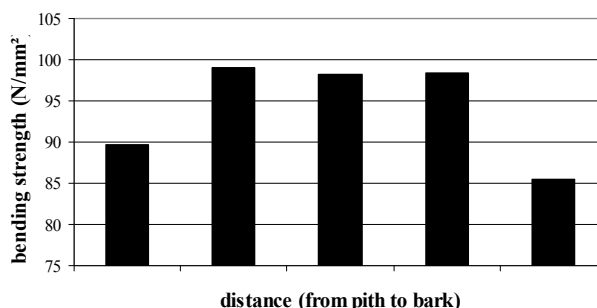


Fig. 3: Mean bending strength across the radius (from pith to bark, 33 samples).

The wood density (measured at the same samples) followed almost the same trend as the bending strength.

The x-ray densitometric analyses of the 10 beams showed not consistent trends (from pith to bark). Some samples confirmed the trends of the wood density determined at the “macro-samples”, showing decreasing density trends (at the outer part of the radius – see Fig. 4.). Other samples seemed to be unaffected (Fig. 5.) by ageing effects. They show increasing wood density (mean latewood density, mean earlywood density and mean ring density).

Comparing the profiles of mean ring density and latewood percentage (Fig. 6) the loss of density due to “ageing effects” can be seen clearly (starting at cambial ring 96). The trend of the mean ring density started to decrease while the trend of the latewood percentage is almost stable.

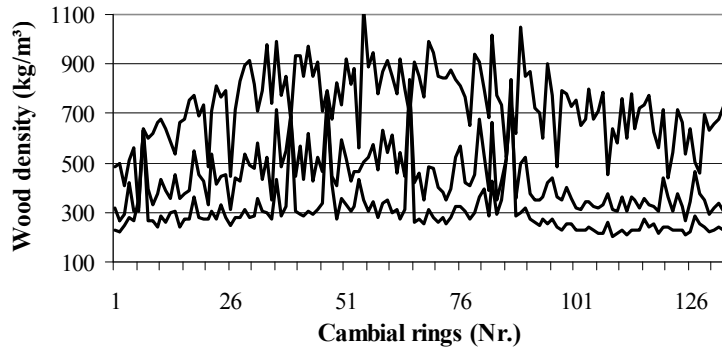


Fig. 4: Density profile from pith to bark. Mean latewood density (top); mean ring density (middle); mean earlywood density (bottom); sample N1

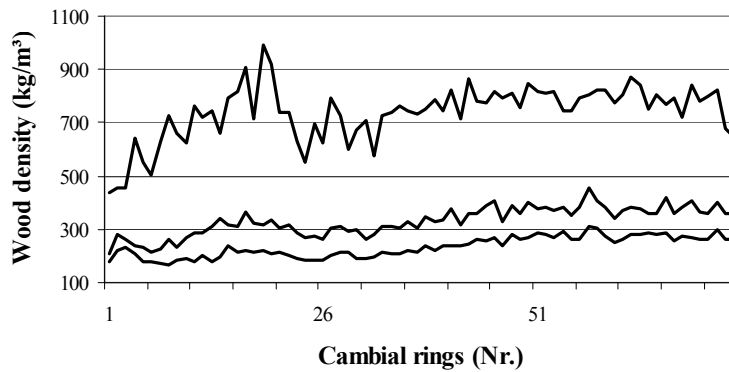


Fig. 5: Density profile from pith to bark. Mean latewood density (top); mean ring density (middle); mean earlywood density (bottom); sample W3

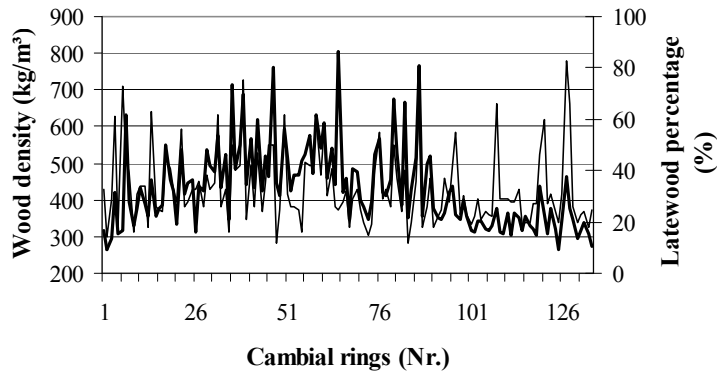


Fig. 6: Mean ring-density (bold line) and Latewood percentage (thin line) profile from pith to bark. (sample N1)



Five from ten samples (beams) showed the divergence between wood density and latewood percentage at the outer part (close to the bark) – like in Fig. 6. The others presented stable density trends and no changes of the relationship to the latewood percentage.

#### 4. Discussion

Five out of ten samples (beams) showed a decreased wood density and consequently a reduced bending strength at the outermost part (close to the bark). The age of the samples (also the origin) were not related to the loss of density. There was no obvious attack of beetles or fungi. Obataya [2] described an increase in bending and compression strength for the first 100 to 200 years. Afterwards the strength is declining. Hoffmann et al. [10] also reported a decline in density and strength with time at water logged wooden findings. Furthermore Bednar and Fengel [11] reported about decreased wood density and strength at sub fossil oak. Schniewind [7] reviewed about a reduction of density up to 20% (compared to recent material). Drdácý [12] found slightly lower density at the surface of about 200 year old Spruce beams than for the whole cross section.

The wooden material of these analyses was about 150 and 300 years old. Because of the long time axes of the reviewed literature the comparison has to be done with caution.

For softwood, latewood percentage is a good predictor of wood density [5, 6, 8, 13, 14]. The relationship between latewood percentage and wood density (mean ring density) was  $R^2 = 0.58$  for European Larch (more than 300 trees) and  $R^2 = 0.58$  for Norway spruce (8 trees – data not shown). The wood-chemical constitution is changing with time [2, 3]. Also crystallinity of cellulose get altered [2, 15]. The wood anatomy and especially the amount of latewood can not be altered due to ageing processes. Therefore, latewood percentage is a good predictor of age-related changes of wood quality.

50% of the samples showed a reduced wood density at the outermost part of the radius. This loss of density was not dependent on the age of the beams. No explanation of this phenomenon can be given at the moment.

#### 5. Conclusions

Studying wood density and mechanical strength across the radius (from pith to bark) showed that at 50 % of the samples the density and strength is lowered at the outer part (close to the bark). Latewood percentage was proved to be a good predictor of changes due to ageing effects.

To understand ageing processes better, more samples – and especially older samples – have to be analyzed. As the method of x-ray densitometry is a destructive method (but low invasive), it's hard to get samples.

#### Acknowledgement

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# HYGRO-LOCK INTEGRATION IN A KELVIN VOIGT MODEL

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## Abstract

This work deals with the modelling of the viscoelastic behaviour of wood components by taking into account environmental interactions. The model is based on the integration of elastic behaviour characterized by a hygro-locking effect in a generalized Kelvin Voigt model. This model requires the memorization of the complete past history of loading and moisture content variations through a double hereditary integral. This approach, treated with an incremental algorithm, can be used to develop models for different scale structural elements and to study the behaviour of joints under moisture content variations.

## 1. Introduction

The creep deformation of timber structures results in a complex coupling between varying humidity and temperature in the surrounding environment, the moisture content changes induced by boundary exchanges characterized by sorption isotherms, mass and heat transfer in wooden material and the viscoelastic process induced by the external load. The coupling between varying moisture content and the creep deformation is referred as the mechano-sorptive behaviour. In this context, if we consider ageing elements like lumbers or joints, see Fig. 1, the coupling between moisture content and loading histories induces apparent increases of deflexion or compliance and a global resistance decrease.

In the European Code for timber design,  $K_{def}$  and  $K_{mod}$  coefficients are introduced to take into account these phenomena. However, the long term behaviour prediction requires the knowledge of the complete past history of moisture content and loading. One solution consists in the development of numerical tools which can model the following effects, assuming linear behaviour restriction in regards to the viscoelastic response under moisture content variations:

- a moisture content dependence on elastic properties expressed by a hygro-lock effect as evidenced during drying phases by Gril [7],
- a moisture content dependence on viscoelastic properties which is characterized by an amplification and an acceleration of creep strains for high level of moisture content,
- swelling and shrinkage effects amplified by the strain level as shown by Ranta Maunus,
- a creep limit observed in the linear domain as demonstrated by Hunt

The first section concerns the development of the hygro-lock concept for elastic response. The adaptation for viscoelastic behaviour is proposed in the second part. Finally, an application example is proposed in order to highlight hygro-lock effect during a creep-recovery test under climatic variations.

## 2. Elastic behaviour and mechano-sorptive stress

In order to isolate each process, this paper deals with a numerical approach about the hygro-lock effect introduced in a viscoelastic model. A first formulation has been developed by Husson who proposed a specific elastic behaviour under moisture content variations  $w$ . For a uniaxial configuration,  $k(t)$  designates the time evolution of elastic rigidity induced by moisture content variations. Considering a time function for  $w$ ,  $k(t)$  can be expressed by:

$$k(t) = k(w(t)) \quad \text{where} \quad \begin{cases} \dot{w} \geq 0 \Rightarrow \dot{k} \leq 0 \\ \dot{w} \leq 0 \Rightarrow \dot{k} \geq 0 \end{cases} \quad (1)$$

In order to model the effect of hygro-lock highlighted by Gril, Husson has shown that the constitutive equations, for elastic response, can be written as follows:

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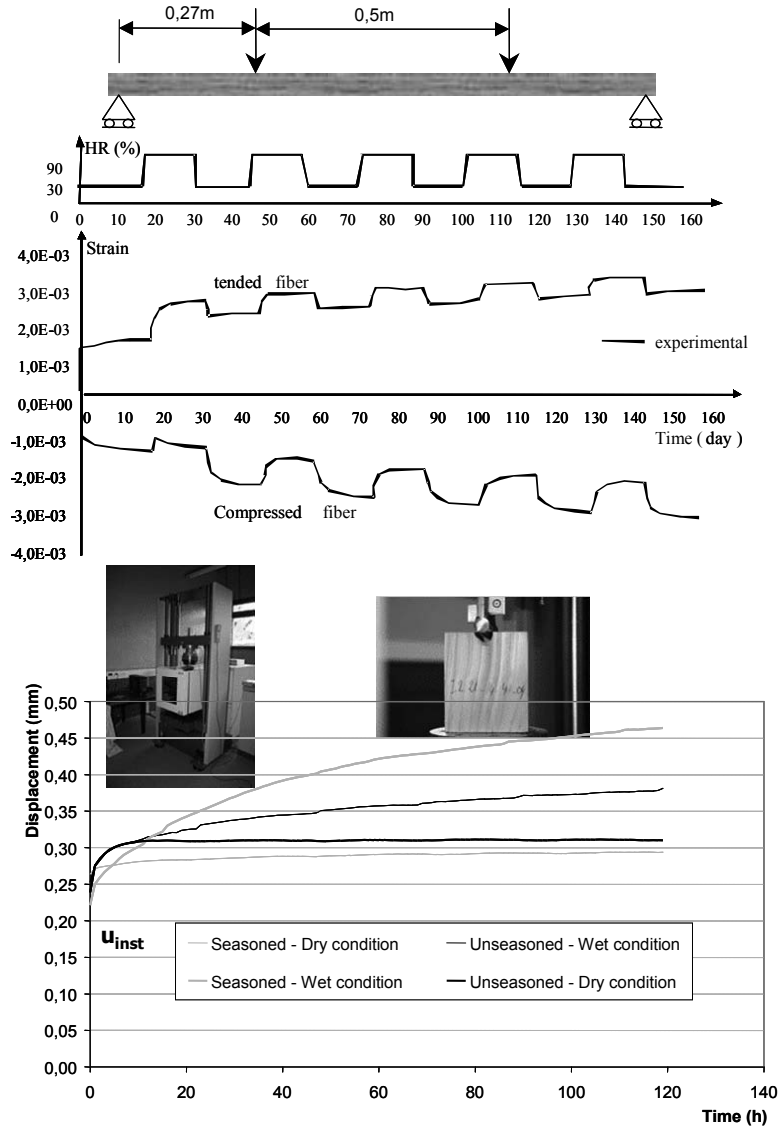


Fig. 1: Response of a lumber and dowel bearing under moisture content variations

$$\varepsilon_e(t) = \int_0^t \frac{1}{k_{\min}(\tau, t)} \cdot \frac{\partial \sigma}{\partial \tau} d\tau \quad \text{with} \quad k_{\min}(\tau, t) = \min_{t' \in [\tau, t]} k(t') \quad (2)$$

$k_{\min}(\tau, t)$  designates the minimum of the elastic modulus between the actual time  $t$  and the specific time  $\tau$ . For a constant mechanical loading, Fig.2 illustrates this definition.

### 3. Generalization for viscoelastic behavior

In order to take into account differed effects, it is necessary to introduce the last hygro-lock effect in a Kelvin Voigt model which is adapted to simulate a creep long term response in timber elements, see Fig. 3.

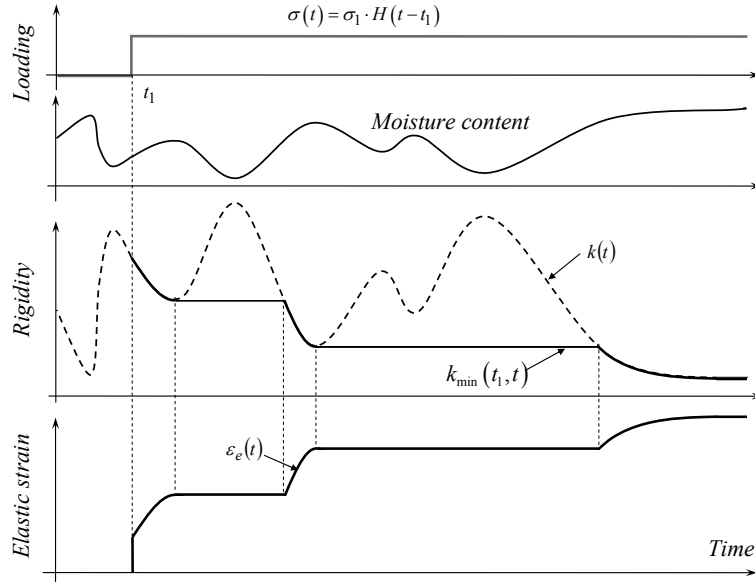


Fig. 2: Response of the hygro-lock model

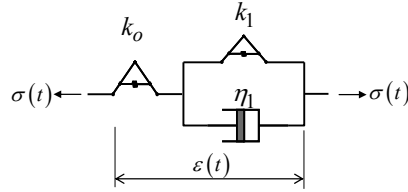


Fig. 3: Kelvin Voigt model with hygro-lock effect

According to the specific hygro-lock effect for each spring, the global behaviour can take this following form:

$$\varepsilon(t) = \int_{0^-}^t J(\tau, t) \cdot \frac{\partial \sigma}{\partial \tau} d\tau \quad \text{with} \quad J(\tau, t) = \frac{1}{k_{\min}^o(\tau, t)} + \int_{\tau}^t \frac{1}{\eta_1(\vartheta)} \cdot \exp\left[-\int_{\vartheta}^t \frac{k_{\min}^1(\tau, \alpha)}{\eta_1(\alpha)} d\alpha\right] d\vartheta \quad (3)$$

This last formulation can be compared with a non linear Boltzman's formulation. In this case, the classical Boltzman's integral is generalized by considering two time integrals. The first expresses loading effects and the second presents the coupling with moisture content effects. Analytic resolutions are performed using an incremental formulation implemented in mathematical formal software. In order to model long term behaviour taking into account more complex and more realistic creep functions this model can be completed by  $N$  hygro-lock Kelvin Voigt cells added with a specific swelling-shrinkage element, Fig. 4. In this context, the relation (3) can be updated as follow:

$$\varepsilon(t) = \int_0^t J(\tau, t) \cdot \frac{\partial \sigma}{\partial \tau} d\tau + \int_0^t \alpha \cdot \frac{\partial w}{\partial \tau} d\tau$$

with

$$J(t, \tau) = \frac{1}{k_{\min}^o(\tau, t)} + \sum_{p=1}^N \int_{\tau}^t \frac{\exp\left[-\int_{\tau}^{\beta} \frac{k_{\min}^p(\alpha) \cdot k_{\min}^p(\tau, \alpha)}{k_{\min}^p(\tau, \alpha) - k^p(\alpha)} d\alpha\right]}{\eta^p(\beta)} d\beta \quad (4)$$

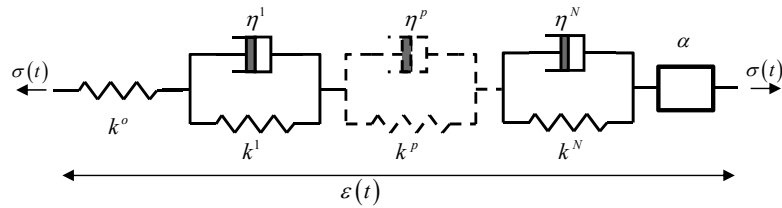


Fig. 4: Generalized Kelvin Voigt model with hygro-lock and shrinkage-swelling effects

#### 4. Application

The mechano-sorptive behaviour, expressions (4), can now be implemented in a formal computation software in order to show some response examples. Its complexity requires a numerical treatment based on a time incremental formulation. An example of application deals with a creep and recovery loading under variable humidity at different levels. The strain response is shown in Fig. 5. Kelvin Voigt properties have been chosen to amplify the decrease of apparent rigidity and creep acceleration during moistening phases. In order to evidence the viscoelastic hygro-lock effects, the swelling-shrinkage response is eliminated. Different stages are highlighted:

1. creep response under low humidity level
2. acceleration of creep process during moistening phase
3. hygro-lock effect during drying phase
4. partial recovery phase under low humidity level
5. acceleration of recovery phase during moistening phase
6. hygro-lock effect during drying phase
7. total recovery at high humidity level

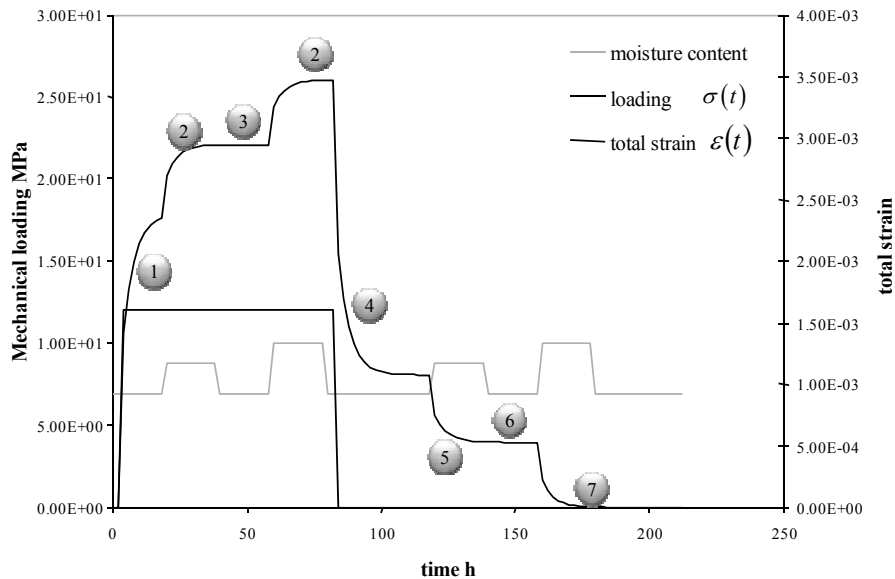


Fig. 5: creep and recovery loading

## 5. Conclusions and perspectives

This work deals with a new mechano-sorptive approach. It is concentrated on mechano-sorptive viscoelastic behaviour. An analytical expression of a pseudo creep function, including the hygro-lock effect, is proposed. This function, generalized for any mechanical load, has been integrated in a generalized Kelvin Voigt model in a new combined activation model. This model has been implemented in an incremental formulation in order to simulate, in the time domain, complex interactions between loading history and moisture content variation.

Firstly, this numerical approach must be coupled with heat and mass transfer algorithm and enables us to simulate the long term response for massive timber elements with moisture content gradients. In a second time, analytic forms can be adapted for beam theory according to stress gradient and swelling-shrinkage effects for no-isostatic configurations.

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# RESEARCH ON THE AGING OF WOOD IN RISH

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## **Abstract**

This paper reviews the research activities of the Research Institute for Sustainable Humanosphere (RISH) on the aging of wood, these are, 1) Collection and identification of wood samples from cultural properties and historical buildings, 2) Characterization of the naturally aging wood, 3) Characterization of the accelerated aging treated wood, 4) Establishment of the database on the wood quality from historical building, and 5) Organizing symposia: Wood Culture and Science series.

## **1. Introduction**

Wood is aged and degraded under the service of any applications. The subsequent changes of the physical, mechanical and chemical properties of wood proceed very slowly and depend on environmental conditions. Japan preserves a great amount of wooden cultural properties and historical buildings and the precise understanding and characterization on the aging of wood is of critical importance for their conservation and restoration.

Since wood culture and science is an interdisciplinary study being involved with many research fields of Museology, Conservation and restoration science, Archaeology, Material science, and Wood science, the collaboration with the network among the various research fields are essential to promote the activities.

Research Institute for Sustainable Humanosphere (RISH) started to collect the wooden samples from cultural properties and historical buildings under the support of the Agency for Cultural Affairs of Japan some ten years ago and now acts as a research core on the aging of wood in Japan in collaboration with the related research organizations, such as National Research Institute for Cultural Properties, Nara (NRICP), National Museum of Japanese History (MJH), and Tokyo National University of Fine Arts (TUFA).

This paper reviews the research activities of RISH on the aging of wood under the research network with the Buddhist sculptors, traditional carpenters as well as the researchers in the fields of art/architectural history, conservation science, and wood science.

## **2. Research Activities of RISH**

The research activities of RISH on the aging of wood are summarized as follows;

- Collection and identification of wood from cultural properties and historical buildings
- Characterization of the naturally aging wood
- Characterization of the accelerated aging treated wood
- Establishment of the database on the wood quality from historical building
- Organizing symposia: Wood Culture and Science series

### **2.1. Collection and identification of wood samples from cultural properties and historical buildings**

RISH collects naturally aging wood samples from the members of cultural properties and historical buildings under the support of the Agency for Cultural Affairs of Japan. The samples are identified the species, then registered at the Xylarium and described with historical, physical, and mechanical

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properties. Fig. 1 shows a part of collections of wood samples from the historical buildings in the Xylarium of RISH [1].



Fig. 1 Collection of wood samples from the historical buildings in the Xylarium of RISH.

## 2.2. Characterization of the naturally aging wood

Since hinoki (*Chamaecyparis Obtusa* Endl.) wood has been mostly used as a construction material in Japan, eight hinoki samples were selected out of naturally aging wood from historical buildings collected in the Xylarium of RISH; those are mainly from Prof. Kohara Collections (ca 200 pieces) and from historical buildings listed in National treasure & cultural properties in Japan and identified by Profs. Hamajima M (MJH), Kubodera S (RICPN) and Yano K (TUFA) from the viewpoints of architectural and art histories.

The oldest samples came from the member of Horyuji temple built in 6<sup>th</sup> century. The year of wood formation of each sample was determined by dendrochronology done by Prof. Mitsutani T (RICPN) and by radioactive carbon chronology by Prof. Imamura M, et. al. (MJH), respectively. The age of the samples ranges from 500 to over 1600 years.

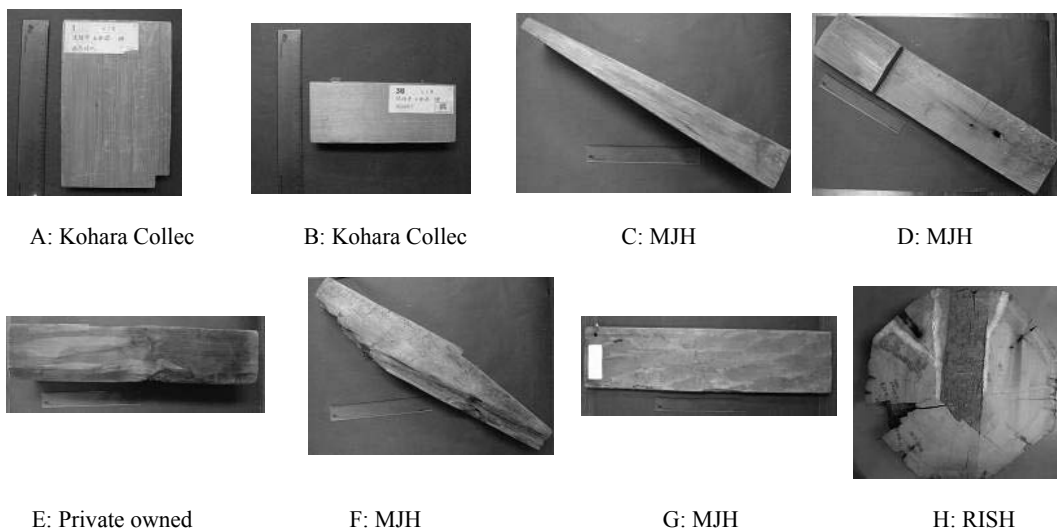


Fig. 2 Hinoki (*Chamaecyparis Obtusa* Endl.) wood samples selected from historical buildings  
Remarks: A-G, samples from Horyuji-temple, H, Senjuji-temple

Specimens cut out from each sample with the dimensions of 2 x 10 x 60 (L and R directions) mm were dried and then the physical (density, equilibrium moisture content, colour properties, etc), mechanical (moduli of rupture and elasticity in bending, yield energy, visco-elastic properties),

thermal (TG, DSC) and chemical (chemical components, FT-IR spectroscopy, X-ray diffractometry) properties are being evaluated. Fig. 3 shows the process flow of the property evaluation.

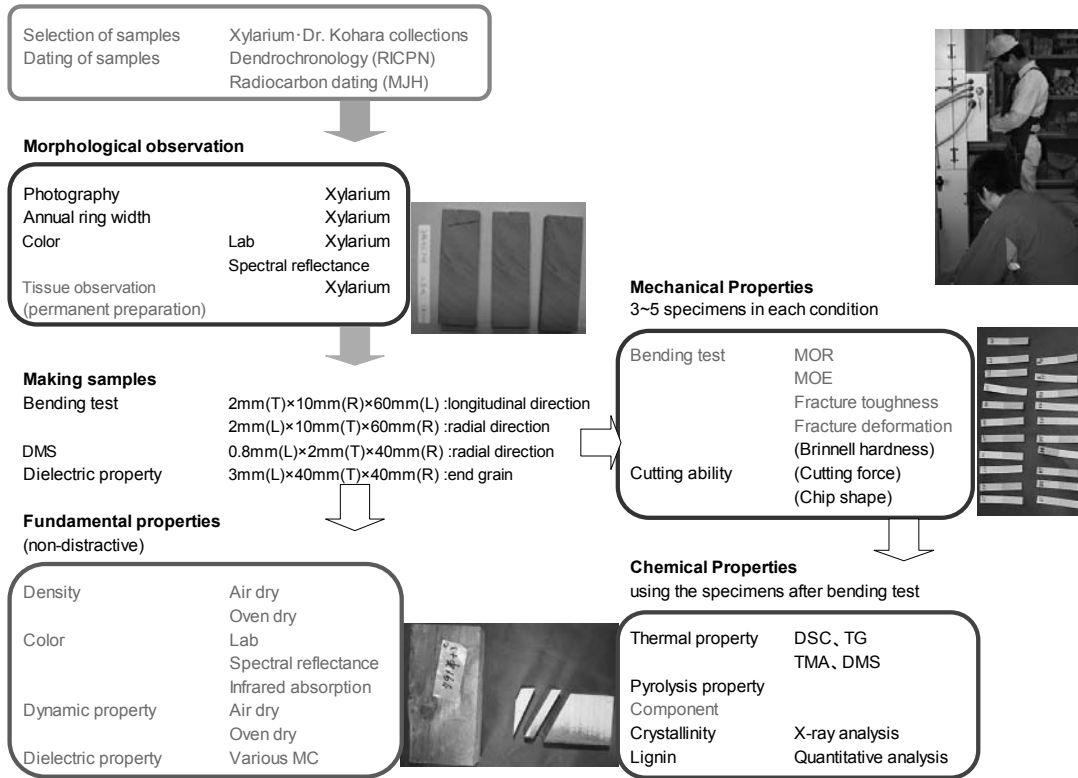


Fig. 3 Process flow for evaluating the naturally aging wood from the cultural properties and historical buildings.

As a part of the results from the physical and mechanical properties, Fig. 4 shows the age of the naturally aging wood samples determined by the dendrochronological dating and their equilibrium moisture contents. The wood substances of aging wood were less accessible to moisture and the equilibrium moisture content of the naturally aging wood decreased with an increase of the aging period, as shown in the figure.

Fig. 5 shows the typical stress-strain curves of the aging wood of hinoki samples in the longitudinal direction. The naturally aging wood samples showed somewhat higher strength and stiffness but less toughness than those of control. It seems that the bending strength in the longitudinal direction of the naturally aging wood samples does not change so much through the aging period over 1600 years, though the values are scattered.

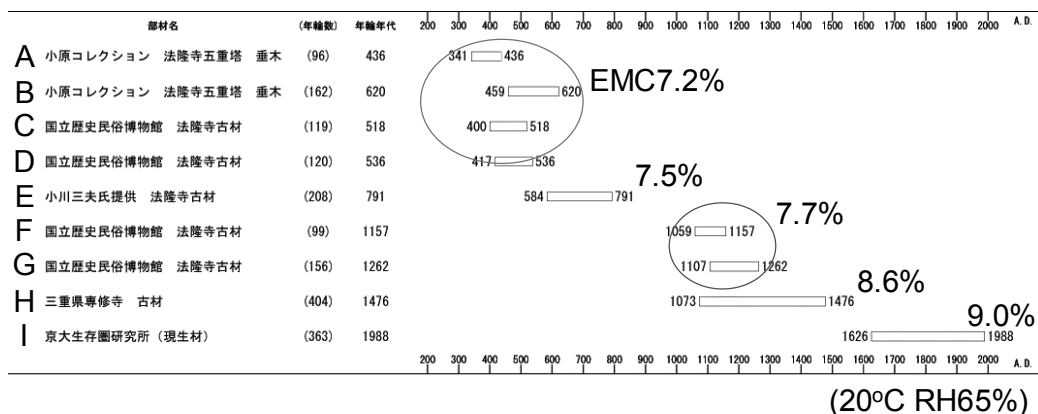


Fig. 4 Age of wood samples determined by dendrochronological dating and their equilibrium moisture contents

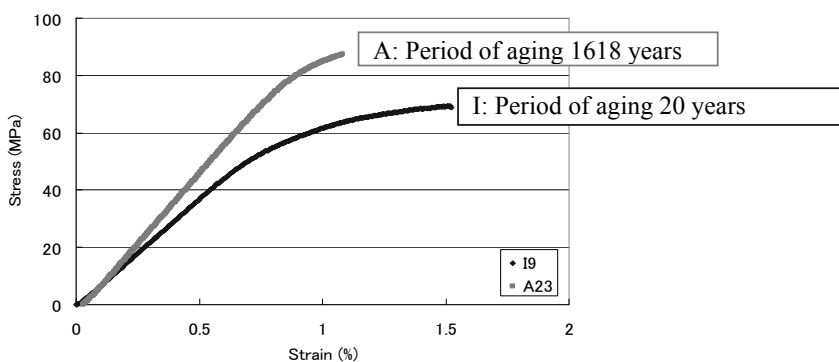


Fig. 5 Typical stress and strain curves of the aging wood of hinoki samples (L-direction).

### 2.3. Characterization of the accelerated aging treated wood

The aging of wood is considered to be a slow thermo-oxidative process caused by oxygen in the air. The hinoki specimens with the highest quality in terms of Buddhist sculptors were subjected to the heat treatment at 90, 120, 150 and 180 C under various treatment times from 0.5 hr to 7 years (to be continued) by normal oven method. Effects of accelerated aging (heat) treatment on the changes of physical, mechanical and chemical properties are now being characterized [2, 3]. The changes of physical, mechanical and chemical properties were analyzed to evaluate the mechanism of the degradation in aging wood. The results from the wood under the heat treatment are compared with those from naturally aging wood.

### 2.4. Establishment of the database on the wood quality from historical buildings

The collected samples and data are listed and preserved in the Xylarium and then partly opened through the on-line access of Humanosphere Database (Xylarium database) on the homepage of RISH as shown in Fig. 6.

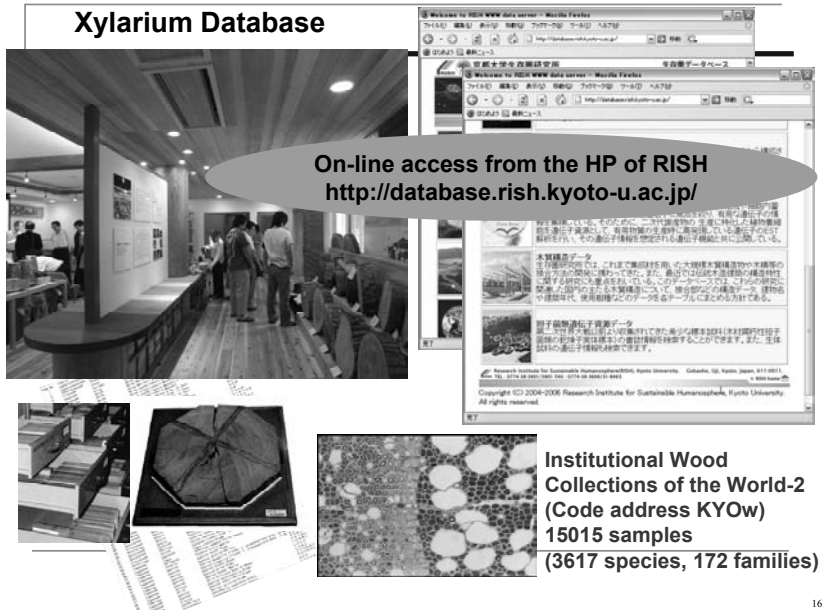


Fig. 6 On-line access of Xylarium database on the homepage of RISH

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# EVALUATION OF THE AGING WOOD FROM CULTURAL PROPERTIES AS COMPARED WITH THE ACCELERATED AGING TREATMENT -ANALYSIS ON COLOR PROPERTIES-

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## Abstract

In this paper, we studied on the color change of wood during aging comparing the naturally aging wood with the accelerated aged wood. Naturally aging samples of Hinoki (*Chamaecyparis obtusa* Endl.) were collected from historical properties. For accelerated aged samples, recently felled down Hinoki specimens were heated in the air at the temperature of 90, 120, 150, and 180°C. Applying kinetic analysis to the measured color data, it was concluded that the color change during natural aging was mainly explained as a mild thermal oxidation process at the ambient temperatures.

## 1. Introduction

In Japan, there are many wooden cultural properties and historical buildings such as Horyuji temple which is the oldest wooden building in the world. The elucidation of aging mechanism of wood is important not only for the preservation and restoration of wooden cultural properties but for the basic research of wood. This is the significant mission of Japan that provides long history of wooden culture.

Though some empirical data suggest that the aging of wood is a mild thermal oxidation at the room temperature [1-3], few papers have reported on the theoretical evaluation and the detail mechanism. On the other hand, sculptors and carpenters have used the color of wood as a criterion of aging. This may be because the color properties directly express chemical change of wood. In this study, color properties were used to evaluate the aging mechanism of wood; color properties of naturally aging wood samples cut out from cultural properties and aged wood samples accelerated by heat treatment were measured and compared. Kinetic analysis was performed based on the relationship between time and temperature.

## 2. Materials and methods

Hinoki (*Chamaecyparis obtusa* Endl.) which is the typical members for traditional buildings and Buddhist sculptures in Japan was chosen as wood species. Eight naturally aging wood samples collected from historical buildings [4] were used as shown in Table 1. The year of wood formation was determined by dendrochronology and radiocarbon dating. The detected years agreed with the old documents. Aging heartwood specimens, being free from bio-degradation and weathering, were cut out with the dimensions of 60mm (L) x 10mm (R) x 2mm (T). For an accelerated aging specimen, 360-year-old air-dried Hinoki from Kiso, Japan was used. Specimens were cut out with the dimensions of 120mm (L) x 20mm (R) x 4mm (T) from the outermost part of the heartwood. The samples were dried at 60°C and then heated by the oven at the temperatures ranging from 90 to 180°C. Table 2 shows the maximum treatment time of the specimens at each treatment temperature.

The color of the specimens was measured with a spectrophotometer (KONICA MINOLTA CM-2600d) under a D65 light source and observer angle of 10 degrees. The sensor head of the spectrophotometer was 8 mm in diameter. The CIELAB color parameters ( $L^*$ ,  $a^*$ ,  $b^*$ ) were used to express the color change. The differences in the lightness ( $\Delta L^*$ ) and color ( $\Delta E$ ) were calculated using the following formulas:

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$$\Delta L^* = L_t^* - L_r^* \tag{1}$$

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \tag{2}$$

where  $L_t^*$  is the lightness of the treated specimen,  $L_r^*$  is the average of 83 untreated specimens as a reference, and  $\Delta a^*$  and  $\Delta b^*$  are the chroma differences based on the untreated specimens. Nine to sixteen and three to five specimens were tested at each condition for naturally aging wood and accelerated aged wood, respectively. Three locations in each specimen were measured, and the average values with standard deviations were calculated.

Table 1: Naturally aging wood samples.

Sample	Temple	Aging time [year]
A	Horyuji, Gojunoto	1618.5
B	Horyuji	1467.5
C	Horyuji, Gojunoto	1548.0
D	Horyuji	1530.5
E	Horyuji	1319.5
F	Horyuji	899.0
G	Horyuji	822.5
H	Senjuji, Mieido	732.5

Table 2: Accelerated aged wood samples.

Treatment temperature [°C]	90	120	150	180
Treatment time [hour]	256~12288*	32~7680	4~960	0.5~120

\*The treatment at 90oC is now in processing and planed up to 61440 hours (approximately 7 years).

### 3. Results and discussion

#### 3.1. Color Changes

Fig. 1 shows the specimens of naturally aging and accelerated aged wood. The color of naturally aging wood and accelerated aged wood were darkened with aging and treatment times, respectively. Fig. 2 shows the changes of the lightness ( $L^*$ ) and the color difference ( $\Delta E$ ) of naturally aging wood and accelerated aged wood. In naturally aging wood,  $L^*$  decreased and  $\Delta E$  increased with aging time. In the case of accelerated aged wood,  $L^*$  decreased and  $\Delta E$  increased with treatment time over all of the treatment temperatures. These behaviors can be described as sigmoidal curves. Similar trends irrespective of treatment temperatures implied that the color changes were caused by the same mechanism.

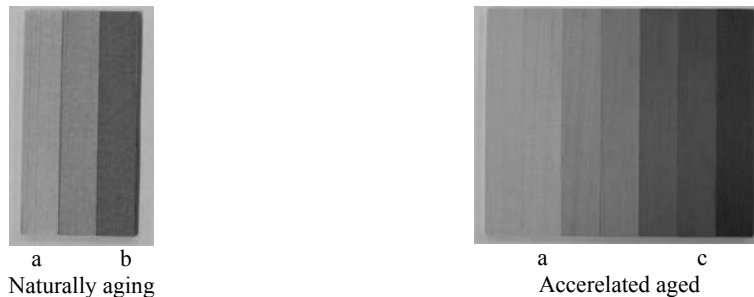


Fig. 1: Color change of natural aging and accelerated aged wood  
a: control (untreated wood), b: aging for 1400 years, c: heat-treated at 120°C for 320 days.

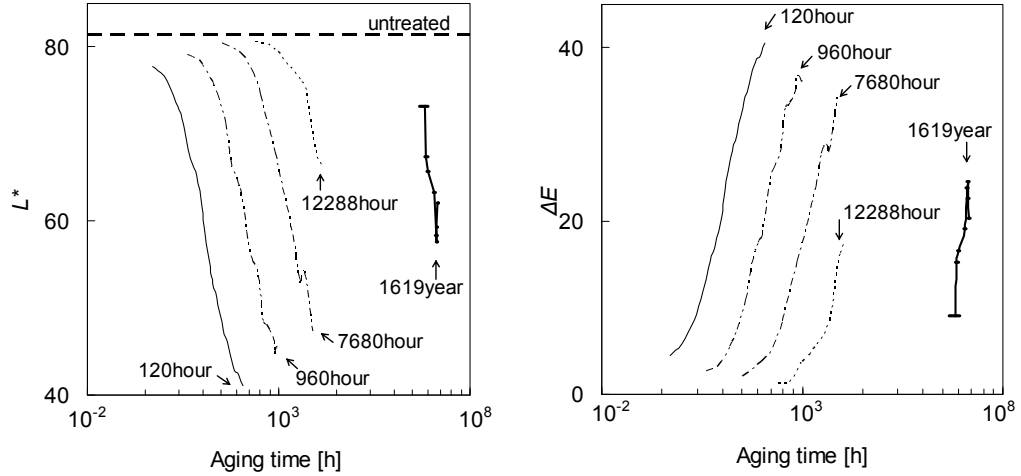


Fig. 2: The relationship between aging time and the lightness ( $L^*$ ) or the color difference ( $\Delta E$ )  
 (.....:90 °C, ----:120 °C, -.-.-:150 °C, —:180 °C, ———:naturally aging).

### 3.2. Kinetic Analysis

The concepts of kinetics are often applied to accelerated aged phenomena of materials to predict the lifetime in natural ageing conditions. It is empirically known that the chemical reaction can be described by Arrhenius equation:

$$k = A \exp\left(-\frac{E_a}{RT}\right) \quad (3)$$

where  $k$  is the rate constant of the chemical reactions,  $A$  the frequency factor,  $E_a$  the apparent activation energy,  $R$  the gas constant and  $T$  the absolute temperature. It represents the dependence of the reaction rate on the temperature and the apparent activation energy. By determining the time which the chemical reaction achieves the arbitrary criterion of properties at different aging temperatures, the reciprocal of treatment temperatures can be plotted versus the logarithm of the determined time. This plot is called the Arrhenius plot. The apparent activation energy is then calculated from the slope of the Arrhenius plot. This allows determining the degradation rate at any temperature by extrapolating the Arrhenius plot.

As mentioned above, the extrapolation procedure generally uses only one processed data point from each accelerated aging temperature curve, eliminating most of the experimental points from analysis. This elimination sometimes makes it difficult to accurately predict the change during natural aging. To determine the activation energy using all of the data, we used the time-temperature superposition (TTSP) method [5]. TTSP is a well-known methodology that is frequently used to describe the mechanical and electrical relaxation behavior of polymers. The Arrhenius approach applying the TTSP method has been successfully used for years in polymers to make predictions of thermal aging at ambient conditions. According to the principle of the TTSP method, both time and temperature are equivalent, i.e. the material parameter values obtained for short times at a given temperature is identical with that measured for longer times at a lower temperature when the curves are shifted on a logarithmic time axis. The curves of the measured material parameter vs. logarithmic loading time at different temperatures can be superimposed by proper scale changes on the time axis. The shift distance along the logarithmic time axis is called the time-temperature shift factor  $a_T$  given by

$$a_T = t_{ref} / t_T \quad (4)$$

where  $t_{ref}$  is the test time at a reference temperature  $T_{ref}$ , and  $t_T$  is the time required to give the same response at the test temperature  $T$ . Combining Eqs. (3) and (4) gives

$$a_T = \exp\left[\frac{E_a}{R} \left(\frac{1}{T_{ref}} - \frac{1}{T}\right)\right] \quad (5)$$

where both  $T$  and  $T_{ref}$  are absolute temperatures. By describing the plot of the reciprocal of treatment temperatures ( $1/T$ ) vs. the logarithm of the shift factor ( $\ln(a_T)$ ), it is also available to calculate  $E_a$  and predict the properties change in the ambient conditions [5-8].

To determine the shift factors, the temperature of 180°C was chosen as the reference temperature  $T_{ref}$ . Some regressions were performed for  $L^*$  and  $\Delta E$  at 180°C expressed as a function of  $t$ . In other words, first- and second-order polynomial expressions, exponential function and some sigmoidal functions were applied to the regression. Judging from the coefficients of determination, the logistic function described as follows was used.

$$y = \frac{a}{1 + b \exp(-cx)} \quad (6)$$

where  $y$  is a color property,  $x$  is  $\log t$ , and  $a$ ,  $b$ , and  $c$  are constants. Because the logistic function requires 0 as the initial  $y$  value,  $L^*$  was analyzed after conversion to the relative reduction [%] as follows:

$$rL_t^* = \frac{100(L_r^* - L_t^*)}{L_r^*} \quad (7)$$

where  $L_t^*$  is the lightness of the treated specimen and  $L_r^*$  is the average of 83 untreated specimens as a reference. The parameters of the logistic function ( $a$ ,  $b$ , and  $c$ ) were estimated using the least squares method for the color properties,  $rL_t^*$  and  $\Delta E$ . The regression equations with detected parameters were the following.

$$rL_t^* = \frac{56.2}{1 + 9.92 \exp(-2.11 \log t)} \quad (8)$$

$$\Delta E_t = \frac{47.5}{1 + 7.94 \exp(-1.89 \log t)} \quad (9)$$

Then the measured color properties of the lower treatment temperatures than 180°C were superposed by  $a_T$  on the time axis. The proper  $a_T$  was determined by the least square method. Fig. 3 shows the superposed data with the regression curve and  $a_T$  at each treatment temperature. Well-superposed curve indicated that the color change was caused by the same reaction irrespective of treatment temperatures. The apparent activation energy was calculated from the relationship between reciprocal of treatment temperatures ( $1/T$ ) and logarithm of the shift factor ( $\ln(a_T)$ ), and then color change in the ambient condition was predicted by extrapolation to the temperature of 15°C, the annual mean air temperature in Japan. The Arrhenius plot of  $L^*$  is shown as open circles (○) in Fig. 4 and the plot of  $\Delta E$  shown the similar features. The regression line showed a good correlation efficient ( $\geq 0.99$ ). The apparent activation energy ( $E_a$ ) calculated from the slope was  $1.17 \times 10^5$  J/mol in  $L^*$  and  $1.14 \times 10^5$  J/mol in  $\Delta E$ . These values were similar to the  $E_a$  obtained from other properties such as the reduction of weight, MOE, MOR and so on ( $1.1 \sim 1.3 \times 10^5$  J/mol) [1-3]. The shift factor of naturally aging data shown as a pasted circle (●) was positioned near to the regression line. These results suggested that color change during natural aging was mainly explained as thermal oxidation. Lower  $a_T$  than the extrapolation meant that the natural color change was somewhat faster than the prediction and indicated that, in addition to thermal oxidation, the color change could be accelerated by other factors such as moisture in the air, repetition of drying and wetting, and repetition of hot and cold.

#### 4. Conclusion

$L^*$  decreased and  $\Delta E$  increased during both the natural and accelerated aging process. The changes of  $L^*$  and  $\Delta E$  during the accelerated aging were described by the logistic function. Applying the kinetic analysis to the color data was valid. According to the kinetic analysis using the TTSP method which allows using all of the data, the color change during the natural aging was explained as the thermal oxidation. The apparent activation energy calculated from  $L^*$  and  $\Delta E$  of the accelerated aged wood using the shift factor was  $1.17 \times 10^5$  and  $1.14 \times 10^5$  J/mol, respectively. The data of naturally aging wood was approximate to the Arrhenius plot.



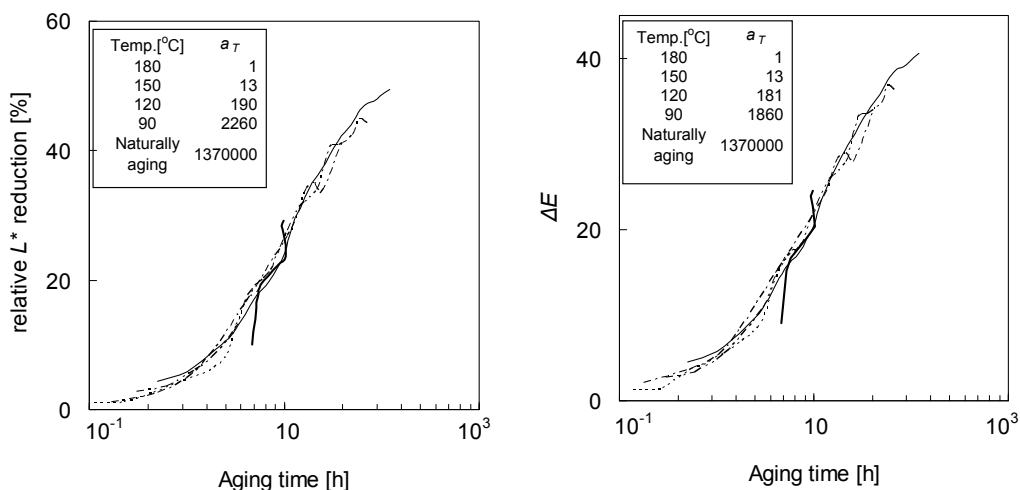


Fig. 3: Time-temperature superposition of the color data from Fig. 2 at a reference temperature of 180°C (-----:90°C, - - - - -:120°C, - · - · - · -:150°C, ———:180°C, ———:naturally aging).

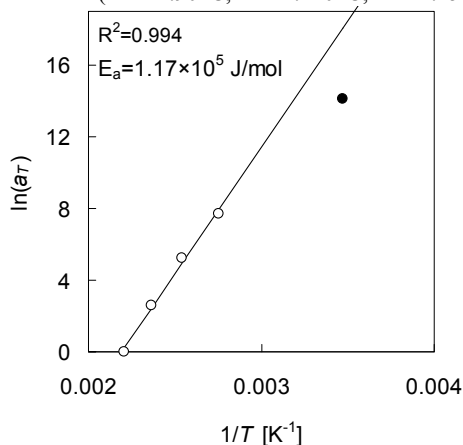


Fig. 4: Arrhenius plot of  $\ln(a_T)$  as a function of  $1/T$  calculated from  $L^*$ . (○: shift factor of accelerated aged data, ●: shift factor of naturally aging data, —:regression line to shift factors of accelerated aged data,  $E_a$ : apparent activation energy obtained from slope of regression line)

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# STRENGTH AND MOE OF POPLAR WOOD (*POPULUS ALBA* L.) ACROSS THE GRAIN: EXPERIMENTAL DATA

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## Abstract

The paper is centred on the mechanical characteristics of poplar wood (*Populus alba* L.), particularly on selected short term loading tests (strength and modulus of elasticity) across the grain. The reason for such work arises from the necessity to deepen the knowledge of the mechanical behaviours of this species to apply it to a better conservation of painted panels. Tension and compression tests perpendicular to the grain, at three different wooden moisture content values, are described. The results show no evident differences at various moisture content values and for different loading tests.

## 1. Introduction

During the Renaissance period (XIII-XV sec.) many artists, such as Giotto, Leonardo da Vinci, Botticelli, Raffaello and Caravaggio (just to cite a few examples), painted their masterpieces on wooden supports. In the Tuscany area most of these supports were made of poplar wood (*Populus alba* L.) because of its availability, its technological characteristics and easy processing [6]. By the time elapsing many problems in conservation of the artworks have developed. Painted panels are really complex objects because of the heterogeneity of the used materials, such as wood, gypsum, animal glues, colours and varnish [6]. Each one contributes to the conservation (or deterioration) of the objects according to its physical or mechanical behaviour. Actually the painted panels, that today we admire in museums or churches, show damages both to the painted layers, such as craquelure or bucklings, and to the wooden supports, such as cupping and cracks [1,2] and the anatomical direction are specially important because the perpendicular to grain ones contribute very much to the phenomena. These damages are due to the climatic conditions cyclic variations and the contemporary presence of mechanical restraints on the painting panels themselves, particularly the crossbeams and the frame, if it is present. When the climatic conditions vary, particularly the relative humidity of the air (RH), also the moisture content (MC) of the painting panels changes, trying to reach the new equilibrium value. The wooden support is usually restrained, as already explained, and it is not able to deform. The inner forces, due to the restraining, produce loss of dimension or cracks. In order to deepen the basis of the mechanism that is responsible of these damages on the wooden supports of the painted panels, a wide research has carried out at DISTAF, and a part of it is described in this paper. The main aim of the whole work is the physical and mechanical characterization of new poplar wood, measuring the density, the restraining/swelling coefficient, the diffusion coefficient, the strength and MOE values, the visco-elastic deformation, the compression set shrinkage across the grain to apply such knowledge to a better conservation of painting panels. This paper is centred on the description of the utilized methods and the obtained results of short term loading tests according to three different equilibrium moisture content.

## 2. Materials and methods

The tested specimens are obtained by new poplar wood (*Populus alba* L.). They are clean, with straight grain, or as much as possible, and cut as cubes (30x30x30 mm) in order to represent a small “unit” of a painted panel. Specimens are cut according to three different directions: perfectly radial (identified by the abbreviation 0 in the following graphs), perfectly tangential (identified by the abbreviation 90 in the following graphs) and 45, that is the intermediate direction between the others (identified by the abbreviation 45 in the following graphs). After cutting, the specimens are put in three different boxes in order to be equilibrated at three different moisture contents. In each box a unique climate is realized: dry climate (30% RH and 30°C), normal climate (65% RH and 20°C) and

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humid climate (85% RH and 30°C). Each one represents a particular state: the humid and dry climates are the extreme values in order to magnify the phenomena, while the normal climate represents the standard one. When the specimens get equilibrated each set reaches the following equilibrium moisture content (EMC): dry specimens 6%, normal specimens 12% and humid specimens 15%.

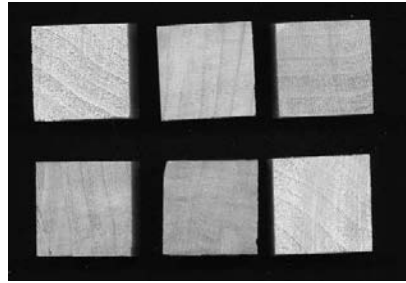
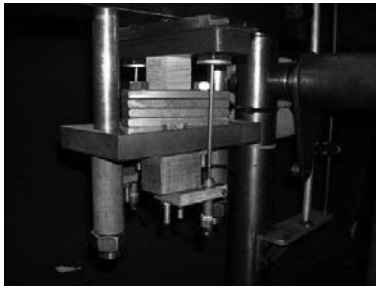
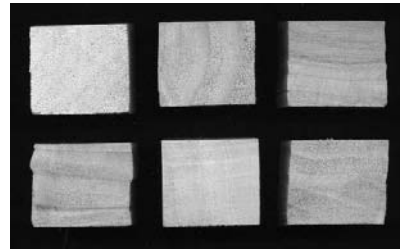


Fig. 1: specimens to be tested.

Tests in tension and compression have been carried out. The specimens have been set between two plates, and a force is applied to realize the compression tests. The compression across the grain do not cause a definite fracture of the material, because as it is well-known wooden cells deform without breaking. Therefore it is necessary to determine a clear strength limit and value, even if it is a calculated instead of a measured one. It has been derived according to the European standard UNI EN 408.

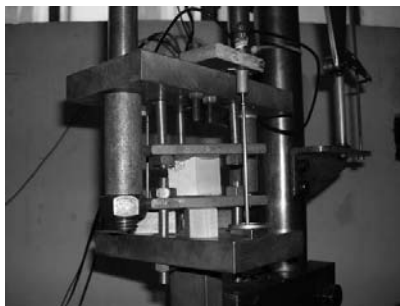


(a)

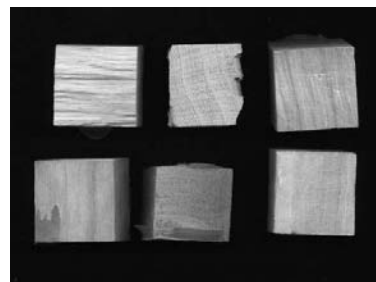


(b)

Fig. 2: compression tests (a), specimens tested in compression (b). The specimens have been glued, using an epoxy resin, on two plates to realize the tension test. The tension tests has been carried out till the fracture of the specimens, so the strength value is measured.



(a)



(b)

Fig. 3: tension tests (a), specimens tested in tension (b)

Both in the tension and compression tests the deformation is measured trough two displacement transducers, one on each side in order to obtain a medium value that could represent the deformative behaviour of the specimen.

### 3. Results

#### 3.1. Compression tests perpendicular to the grain

The calculated strength values are grouped in an homogeneous way (see fig. 3). They are between 2,50 and 5,50 MPa, 3,96 MPa on the average. No influence of the anatomical directions is evident. Analysing the strength values according to the EMC, we observe that as the moisture content increases, the strength decreases.

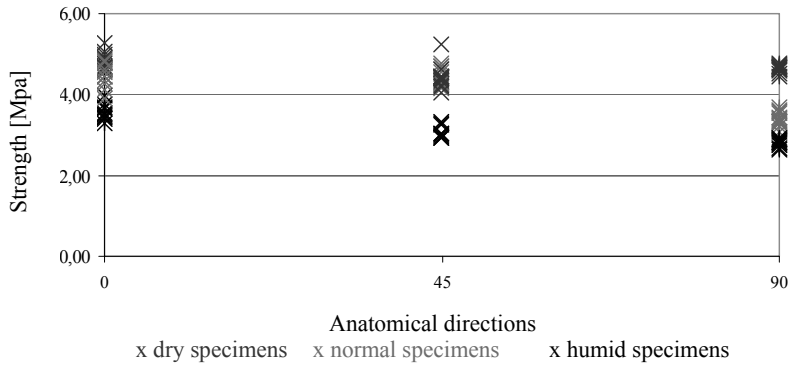


Fig. 3: Strength vs loading direction

The modulus of elasticity (MOE) is the measure of the material stiffness: the bigger the value is, the stiffer the material is (see fig. 4). The calculated values are between 150 and 750 MPa, on the average 328 MPa. As it is evident by the graph (fig. 4), the MOE values are related to the moisture content of the specimens: as the MC increases, the stiffness decreases. Moreover it is greater in radial direction ( $> 400$  MPa for dry specimens) and smaller in T direction ( $< 300$  MPa). A further comment is requested for the radial tests. Indeed the MOE values show a greater variability, probably due to influence of the rays. In the radial tests, the rays are parallel to the loading direction, and it leads to a biological non-homogeneity of the specimens, that affects the MOE values. The presence of rays is not significant for the other tests that give more homogeneous results.

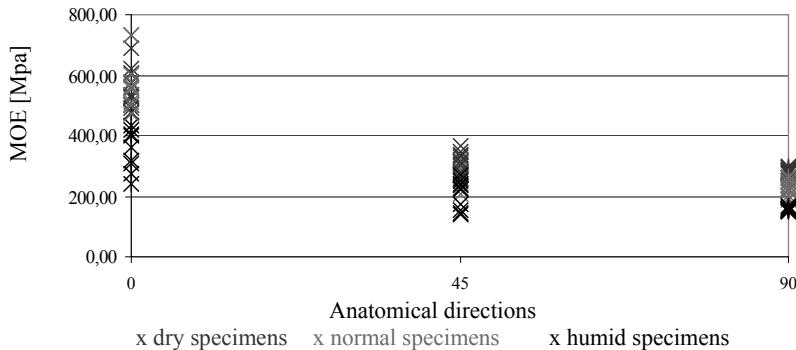


Fig. 4: MOE vs loading direction

#### 3.2. Tension tests perpendicular to the grain

The strength values are measured (see fig. 5), and they are between 2,00 and 6,20 MPa, 3,30 MPa on the average.

As shown for the compression tests (see § 3.1), the biological variability of the specimens is evident for the radial direction. Once again it depends on the direction of the rays compared to the loading direction. Another aspect needs to be discussed: the normal specimens show greater values than the dry and humid ones. Actually the largest values are expected for the drier specimens. This behaviour

has been pointed out by many authors [3,4,5], and it could be due to a chemical or physical deterioration of the wood as a consequence of low moisture content ( $EMC \leq 6\%$ ).

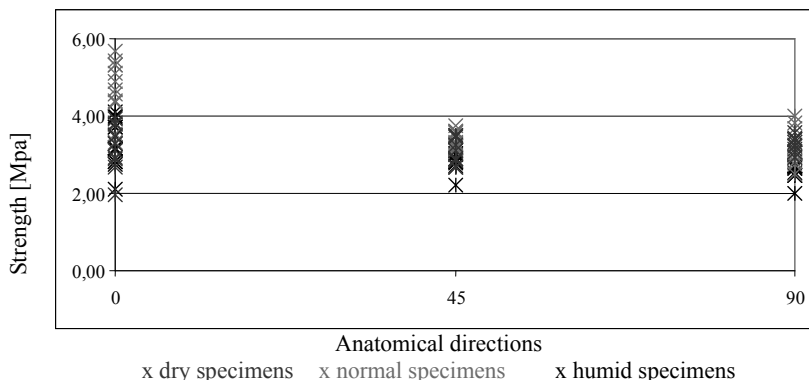


Fig. 5: Tensile strength vs loading direction

The MOE values (fig. 6) are between 150 and 550 MPa, on the average 288 MPa. The values are related to the EMC, exactly as for the compression tests (see § 3.1), as the moisture content increases the stiffness decreases. It is greater in radial direction ( $> 300$  MPa) than in tangential one ( $< 300$  MPa), and the variability along the radial direction is still present.

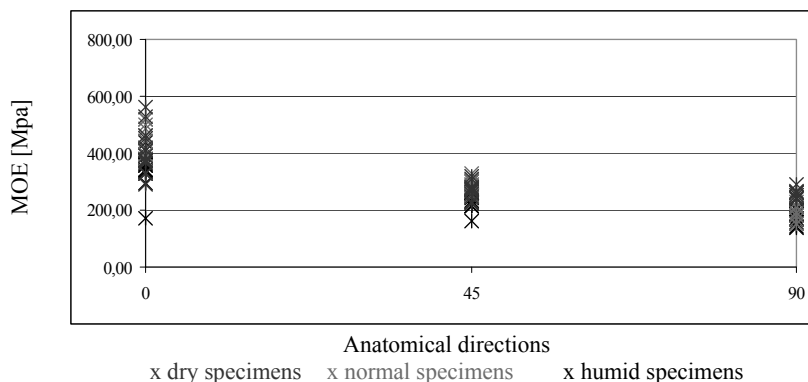


Fig. 6: MOE vs loading direction

#### 4. Conclusions

The tests have shown no evident differences when comparing all the results. The moisture content does not influence significantly the strength and the modulus of elasticity. The same can be said for the anatomical directions. From this homogeneity arises the choice to calculate average values, both for strength and MOE, including the different EMC and loading directions (see Table 1).

Table 1: strength and MOE average values.

	Compression values	Tension values
MOE	328 MPa	288 MPa
$\sigma$	3,96 MPa	3,30 MPa

A comment, that could be a future investigation of the wood behaviour, is requested for the strength value of normal specimens in tension tests, that is greater than the others.

### **Acknowledgements**

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# PHOTODEGRADATION AND THERMAL DEGRADATION OF OUTDOOR WOOD

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## Abstract

In this study, wood samples were exposed to thermal treatment in dry- and humid conditions and to light irradiation. The results showed that neither xenon lamp nor mercury lamp can simulate properly the effect of sunlight. Light irradiated samples put behind an aluminium plate also suffered considerable chemical changes, measured by infrared technique and colour measurement. Degradation of lignin was inconsiderable in the shadow. Carbonyl groups generated by sunlight were partly contrary to those generated in shadow. The latewood suffered considerably less photodegradation than earlywood. Thermal degradation was much greater in humid condition than in dry condition.

## 1. Introduction

The main factor that causes the greatest changes in the surface properties of wood during outdoor exposure is sunlight. Resistance of degraded surface to water movement and fungi attack decreases by weathering. Careful investigation of such wood degradation is difficult using outdoor exposure, because weather conditions are not repeatable, and there are many other factors beside sunlight influencing the results. Therefore light-induced degradation of wood is usually investigated under artificial conditions. The most frequently used artificial light source is the xenon lamp. Because of the thinning of earth's ozone layer, nowadays more ultraviolet (UV) radiation reaches the earth's surface than before. Therefore the UV B wavelength region (280-315 nm) has to be taken into consideration as a strong degrader of biological molecules. Since xenon lamps have no emission in the UV B region, a new light source (or combination of light sources) should be introduced to simulate the sunlight.

Besides photodegradation, surface thermal degradation has to be taken into account, too. Surface temperature of sun-irradiated outdoor wood can reach 60°C, or even 90°C if it is dark enough. This local temperature is high enough to produce chemical degradation. In the scientific literature, similar investigation can hardly be found.

To protect our outdoor wooden cultural heritages, first of all the mechanism of photodegradation and thermal degradation of wood has to be clearly understood.

## 2. Experimental

The following hardwood species were investigated: beech (*Fagus crenata* Carr.), black locust (*Robinia pseudoacacia* L.), poplar (*Populus x caulesceus*), and zelkova (*Zelkova serrata* Thunb.), the softwood samples were: Japanese cedar (*Cryptomeria japonica* D. Don), Japanese cypress (*Chamaecyparis obtuse* Conf.), Scots pine (*Pinus sylvestris* L.), spruce (*Picea abies* Karst.) larch (*Larix decidua* L.) and as a unique wood bamboo (*Phyllostachys pubescens*) also was investigated. Planed surfaces with a tangential orientation were prepared. The sample size was 50x10x2 (mm). Samples of different series were prepared using the same board. All species were represented by a series of 2 samples, and 5 points of fixed location were measured. The data presented in this study are the average of 10 measurements. The natural sunlight irradiations were carried out between 5th of May and 19th of August, 2003 (air temp. varied 16-41 °C, max. RH 80% and the daily average of total solar power density were between 436-459 W/m<sup>2</sup>). In order to determine the effect of the sunlight alone, samples were exposed outside only on sunny days. The other series of specimens were irradiated with a xenon lamp, in a commercial chamber (SX-75: Suga Test Instruments Co. Ltd., Tokyo). A mercury lamp as a strong UV light emitter was also used to irradiate specimens (HAL 800NL, installed into a KBP.659 Nippon Denchi Co. Ltd. chamber.). Total irradiation time was 200

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hours for sunlight and xenon light, and 20 hours for mercury light. One part of the samples was covered with a 2 mm thick aluminium plate in all experiments. These surfaces were used to determine the thermal degradation caused by the applied irradiation.

Earlywood and latewood surfaces were investigated parallel in order to find the photodegradation differences. To determine the thermal degradation solely, samples were stored in total darkness at 90°C under both dry and humid conditions up to 36 days.

The colour and the infrared (IR) spectra of wood specimens were measured before and after irradiation. Exposures were interrupted after 5; 10; 20; 30; 60; 120 and 200 hours to obtain colour and IR data. Colour measurements were carried out with a colorimeter (SE-2000 Nippon Denshoku Industries Co. Ltd., Tokyo).  $L^*$ ,  $a^*$ , and  $b^*$  colour co-ordinates were calculated based on  $D_{65}$  light source. The IR spectra measurements were performed with a JASCO FTIR double beam spectrometer equipped with a diffuse reflectance unit (JASCO: DR-81). The resolution was  $4\text{ cm}^{-1}$  and 64 scans were obtained and averaged. The background spectrum was obtained against an aluminium plate. The spectral intensities were calculated in Kubelka-Munk (K-M) units. Two-point baseline correction at  $3800\text{ cm}^{-1}$  and  $1900\text{ cm}^{-1}$  was carried out. Difference spectra were calculated by subtraction of no irradiated from the irradiated.

### 3. Results and discussion

Ultraviolet (UV) photodegradation of wood is a widely investigated phenomenon [1] [2] [3], however thermal degradation occurring during photodegradation is hardly investigated. It is usually thought to be negligible by researchers. New results show, that the thermal degradation of wood is faster if the sample got UV irradiation previously [4] [5] [6]. This finding highlights that this problem needs further investigation. Therefore, this study discusses these two phenomena separately. One part of all samples was covered by an aluminium plate to filter the direct UV radiation. With this method, only the minimum of the thermal degradation could be determined. The real surface temperature is higher than behind the aluminium plate and the thermal degradation depends on the temperature exponentially.

The colour changes of beech wood are presented in Fig. 1. The darkening caused by sunlight was fast in the first 10 hours. After 30 hours the lightness decrease was moderate but it decreased linearly.

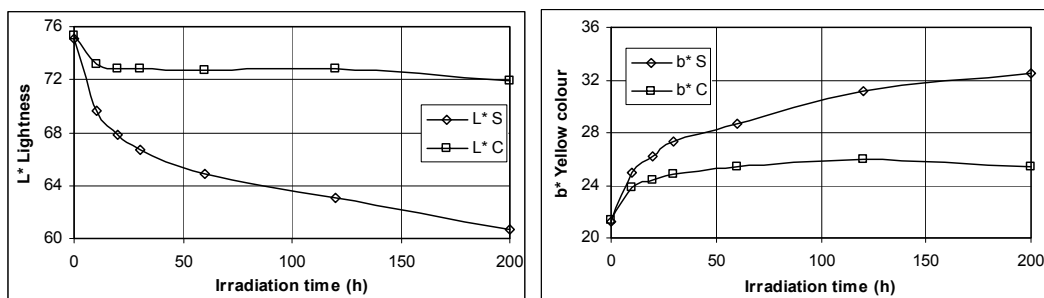


Fig. 1 Colour change of beech wood caused by direct sunlight radiation (S) and behind an aluminium plate (C)

The covered area suffered considerable lightness decrease during the first 10 hours of exposure, and then the lightness remained constant. The yellowing (increase of  $b^*$  colour co-ordinate) showed similar changes but in the opposite direction. The rates of the changes were different. The lightness decrease of the exposed area was 2.6 times higher than that of the covered area after 10 hours exposure. The rate of the yellowing was only 1.5.

The colour change is a sensitive indicator of photo- and thermal degradation of wood but it does not show the chemical changes. The IR spectrum gives information about the chemical structure. The difference IR spectra of spruce earlywood are presented in Fig. 2. The positive peaks represent the absorption increase while the negative peaks represent the absorption decrease caused by the treatment. The band assignment can be found in a previous work [1]. After light irradiation, the carbonyl band between  $1680$  and  $1900\text{ cm}^{-1}$  increased and the peak of the aromatic skeletal vibration



arising from lignin ( $1510\text{ cm}^{-1}$ ) decreased together with the guaiacyl vibrations at  $1275\text{ cm}^{-1}$ . There is an absorption decrease at  $1174\text{ cm}^{-1}$  because of the ether splitting.

Since there is no absorption change at  $1510\text{ cm}^{-1}$ , the lignin did not suffer degradation in the shadow. The aluminium plate protected it against light irradiation. The ether band was split although

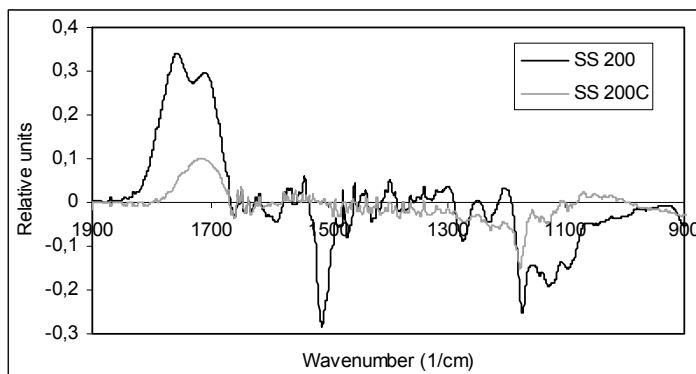


Fig. 2 Difference IR spectra of spruce earlywood (S) after 200 hours sunlight (S) irradiation and behind an aluminium plate (C)

it is about half part compared to the effect of sunlight. The carbonyl groups generated in shadow are different to those generated by sunlight. The band at  $1764\text{ cm}^{-1}$  (unconjugated ketones, carboxyl groups and lactones) is completely missing. It means that the unconjugated ketones, carboxyl groups and lactones are generated after the splitting of the aromatic ring of lignin. The number of generated carbonyl groups absorbing around  $1720\text{ cm}^{-1}$  is much less in shadow than in the exposed area. It can be concluded, that the outdoor wood suffers degradation in shadow, too. This chemical change is different from that caused by the sunlight. Our results show the importance of thermal degradation during outdoor exposure.

Experiments in total darkness were carried out to determine the effect of thermal degradation. Samples were placed in two desiccators, both over  $\text{P}_2\text{O}_5$  (dry condition) and over water (wet condition). The desiccators were placed in a drying chamber at  $90^\circ\text{C}$ . The results of black locust wood are presented in Fig. 3. In dry condition, samples colour shifted towards red (increase of  $a^*$  co-ordinate). Under wet condition the colour change was more intensive. The red colour co-ordinate increased and the yellow ( $b^*$ ) decreased. After 120 hours treatment, the red component also decreased. (Here, the data are presented up to 200 hours, but this test was continued up to 36 days.) The saturated vapour removed the coloured degradation products from the samples. This removal is confirmed by the IR spectra as well. (These spectra are not presented here.) The decrease of  $b^*$  shows the removal of the water-soluble extractives. Black locust is rich in extractives creating its unique yellow colour. The decrease of  $b^*$  was less pronounced in case of the other samples. Real weather conditions are between the simulated dry and wet conditions. Air humidity plays an important role in thermal degradation.

Xenon lamps have no emission in the UV B region while the emission spectrum of mercury lamp has wider range of UV light. To simulate the effect of this UV B radiation, a mercury lamp may be used. The disadvantage of the mercury lamp is that it emits in UV C region as well, however, the UV C radiation of sun does not reach the surface of the earth. Both light sources were tested. It is difficult to compare the light sources because its light emission power and spectrum are individual. These data of light sources are usually partly presented by the suppliers. That is why the absolute intensities of the generated changes are not suitable for comparison. The tendency of changes has to be compared.

The colour changes caused by the artificial light sources are presented in Fig. 3. Both lamps had the same electric power (300 Watt). The mercury lamp produced continuous and parallel yellow and red colour changes. Colour shift occurred only after 30 hours of irradiation in case of xenon lamp. The control samples were stored in the laboratory on an open place ( $20^\circ\text{C}$ ; 45-50% RH). Their colour also shifted towards red a little (marked as "Nonirr" in Fig. 3.).

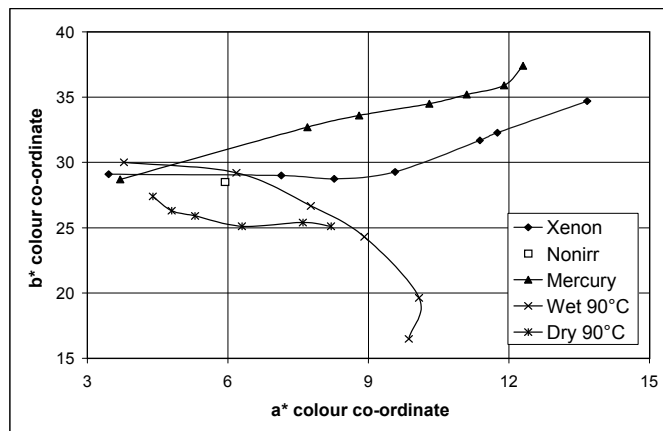


Fig. 3 Colour change of black locust wood caused by different treatments (treatment duration was 200 hours)

For comparison of light source, IR spectra give more detailed data. These different spectra of poplar wood are presented in Fig. 4-5. The large positive peaks in Fig. 5 around  $1100\text{ cm}^{-1}$  do not represent absorption increase. This is a distortion caused by the applied Kubelka-Munk theory. In this experiment, the electric power consumption of mercury lamp was about 10 times higher than that of xenon lamp, therefore the irradiation times are different. The effect of the two irradiation types (*i. e.* sunlight and xenon light) differed on peaks in the  $1900$  and  $1680\text{ cm}^{-1}$  wavenumber region. Usually two peaks develop in this region during the exposure of wood to UV radiation. The xenon light causes a slightly greater increase in the peak at  $1716\text{ cm}^{-1}$  wavenumber than at  $1775\text{ cm}^{-1}$ . As a single measure of the deviation between these two peak intensities, their ratio was calculated on the basis of the higher wavenumber. The  $1716\text{ cm}^{-1} / 1775\text{ cm}^{-1}$  ratio after the exposure to xenon light was 1.05. On the other hand, exposure to sunlight for 200 hours created the opposite change in intensity, the  $1716\text{ cm}^{-1} / 1775\text{ cm}^{-1}$  ratio was 0.81. These ratios changed during exposure. In the first hours of exposure, deviation in the peak intensity ratio increased, mainly because of the rapid increase of the peak at  $1716\text{ cm}^{-1}$  in case of xenon light. After 60 hours, the deviation decreased. The increase of the band at  $1716\text{ cm}^{-1}$  was rapid at the beginning of xenon light irradiation and stayed the same after 60 hours of irradiation. In contrast this band increased continuously during the 200 hours sunlight irradiation. Our experiments suggest that the xenon light can simulate the effects of sunlight only after long-term irradiation.

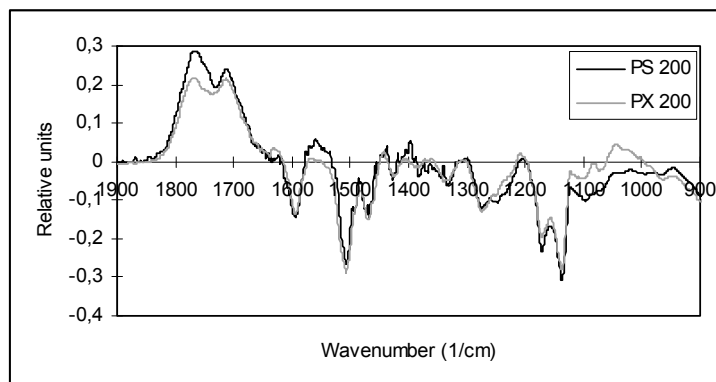


Fig. 4 Difference IR spectra of poplar wood (P) after 200 hours sun (S) and xenon lamp (X) irradiation

The differences between mercury lamp and sunlight irradiation can be seen mainly in the carbonyl groups region (Fig. 5). In the unconjugated carbonyl groups region, the mercury lamp created a large united peak with a maximum at  $1750\text{ cm}^{-1}$ . In contrast, the sunlight created two well separated peaks.

These two peaks were also visible in the case of mercury light irradiation up to 1 hour of irradiation. After 3 hours irradiation they were completely integrated within one peak. In the conjugated carbonyl groups region ( $1620-1690\text{ cm}^{-1}$ ), absorption decrease was found only in the case of mercury light irradiation. Probably it is created by the UV C part of irradiation. It can be concluded that the mercury lamp is not able to simulate the degradation caused by sunlight. Further investigation is needed to clarify the effect of mercury lamp if the UV C part is filtered out.

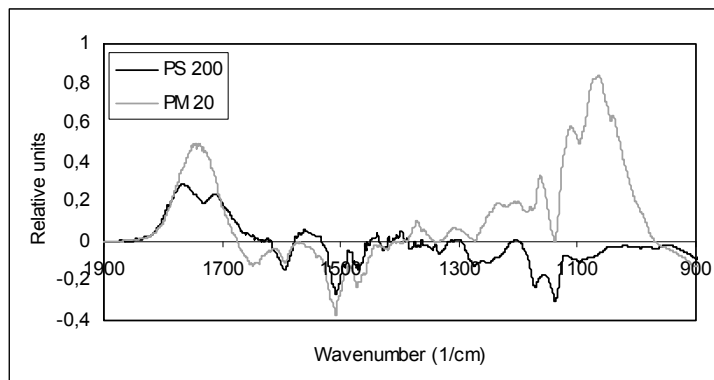


Fig. 5 Difference IR spectra of poplar wood (P) after 200 hours sun (S) and 20 hours mercury lamp (M) irradiation

The sensitivity of earlywood and latewood within the heartwood and sapwood was investigated, too. The results of 120 hours sunlight irradiation of Japanese cedar are presented in Fig. 6. The obvious changes are visible in the aromatic ring region around  $1510\text{ cm}^{-1}$  and in the carbonyl group region ( $1680-1850\text{ cm}^{-1}$ ). The latewood suffered the same changes independently on its position within the wood. The smallest degradation was found in the case of larch latewood. The earlywood suffered much greater degradation than the latewood. Comparing the sensitivity of the two types of earlywood, it can be seen that within the sapwood the sensitivity of earlywood is a little greater than within heartwood. It means that the latewood surface gives protection for the other layers in the case of outdoor roundwood.

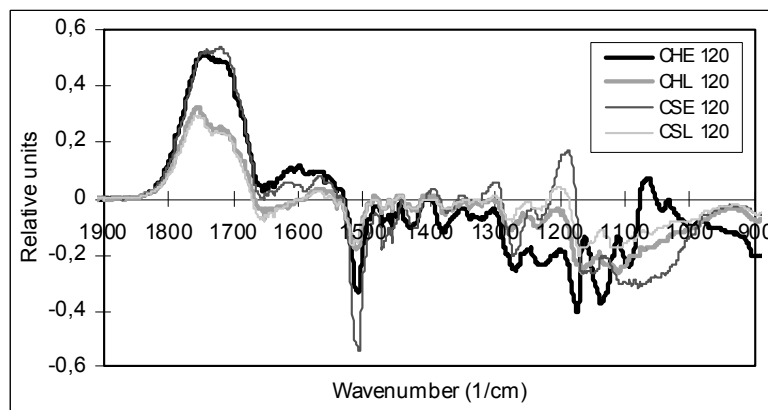


Fig. 6 Difference IR spectra of Japanese cedar (C) heartwood (H), sapwood (S), earlywood (E) and latewood (L) after 120 hours sunlight irradiation

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## (B) BIODETERIORATION



# ROMANIAN WOODEN CHURCHES WALL PAINTING BIODETERIORATION

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## Abstract

The Romanian wooden churches are a category of monuments which belong to the great family of European wood architecture. The inside decoration based on tempera painting technique is the most vulnerable part of these monuments. Based on the biological examination for a number of over 300 wooden churches, the paper presents the causes of the decay for this artistic component. The most extended and irreversible decays are the infiltrations of rain water, followed by biological attacks produced by fungi and insects. The most important species of fungi and insects are presented, their localization and peculiarity of attacks. We have concluded with some results and proposals to prevent this kind of decay.

## 1. Introduction

Wooden churches are a category of monuments widely spread all over the space inhabited by the Romanians [5]. They are to be found in great numbers not only in Maramureș, but also in Transylvania, Moldova, Muntenia, Oltenia, Banat and Crișana (Fig. 1-2). Being considered a specific achievement, they belong to the great family of European wood architecture [5]. Out of the 1250 wooden churches declared historical monuments, 8 are included in the UNESCO Patrimony (Surdești, Plopiș, Bârsana, Budești, Desești, Rogoz, Ieud Deal, Poienele Izei).



Fig. 1 Wooden churches: (a) abandoned; (b) restored

The inside decoration with these churches was based on the tempera painting technique, taken over from the Byzantine space and perpetuated up to the present day. The painted scenes entirely cover the walls, the vaults and the ceilings of the narthex, nave and altar (Fig. 2). The support of the painting, at the wall level, is made of the surfaces of the wooden beams, between which strips of interstitial canvas was stuck with animal or vegetal glues. The vaults can be made of beams or planks, between which the strips of canvas were applied. Calcium carbonate and glue were used to make the start of preparation. The pigments, based on mineral oxides, were applied over the ground by means of organic binders, especially egg.

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Fig. 2 Painted vaults

## 2. Submission

After 1994, I was involved in several national and international projects, whose purpose was the wooden churches restoration. Thus, I had the opportunity of analysing and elaborating the biological examinations for a number of over 300 wooden churches from different geographical areas of Romania. According to my experience, I understood that the most vulnerable part of these monuments is their painting. We might replace without much difficulty the roofs, the beams, or even entire walls, but we could not replace a lost painting. Whatever we can do is to evaluate the reasons of the decays and to try to prevent them. Among the causes that produce the most extended and irreversible decays, infiltrations of rain water, followed by biological attacks rank first.

The clapboards are the constituent parts whose degradation takes the shortest time and if they are not replaced in good time (35-40 years) [1], infiltrations of rain waters will wash away a good part of the inside paintings or leave dirt stains difficult to remove. At the same time, moist wood is attacked by biological factors (micro-organisms, fungi and wood-boring insects). In the first stage, the interstitial canvases are affected by infiltrations of rain water and their moisture leads to the appearance of micro-organisms attacks (bacteria and moulds), followed by the textile fibre rotting. There were quite rare cases in which I came across the period during which the microfungi produce colonies that are visible without effort. An eloquent example in this aspect is the Wooden Church of Spălnaca/county of Alba, where white colonies, having a diameter of 20 to 50mm developed abundantly on the painting at the level of interstitial canvas. With laboratory colonies, out of the drawn samples, four species of microfungi have been identified, belonging to *Rhizopus*, *Penicillium* and *Aspergillus* genres (Fig. 3).

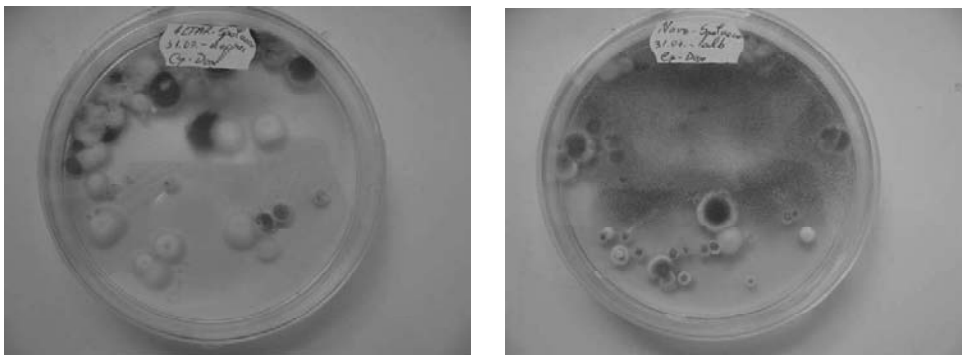


Fig. 3 Microfungi identified in laboratory, out of the drawn samples



The restriction of the decay to the canvas only indicates the preference of these species for the glue used to stick them. The effect of the decay on the canvas which gets rot very rapidly was also noticed. The cause of the massive decay is the infiltrations of rain waters through the degraded wrapping, which led to the rapid growth of the humidity of the support. When the wood stays wet for a long time, the xylophagous macrofungi will develop. The attacks produced by macrofungi are not visible in their first stage, but when the fruit bodies appear, the timber has already lost part of its mechanic strength. Their evolution is fast and will lead, in a few years, to collapse. On the level of painted elements, I have identified 15 species of macrofungi, among which the most frequent are: *Coniophora puteana*, *Fibroporia vaillantii*, *Phellinus cryptarum*, *Phellinus contiguus*, *Dacrymyces stillatus*, *Hyphodontia breviseta*, *Fomitopsis rosea*, *Gloeophyllum abietinum*, *Gloeophyllum sepiarium*, *Schizopora paradoxa*, *Grandinia arguta*, *Hyphoderma puberum*, *Vesiculomyces citrinus* [2]. *Coniophora puteana* has the highest occurrence both on resin wood and on sapwood on the studied monuments [2]. The fruiting bodies are spread on the surface of the painting, attached tightly and are especially frequent with vaults. The decayed wood is degraded in its depth, under the form of prismatic brown rot (Fig. 4).



Fig. 4 Vault decayed by the *Coniophora puteana* and other fungi

*Phellinus cryptarum* is frequent and specific of oak and it was recorded by us for the first time in Romania [4]. The fruiting bodies are quite widely spread, adherent to the support and the painting beneath them is no longer recoverable. The decayed wood is degraded in its depth, as white rot.

*Phellinus contiguus* is specific of resin woods. The fruiting bodies are widely spread, adherent to the support and the painting beneath them is no longer recoverable. The decayed wood is degraded in its depth, as white rot.

*Gloeophyllum abietinum* and *Gloeophyllum sepiarium* and *Fomitopsis rosea* were noticed in but few cases, with churches having already reached a high level of degradation. They are specific of resin wood.

*Hyphoderma puberum*, *Hyphodontia breviseta* and *Grandinia arguta* occur with wood already degraded by the above mentioned species, when the mechanic resistance of the support is almost entirely lost.

The next stage in wood decay is that of the occurrence of the mixofungi of the *Stemonitis* and *Arcyria* type, which contribute to its turning to clay. There are successions of the fungi species and the combined attacks of fungi and wood-boring insects which at the same time indicate the state of the timber [3]. The attack of insects depends on the wood essence, the percentage of sapwood, its position in the trunk, the moisture content, previous or simultaneous fungi attacks, the contribution of organic substances from glues or bird excreta. With the same wall, an elm or sycamore maple element will be strongly attacked at its heartwood level, as compared to one of oak wood that does not present any attack, except for possibly in its sapwood zone. It is common knowledge that the attacks of *Anobium*

*punctatum* are more intense on the peripheral areas of the wood, but we can often notice them under the interstitial canvases that were glued or in areas with infiltrations that brought organic substances (Fig. 5).



Fig. 5 *Anobium punctatum* attacks: (a) areas with infiltrations; (b) under interstitial canvases

With higher intensity attacks (which we appreciate by the number of holes of flight per 100 cm<sup>2</sup>), the wood becomes fragile, brittle or breakable. With some churches we came across nave vaults made of beech wood (*Fagus sylvatica*), which had been massively decayed by *Ptilinus pectinicornis* and the fragile wood easily broke under mechanic pressure. *Xestobium rufovillosum* can only affect the sapwood areas of oak wood but when in combination with fungi decay, it can extend to the heartwood, too. With beams component of vaults and those inside the walls, where infiltrations of water were of long duration, we came across combined attacks of fungi and insects, which compromised the wood in its depth [3]. In the case of combined attacks of *Anobidae* and fungi, the wood becomes spongy and extremely fragile. Consolidation by means of injection with Paraloid B 72 or other products is not enough to give back its mechanic resistance, especially in the case of planks or vault beams. *Hylotrupes bajulus*, specific of resin wood, can only attack sapwood. The larvae make wide galleries, parallel to the surface of the wood, which gives in easily and comes off together with the painting strata. Those degradations occur especially with the side walls of the nave. In the case of wooden churches, the parts of the building most vulnerable to biodegradation are the ceilings and the vaults. By their position, they are directly exposed to infiltrations of rain waters, the moment when the cover are degraded. The upper part of the vaults, the beams at their basis and the arches sustaining them are the components where water from infiltrations can stagnate. At these levels, the first fungi attacks occur and decays progress rapidly. The arches and the planks come off or break, the beams subside and the vault collapses (Fig. 6)



Fig. 6 Vault in collapse

### 3. Conclusions

We study the biologic agents, but they are not the main cause of the loss of these values. The most important factor of degradation is human negligence. Where communities have built new brick churches, the old wooden ones were abandoned. The priests are not aware of the value of the latter and, unfortunately, they are the first to leave them. The communities treat them as old people having reached the age of death. In this case, the solutions we must look for are not the chemical ones (fungicides or insecticides), but the change of people's mentalities.

The funds allotted by the Ministry of Culture for the restoration of wooden churches, quite a lot in the recent 18 years, cannot cope with the rhythm of degradations. A former student of ours, graduate of Theology Studies and then Conservation Studies, wrote his diploma paper on a group of wooden churches. I succeeded in making him aware of the value of these monuments and talking him into the ways by which we could act so as to have an influence on the local community. The spur we have given has developed and has now turned into a project for 80 churches in the Gorj area, and succeeded in involving the County Inspectorate for Culture and the Metropolitan Church of Craiova, in gathering and instructing the local priests, in editing a photo album, organizing a conference and initiating a project of cataloguing and monitoring. These modest attempts can give us a hope that positive examples will have a chance to increase and we will be able to rescue most of these monuments, which, at present, are part of a lost patrimony in most European countries. In the new context of getting financial support for the cultural projects and through the possibility of accessing the European Structural Funds, we hope to succeed in restoring a greater number of the wooden churches which are historical monuments. Consequently, we are considering the following measures to take:

1. The setting of standards (norms) for settling a deadline within which the replacement of the roof clapboards is obligatory and the type of roof which can be manufactured.
2. The introduction of supplementary restoration norms, concerning the painting protection measures, during the restoration intercessions on the building.
3. The design of an insulation system of the vaults for the churches in function, in order to reduce warmth loss, but to prevent condense and to offer an additional, long term protection of the vaults

and side walls paintings. We would like to prevent in this way improvised solutions made by parishioners that could produce condense or maintain moisture and the appearance of biological attacks.

4. Making the priests aware and through them the flock of the value of these monuments, which could become touristic assets.

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# DISINFECTION AND CONSOLIDATION BY IRRADIATION OF WOODEN SAMPLES FROM THREE ROMANIAN CHURCHES

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## Abstract

Studies on application of ionizing radiation for disinfection and consolidation have revealed that wood objects, lacquer, textiles, paper, objects made of stone and gypsum can be considered for conservation purposes. In this work, first were assessed the biodeterioration factors (insects and moulds) and their effects, and properties of wooden samples from three Romanian churches. After treatment, the properties of the impregnated samples were assessed in order to prove that the gamma irradiation process brings a real improvement in wood condition, in terms of disinfection and structure consolidation. As conclusion, the irradiation treatment is considered proper to be applied to different wooden cultural heritage objects only for their disinfection or both for their disinfection and consolidation.

## 1. Introduction

Disinfection and conservation of archaeological artifacts and art objects by radiation treatment (gamma rays or electron beam) appears to have perspective. Studies on application of ionizing radiation for consolidation have revealed that wood objects, lacquer, textiles, paper, objects made of stone and gypsum can be considered for conservation purposes. Radiation treatment of cellulose materials for disinfection is well known and proven. Additional extensive research is needed to develop treatment methods to lower the radiation dose (1).

The susceptibility to the microbial attack of wood is depending on its moisture content. Microbial deterioration, operated mainly by moulds, can start when the water content is above 20%. These can develop on surface or within internal structures inducing, through the production of exoenzymes, change in cell integrity. The role of bacteria and actinomycetes in deterioration of wood is less important because they require higher water content. They have been found mainly in outdoor and marine environments.

Insects, however, are the most serious source of damage for wooden objects kept in museums, in indoor or outdoor environments. They use wood as a nutrient source, for shelter and egg deposit. In feeding, some insects utilize only the compounds obtained from the cell contents (sugars and starch) while others utilize even the cellulose.

Waterlogged wood is the term used to describe wood kept under wet soil or water, for example archaeological wood such as shipwrecks or pile-dwellings. Under these particular conditions (high water content and lowered oxygen pressure), wood can easily be attacked by microaerophilic and anaerobic heterotrophic microorganisms and, in sea sites, by some marine organisms (2).

Gamma rays, a form of electromagnetic radiation, are extensively used for sterilizing micro flora and killing insects, especially on organic materials. A dose of at least 500 Gy is required to kill larvae and to prevent the emergence of adult insects. Moulds are less sensitive to ionizing radiation than insects, and different strains show different levels of sensitivity. Generally most fungi are killed by a total dose of 10 kGy (3). Despite its power, gamma irradiation does not induce any secondary radioactivity and penetrates completely into the objects. Moreover, a large quantity of materials can be treated at one time.

Gamma rays have also been employed for the polymerization of resins used to consolidate decayed wooden objects by impregnation. For the complete polymerization of the resin, the requested dose is

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about 20-30 kGy. Radiation polymerization has a great advantage over conventional polymerization by chemical catalysts as the heat rise can be perfectly controlled by varying the radiation dose rate. The other advantage of radiation polymerization is that the excess resin from an impregnation can be reused, due to the absence of chemical catalysts in the resin storage. The disadvantage of this consolidation treatment in comparison to other consolidation methods of cultural heritage objects is that the process is not reversible. However, it can save from destruction artifacts which present a very high degree of deterioration (4).

## 2. Materials and methods

### Samples

The studied wooden samples came from three Romanian churches:

- A. piece from wooden ceiling of an evangelical church in Sibiu county,
- B. piece of sycamore maple from resistance structure of an orthodox wooden church,
- C. piece of fir from resistance structure of an orthodox wooden church in Dretea, Cluj county, which was build in 1690, painted in 1770 and now is rebuild at "Astra" Traditional Civilization Museum in Sibiu.

### Treatment

The consolidation of the studied samples was carried out using a standard resin of styrene-unsaturated polyester type tetrahydrophthalic. The samples were treated at the irradiation facility of Regional Conservation Workshop Nucléart (ARC-Nucléart), using a gamma source of Co-60. The irradiation was done at room temperature in open air. The delivered mean irradiation dose was about 24 kGy, enough to disinfect the samples from insects and moulds, and consolidate them through polymerization of the resin. The irradiation dose rate was set up as to not exceed a polymerization temperature of 50-60°C. After treatment, different properties of the impregnated samples were assessed in order to prove that the gamma irradiation process brings a real improvement in wood condition, in terms of disinfection and structure consolidation.

### Characterization and testing

First were assessed the biodeterioration factors (insects and moulds) and their effects. Using a photo camera and a stereomicroscope, were taken pictures before and after treatment and assessed the biodeterioration factors (insects and moulds) and their effects on wooden samples. Samples were also weighed before and after consolidation treatment.

A HunterLab Miniscan XE Plus portable spectrophotometer has been used for colour measurements. The geometry of measurement is  $d/8^\circ$  with a view area of 6 mm in diameter, specular component is included, combination illuminant - standard observer is  $D_{65}/10^\circ$ . All values are reported in CIELAB and CIELCh colour spaces. Every value is obtained averaging 30 measurements.

Mechanical testing has been carried out using Zwick / Roell equipments. For dynamical test (impact test) has been used a Zwick / Roell Pendulum model 5113. The test parameters were: pendulum of 25 J, Charpy impact test, distance between sample holders 35 mm, impact speed 3.85 m/s and angle of pendulum launching  $160^\circ$ . Through Charpy impact test has been measured the impact energy E (J). For statically tests (penetration and bending) it has been used a Zwick / Roell Universal Testing Device model Z005. The penetration test has measured the force F (N) necessary to push 1 mm in the sample a metallic ball of 6 mm diameter. The bending test has measured the bending tensile strength  $\sigma$  (MPa) necessary to bend 2 mm a sample. The distance between sample holders was 60 mm.

For electron spin resonance (ESR) testing was used a Magnettech Miniscan MS200 X band spectrometer. The measurement parameters were: Microwave radiation – 9,3-9,6 GHz, Power – 0.8 mW, Centre field – 335 mT, Sweep width – 15 mT, Modulation amplitude – 0.5 mT, Sweep time – 60 s, Steps – 4096, Pass number – 5, Temperature – room temperature. Gain varied according to the signal intensity of the measured sample. A single signal ( $g_{\text{symm}}=2,004$ ) is observed in the ESR spectra of all samples containing cellulose, including unirradiated samples. In the case of irradiated samples, the intensity of this signal is usually much greater. The intensity of signal is proportional with the quantity of cellulose free radicals present in the sample.

### 3. Results and discussion

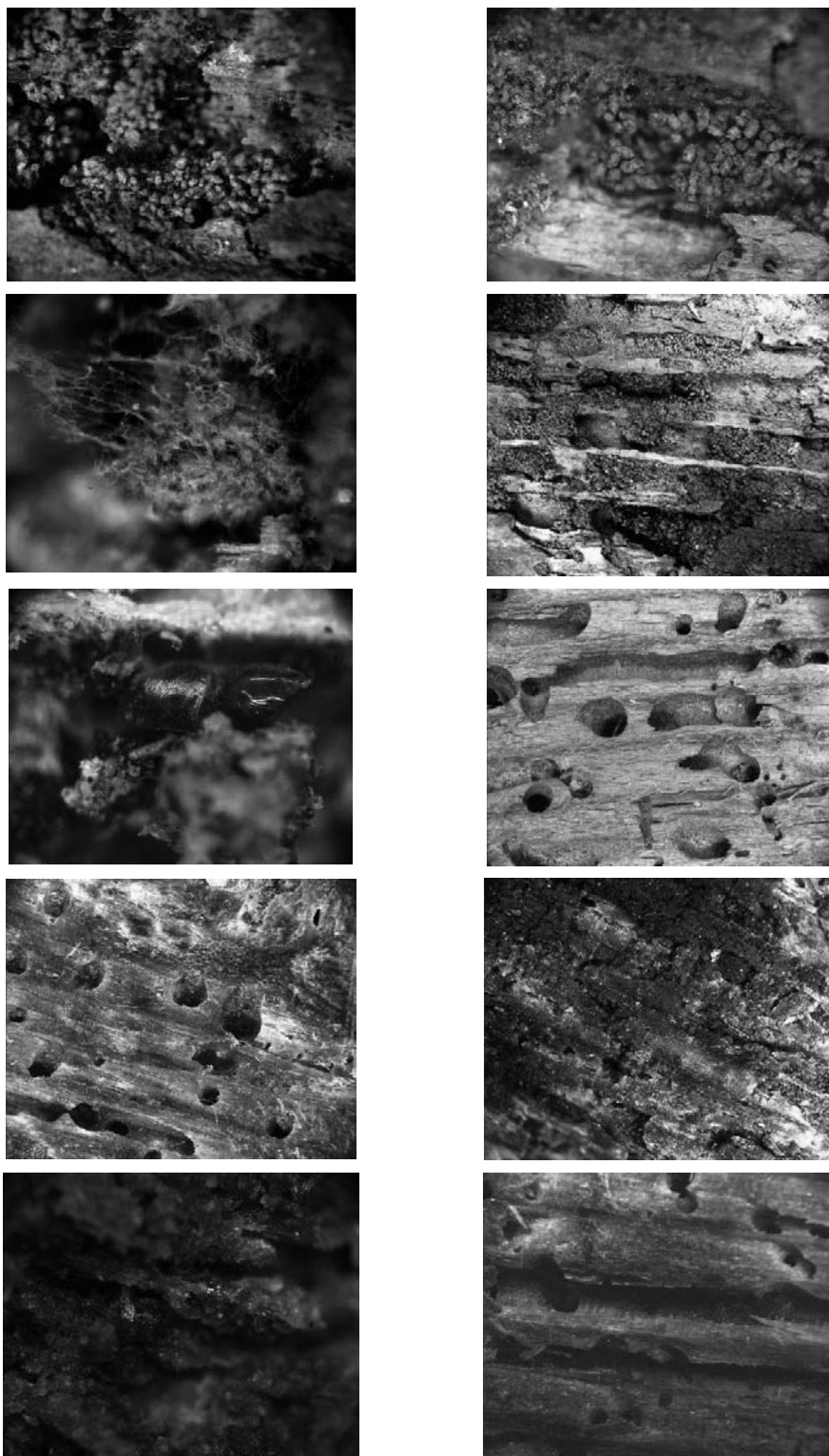


Fig. 1. Pictures of samples for biodeterioration assessment and consolidation evaluation

At examination of samples with a stereomicroscope there were established the biodeterioration factors taking relevant pictures of samples. After impregnation treatment there were taken pictures of treated samples in order to evaluate the consolidation treatment. In Fig. 1 there are presented different pictures taken from samples before and after consolidation treatment.

The samples have been weighed before and after consolidation treatment. Table 1 shows their masses. It can be seen from the table that sample A increased its weight more than 3 times, samples B almost doubled their mass and samples C had more than double mass.

Table 1. Masses of samples before and after consolidation treatment

	Mass of samples (g)							
	A2	B2	B4	B5	C2	C4	C7	C9
Before	22.0	11.6	10.3	79.6	109.4	71.1	95.7	91.9
After	73.6	20.2	17.5	140.4	245.5	167.9	200.7	210.3

In Fig. 2 there are presented pictures of samples before and after impregnation treatment. It can be seen easy the color change of samples.

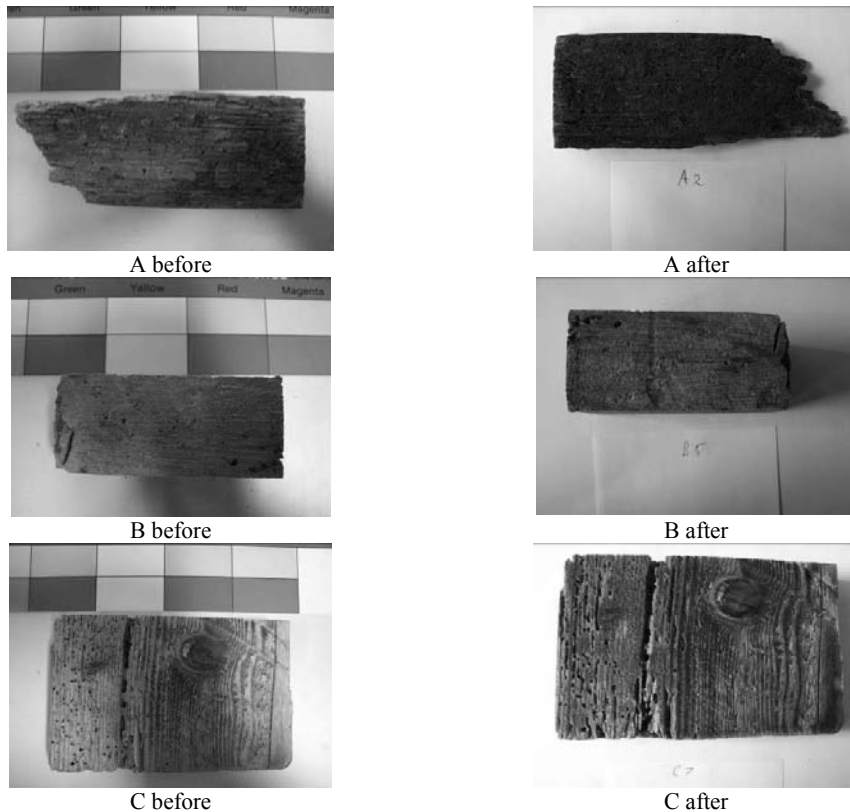


Fig. 2. Pictures of samples before and after impregnation treatment

As can be seen from the Table 2, for the colorimetric measurements all samples showed a decrease in lightness ( $L^*$ ) after treatment. The colour differences between untreated and treated samples are strong and visible. Sample A became less red and less yellow after treatment, this means a significant decrease in chroma ( $C^*$ ) and a strong variation in hue (h). Sample B became redder and yellower after treatment, this corresponding to a small increase in chroma and a strong variation in hue. For sample C, only a decrease in  $b^*$  (less yellow) value can be observed that corresponds to a decrease in chroma and a small variation of hue.



Table 2. Colour values of untreated and treated wood samples

sample	untreated					treated					dE*
	L*	a*	b*	C*	h	L*	a*	b*	C*	h	
A	39.21	4.47	13.71	14.42	71.95	23.81	3.51	6.19	7.11	60.48	17.16
B	60.81	7.47	21.77	23.02	71.06	44.69	13.21	22.71	26.27	59.81	17.14
C	54.55	12.70	31.01	33.51	67.72	42.29	12.14	26.14	28.82	65.10	13.20

Mechanical tests have been carried out in order to prove the mechanical properties improvements brought by impregnation. After applying Charpy test it can be said that no improvement has been noticed because of consolidation treatment. More of this, only consolidated sample A kept its impact resistance, samples B and C lost some of their impact resistance. This means that the wood fiber impregnated with styrene resin is equal or less resistant at impact.

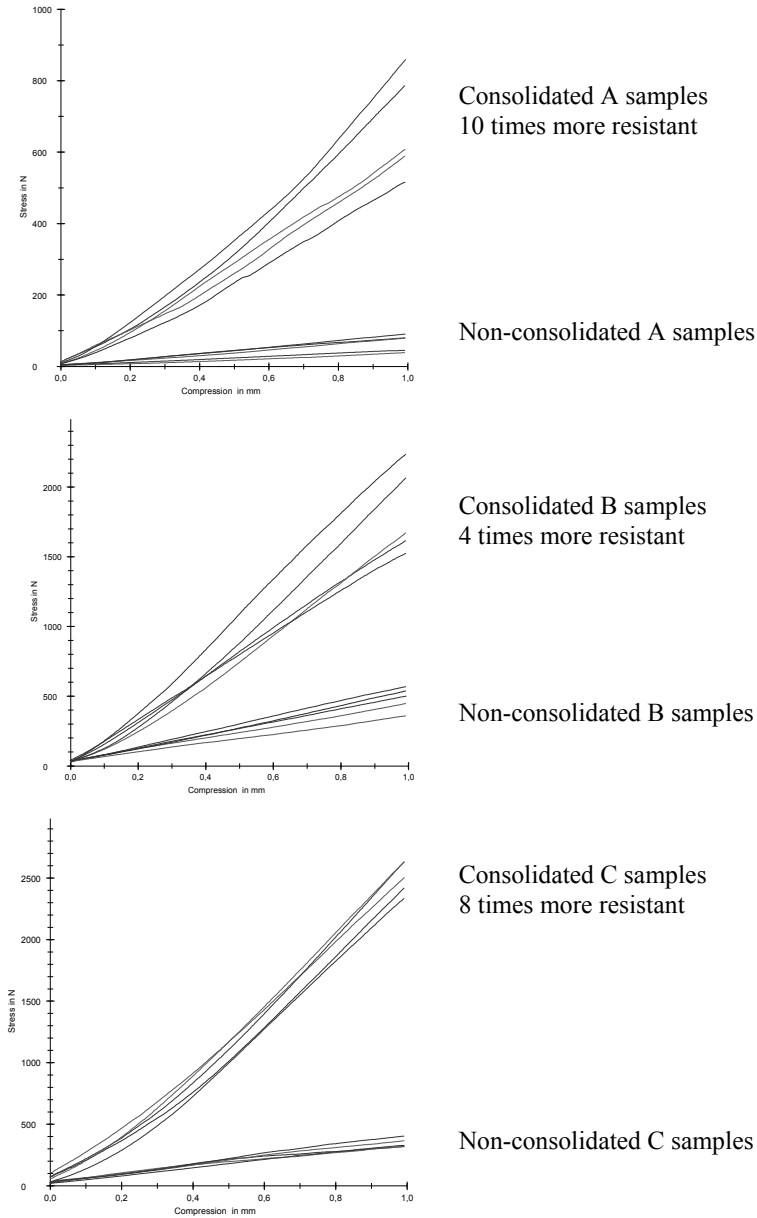


Fig. 3. Diagrams for application of penetration test

Fig. 3 shows the diagrams for penetration test applied at sets of consolidated and non-consolidated samples. From all above diagrams it can be noticed that the consolidated samples have higher resistance to penetration in comparison with the non-consolidated samples. This means that the wood fiber impregnated with styrene resin is more resistant at penetration.

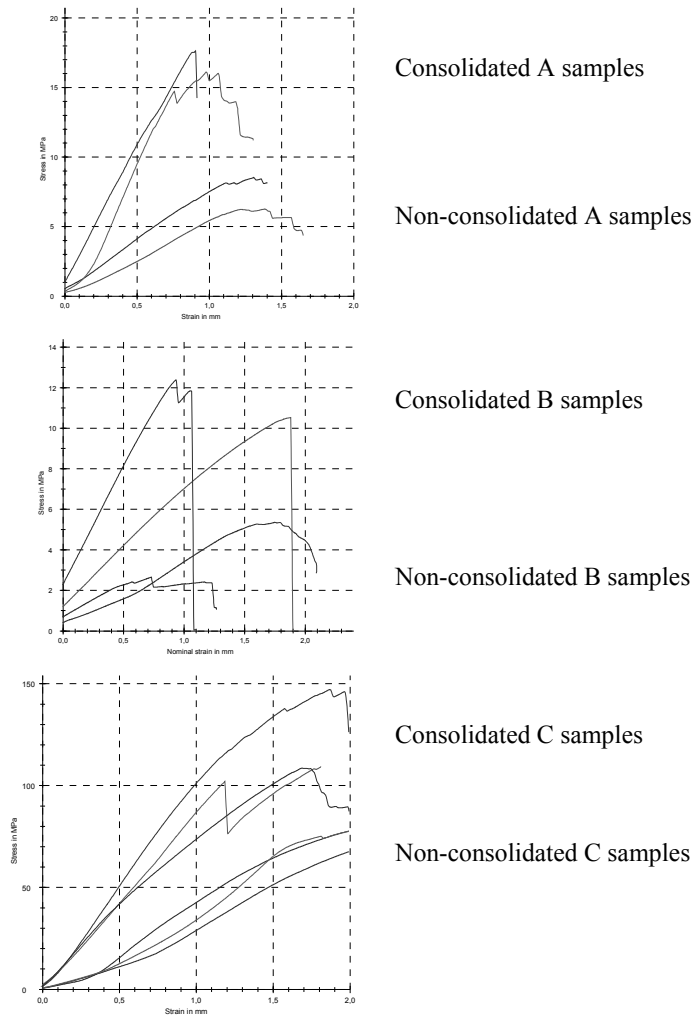


Fig. 4. Diagrams for application of bending test

Fig. shows 4 the diagrams for bending test applied at sets of consolidated and non-consolidated samples. From all above diagrams it can be noticed that the consolidated samples have higher resistance (about 2-3 times) to bending in comparison with the non-consolidated samples. It can be also noticed that consolidated samples crush faster than non-consolidated samples after the appearance of first small crushes in their structure. This means that the wood fiber impregnated with styrene resin is more resistant at bending and after the appearance of first small crushes in structure the flow to total crush is short.

The presence of any free radical in an object gives a potential risk of its damage. Objects containing cellulose have natural free radicals. Irradiation treatment induces in any object free radicals and their persistence could constitute a damage risk. Considering these facts, the intensity of ESR signal of cellulose free radicals it has been used in order to evaluate the potential risk of damage for consolidated and non-consolidated samples. In order to be able to compare them, the intensities were brought at same gain. As can be seen in Table 3, the treated samples have shown three months after

treatment about half intensity per mass unit of the untreated sample. Five months after treatment the intensity per mass unit for treated samples was higher than that after three months but lied between half and one third of the untreated samples. This shows that in addition to consolidation effect, the impregnation with the styrene resin has also a protective effect against natural cellulose free radicals.

Table 3. Semnal RES

Sample	Three months after treatment		Five months after treatment	
	Intensity (a.u.)	Mass normated intensity	Intensity (a.u.)	Mass normated intensity
A untreated	54738	1766	104859	3383
A treated	78894	974	133497	1648
B untreated	85968	1719	198648	3973
B treated	58059	907	92979	1453
C untreated	39149	1223	112194	3506
C treated	97551	938	126054	1212

#### 4. Conclusions

The delivered dose of 24 kGy was more than enough to disinfect the studied samples from insects and moulds. In addition, this dose is very close to the usual dose used for sterilization. So, after treatment the samples were almost sterile. Using this irradiation dose for disinfection, proper packaging of samples and post-irradiation handling procedures of them, after the disinfection treatment the conservator can seal first a totally clean object of biodeterioration factors, and after that proceed to its restoration. Irradiation doses up to 10 kGy can be used for prevention from the action of different biodeterioration factors or for first step of remediation treatment.

In case of consolidation by impregnation with styrene resin, the wooden object becomes heavier and more resistant at penetration and bending. The treated object contains less free radicals than the untreated one. Also, the object changes its colour, generally getting darker, and does not get better impact resistance. When a conservator considers that disadvantages such application of a non-reversible consolidation treatment, darker colour and less impact resistance are not of crucial importance for a certain cultural heritage object then this treatment method can be a very good choice.

As a general conclusion, the irradiation treatment is considered proper to be applied to different wooden cultural heritage objects only for their disinfection (as prevention treatment or as first step of remediation treatment aiming at conservation) or both for their disinfection and consolidation (as remediation treatment aiming at conservation).

#### ACKNOWLEDGEMENTS

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# CHANGES OF MECHANICAL AND CHEMICAL PROPERTIES OF WOOD AFTER BROWN-ROT DECAY AND BLUE STAINING

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## Abstract

Wood inhabiting organisms can cause significant damage on novel and old constructions. Brown rot fungi were chosen as they are known as the most important decay organisms in historical objects. Blue stain fungi are frequently present on wood as well, but there is a general opinion that they do not influence mechanical properties. However, some opposite results have been published as well. Therefore decay patterns of brown rot and blue stain fungi were investigated using nondestructive mechanical tests, FTIR spectroscopy and colour measurements. The results showed that blue stain fungi do not cause significant losses in MoE. However, considerable changes in colour, FTIR spectra and mechanical properties were determined in initial stages of brown rot decay.

**Key words:** wood inhabiting fungi, brown rot, blue stain, mechanical properties, FTIR, mass loss

## 1. Introduction

Norway spruce wood (*Picea abies*) and Scots pine (*Pinus sylvestris*) were and still are the most important wood species for construction applications in Slovenia and most of Central Europe. Particularly sapwood of those two species is extremely susceptible to colonization by wood inhabiting fungi [1].

Damage caused by brown rot fungi is the most frequent reason for failure of historical and novel wooden constructions. Besides wood decay, wooden artifacts are very frequently damaged by blue stain fungi as well. Among them *Aureobasidium pullulans* and *Sclerophoma pithyophila* are reported as the most important staining organisms [2]. In the previous studies there was a general opinion that blue-stain fungi do not influence mechanical properties. However, some opposite results have been published as well [3]. Understanding of decay patterns is of significant importance, to prevent decay and to develop effective and environmentally acceptable solutions for wood conservation.

There are several methods available for elucidation of decay processes in wood. Among them mass loss is one of the easiest and frequently used techniques for determination of changes in wood structure. However, this technique is relatively insensible, as it gives us only rough information on the processes going on in wood. It is well known, that particularly during initial stages of decay, mass loss measurements are not the most reliable option. Chemical analysis or mechanical tests are much more indicative. Some information can be gained from colour as well. In this study, changes in chemical and mechanical properties of wood exposed to brown rot fungi and blue stain species were determined.

## 2. Material and methods

### 2.1. Sample preparation

Samples ( $0.5 \times 1.0 \times 20.0$  cm<sup>3</sup>) were made of Scots pine sapwood (*Pinus sylvestris*) or Norway spruce wood (*Picea abies*). Orientation and quality of the wood met requirements of the standard EN 113 [4]. The samples were exposed to wood inhabiting fungi for the periods between 2 and 8 weeks. Adopted standard procedure EN 152-1 [5] was used for blue stain exposure and EN 113 [4] for brown rot. In this experiment, two blue stain (*Aureobasidium pullulans* and *Sclerophoma pithyophila*) and two brown rot fungi (*Antrodia vaillantii* and *Gloeophyllum trabeum*) were used.

## 2.2. Evaluation of modulus of elasticity

Modulus of elasticity (MoE) was determined before and after fungal exposure. Specimens were oven dried prior MoE measurements. Because of difficulties encountered in measuring the axial vibrations, flexural vibration modes were used to characterize elastic parameters. Considering the hypothesis of the homogeneity of geometrical and mechanical properties along the sample, basic dynamics theorems can be applied to obtain the motion equation of first vibrations. Analysis was performed on specimen with clamped–free end conditions. During the test the lateral displacements of vibrating sample in damped vibration with known vibration mode was measured. As, an inductive proximity sensor was used, a small piece of metal foil of neglected mass was glued on the surface of each sample. The damped frequency was obtained by FFT analysis of the exponentially decayed displacement signals detected in time domain. For determination of Young modulus of samples we used the frequency equation deduced from Bernoulli model, which was assumed as acceptable because of the relatively high length-to-depth sample ratio, ( $E$  - Young modulus (N/m<sup>2</sup>),  $v$  - natural frequency (s<sup>-1</sup>),  $C = 3.51563$  – constant derived from Bernoulli equation,  $\rho$  – density (kg m<sup>-3</sup>),  $l$  – free sample length (m),  $h$  – sample height (m). Measurements were performed on seven replicates.

## 2.3. Chemical analysis (CNS) of wood

Prior to nitrogen and carbon analysis, wood blocks, that was used for MoE measurements, were milled into particles (MESH 80) and homogenized. Approximately, 0.15 g of an oven dry sample was combusted in the oxygen atmosphere at 1350°C in LECO 2000-CNS analyzer to determine carbon and nitrogen content.

## 2.4. FTIR and colour measurements

FTIR spectra were recorded with the Perkin Elmer FTIR Spectrum One Spectrometer, using Abrasive Pad 600 Grit-Coated, PK/100 (Perkin Elmer) paper. DRIFT spectra of wood samples were recorded between 4000 cm<sup>-1</sup> and 450 cm<sup>-1</sup>. Colour of the specimens was recorded with HP Scanjet 4800 scanner. Scanner was chosen, as specimens were too narrow for measurements with colorimeter. Colour obtained with scanner and colorimeter gives comparable results. The reported values are average value of seven replicate measurements. The colour was expressed in Ciel\*a\*b\* format.

## 3. Results and discussion

### 3.1. Brown rot fungi

Eight weeks of exposure to brown rot fungi resulted in notable mass loss of spruce wood specimens, indicating that both brown rot fungi were active. The decay of control spruce wood by *G. trabeum* caused higher mass loss than did exposure to *A. vaillantii*, as expected from previous studies [6]. After eight weeks of exposure to *G. trabeum*, a mass loss of 28.0% was detected, while the mass loss of spruce wood exposed to *A. vaillantii* was only half as much (14.3%). This difference was not consistent across all exposure times. For example, neither fungus caused detectible decay within the first week of exposure, but two weeks of decay resulted in almost two times higher mass loss by *A. vaillantii* (4.5%) in comparison to specimens decayed by *G. trabeum* (2.6%). After four weeks of decay, mass loss caused by *G. trabeum* (13.0%) surpassed those of *A. vaillantii* (10.2%) and then doubled again by week 8 (Tab. 1).

Measurements of modulus of elasticity (MOE) revealed similar trends as those reported for mass loss data with one significant difference. The first sign of incipient decay was clearly seen even after one week of exposure of specimens to both fungal strains. This proves that a nondestructive measurement of MOE loss is a very sensitive tool. After one week of exposure, *G. trabeum* reduced MOE by 7.4% and *A. vaillantii* by 8.3%. Comparable findings are reported in the literature as well [7,8]. As shown with the mass loss measurements, changes in MOE demonstrated that *A. vaillantii* was more effective during the first two weeks of exposure and *G. trabeum* during the remaining period. The remarkable decay capacity of *G. trabeum* can be clearly seen from MOE losses after eight weeks of exposure of control, un-impregnated, specimens where *G. trabeum* caused MOE losses of more than 75%. On the other hand, eight weeks of exposure of the un-impregnated specimens exposed to *A. vaillantii* resulted

in only a 40.5% loss of MOE (Tab. 1). Thus, the data from the control specimens show that *A. vaillantii* degrades wood faster during initial stages of decay, but afterwards its ability to degrade wood decreases. On the other hand, *G. trabeum* proved again that it is one of the most capable brown rot fungi. Curling with coworkers [9] showed that hemicelluloses are decayed initially during incipient brown rot decay, and that degradation of those components significantly influences mechanical properties. Hemicelluloses form an encrusting envelope around cellulose microfibrils, therefore degradation of cellulose depends on the prior removal of hemicelluloses. Thus, it seems that *A. vaillantii* has developed mechanisms to rapidly and effectively degrade hemicelluloses and easily accessible cellulose. On the other hand, *G. trabeum* degrades hemicelluloses, cellulose, and even lignin [10].

Table 1: Mass losses and modulus of elasticity (MOE) losses of Norway spruce wood specimens exposed to brown rot fungi. Standard deviations of five replicates are given in the parenthesis.

Weeks of exposure	<i>G. trabeum</i>		<i>A. vaillantii</i>	
	Mass loss (%)	MoE loss (%)	Mass loss (%)	MoE loss (%)
0	0.0 (0.0)	2.3 (1.0)	0.0 (0.0)	2.3 (1.0)
1	-0.1 (0.2)	7.4 (1.8)	0.1 (0.0)	8.3 (3.4)
2	2.6 (1.2)	12.0 (3.6)	4.5 (1.1)	16.4 (6.2)
4	13.0 (3.6)	47.5 (7.4)	10.2 (1.8)	38.6 (5.9)
8	28.0 (8.8)	76.0 (10.9)	14.3 (3.2)	40.5 (8.3)

Table 2: Relationship between intensities of FTIR absorption peaks that are assigned to C-O stretching in cellulose and hemicellulose ( $1030\text{ cm}^{-1}$ ) and aromatic skeletal vibrations at lignin ( $1600\text{ cm}^{-1}$ ), determined from the surface of wood exposed to wood decay fungi. Standard deviations are given in the parenthesis.

Weeks of exposure	<i>G. trabeum</i>	<i>A. vaillantii</i>
0	18.2 (0.2)	18.2 (0.2)
1	16.9 (1.1)	13.3 (1.4)
2	22.7 (1.8)	9.9 (2.1)
4	22.1 (3.7)	8.3 (2.4)
8	*	8.1 (1.9)

\* The specimens were too degraded; therefore we were not able to determine exact spectra

These data are further supported by FTIR measurements. The relationship between the intensities of IR absorption peaks that are assigned to C-O stretching in cellulose and hemicelluloses ( $1030\text{ cm}^{-1}$ ) and aromatic skeletal vibrations (stretching) of lignin ( $1600\text{ cm}^{-1}$ ), decreases during the first four weeks of exposure to *A. vaillantii* and then remains almost constant during the last four weeks. Data presented in Tab. 2 shows that the amount of cellulose and hemicellulose decreases faster than lignin content, indicating that *A. vaillantii* does not have mechanisms to degrade lignin. On the other hand, there was a different relationship between the IR absorption peaks of specimens exposed to *G. trabeum*. During the first week, the amount of cellulose and hemicellulose decreased, but afterwards the relationship between cellulose and hemicellulose peaks increased, indicating that there must be some partial degradation or change of lignin (Tab. 2). It is likely that these relationships are clear evidence that *G. trabeum* uses more sophisticated mechanisms to degrade wood, and that this fungus is able to utilize a higher percentage of wood components than *A. vaillantii*. However, it must be considered that these spectra were determined from the surface of exposed specimens, and that the decay of surface layers is usually faster than decay of the interior portions of the specimens.

Colour of the specimens is one of the parameters that reflect the changes in chemical composition in wood. Exposure to both fungi resulted in a darkening of the wood, *A. vaillantii* from the beginning of the exposure, while *G. trabeum* initially caused a lightening of the wood within the first four weeks followed by a darkening of the specimens (Tab. 3).

Table 3: Colour of Norway spruce wood specimens after exposure to wood decay fungi for the period between one and eight weeks.

Weeks of exposure	<i>G. trabeum</i>			<i>A. vaillantii</i>		
	L*	a*	b*	L*	a*	b*
0	89.0	2.4	10.4	89.0	2.4	10.4
1	68.6	5.2	13.2	74.9	5.7	13.2
2	64.7	6.1	13.2	68.2	6.6	13.2
4	58.0	6.6	13.7	64.3	7.1	13.2
8	48.2	6.6	12.8	67.8	6.6	13.2

### 3.2. Blue stain fungi

As expected, exposure of Scots pine wood specimens to blue stain fungi, resulted in considerable colour changes. The first signs were visible after the second week of exposure. However, colour changes of specimens exposed to *S. pithyophila* ( $\Delta E = 18.7$ ) for two weeks, were more prominent than the ones exposed to *A. pullulans* ( $\Delta E = 2.6$ ). *S. pithyophila* remained more active all the time of exposure. Within time of exposure, the specimens became darker, less reddish and yellowish and more bluish and greenish (Tab. 4). Maximum colour change was observed after the sixth week at *S. pithyophila* and after eight weeks at *A. pullulans*. The most important reason for observed changes is melanin excreted by those two staining fungi [3].

Table 4: Colour changes of Scots pine sapwood exposed to blue stain fungi

Weeks of exposure	<i>Sclerophoma pithyophila</i>				<i>Aureobasidium pullulans</i>			
	L*	a*	b*	$\Delta E$	L*	a*	b*	$\Delta E$
0	85.1	3.8	13.7	0.0	85.1	3.8	13.7	0.0
2	66.7	3.1	10.7	18.7	82.6	3.8	14.6	2.6
4	63.8	2.6	9.0	21.8	79.7	2.8	12.8	5.6
6	57.5	2.5	7.5	28.3	71.9	3.1	11.3	13.4
8	58.4	2.5	7.7	27.4	70.1	3.0	10.1	15.4

Mass changes of wood specimens exposed to blue stain fungi were insignificant. In none of the cases mass losses were observed. In contrary, all specimens gain some weight; firstly because specimens were prior fungal exposure immersed to malt agar suspension, as proposed by EN 152-1 standard [5], and secondly, as fungi contributes to the mass of the specimens with their biomass and excreted compounds like melamine. However, changes of mass are relatively small and are always smaller than 1 % (Tab. 5). Those measurements are in line with data of Highley [1].

Table 5: Modulus of Elasticity, mass changes and carbon and nitrogen content in wood after exposure of Scots pine sapwood to blue stain fungi

Weeks of exposure	<i>Sclerophoma pithyophila</i>				<i>Aureobasidium pullulans</i>			
	$\Delta m$ (%)	$\Delta MoE$ (%)	C (%)	N (%)	$\Delta m$ (%)	$\Delta MoE$ (%)	C (%)	N (%)
0	0.8	1.2	47.63	0.0352	0.8	1.2	47.63	0.0352
2	0.6	2.8	46.33	0.0205	0.2	1.7	47.00	0.0160
4	0.3	3.6	46.55	0.0181	0.0	1.5	47.02	0.0163
6	0.2	2.5	46.48	0.0175	0.1	2.9	45.60	0.0164
8	0.8	2.7	46.27	0.0181	0.8	1.7	45.97	0.0162

Nitrogen content in uninfected pine sapwood was 0.0352%. Immediately after exposure, nitrogen content dropped significantly to 0.0205% at *S. pithyophila* and to 0.0160% at *A. pullulans* and remained almost constant within time of exposure (Tab. 5). This indicates that blue stain fungi rapidly consumed nitrogen within the first two weeks of exposure. This nitrogen was translocated from wood and utilized for fungal growth in wood as well as it in soundings. The remaining nitrogen in wood is either in biologically unavailable form or it was used for chitin and protein synthesis and is present in

wood as a constituent of fungal hyphae. Similar as nitrogen content, carbon content decreased as well from initial 47.63%, to final 46.27% at *S. pithyophila* and to 45.97% at *A. pullulans*.

Unexpectedly, modulus of elasticity (MoE) of blue stained specimens did not decrease but even slightly increase (Tab. 5). The first increase of MoE was observed immediately after sterilization, even at specimens that was not exposed to wood decay fungi at all. Similar phenomenon is reported for heat treated wood, where MoE firstly increases with temperature of treatment, but when temperatures reach over 150°C, MoE starts decreasing [11]. This increase of MoE is assigned to fact that after water evaporation, cellulose hydroxyl groups form hydrogen bonds between neighbouring micro-fibrils, which results in improved MoE of steam-sterilised wood. Another possible explanation for increased MoE values of blue-stained wood is the presumption that melanin could interact with wood functional groups and additionally crosslink wood components. However, this is only presumption which is very difficult to prove.

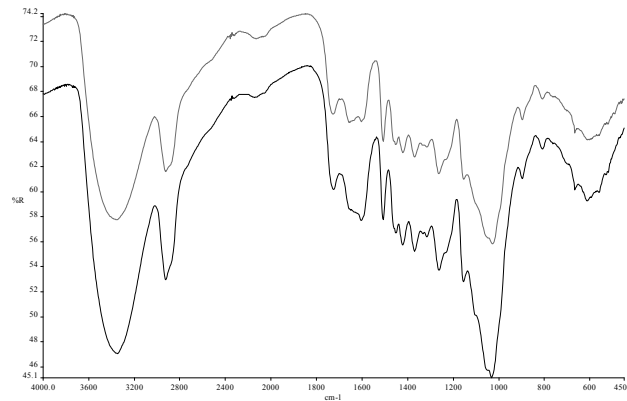


Fig. 1: FTIR spectra of control (lower line) *Pinus sylvestris* wood and wood exposed to *Sclerophoma pithyophila* for eight weeks (upper line)

Infra red spectra confirms, previously observed results. There were no differences in IR peaks between control unexposed pine wood and blue-stained wood even after eight weeks of exposure (Fig. 1). This confirms that blue stain fungi did not change lignin, cellulose and hemicelluloses structure. Additionally, it seems that the amount of melanin in wood is too low to be observed from FTIR spectra. Secondly, as functional groups of melanin are relatively similar to lignin functional groups, melanin can not be resolved from FTIR spectra [12].

These data are important from application point of view. Blue stained wood can be therefore used for various construction applications. And secondly, if albino blue stain fungi are utilised for biocontrol applications [13], it could be presumed that these fungi will not significantly influence mechanical properties of colonised wood.

#### 4. Conclusions

Comparison of results obtained with different experimental techniques gives very interesting details on brown rot decay of Norway spruce wood. Decay caused by the *A. vaillantii* is faster during the initial stages, but then decreases compared to decay by *G. trabeum*. FTIR analysis of decayed specimens resolved that *A. vaillantii* decays predominately hemicelluloses and cellulose and that lignin remains almost unaffected. On the other hand, *G. trabeum* decays hemicelluloses and cellulose, as well as lignin, which results in significantly higher mass losses compared to decay caused by *A. vaillantii*.

*Aureobasidium pullulans* and *Sclerophoma pithyophila* significantly blue-stained pine wood specimens. However, the results of the experiments showed that this change is only aesthetic and does not influence neither weight or mechanical properties of blue-stained wood.



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# WOOD DECAY FUNGI IN LATVIAN BUILDINGS INCLUDING CULTURAL MONUMENTS

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## Abstract

During a period of 12 years (1996 – 2007), a total of 300 private and public buildings as well as more than 20 cultural monuments had been inspected in Latvia regarding the damage by wood decay basidiomycetes. The culture heritage sites included castles, manors, churches and old buildings. The total number of fungal occurrences in wooden constructions comprised 338. Brown-rot damage occurred more frequently (78.1%) than white-rot (21.9%). A total of 60 species of wood decay fungi were identified on the constructive and decorative materials. *Serpula lacrymans* (46.7%), *Antrodia* spp. (12.7%), *Coniophora* spp. (5.9%) and *Gloeophyllum* spp. (2.9%) were the most frequently recorded fungi. Majority of decay fungi were found on wood indoors (83%), while wood outdoors was damaged only in 17% cases. Cultural heritage sites were decayed in 91 (27%) cases.

## 1. Introduction

Latvian cultural monuments, which include archaeological monuments, monuments of urban development, architectural monuments, monuments of art and historical monuments, are a part of the world's cultural historical heritage. The inclusion of the historical centre of Riga in the UNESCO world cultural heritage list in 1997 testifies the significance of the Latvian cultural heritage. In Latvia, there are 8517 state protected cultural monuments including 76 medieval castles or ruins, 142 manors, 136 Lutheran churches, 68 Catholic churches, 36 Orthodox churches and 29 homes of prominent persons [17]. Wood is probably the oldest construction and decorative material used in buildings, and is a common material widely used in Latvian buildings. In Latvia, forests still occupy about 50% from the total area. To preserve these resources, rational utilisation of wood, including the extension of its service life by preventing against biodegradation, is of crucial importance.

In appropriate environmental conditions, wood outdoors and indoors can be degraded by brown-rot, white-rot, or soft-rot fungi. The most important wood-degrading fungi within buildings in Europe and North America are fungi that cause brown-rot in conifers. White-rot fungi, which preferentially attack hardwoods, are less common in buildings [16]. Only three fungal species are often mentioned as most frequent house-rot fungi in Europe: *Serpula lacrymans*, *Coniophora* spp., and *Antrodia* spp. [14].

The aim of our study was to examine Latvian buildings including cultural monuments in terms of biodeterioration caused by wood decay fungi.

## 2. Materials and methods

### 2.1. Inspected buildings

During a period of 12 years (1996 – 2007), a total of 300 private and public buildings as well as more than 20 historical and old buildings had been inspected in respect to the damage by wood decay basidiomycetes in Latvia. The cultural heritage sites are listed below:

Castles: Araiši lake fortress (castle) (reconstruction of an ancient Latgalian residential site of the 9<sup>th</sup> century), Lielvarde wooden castle (reconstruction of the castle dating from the 12<sup>th</sup> century), Turaida castle (13<sup>th</sup> century).

Manors: Dikli manor (15<sup>th</sup> century), Malpils manor (18<sup>th</sup> century), Svente manor (20<sup>th</sup> century), Vainiži manor (18<sup>th</sup> century), Zeluste manor (18<sup>th</sup> century).

Churches: Bikeri Lutheran church (18<sup>th</sup> century), Dundaga Lutheran church (18<sup>th</sup> century), Feimani Roman Catholic Church (18<sup>th</sup> century), Gulbene Lutheran church (19<sup>th</sup> century), Jesus Lutheran church in Riga (17<sup>th</sup> century), Cathedral of Holy Trinity in Liepāja (18<sup>th</sup> century), Malpils Lutheran church

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(18<sup>th</sup> century), Rezekne Lutheran church (20<sup>th</sup> century), Riga Dome (13<sup>th</sup> century), Sabile Lutheran church (16<sup>th</sup> century), Sloka Lutheran church (19<sup>th</sup> century), Vecpils Roman Catholic Church (18<sup>th</sup> century).

Old buildings: Desele watermill (1920), Liepaja railway station (19<sup>th</sup> century), Ventspils Latvian Society building (1912).

Our attention was paid both to the internal woodwork (floors, walls, ceilings, roof inner portions etc.) as well as external woodwork (windows, stairs, walls, roofs, fences, bridges, benches). The research consisted of damage diagnosis *in situ*, identification of fungi, and examination of the attacked materials.

## 2.2. Identification of microorganisms

Fungal material (mainly fruiting bodies, in some cases mycelium and strands) was collected in paper and plastic bags, brought to the laboratory, dried at room temperature and prepared for microscopy. The identification of wood decay fungi was performed by using light microscopy and reagents (Melzer, 5% KOH, cotton blue, sulphovanillin). Wood decay fungi were identified according to the keys [2] [3] [4] [5] [7] [10] [13]. All corticoid species were referred to the group of Corticiaceae s. lat. [3]. In several cases only available material was brown-rotted wood. In these cases wood samples were examined visually and recorded as unidentified brown-rot.

## 3. Results and discussion

### 3.1. Overview on wood decay fungi in Latvian buildings

338 cases of fungal degradation in private/ public buildings and cultural heritage were recorded during our inspections. Cultural heritage sites were decayed in 91 (27%) cases.

Table 1 lists the species of wood decay fungi, the type of rot and the species occurrence in buildings. In few cases, the corticoids and brown-rot fungi were not identified to the species level. In Table 1, they appear as "Unidentified Corticiaceae s. lat." and "Unidentified brown-rot". All unidentified species were also added to the number of occurrences. The number of cases fluctuated yearly, in the average, 28 cases per year were recorded. A total of 60 fungal species were identified; 21 of them belonged to brown-rot species, while 39 were white-rot producers. This demonstrates a higher diversity of white-rot species in buildings.

Table 1: Wood decay basidiomycetes, type of rot and occurrence in Latvian buildings.

Basidiomycetes	Occurrences in objects		Total occurrences	
	CM*	PP**	No	%
Brown-rot				
<i>Antrodia</i> spp.:	17	26	43	12.7
<i>Antrodia serialis</i> (Fr.) Donk				
<i>Antrodia sinuosa</i> (Fr.) P. Karst.				
<i>Antrodia sordida</i> Ryv. & Gilb.				
<i>Antrodia vailantii</i> (DC : Fr.) Ryv.				
<i>Antrodia xantha</i> (Fr.: Fr.) Ryv.				
<i>Coniophora arida</i> (Fr.) P. Karst.	1	-	1	0.3
<i>Coniophora puteana</i> (Schum.) Karst.	5	14	19	5.6
<i>Fomitopsis pinicola</i> (Sw.) P. Karst.	1	-	1	0.3
<i>Gloeophyllum</i> spp.:	6	4	10	2.9
<i>Gloeophyllum abietinum</i> Fr.: Fr.				
<i>Gloeophyllum sepiarium</i> (Wulf.: Fr.) P. Karst.				
<i>Lentinus lepideus</i> (Fr.: Fr.) Fr.	-	1	1	0.3
<i>Leucogyrophana pinastri</i> (Fr.) Bond.	-	2	2	0.6
<i>Leucogyrophana pseudomolusca</i> (Parm.) Parm.	-	1	1	0.3
<i>Oligoporus caesius</i> (Schrad.: Fr.) Gilb. & Ryvarden	1	-	1	0.3
<i>Oligoporus placentus</i> (Fr.) Gilb. Ryvarden	-	2	2	0.6

<i>Paxillus panuoides</i> (Fr.: Fr.) Fr.	2	3	5	1.4
<i>Phaeolus schweinitzii</i> (Fr.) Pat.	1	-	1	0.3
<i>Postia stiptica</i> (Pers.) Jülich	1	-	1	0.3
<i>Pycnoporellus fulgens</i> (Fr.) Donk	1	-	1	0.3
<i>Serpula himantoides</i> (Fr.: Fr.) Karst.	-	1	1	0.3
<i>Serpula lacrymans</i> (Wulf.: Fr.) Schroet.	8	150	158	46.7
Unidentified brown-rot	3	13	16	4.7
Subtotal	47	217	264	78.1
White-rot				
<i>Antrodiella</i> sp. Ryv. & Johan.	-	1	1	0.3
<i>Armillaria ostoyae</i> (Romagn.) Herink	-	1	1	0.3
<i>Asterostroma cervicolor</i> (Berk. & M.A. Curtis) Massee	-	1	1	0.3
<i>Athelia epiphylla</i> Pers.	1	-	1	0.3
<i>Athelia</i> sp.	1	-	1	0.3
<i>Auricularia mesenterica</i> (Dicks.: Fr.) Pers.	1	-	1	0.3
<i>Bjerkandera adusta</i> (Willd.) P. Karst.	1	-	1	0.3
<i>Botryobasidium candicans</i> John Erikss	2	-	2	0.6
<i>Ceriporia excelsa</i> (Lund.) Parm.	-	1	1	0.3
<i>Ceriporia purpurea</i> (Fr.) Donk	1	-	1	0.3
<i>Ceriporia reticulata</i> (Hoffm.) Domański	1	-	1	0.3
<i>Crepidotus mollis</i> (Schaeff.) Staude	1	-	1	0.3
<i>Cylindrobasidium evolvens</i> (Fr.) Jul.	-	6	6	1.8
<i>Dacryobolus sudans</i> Fr.: Fr.	1	-	1	0.3
<i>Gloecystidiellum cf. luridum</i> (Bres.) Boidin	1	-	1	0.3
<i>Hyphoderma obtusum</i> J. Erikss.	2	-	2	0.6
<i>Hyphoderma praetermissum</i> (P. Karst.) Erikss. & Strid	2	-	2	0.6
<i>Hyphoderma puberum</i> (Fr.) Wallr.	1	2	3	0.9
<i>Hyphodontia alutacea</i> (Fr.) John Erikss.	1	1	2	0.6
<i>Hyphodontia aspera</i> (Fr.) John Erikss.	2	1	3	0.9
<i>Hyphodontia crustosa</i> (Pers.) J. Erikss.	1	-	1	0.3
<i>Hypholoma fasciculare</i> (Huds.) P. Kumm.	1	-	1	0.3
<i>Hypohnicium bombycinum</i> (Sommerf.) J. Erikss.	1	-	1	0.3
<i>Laeticorticium roseum</i> (Pers.) Donk	1	-	1	0.3
<i>Meruliopsis corium</i> (Pers.) Ginns	1	-	1	0.3
<i>Peniophora cinerea</i> (Fr.) Cooke	1	-	1	0.3
<i>Peniophora incarnata</i> (Pers.: Fr.) P.Karst.	-	1	1	0.3
<i>Phanerochaete tuberculata</i> (Karst.) Parm.	2	-	2	0.6
<i>Phanerochaete velutina</i> (Fr.) P. Karst.	-	1	1	0.3
<i>Phlebiopsis gigantea</i> (Fr.: Fr.) Jul.	3	1	4	1.2
<i>Pluteus semibulbosus</i> (Lasch.) Gill.	1	-	1	0.3
<i>Resinicium bicolor</i> (Fr.) Parm.	2	1	3	0.9
<i>Schizopora paradoxa</i> (Schröd.) Donk	1	-	1	0.3
<i>Scytinostroma cf. odoratum</i> (Fr.) Donk	1	-	1	0.3
<i>Sebacina calcea</i> (Pers.) Bres.	1	-	1	0.3
<i>Shizophyllum commune</i> Fr.: Fr.	1	4	5	1.5
<i>Skeletocutis carneogrisea</i> A. David	1	-	1	0.3
<i>Stereum sanguinolentum</i> (Alb. & Schwein.) Fr.	2	-	2	0.6
<i>Trichaptum abietinum</i> (Pers. in Gmelin) Ryv.	3	1	4	1.2
<i>Vesiculomyces citrinus</i> (Pers.) Hagstrom	-	1	1	0.3
Unidentified Corticiaceae s. lat.	1	6	7	2.1
Subtotal	44	30	74	21.9
Total	91 (27%)	247 (73%)	338	100

\*CM – cultural monuments; \*\*PP - private and public buildings

The occurrence of brown-rot fungi in Latvian wooden constructions was more frequent than that of white-rot fungi. The frequency of brown-rot fungi comprised 78.1% of the total recorded cases, while white-rot fungi made up 21.9%. We suppose that the prevalence of brown-rot was related to the wide use of coniferous (pine, spruce) timber in buildings. Most frequent fungal species were *Serpula lacrymans* (46.7%), *Antrodia* spp. (12.7%), *Coniophora* spp. (5.9%) and *Gloeophyllum* spp. (2.9%). Brown-rot is reported as the most common decay type in the buildings of temperate climate. The typical brown-rot fungi are *Serpula lacrymans*, *Coniophora puteana*, different *Poria* species, *Gloeophyllum* sp., *Paxillus panuoides* and *Lentinus lepideus* [18]. Our data showed some differences in comparison with other European countries. For example, in Danish buildings, *Coniophora puteana* is reported as a dominant species comprising 50% of the total wood damages [2] [16], while Norwegian data showed that *Antrodia* species were the most frequent (18.4%) in the period of the study [1].

### 3.2. Wood decay fungi in cultural monuments

23 cultural monuments including castles, manors, churches and old buildings were examined in respect to the damage by wood decay fungi. The number of the inspected culture heritage sites comprised 27% of the total number of inspections carried out in the given period.

Table 1 demonstrates that brown-rot prevailed (47 cases) over white-rot (44 cases) in the cultural monuments. The dominant fungal genus in wood constructions was *Antrodia* (17 cases), followed by *S. lacrymans* (8 cases). The third most abundant genera were *Gloeophyllum* and *Coniophora* (6 cases). In 29 cases, the corticoid fungi were recorded in the cultural heritage sites with the totally identified 19 species.

The most abundant fungal material was collected in Araishi lake fortress (castle) and Lielvarde wooden castle. A total of 41 species of wood decay fungi were recorded in both locations. All species were saprobes growing on wooden constructions - logs, bridges, fences, tables, stairs, walls, roofs, and ceilings. The majority of the recorded species were characteristic of conifers, which was expected, because spruce was used as the primary (Lielvarde) or the sole construction material (Araishi).

### 3.3. Decayed structural parts of buildings

A total of 456 fungal occurrences in interior and exterior wood were recorded during our inspections (Table 2). In numerous cases, the same fungal species were identified in different structural parts of buildings. This resulted in a higher number of occurrences in structural parts (Table 2) than the number of occurrences listed in Table 1.

The examination of decay localities demonstrated that wooden structures indoors were damaged more frequently than outdoors (Fig. 1). In 372 cases (83%), fungal damage was recorded in interior wood, while exterior wood was decayed in 78 cases (17%).

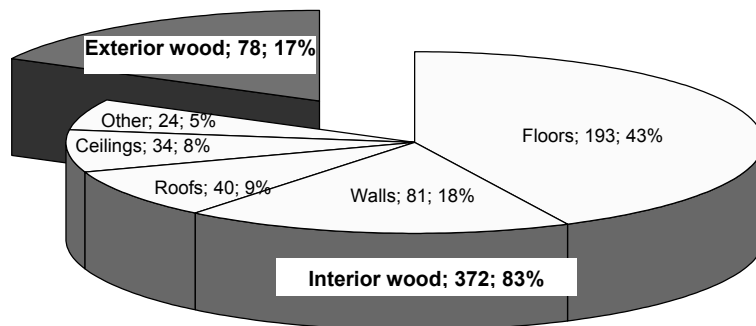


Fig. 1 - The number and percentage of fungal occurrences in wooden structural parts

Table 2: Occurrence of wood decay basidiomycetes in structural parts of damaged buildings.

Fungi	Interior wood, No (%)					Exterior wood, No (%)
	Floors	Walls	Roofs	Ceilings	Other*	
<i>Antrodia</i> spp.	18 (9.3)	2 (2.5)	10 (25.0)	4 (11.8)	1 (4.2)	13 (16.7)
<i>Antrodiella</i> sp.	-	-	-	-	1 (4.2)	-
<i>Armillaria ostoyae</i>	-	-	-	-	-	1 (1.3)
<i>Asterostroma cervicolor</i>	-	-	-	1 (2.9)	-	-
<i>Athelia</i> spp.	-	-	-	1 (2.9)	-	2 (2.6)
<i>Auricularia mesenterica</i>	-	-	-	-	-	1 (1.3)
<i>Bjerkandera adusta</i>	-	1 (1.2)	-	-	-	1 (1.3)
<i>Botryobasidium candicans</i>	-	-	-	1 (2.9)	-	1 (1.3)
<i>Ceriporia</i> spp.	-	-	1 (2.5)	-	-	2 (2.6)
<i>Coniophora</i> spp.	7 (3.6)	2 (2.5)	9 (22.5)	4 (11.8)	-	1 (1.3)
<i>Crepidotus mollis</i>	-	-	-	-	-	1 (1.3)
<i>Cylindrobasidium evolvens</i>	-	-	3 (7.5)	2 (5.9)	-	1 (1.3)
<i>Dacryobolus sudans</i>	-	-	-	-	-	1 (1.3)
<i>Fomitopsis pinicola</i>	-	-	-	-	-	1 (1.3)
<i>Gloeocystidiellum cf. luridum</i>	-	1 (1.2)	-	-	-	-
<i>Gloeophyllum</i> spp.	-	-	2 (5.0)	-	-	9 (11.5)
<i>Hyphoderma</i> spp.	-	-	1 (2.5)	2 (5.9)	-	4 (5.1)
<i>Hyphodontia</i> spp.	-	1 (1.2)	1 (2.5)	1 (2.9)	-	3 (3.8)
<i>Hypholoma fasciculare</i>	-	1 (1.2)	-	-	-	1 (1.3)
<i>Hypohnicium bombycinum</i>	-	-	-	-	-	1 (1.3)
<i>Laeticorticium roseum</i>	-	-	-	-	-	1 (1.3)
<i>Lentinus lepideus</i>	-	-	-	-	1 (4.2)	-
<i>Leucogyrophana</i> spp.	-	-	-	1 (2.9)	-	2 (2.6)
<i>Meruliopsis corium</i>	-	-	-	-	-	1 (1.3)
<i>Oligoporus</i> spp.	1 (0.5)	-	-	-	1 (4.2)	2 (2.6)
<i>Paxillus panuoides</i>	-	1 (1.2)	1 (2.5)	1 (2.9)	-	2 (2.6)
<i>Peniophora</i> spp.	-	-	-	1 (2.9)	-	1 (1.3)
<i>Phaeolus schweinitzii</i>	-	-	-	-	-	1 (1.3)
<i>Phanerochaete</i> spp.	-	-	-	-	-	3 (3.8)
<i>Phlebiopsis gigantea</i>	1 (0.5)	1 (1.2)	-	-	-	2 (2.6)
<i>Pluteus semibulbosus</i>	-	-	-	-	-	1 (1.3)
<i>Postia stiptica</i>	-	-	-	-	-	1 (1.3)
<i>Pycnoporellus fulgens</i>	-	-	-	-	-	1 (1.3)
<i>Resinicium bicolor</i>	-	-	-	2 (5.9)	-	1 (1.3)
<i>Schizopora paradoxa</i>	-	1 (1.2)	-	-	-	1 (1.3)
<i>Scytinostroma cf. odoratum</i>	-	-	1 (2.5)	-	-	-
<i>Sebacina calcea</i>	-	-	1 (2.5)	-	-	-
<i>Serpula himantioides</i>	-	-	-	-	-	1 (1.3)
<i>Serpula lacrymans</i>	158 (81.9)	66 (81.5)	-	8 (23.5)	18 (75.0)	4 (5.1)
<i>Shizophyllum commune</i>	-	-	1 (2.5)	1 (2.9)	-	2 (2.6)
<i>Skeletocutis carneogrisea</i>	-	1 (1.2)	-	-	-	-
<i>Stereum sanguinolentum</i>	-	-	-	-	-	2 (2.6)
<i>Trichaptum abietinum</i>	-	-	1 (2.5)	-	-	3 (3.8)
<i>Vesiculomyces citrinus</i>	-	-	-	-	-	1 (1.3)
Unidentified Corticiaceae s. lat.	-	-	4 (10.0)	2 (5.9)	-	-
Unidentified brown-rot	8 (4.1)	3 (3.7)	4 (10.0)	2 (5.9)	2 (8.3)	1 (1.3)
Total No.	193	81	40	34	24	78

\*Other: doors, stairs, windows.

The results on occurrence of decay fungi in particular structural parts showed that the floors were infected most frequently (43%). The walls were attacked in 18%, inner roof portions in 9%, while ceilings in 8% of the total number of damage. The remaining 5% of the interior wood decay was attributed to damage of other structures (doors, stairs, and windows).

### 3.4. Most frequent fungal species in Latvian buildings

*Serpula lacrymans* is the most dangerous destroyer of structural wood indoors. The fungus is found in buildings in the moderate climate of central, Northern and Eastern Europe, but does not occur in tropical or desert regions [15]. Dry rot is reported to be common for the coolest regions of Japan and Southern Australia [9]. Our previous findings have testified that *S. lacrymans* is also common in Latvia's buildings [8].

According to our data (1996 – 2007), *S. lacrymans* had been found almost in all regions of Latvia. The occurrence of *S. lacrymans* comprised 47% from the total number of wood decay fungi in buildings (Tab. 1). The cases varied yearly owing to the fluctuations in climatic conditions. The occurrences of *S. lacrymans* grew especially in humid summers, when the wetting capability of building structures tended to increase. Most of the cases were observed from March to October, with a maximum in August. For example, in Switzerland, the occurrence of dry rot comprised 45% and in Denmark 20% [9], while in Finland about 50% among other fungal species in buildings [12]. The wide distribution of *S. lacrymans* in Latvian buildings can be explained by the most serious damage of wood compared with other house fungi. As a result, our laboratory had received more samples and calls for identification and eradication of *S. lacrymans*.

The dry rot fungus dominated in all categories of infected structural parts indoors: the floors were damaged in 81.9%, walls in 81.5%, ceilings in 23.5%, and other interior wood parts in 75% cases. *S. lacrymans* was not found in roof constructions. It should be mentioned that *S. lacrymans* as a typical indoor fungus in our inspections was found 4 times outdoors. In Table 2 these cases were included in the column of the exterior wood damage, although the fruiting bodies were developed on building foundation or 50 - 70 cm from the foundation on the ground.

The second most frequent fungi in Latvian buildings were the genus *Antrodia* (12.7%) with five species: *Antrodia serialis*, *A. sinuosa*, *A. sordida*, *A. vailantii*, and *A. xantha* (Tab. 1). The decayed structural parts of buildings revealed that *Antrodia* occupied the second place in floor damage (9.3%) (Tab. 2). *Antrodia* was the most frequent genus in roofs (25.0%) and exterior wood (16.7%).

*Coniophora* spp. comprised 5.9% of fungal occurrence in buildings (Tab. 1). Two species, *C. puteana* and *C. arida*, were identified. *C. arida* was identified only once in wall, while *C. puteana* was the third most frequent species in floors (3.6%) and the second in roofs (22.5%).

Two *Gloeophyllum* species were recorded in construction wood, namely, *G. sepiarium* and *G. abietinum*, comprising 2.9% of occurrences (Tab. 1). *Gloeophyllum* was more common in exterior wood (walls, roofs, fences, bridges, benches). In Latvia, *Gloeophyllum* has not been found in window structures, while in other European countries, these species are the most important destroyers of windows that had accumulated moisture due to inappropriate window construction [2] [14]. The modern windows with wooden lists holding the pane are reported to be totally damaged after 5 years. Another species, *G. trabeum*, an obligatory fungus of the European Standard EN 113 [6] has not been found in wooden constructions in Latvia. This is considered as a very rear species in Latvia [11].

The majority of the identified white-rot species belonged to corticoid fungi. In our study, most of the corticoids were identified to the species level to demonstrate the high diversity of this group in wooden constructions. The occurrence of corticoids in buildings comprised 15.6%, with the total number of 25 species (Tab. 1).

The development of wood decay basidiomycetes in buildings was fostered by poor maintenance, non-professional reconstruction/repair or improper building construction. The fungi were usually found in places with a regular wood moistening and the lack of ventilation favoured by leaking roofs, damp cellars, humid premises, damaged water pipes, or places with water condensation.

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# FUNGAL DECONTAMINATION BY COLD PLASMA: AN INNOVATING PROCESS FOR WOOD TREATMENT

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## Abstract

Cold plasma technology offers an environmental approach to decontaminate wood surfaces, alternative to conventional methods such as biocides applications. Decontamination effect of plasma discharge is evaluated on a blue-stain fungus *Aureobasidium pullulans*, responsible for bluish discoloration of wood material causing aesthetical and economical damages. Several gas mixtures and exposure times were assessed. These results suggest that fungal spores are inactivated after an afterglow exposure of 15 minutes.

## 1. Introduction

In the last years, biological decontamination by afterglows issued from plasma devices has shown promising results [1-3]. Efficiency of plasma discharges as a sterilisation process has been proved on bacteria contamination, in several industrial activities.

Wood material exposed to natural outdoor conditions is subject to biological development. In the case of fresh maritime pine (*Pinus pinaster*), bluish discoloration due to blue-stain fungi development causes aesthetical damages, limiting its use according to safety issues. Nowadays, preventive chemical treatments are performed to delay fungal growth, but new environmental issues impose to develop alternatives to conventional biocides.

According to industrial requirements, flowing afterglow which is a clean dry process offers numerous advantages and may answer to specific needs regarding preservation of cultural heritage.

The present study focuses on the assessment of atmospheric pressure afterglow efficiency as a curative treatment. In order to better understand the mechanisms involved between the excited gas and microorganisms, efficiency of plasma afterglow is initially assessed directly on fungal cultures of *Aureobasidium pullulans*, set on a membrane filter. After recalling plasma characteristics, the main objective is, in the first place, to set up the experimental device and the characterisation means to find better plasma parameters to inactivate *Aureobasidium pullulans*. Finally, first promising results are presented.

## 2. Gas plasma

### 2.1. Definition and characteristics of gas plasmas

Cold plasmas, in reference to their low gas temperature, are partially ionised gases generated from the introduction of sufficient energy to ionise a molecular or atomic gas. For instance the application between two electrodes, in the gas, of an electric field leads to such environments. Thanks to the supplied energy, numerous reactive species are created, resulting from collisions between electrons and present neutral species. As a consequence, a cold plasma is composed of electrons, ions, molecular and atomic neutral particles. Moreover, ions and neutral particles are constituted of a very large number of excited states, themselves responsible for the formation of new elements as photons, radicals, etc. This complex composition suggests that cold plasmas are potential energy tanks which may interact with material surfaces in order to obtain physical and/or chemical modifications.

The Dielectric Barrier Discharge (DBD) technology provided for the experiments is a particular way to generate cold plasmas. In this system, a dielectric coating on one or more electrodes is realised in

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order to limit the electrical current and thus allow the generation of electrical discharges in gases at atmospheric pressure.

## 2.2. The flowing afterglow

The experimental device uses the flowing afterglow to process on samples. Many advantages of using afterglows are recognised. First of all, afterglow is only composed of neutral species (atoms, molecules, radicals and photons), and depending on the experimental conditions the gas temperature can be made close to ambient temperature, an important factor to treat heat-sensitive materials. Moreover, studies have demonstrated that neutral species [4], in large amounts in the afterglow, play the major role in the sterilisation process. Operating in the discharge itself is thus not required and anyway the little gas gap (1mm) does not allow to introduce large samples in the reactor. Then, the afterglow can fill large chamber volumes to process on large pieces, and can be carried out via a tube directly through a contaminated surface, to be one day directly supplied with a portable equipment. Finally, interactions between the contaminated surface and neutral species from the afterglow can be considered as a phase gas chemical process, thereby ensuring material integrity and protecting the material intrinsic properties. Consequently, decontamination process by afterglows issued from DBD answers to specifications required regarding the preservation of cultural heritage.

## 3. Experiment

### 3.1. Isolation and identification of fungal strains

To isolate fungi responsible for maritime pine biodeterioration, samples were taken from decayed wood and deposited on malt agar plates. After 10 days of incubation, subcultures of single colonies were carried out on fresh agar plates in order to isolate and purify the different fungal strains. Once isolated, identification could be performed. Mains species identified are moulds: *Penicillium sp.*, *Gliocladium sp.* and *Trichoderma sp.* and blue-stain fungi: *Ceratocystis sp.* and *Aureobasidium pullulans*.

### 3.2. Samples preparation

The fungus *Aureobasidium pullulans* is cultured on malt agar medium for ten days at 24°C. A fungal suspension is prepared adding 20 mL of sterile water to the plate culture and then filtered through a glass filter in order to recover only fungal spores (mycelium is retained in the filter). An aliquot of the suspension is next spread on sterile nitrocellulose membrane filters stuck on a glass slide and let to dry at ambient temperature before being exposed to the afterglow. Initial spore concentration of the fungal suspension is evaluated. Dilutions in sterile water are then plated on malt agar plates and incubated 5 days at 24°C. The number of colonies formed are counted, and thus initial concentration is assessed.

### 3.3. Afterglow exposure

The DBD reactor (Fig. 1) is an industrial reactor (AXCYS Technologies) electrically supplied by a generator which delivers chopped quasi sinusoidal voltage and current waveforms in a frequency of 125 kHz. Total delivered power was fixed to 900 Watts, and the duty cycle was adjusted to 10% in order to keep the temperature inferior to 40°C so that thermal effect of the gas is not to be considered.

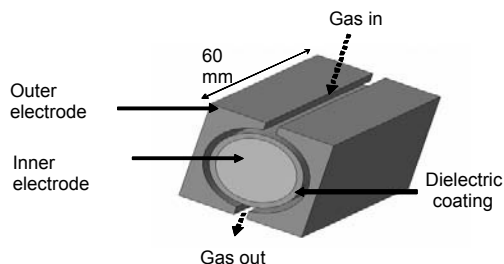


Fig. 1 - Schematic representation of the industrial reactor used for the process (AXCYS Technologies).

An adapter is added to the reactor in order to collect the gas exiting from the reactor and guide it in a quartz tube of 1 cm of diameter. Glass slides are placed at 1 cm from the exit of the tube. The sample moves thanks to a custom-made conveyor belt so that the treatment is dynamic and the entire surface

can be treated. The duration of the treatment is then defined by the velocity of the conveyor belt. In this work, three times of treatment were studied: 1, 2 and 15 minutes. Moreover, used gases are mixtures of nitrogen and oxygen, the oxygen percentage being equal to 0, 5 and 20%.

### 3.4. Characterisation means

#### Fluorescence microscopy

Fluorescence microscopy observations are performed directly on membrane filters. A double staining procedure, with two fluorescent dyes: fluorescein diacetate (FDA) and propidium iodide (PI), gives information about cells viability [4]. FDA stains viable cells because of the enzymatic activity and the integrity of membrane cells. On the contrary, PI bounds DNA of dead cells emitting red fluorescence ; this stain cannot cross the membrane of viable cells. In this way, when spores are observed by fluorescence microscopy, viable and non viable cells respectively fluoresce green and red [5]. Such characterisation mean is used as a qualitative one.

#### Quantitative plate count

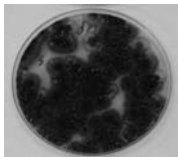



To measure growth inhibition after afterglow treatment, colonies on plate cultures are counted to estimate the number of survivors. Filters are rinsed in 10mL sterile water and suspensions are vortexed for 1 minute in order to recover the spores. Dilutions in sterile water are then streaked on malt agar plates. The number of CFU (Colonies Forming Unit) is determined after incubation for 5 days at 24°C. Inhibition percentage of fungal growth is calculated as indicated in (1):

$$\% \text{ inhibition} = \frac{\text{CFU}_{\text{control}} - \text{CFU}_{\text{after treatment}}}{\text{CFU}_{\text{control}}} \times 100 \quad (1)$$

## 4. Results

Three experiments were realised. Results of the first run are presented in Table 1. Gas mixtures containing 0, 5 and 20% of oxygen were tested and the treatment duration was equal to 15 minutes. Results show that after 15 minutes of afterglow exposure, spores are inactivated for all gas mixtures.

Table 1: Agar plate cultures after different gas afterglow exposures (first experiment).

Gas Mixture N <sub>2</sub> /O <sub>2</sub>	control	100/0	95/5	80/20
Agar plate cultures				
Percentage inhibition	-	100%	100%	100%

Observations by fluorescence microscopy of exposed filters confirm agar plate cultures results: dead spores could be observed, whereas viable spores were not detected (Fig. 2b). On the contrary, the presence of viable spores assessed on control filters (Fig. 2a), correlated with the high number of colonies counted on plates, confirms the decontaminating properties of afterglow on fungal spores.

For the two following runs, the same process and experimental conditions were used. Three exposure times (1, 2 and 15 minutes) and gas mixtures (N<sub>2</sub>/O<sub>2</sub> 80/20, 95/5 and 100/0) were tested. Fig. 3 represents the number of survivors after different treatment duration. Results show a reduction of the initial spore population as a function of exposure time for all gas mixtures, first significant results are obtained after 2 minutes of exposition. After 15 minutes of afterglow treatment, inactivation of fungal spores can be considered as total (inhibition percentage = 100%) and confirmed the first run results.

Results obtained by fluorescence microscopy confirm the evolution revealed by the numeration on agar plates. However, they were not totally conclusive, since glass slides preparation was not optimised. Nevertheless, few viable spores were observed on untreated filters and short exposure-times treated samples, whereas the number of dead spores increases as a function of exposure time.

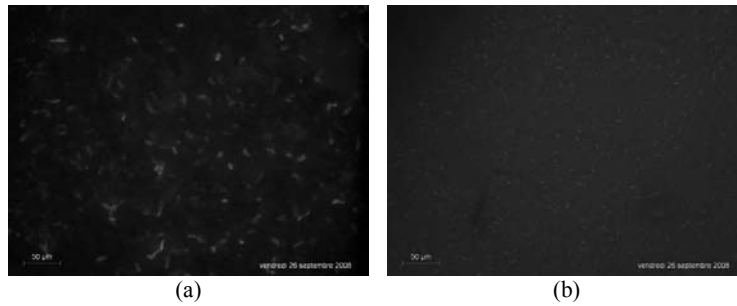


Fig. 2 - Fluorescence microscopy images: (a) untreated control; (b) spores exposed to the afterglow for 15 min.

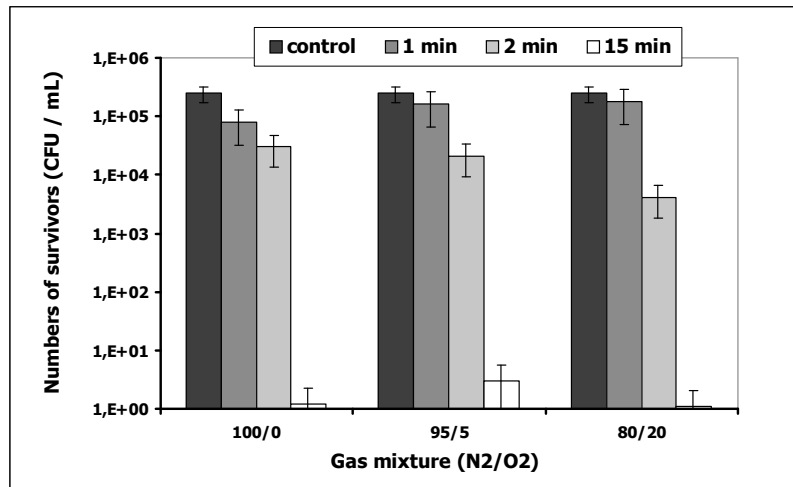


Fig. 3 - Evolution as a function of exposure time and gas composition of the spore population submitted to the afterglow. (Mean values of three agar plates).

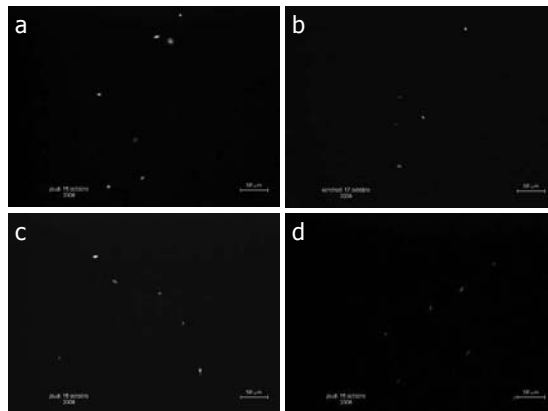


Fig. 4 - Fluorescence microscopy observations of fungal spores after different exposure times: (a) control, (b) 1 min, (c) 2 min, (d) 15 min.

## 5. Discussion

This work showed first very promising results. Efficiency of afterglows to inactivate fungal spores has been shown for treatment duration of 15 minutes. Moreover, for shorter exposure times (2 minutes), an effect has been assessed too, denoting a great interest of such environments.

Quantitative plate counts provide results which can be reproduced, considering the low bar errors, and give information relative to the percentage of growth inhibition according to exposure time. More experiments would supplement first data and so the shorter time necessary to inhibit all fungal spores would be determined.

Moreover, according to first results, the nature of gas mixture does not seem to be a preponderant parameter in the decontamination process. No significant differences were observed by increasing the percentage of oxygen to 0 to 5 and 20%. Some more runs with different treatment duration and gas mixtures may reveal the most effective plasma parameters.

Otherwise, fluorescence microscopy observations only give few qualitative information as far as spore viability is concerned, due to a problem of glass slides preparation. This characterisation mean is now being improved in order to avoid this problem.

## 6. Conclusion and perspective

To conclude, this study emphasizes the efficiency of a new curative anti-fungal treatment realised at atmospheric pressure.

Further experiments may improve these first promising results. Influence of several parameters has to be studied such as the exposure time and the gas mixture. Moreover, new characterisation means could give more information about spore viability, for example, scanning electron microscopy may show the aspect of spores after plasma exposure.

Furthermore, to better understand mechanisms, reactive species composing the afterglow have to be analysed more precisely.

Finally, best plasma parameters once defined, assessment of the efficiency of the afterglow will be investigate on contaminated wood.

## Acknowledgements

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# DIAGNOSIS OF PAINTED WOOD CEILING PLANKS FROM AN EIGHTEEN CENTURY BUILDING

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## Abstract

Our studies were conducted on three panels of decorative cover ceiling planks in chestnut wood with paintings from the eighteen century. This work has been developed within the scope of the final project of the Specialized Technical Courses in “Conservation and Restoration of Wood and Wood Furniture” and “Conservation and Restoration of Wood Paintings” carried out by FRESS in Lisbon, Portugal. The three panels are formed by separated planks in a reasonable conservation state though with signs of degradation by wood-boring beetles whenever sapwood was present, staining, warp and metal corrosion due to the fasteners. The combined action of the wood-boring beetles and the presence of moisture at some point in the life history of the panels destroyed parts of the paintings support. The diagnostic of the state of the wood is presented in this paper, as well as the possible lines of intervention to restore the supports.

## 1. Introduction

Within the scope of the final project of the Specialized Technical Courses in “Conservation and Restoration of Wood and Wood Furniture” and “Conservation and Restoration of Wood Paintings” carried out by FRESS in Lisbon, Portugal, three panels of decorative cover ceilings planks in chestnut wood (*Castanea sativa* Miller) with paintings from the eighteen century have been studied. The project includes the diagnosis of the structure with description of the anomalies present and the preliminary definition of an intervention proposal to improve the conservation state of the wood. Details of the conservation of the painting will not be discussed in the present paper.

These panels have been out of use for sometime and belong to the collection of the Museum of *Fundação Ricardo Espírito Santo Silva*. Due to lack of available exhibition space, the panels were on storage for more than ten years not always under the best conservation conditions which might have led to the aggravation of some pre-existent problems, namely warp and insect infestation.

The ceiling panels can be assigned, taking into account the drawings and the colours present, to the Portuguese early baroque style. This style is called *brutesco*, and is characterized by massive ornamentation, with general vegetal motifs, like flowers and large leaves, and *ferroneries* [1]. The dominant colours of the paintings from this period also found in the studied panels were red, blue, yellow, green and black.

The three panels under study are formed by separated boards, two of them have overlapping joints (*saia-camisa*) and the other one has a single joint structure (*junta seca*). All of them are in a reasonable conservation state, however with signs of degradation by wood-boring beetles whenever sapwood was present, staining, warp and metal corrosion near the fasteners.

The combined action of the wood-boring beetles and the presence of moisture at some point in the life history of the panels destroyed parts of the paintings support. The diagnostic of their state of conservation is presented as well as some ideas about the way forward in the conservation of these interesting objects.

## 2. Diagnosis

The initial step of the diagnosis was the identification of the wood used and a preliminary evaluation revealed that all ceiling panels were made of chestnut (*C. sativa*). They show signs of degradation not only from the time they were in use but also linked to the far from ideal conditions of storage of the last few years.

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The chestnut wood planks show a significant number of cracks and fractures both caused by the action of xylophage insects with consequent lost of ligneous material and the intermittent exposure to moisture. In association with variations of moisture levels of the wood, bow, severe warp and cracks developed in some of the planks. These are in clear risk of collapsing thus putting in serious risk the interesting characteristics of the paintings. Staining due to moisture or corrosion by metal is also present in all panels. Large deposits of dirt sediments are also present in all panels.

Prior to the intervention all planks were treated by fumigation to stop all possible insect activity.

### 2.1. Ceiling panel nº1

This structure, of approximately 2.16 x 1.65 m, is formed by nine planks with polychromes and the usual decoration motifs of the “*brutesco*” (Fig. 1).

The planks, in Portuguese chestnut wood, are made mostly of heartwood in reasonable state of conservation though with the already referred problems of bow, warp, cracks and loss of material due to insect activity (Fig. 2).

The planned intervention in this panel, in what respects the conservation of the wooden substrate, will involve the careful cleaning of the surfaces and the posterior use of two complementary techniques: application of small grafts of wood from the same species to restore the volumetric dimensions of the initial panel and consolidation of the unpainted surface with an acrylic polymer in organic solvent. Paraloid B72 is one of the materials most used for consolidation of wood objects [2] and it will be the most probable choice for this work.



Fig. 1 – Front view of ceiling panel nº 1

### 2.2. Ceiling panel nº2

This structure, of approximately 2.03 x 1.70 m, is formed by eight planks with polychromes with the same usual motifs of the “*brutesco*” framed by a gold and yellow painted border (Fig. 3).

The planks, in Portuguese chestnut wood, are made again mostly of heartwood in reasonable state of conservation. Fig. 4 shows some details of the rear view of the ceiling plank underlying the most important problems found in panel nº2. In this case, and due to the very small amounts of sapwood used, there were very few signs of insect degradation and considerably less loss of material.

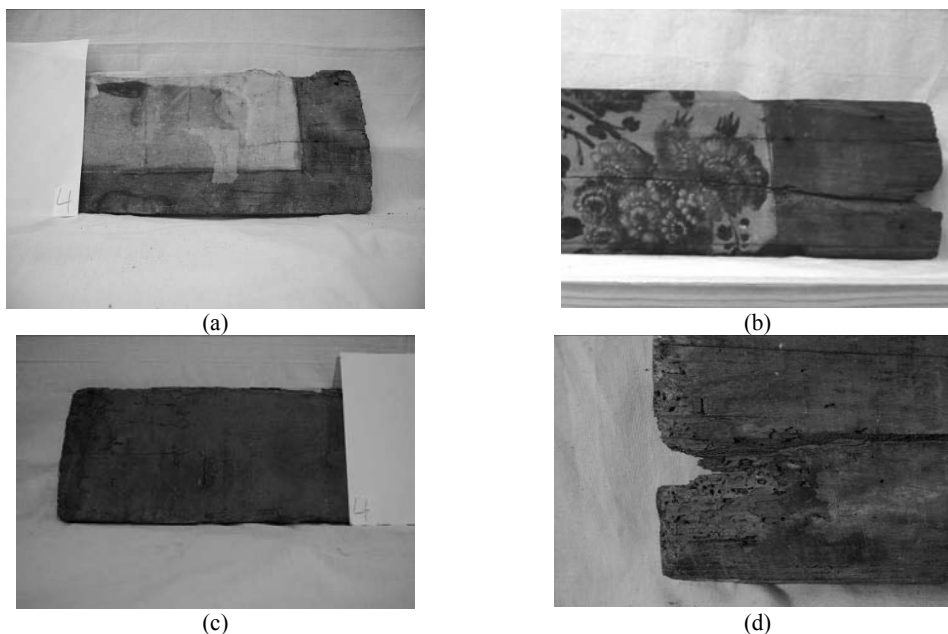


Fig. 2 – Ceiling panel n° 1. Details (a) zone with detachment of the painting; (b) severe cracking; (c) moisture stain better seen in the back view and (d) insect damage and consequent loss of material

The planned intervention in this panel, in what respects the conservation of the wooden substrate, will again involve the careful cleaning of the surfaces followed by spot impregnation, namely of the holes caused by metal elements, with an acrylic resin in organic solvent.



Fig. 3 – Front view of ceiling panel n° 2



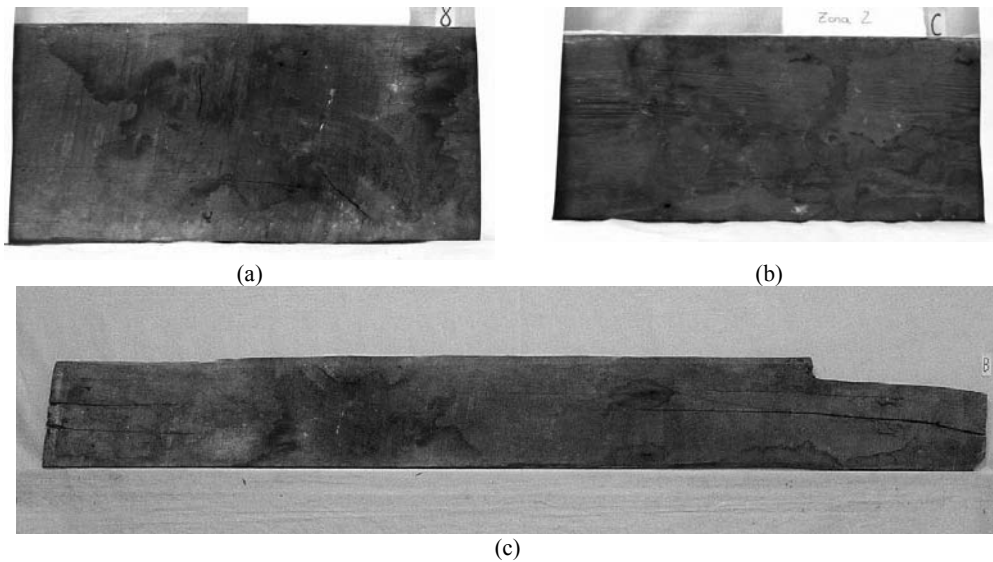


Fig. 4 – Ceiling panel n° 2. Details of rear face (a) dust deposits and moisture stains; (b) dust deposits and corrosion stains; (c) bow, severe cracks and moisture stains

### 2.3. Ceiling panel n°3

This structure, of approximately 2.05 x 1.673 m, is formed by nine planks with polychromes and the usual decoration motifs of the “*brutesco*” in this case very similar to the ones applied in ceiling panel n° 1 (Fig. 5) .

The planks, of Portuguese chestnut wood, are made mostly of heartwood in reasonable state of conservation though with problems similar to those described for ceiling panel n° 1. The loss of material was, in the case of this panel, more evident and had a stronger impact in the painted surface (Fig. 6).

The planned intervention in this panel, in what respects the conservation of the wooden substrate, will involve again the careful cleaning of the surfaces and the subsequent use of the two complementary techniques described: application of small grafts of wood and consolidation of the unpainted surface with an acrylic polymer in organic solvent.

### 3. Future work

The intervention described, done within the final year project of the Specialized Technical Courses carried out by FRESS, was considered important not only to preserve the interesting features of the panels but also to allow their display in future exhibitions planned for the Museum of *Fundação Ricardo Espírito Santo Silva*.

The present paper dealt with the initial steps of the restoration of the wooden supports of three painted ceilings from the eighteenth century. In parallel, measures were also taken to clean and restore the paintings. As conclusion of this work it will be necessary to build a new wood structure to support each panel. These new structures will be made in a softer wood to sustain the panel’s movements and to avoid further cracking and will allow their planned display.

### Acknowledgements

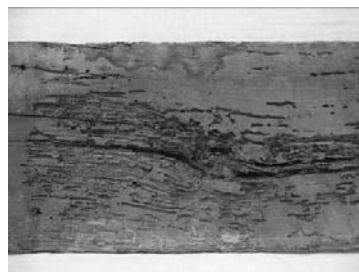
To all students and teachers of the Specialized Technical Courses in “Conservation and Restoration of Wood and Wood Furniture” and in “Conservation and Restoration of Wood Paintings”. To Museum of *Fundação Ricardo Espírito Santo Silva* for allowing the use of the objects and to *Instituto de Artes e Ofícios* where all the restoration work is ongoing.



Fig. 5 – Front view of ceiling panel n° 3



(a)



(b)

Fig. 6 – Ceiling panel n° 3. Details (a) front view with loss of material due to insect attack  
(b) back view of the same zone

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# STUDIES ON INSECT DAMAGES OF WOODEN CULTURAL HERITAGE IN LATVIA

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## Abstract

Wooden materials are frequently attacked by wood-destroying organisms, such as insects, fungi and bacteria. With respect to wooden heritage in Latvia it was for the first time attempted to clarify the situation of attack by wood-boring insects: collections of objects (buildings, furniture, other wooden artefacts) in Riga's Brivdabas Muzejs were thoroughly studied; churches in Riga, Kuldīga, Liepāja, Bauska, etc. and castles (Rundale, Tukums) as well as other buildings were investigated, starting in 2004. An overall evaluation of the on-going survey is presented with respect to attack by beetles *Anobium punctatum* (DeGeer), *Hadrobregegmus pertinax* (L.), *Xestobium rufovillosum* (DeGeer), *Hylotrupes bajulus* (L.).

## 1. Introduction

Based on initial studies in England [1-4] and parallel surveys in Germany, especially at the Westphalian Open Air Museum in Detmold [5, 6], approaches were made with regard to a conceptual application of the integrated pest management (IPM) to wood-destroying insects.

The first step of IPM is monitoring of the species damaging the timber [7, 8, 9, 11], including degree of their occurrence and categorization of the respective damages. Monitoring in this context is supervising the occurrence or the density of occurrence of populations of pest organisms, among them the wood-destroying animals. The next step of IPM [4] includes the evaluation of adequate strategies for the control measures and their application [10, 12]. Finally, the ecological parameters (i.e., conditions in the respective building) responsible for the attack need to be modified, e.g. moisture content of the specific wooden material.

In the frame of a European Community project "Integration of the Latvian State Institute of Wood Chemistry in the European Research Area", called "Evaluation of the condition of the historical and cultural wooden heritage", the first attempt was made on evaluation of wood-boring insects in Latvia and application of monitoring measures in Latvian historical wooden buildings, structures and artefacts.

The objects including one open air museum, several castles, a multitude of churches, and several other buildings (i.e., farm houses, mansions; Figs. 1, 5, 13, 15-17; Table 1) were investigated. Initial monitoring measures were applied depending on the capacity of the people in charge. Additionally, other objects will be included in the future. If not already done so, control measures will be discussed and advised to the administrators.

## 2. Material and methods

Table 1 summarizes the objects which have been visited, revisited and studied to different extent since the beginning of the year 2004.

Besides handing out information about the insects, their biology and typical characters of attack to the people in charge of the different objects, simple monitoring measures were explained and advised. These included: clean respective insect attacked areas on and around the objects, collect emerged insects and/or use white papers underneath suspected areas, in order to prove without doubt recent activity of wood-boring insects (Fig. 9).

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In Durbe's Castle specific care and awareness was advised for some storage areas with activity by AP (Fig. 13). In Rundale for the stored parts of Lestene's Church the local restorers were advised to use sticky traps with special insect glue, which were cut individually for certain areas with insect attack (Fig. 10). Furthermore, they were asked to control the beetle emergence at a frequency of two weeks (i.e., March to June).

So far, other monitoring measures, which were used in Detmold, like large-scale beetle collection, frass analyses, trapping with pheromone-, light-, or combined sticky traps, paper covers of infested timbers or other devices with sticky material were not applied in the scope of this project.

### 3. Results and discussion

Active attack by the most important wood-destroying insects furniture beetle (*Anobium punctatum* (DeGeer); AP), Deathwatch beetle (*Xestobium rufovillosum* (DeGeer); XR), Old house borer (*Hylotrupes bajulus* (L.); HB), and the anobiid species *Hadrobregmus pertinax* (L.) (HP) could be shown for many of the investigated sites in Latvia, of course with respect to specific wood species used (oak, pine, spruce, etc). Unsurprisingly, old historical and/or recently died-out infestations (Figs. 4, 6) were found in almost all artefacts (agricultural and household materials, furniture), constructions (block houses, roofs), and sacral artefacts (altars, benches, organs, pulpits). Attack by fresh-wood insects was encountered in many buildings of the Latvijas etnogrāfiskais brīvdabas muzejs, proving the use of freshly cut trees in past times for the constructions of houses, yet, of only destructive importance in the first two years after construction.

The degree of attack varied in the objects (see Table 1).

1) Only in 17 of 68 thoroughly inspected buildings of the Open air museum in Riga active attack was found (the remaining buildings [ $n \geq 30$ ] could only – if at all – be investigated in parts. Furniture and agricultural artefacts were attacked mainly by the Anobiid beetles AP and sometimes by HP. In roof beams attack by HB was encountered. Only little XR attack was found (storage room, K36, Fig. 3).

2) The situation of several churches is alarming. Bauska's Sveta Gara luteranu church (Figs. 11, 12, 17), Kuldiga's Svetas Trīsvienības church (Fig. 14, 16) and Sv. Annas luterāņu church and Liepāja's Sveta Annas church are in a terrible condition regarding overall AP attack. Other churches, including Rīgas Doms which needs to be investigated more thoroughly, seem to have only little attack by wood-boring insects. This may be due to recent renovations (Kuldigas Katrīnas, Valmieras Sīmaņa and Dzierciems churches) and/or former use of wood preservatives.

3) Parts of Lestenes church which are stored in one of the front buildings of Castle Rundale exhibit massive attack by the Deathwatch beetle XR and the Furniture beetle AP (Figs. 8-10). For the sculptures, the altar and other parts a control measure needed to be carried out. After discussing the possible measures it was decided to eradicate the wood-boring insects using the fumigant methyl bromide in October of 2005.

4) In the roof of Rundale's Castle extinct attack by HB (Fig. 7) and XR was found in different construction parts. Oak supports in the roofs need to be controlled with regard to activity of XR.



Fig. 1. Rīga, Latvijas etnogrāfiskais brīvdabas muzejs (LEM), Fisherhouse (K21)



Fig. 2. Rīga, LEM, infested Furniture (K21)

Table 1: Objects visited and studied in the frame of the project and categorization of insect attack. Abbreviated: ✓ visited, revisited, studied; 0/+//+++ degree of insect attack; ? to be evaluated; \* visited before 2004; ren. Renovated/restored.

Location	Object(s)	visit	study	attack
Rīga	Latvijas etnogrāfiskais brīvdabas muzejs		✓	+++/0
Āraiši	Āraišu ezerpils		✓	+/fungi!
Rundāles	Rundāles pils		✓	+
Tukums	Durbes muiža	✓		+
Cēsis	Cēsu pilsdrupas		✓	
Cēsis	Jaunpils			???
Ēdole	Ēdoles muiža			
Rīga	Rīgas Doms	✓		0/?
Rundāle	Lestenes baznīca		✓	+++
Bauska	Bauskas Sv. Gara luterāņu baznīca		✓	+++
Kuldīga	Kuldīgas Sv. Trīsvienības baznīca		✓	+++/+
Kuldīga	Kuldīgas Sv. Katrīnas luterāņu baznīca		✓	+/0
Kuldīga	Kulīgas Sv. Annas luterāņu baznīca	✓		++/+
Ēdole	Baznīca	✓		0/?
Liepāja	Liepājas Sv. Annas luterāņu baznīca		✓	++
Liepāja	Liepājas Sv. Trīsvienības luterāņu baznīca		✓	+++
Cēsis	Cēsu Sv. Jāņa luterāņu baznīca	✓		?
Iģene	Iģenes Luterāņu baznīca	✓		ren.+/0
Valmiera	Valmieras Sv. Sīmaņa baznīca		✓	ren.0/?
Vecpils	Vecpils katoļu baznīca	✓		+++
Krimulda	Krimuldas luterāņu baznīca	✓		0/?
Turaida	Turaidas luterāņu baznīca	✓		0/?
Dzierciems	Baznīca	✓		ren.0
Āraiši	Baznīca	✓		0/?
Zeme	Baznīca			ren.
Aloja	Alojas luterāņu baznīca			???
Turlava	Lipaiķu luterāņu baznīca			+++
Durbe	Durbes luterāņu baznīca			???
Liepa	“Kalāči”		✓	++
Veselava	Veselavas muiža	✓		+/0
Dzirciems	Pastariņa muzejs	✓		+
Bīriņi	Bīriņu muiža *	✓		+/?
Cesvaine	Cesvaines muiža *	✓		+
Piebalgas r.	Kalna Kaibēni, Kaudziņu muzejs*	✓		+

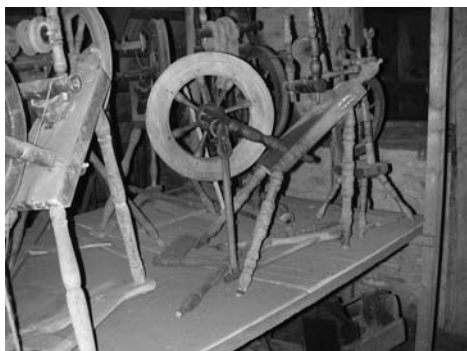


Fig. 3. LEM, storage house (K36)



Fig. 4. LEM, wagon in stable (V60)



Fig. 5. Rundāles pils (RP), internal front



Fig. 6. RP, stored infested material



Fig. 7. RP, attack by Old house borer



Fig. 8. RP, stored parts of Lestene church



Fig. 9. RP, frass from infested sculpture



Fig. 10. RP, cardboard with glue



Fig. 11. Bauska/ Gara luterāņu church, frass at alta



Fig. 12. Frass at construction



Fig. 13. Durbes castle, storage room



Fig. 14. Kuldīga/ Trīsvienības church, organ



Fig. 15-17. Kuldīga/ Katrīnas luterāņu and Trīsvienības churches, and Bauska/ Gara luterāņu church

5) Although the two latter mentioned Anobiid beetles are of main importance, *Hadrobregmus pertinax* was found with a massive infestation in one of the buildings in Kalaci as well as in the fisherman's house (K21) in the brīvdabas muzejs (Figs. 1, 2). 6) Here fresh-wood insects like *Callidium violaceum* (L.), found in building (K8) in 2004, could be attributed to freshly cut wood for heating in and near the house. Similar to the latter was the origin of frass derived from different introduced artefacts in other houses of the museum (e.g. V64).

#### 4. Outlook

Regarding the objects studied so far in the EC-project WOODPRO (QLAM-2001-00360, "Integration of the Latvian State Institute of Wood Chemistry in the European Research Area") it became obvious

that there is a great need of increasing the number of thorough investigations of other building types with reference to age and geographical region, including the use of monitoring measures.

Furthermore, curative control measures are required. Curative wood preservatives are still commonly used. Fumigations or humidity-regulated heat treatments would be required for several massively attacked buildings, especially churches, for either overall treatment or treatment of specific sections. For portable small artefacts or larger ones up to the size of wagons (Fig. 4) it may be advised to use a mobile or stationary thermal chamber which could be used by several institutions demanding treatment of infested objects.

### Acknowledgements

Uwe Noldt thanks for all cooperation: Dr. Martins Kuplais (Latvijas etnogrāfiskais brīvdabas muzejs, Riga); Dr. Hubertus Michels, Daniel van't Hull, Oliver Junk (WFM Detmold); Helmut Maintz (Domkapitel Aachen); Dr. Alexander Niederfeilner (MBW Tittling); Tilo Haustein (Hochschule Wismar).

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# TERMITE INFESTATION RISK IN PORTUGUESE HISTORIC BUILDINGS

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## Abstract

In mainland Portugal, subterranean termites of the genus *Reticulitermes* are indigenous and a well-established pest of wood in service. This has particular relevance in the maintenance of historic buildings. Dry wood termites of the genus *Cryptotermes* are now also recognized as a major problem in the conservation of old buildings in the islands of Madeira and in several of the Azorean Islands. The present recorded distribution of dry wood and subterranean termites in Portugal is discussed. The very recent discovery of *Cryptotermes brevis* in Lisbon is highlighted and the possible impacts of this finding discussed.

## 1. Introduction

Termites are social insects found in a wide range of terrestrial environments and are distributed throughout the warmer regions of the world. They are very important organisms ecologically as they significantly contribute to the organic decomposition process either by direct consumption of decomposing plant materials, by physical and chemical conditioning the soil they inhabit and by nitrogen fixation. They feed on a very wide variety of organic detritus like dry grass, decaying leaves, animal dung, humus and living or dead wood.

As termites forage on cellulosic resources they can cause damage to living trees and many crop plants, but the fact that they can use dead wood makes them a major pest for timber used for construction purposes both outdoors and inside buildings. They are known to cause damage to buildings throughout the tropics, sub-tropics and temperate regions and have an increasing economic impact when present [1].

Several factors are suggested to explain their special distribution but the absence of a sufficiently high temperature for long periods of the year seems to be chiefly responsible for the absence of termites. However, an increase in the demand for wood products for construction linked with the growth of human population and the much discuss “global warming”, will most likely favor the spread of the pest species well beyond their natural distributions. Anthropogenic climate changes and the increased use of central-heating systems can already be linked to the establishment of *Reticulitermes sp.* in more northerly cities such as Paris, Hamburg and Toronto, or in Saunton, U.K. [2].

In Portugal, the first known reference to the presence of subterranean termites, *Reticulitermes lucifugus* (Rossi), dates back to the end of the XIX century [3]. They occur naturally over the entire country, and are a well-established pest to wood in service particularly linked to historic buildings [4, 6, 7, 10]. A second termite species can be easily found on the mainland of Portugal though more often in the natural habit than linked to applied wood, the drywood termite *Kalotermes flavicollis* Fabricius.

In the Portuguese islands of Madeira and Porto Santo, Mateus and Goes [8] reported the presence of another drywood termite, in this case also a known building pest, *Cryptotermes brevis* (Walker). Carvalho [9] referred to the existence of two endemic species (not linked to construction) in the Island of Madeira, *Cryptotermes barretoii* (Grassé) and *Neotermes precox* (Hagen). *C. brevis* was also recently identified in the Islands of the Azores [10, 11]. Further details of the distribution of termites in Portugal are given in the following chapter.

## 2. Distribution

### 2.1. Subterranean termites

In continental Portugal, subterranean termites are a well-establish pest of wood in service and all known infestations involve termites from the species formerly referred to as *Reticulitermes lucifugus*

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(Rossi) and presently known as *Reticulitermes grassei* Clément [12] which is indigenous. The same species was also identified in the city of Horta in the Azorean Island of Faial [11, 16].

Fig. 1 shows the present known distribution of *R. grassei* in continental Portugal, considering all municipalities where at least one record of the presence of termites exists. The map is based on LNEC's data base of termite occurrence, mostly in buildings but also in the natural environment. At present, the data base has close to 1000 records.

In particular, the region of Lisbon and its surroundings is considered severely infested, showing the highest total number of reported infestations. Fig. 2 shows the present known distribution of *R. grassei* in Lisbon, considering all boroughs (48 out of 53) where at least one record of the presence of termites exists.

Taking into account the biology of the species and the present known distribution, *R. grassei* seems to have a wide distribution, apparently without latitudinal, longitudinal or altitudinal patterns [4, 7]. Municipalities or boroughs without records should not be seen as zones without termites just zones where termites have not yet been identified.

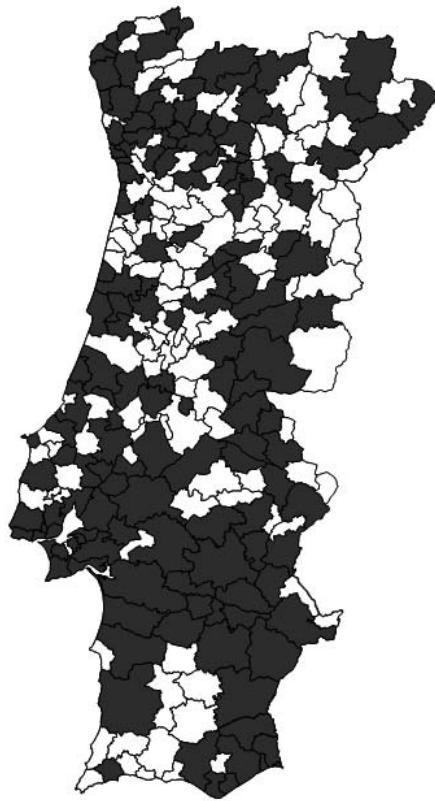


Fig. 1: Subterranean termites distribution in Continental Portugal. Municipalities with at least one recorded case of *Reticulitermes grassei* presence.

The presence of termites in buildings follow a general pattern closely linked with the amount of susceptible timber used in the construction and eventual building deficiencies occurring during the life of the construction and causing increased moisture contents of the applied wood.

In what concerns the presence of subterranean termites in heritage buildings or building sites, no systematic studies have been so far conducted but the presence of termites has been already recorded in several important World Heritage sites like the Monasteries of Jerónimos in Lisbon and Santa Maria of Alcobaça, the Convent of Christ in Tomar and a number of buildings in the city of Évora like the old University and the churches of Santa Clara and Misericórdia.

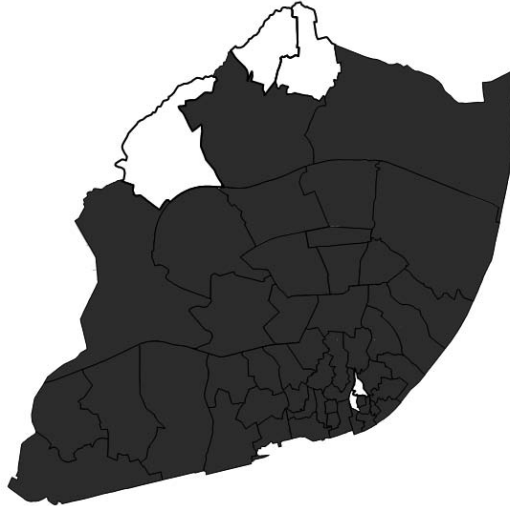


Fig. 2: Subterranean termites distribution in the city of Lisbon. Boroughs with at least one recorded case of *Reticulitermes grassei* presence.

These buildings have all undergone in recent years large interventions, to correct mainly moisture problems, which were likely to contribute to the control of subterranean termites. The rehabilitation and maintenance works should have had taken into account the presence of the insects and were hopefully adequate to their control.

Nevertheless, in many other rehabilitation works the termite risk is often overlooked and it is not unusual to find situations where infested timber is left as rubble in hidden parts of the repaired constructions and new susceptible timbers are used without any preventive treatments or control measure that would prevent termite re-infestation.

## 2.2. Drywood termites

Drywood termites are social insects as well, but unlike subterranean termites, they live inside the wood they infest. This characteristic facilitates their dispersion, thus being easily transported with infested materials and, once introduced in suitable environments, colonies can survive and establish. *Cryptotermes brevis* is considered to be the termite species that has suffered more introductions and the most important drywood termite with pest status.

*C. brevis* possess a lifecycle with caste division (reproductives, nymphs, pseudergates and soldiers) as all social insects. Every year (usually starting in June), the primary reproductives or alates emerge and are responsible for the colonization of new areas. Due to their feeding habits on drywood, all untreated timbers can become a potential food source and suffer destruction.

*Cryptotermes brevis* is native of the Caribbean region, being present in all islands of the Greater and Lesser Antilles. It occurs in southern Florida and coastal regions of Mexico, Central America and northern South America. It has been introduced to Pacific islands including the Galapagos, Fiji, Hawaii, Marquesas, New Caledonia and Easter Island. It is known to be also present in the Atlantic Islands of Ascension, Bermuda and St. Helena. It has also been introduced to the coastal areas of continents including several locations in Australia, China, Madagascar, Gambia, Ghana, Nigeria, Senegal, Sierra Leon, South Africa, Uganda and Zaire [13,14]. In Europe, references exist on the presence and establishment of *C. brevis* in Canary Islands [8], Madeira Island [8], Naples, Liguria and Sicily [15].

*C. brevis* was identified in the Islands of Azores in 2002 though alates and wings had been noted by a number of people in the previous years without clear recognition of their origin. An assessment of the

situation on the several islands of the Azorean archipelago has already begun and so far well established populations of this pest termite were found in the Islands of S. Miguel, Terceira, Faial and Santa Maria [11, 16].

In Angra do Heroísmo, a World Heritage Site, Borges and co-workers [17] refer a progressive degradation, which is often alarming to the architectural heritage, showing the impact of this termite species as a building pest. The data available indicates that 43 % of the buildings in Angra do Heroísmo were infested. It is also important to mention that 50% of the infested cases presented severe infestation and destruction.

The most infested parts of the buildings are usually the roof structures. Drywood termites may enter through attic ventilations or other entries that might exist (doors or windows). Once installed inside the wood (possibly using holes already made by wood borers like *Anobium* spp.) the holes to the exterior are sealed and the identification of their presence becomes even more difficult.

The origin of the termites in the Islands of Azores is still undetermined, though their distribution in Angra do Heroísmo (heavy infestation extending from the harbour) suggests an entry in the port area at least several decades ago, possibly at the time of the last major earthquake (in 1980) and associated with the extensive rebuilding that took place at that time [10].

Buildings in the Azorean cities historical centres usually are masonry buildings, most of them three and four stories high, with timber floor and roof structures. Each building may share, with the adjacent buildings, its lateral two walls, and several buildings in these conditions form a city block acting in the urban panorama as an unitary building structure.

This kind of construction is common in buildings constructed after the XIX century and can also be found in several cities in Continental Portugal and particularly in Lisbon (Fig. 3). Lisbon shares also with the most termite infested cities of the Azores, the proximity to the sea and the continuous flow of wood, mostly cryptomeria, that arrive at its port from those islands. Infested furniture is traditionally one of the most important means of introducing the pest and should also be considered as a serious risk taking into account the normal circulation of people and products between Portugal and the Azores.

It was therefore not very surprising the recent identification by the author of a well established infestation by *Cryptotermes brevis* (Fig. 4) in a building located in a Lisbon street (Rua da Junqueira) not far from the river Tagus and the port area but also very close to important sites like the Monastery of Jerónimos in Belém.

Further work is needed to determine whether the presence of *C. brevis* in this building is just the result of a recent hazard introduction or the first symptom of a bigger problem in the city of Lisbon



Fig. 3: Typical Portuguese masonry buildings from the late XIX<sup>th</sup> century or the early XX<sup>th</sup> century in Rua da Junqueira, Lisbon.

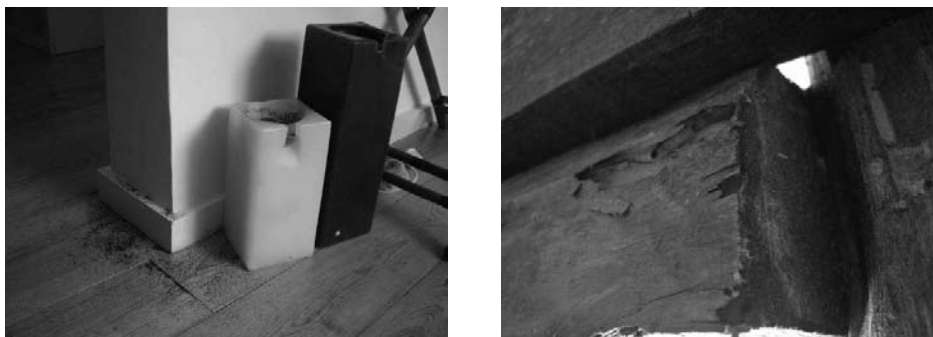


Fig. 4: Details of the infestation by *C. brevis* in Rua da Junqueira, Lisbon. Termite fecal pellets on the floor and aspect of an infested timber beam.

### 3. Final considerations

The growth of urban populations of pest termite species is an increased matter of concern. The importance of termite attack risk assessment in any scheme of monitoring, prevention and control of the condition of timber in buildings cannot be understated. The determination of potential high-risk areas where termites can establish and multiply themselves is a necessary task to be undertaken in order to prevent further spreading of the urban pest species.

Understanding the range of subterranean termite infestation problems in order to predict (to some extent) the occurrence and severity of potential problematic areas in countries like Portugal, where termites have a wide range, not obviously defined by temperature, humidity, vegetation or soil type, the mapping of risk areas needs to consider variables specific to the construction type and its immediate environment.

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# BIODETERIORATION OF CULTURAL HERITAGE IN ESTONIA

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## Abstract.

There are about 5000 buildings of some historical value in Estonia with more than 90% of them involving wooden elements or timber structures. The present paper provides data obtained by a large-scale survey of hundreds of buildings of cultural value to assess the extent of fungal decay. The survey found the dry rot fungus (*Serpula lacrymans*) to be the most invasively spread fungus that buildings all over Estonia have been infested by. *Coniophora puteana* is the second most widely spread fungus, though it's significantly less invasive than *S.lacrymans*. Beside the above two fungi, *Leucogyrophana spp.*, and *Antrodia spp.* are extensively present in Estonia. In addition to wood decay fungi, there are moulds that can also attack buildings and elements of cultural value. Although the mould used to be largely neglected in Estonia due to the lack of expertise, since the import of the relevant know-how from Finland and Sweden the moulds such as *Aspergillus spp.*, *Cladosporium spp.* and *Penicillium spp.* have been identified and counter-measures are being taken. The insect damage was also found to be rather extensive. While in the continental Estonia *Anobium punctatum*, *Anobium pertinax* and *Buprestis haemorrhoidalis* are the most widely spread, the islands have a fourth species, i.e. *Hylotrupes bajulus* as well. Further studies will involve measuring the strength of structural timber, assessing the extent of fungal decay and elaborating recommendations for renovating the buildings of cultural value. The results of the study can be applied by the renovation companies and the administrators of the buildings of cultural value.

**Keywords:** Cultural Heritage, Dry rot, Wood-destroying fungi, Wood-disfiguring fungi, *Serpula lacrymans*, *Coniophora puteana*

## 1. Introduction

As of 01.10.08, 23 931 items, including 12 966 items of arts heritage, have been officially registered as items of cultural heritage of the Republic of Estonia (See the classification in Fig. 1). The majority of the items of arts heritage are deposited in and protected by museums and arts depositories, with KUMU in Tallinn being the largest among them. While the depositories have the facilities to maintain appropriate conservation conditions and major problems with biodeterioration can be prevented, the items of architectural heritage (5277 in total) run the highest risk of biodeterioration. Across the centuries, timber and limestone have featured as the main building materials in North, North-East and West Estonia (including islands), and stones and clay in Central and South Estonia. Therefore, in most of the buildings timber is either the material applied throughout or in certain parts of the building, while pine and spruce as the species indigenous of this latitude are the most frequently resorted to. The main types of deterioration include the fungal attack, beetle infestation and chemical and physical change [1] of which the present paper addresses the first two detected in the timber parts and elements of 938 buildings (including 136 items of architectural heritage) surveyed in Estonia in 1999-August 2008.

The fungal attack can be caused either by wood-destroying fungi or by wood-disfiguring fungi [2]. Although the surveyed timber items of cultural heritage featured both of the above types, the former appears to be substantially more destructive. The wood-destroying fungi can be further divided into basidiomycete wood-rotting fungi and soft rot fungi, and the wood-disfiguring fungi into bluestain fungi and moulds, respectively [2]. In addition to the fairly rough classification described, Schmidt (2006) in "Wood and Tree Fungi" provides a more complex taxonomy which distinguishes between wood rot fungi including Brown Rot, White Rot and Soft Rot, and wood discoloration fungi including Moulds, Bluestain and Red streaking, respectively [3].

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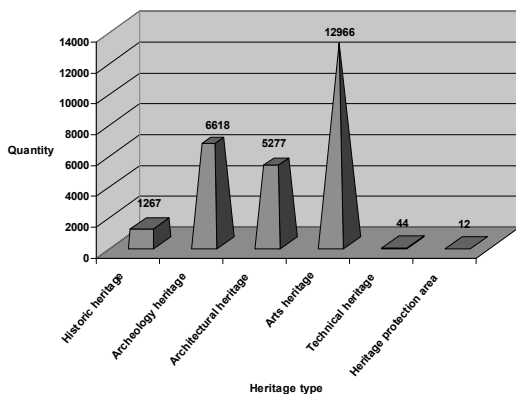


Fig. 1. Cultural heritage of the Republic of Latvia

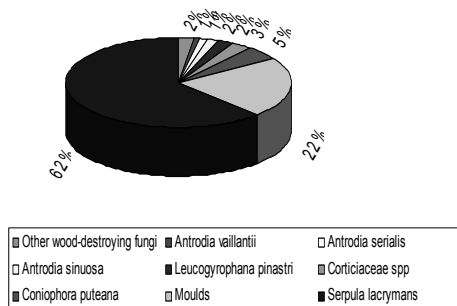


Fig. 2. Wood destroying fungi

## 2. Methods and equipment

Different methods are applied to identify different species of fungi and scope of damage. Wood-destroying fungi are identified from dust and/or wood samples. Micro-items isolated from the dust sample collected from items or materials are inspected by the microscopic examination of 600-fold enlargement. The above described method is used to detect variation among the isolated spores in terms of shape, colour and number to identify the most common wood-destroying fungi present in a building. The wood samples including mycelial strands and/or fruitbodies are collected from the damaged material and the fungi are identified on the basis of morphology, colour and development (conidiogenesis) of conidio and conidiogenous cells. This is the method widely applied in other research centres. For further refinement, there are DNA-based techniques available, of which the PCR method is most often resorted to. In several cases the identification can be performed by visual survey of the shape and colour of the mycelial strands and fruitbodies (Photo 1).

Photo 1. Typical fruitbody of *Serpula lacrymans*.

Moulds are identified by material sample (in the presence of distinguishable colonies) and/or air sampling by plating it (most commonly on malt-extract agar MEA). When quantitative data are sought, Loreco Impaktor-FH3 is used to analyze air volumes of 100 to 500 litres. The samples are incubated for 7 days at 25°C and the number of moulds is counted in CFUs. The species are identified by microscopic examination after the sample is treated with Methylthionium chloride. The fungi are identified on the basis of morphology, color and development (conidiogenesis) of conidia and conidiogenous cells.

The beetles are identified by measuring the size of cavities, visual examination of the damage patterns, and collecting sample species or eggs-larvae from inside the wood [5].

The relative air humidity and the absolute moisture content of the materials are determined with Protimeter Moisture Measurement System and GANN Electronic Moisture Meter Hydromette UNI 2.



The survey on enclosed structures was performed with Olympus Fiberoptic device R080-063-045SW115-50.

### 3. Fungal decay of wood in architectural heritage of Estonia

The distribution of detected fungal decay of wood is presented in Fig. 2 where *Serpula lacrymans* and moulds account for 62% and 22%, respectively. As the survey was performed on culturally valuable buildings at risk rather than at buildings selected at random, Fig. 2 reflects the distribution of fungal decay recognized in the buildings examined. The decay caused by *Coniophora puteana* is most widely recognized because of its high rate of growth and extensive scope of damage.

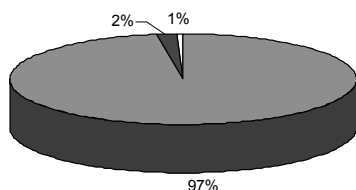


Fig. 3 Types of wood rot

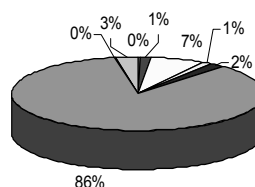


Fig. 4 Brown rot fungi

#### 3.1. Wood-destroying fungi

The majority of basidiomycete wood-rotting fungi present in the buildings in Estonia account for the development of brown rot (Fig. 3). White rot has been detected in and around the foundation and in the structures placed below the ground level. Soft rot fungi have only been identified in 3 cases in the Timber Foundations of the Old Town of Tartu. The most frequently recognized causes of brown rot include *Serpula lacrymans* and *Coniophora puteana*, [4] along with *Leucogyrophana spp.* and *Antrodia spp.* [8] (Fig. 4). The data in Table 1 suggest that all the above fungi primarily affect the softwood inside the buildings. *Serpula lacrymans* is common in cellars and in the ground floor structures and less common in the wall structures and upper floor structures. In the latter case, the structures built in 1700-1930 are most extensively infested as these were stuffed with loam for insulation and fire protection. This type of filler keeps wet and provides fungi with alkaline and acid elements to maintain the PH-level that promotes their growth. *Coniophora puteana* mainly affects cellars and the ground floor structures (mostly when the cellar is missing). *Antrodia spp.* is bound to flourish around the leaks in the roofs and window openings though different species of *Antrodia* can also inhabit the areas around leaking pipes or rooms of high humidity. Different species of *Leucogyrophana* are also present in cellars and around the leaks in pipes but they can spread all over the building. The *Corticaceae* appear to be attracted to leaks in floor structures.

Table 1. Some common brown-rot fungi [3]

Fungus	Predominant occurrence				
	standing tree	timber outdoors	timber indoors	softwood	hardwood
<i>Laetiporus sulphureus</i>	x				x
<i>Phaeolus schweinitzii</i>	x			x	
<i>Piptoporus betulinus</i>	x				x
<i>Sparassis crispa</i>	x			x	
<i>Gloeophyllum spp.</i>		x		x	
<i>Daedalea quercina</i>		x			x
<i>Lentinus lepideus</i>		x		x	
<i>Paxillus panuoides</i>		x		x	
<i>Antrodia spp.</i>			x	x	
<i>Coniophora spp.</i>			x	x	
<i>Serpula lacrymans</i>			x	x	
<i>Meruliporia incrassata</i>			x	x	x

### 3.2. Wood-disfiguring fungi

As the present paper is focusing on the heritage of architecture, the problems generated by the blue stain fungi found in fresh sawn timber and recently placed structural timber are ignored. It is the mould that is responsible for most of the damage in buildings and items of cultural value. Due to the lack of expertise they used to be largely neglected in Estonia till the beginning of this century although the influx of relevant know-how from Finland and Sweden has enabled to identify damage by moulds and take measures against it.

Moulds primarily affect items and the surface of materials in buildings in three ways. Firstly, the materials can deteriorate due to the spread of mycelium. As the development of mould mycelium and the amount of organic nutrients is many times smaller than that of wood-destroying fungi, destruction of materials is a problem when the long-term conservation of materials is an issue. Secondly, the deterioration can be induced by the pigments imparted by moulds to the surrounding materials, whereas this damage is most substantial in art heritage because of the change in colours it leads to. Thirdly, there is the chemical deterioration, which is instigated by the new compounds resulting from the chemical reaction involving the substances imparted by the mycelium on the one hand and the substances present in the surrounding materials on the other [6]. The described process can lead to the changes in the chemical contents of the coloured surfaces.

Moulds can have ill effects on the health of the inhabitants most of which are responsible for allergy and hypersensitivity reactions. The four types of reactions distinguished are as follows:

1. Allergic reaction whereby the spores of fungi inhaled induce the generation of antibodies. If the person encounters the same antigens, the latter and antibodies interact and certain activating agents released can induce symptoms like sensitive mucosa or dyspnoea.
2. Cytotoxic reaction is an allergic condition whereby the cells fight other cells of the body. This type is of low prevalence and occurs e.g. in infants with rhesus-conflict.
3. Immune complexes are developed as a result of recurrent and frequent inhalation of antigens (the volatile metabolites of an allergen, e.g. fungal spores or micro-organisms). [4]
4. Contact hypersensitivity due to the recurrent contact of skin with nickel, chrome etc. elements. The powerful allergens in Estonia include some species of *Cladosporium* and *Aspergillus*, e.g. *Cladosporium cladosporoides*.

In addition to causing allergic reactions some moulds produce endotoxins and mycotoxins [7] that spread through air and cause various ill-effects. *Stacybotrus chartarum (atra)* is the species of mould active in Estonia. Beetle infestation also has a substantial share among the causes of deterioration of cultural heritage. Beside the *Anobium punctatum*, *Anobium pertinax* and *Buprestis haemorrhoidalis* most widely spread in continental Estonia, the islands also have the House longhorn beetle (*Hylotrupes bajulus*) (Photo 2).



Photo 2 Beetle attack in the old church in Ruhnu

#### 4. Causes of biodeterioration

Over the last century, the basidiomycete wood-rotting fungi and beetle infestation have seen two periods of high activity in Estonia. After World War Two when lots of buildings including those of cultural value had to be renovated and repaired, the wood-destroying fungi easily spread all over Estonia. Because of the substandard building materials applied, the timber used often had inadequately high moisture content. The aftermath of this period lasted up until mid-1960s. The beginning of another period of high fungal activity dates back to late 1980s and early 1990s and occurred due to the gaps in the building code and lack of supervision. The second period mainly affected private houses, cottages and auxiliary buildings and no extensive damage to the cultural heritage was recognized. Research on wood-destroying fungi has established a clear-cut relationship between the rate of wood deterioration and age of wood in the building. In many buildings the old slightly damaged elements were and are replaced by new elements of the same size and shape whereby the growth of destroying fungi is further enhanced as the spread rate in fresh cut timber is manifold higher than in the old one.

Causes of fungal attack and beetle infestation in Estonia, Latvia and Lithuania differ from those in the rest of EU countries. During the 50 years of Soviet rule the inadequate renovation and maintenance system and low-quality performance in construction companies resulted in unreasonable and technically inadequate changes in architectural heritage which account for the following causes of fungal deterioration:

Changes in the moisture pattern of the ground floors, where due to the elevated road and surrounding areas the vent pipes have been stopped up and this leads to the changes in the moisture pattern in the ground floor and lower part of walls.

The floor structures changed during renovation, e.g. replacing the floor boards (damaged by fungal attack) by concrete floors in turn resulted in changes to the moisture pattern in the lower part of walls and indirectly affected all the building. Placing concrete floors in some parts of the building deteriorated the performance of under-floor ventilation and the remaining wood flooring was easily attacked by fungi. Besides, the timber floors were often replaced by low-quality flooring, which accelerated the process of fungal decay.

Changes in the function of buildings and rooms, e.g. a church performing as a sports hall or chapels and manor houses functioning as warehouses in agricultural production. These changes did not only affect the moisture regime, they also involved extensive changes to the building structure itself, thus leading to substantial changes to the exterior looks of the buildings of historic value.

Building damp rooms (shower-rooms, saunas, pools, etc.) in old-age buildings the initial design of which did not feature these facilities. The damp rooms were insulated by various plastics that substantially contributed to the deterioration of the surrounding timber elements.

Thermal insulation installed at historic buildings caused changes in the moisture and thermal regime of the buildings resulting in condensation water that in its turn increased the moisture content of the surrounding timber elements.

Inadequate performance of roof and rainwater runoff occurred as a rule due to the inadequate or missing maintenance of buildings and resulted in the deterioration of the whole building and the longer was the period of neglect, the more extensive was the damage incurred.

#### Summary

Humidity control is the best measure to prevent biodeterioration in buildings of cultural heritage and requires the installation of temperature and humidity meters in all the buildings of architectural heritage and places that house items of art heritage. The detected increase in humidity calls for an effective analysis suggesting the measures to be taken.

Research on wood-destroying fungi has established a clear-cut relationship between the rate of wood deterioration and age of wood in the building. In many buildings the old slightly damaged elements were and are replaced by new elements of the same size and shape whereby the growth of destroying fungi is further enhanced as the spread rate in fresh cut timber is manifold higher than in the old one.

Therefore, if the apparently damaged elements pass the tests for strength, the elements should remain in the structure and require treatment by fungicides to stop the process of decay.

Renovation plans of timber structures require major addenda to reflect the new developments such as testing of timber elements for strength, determining the extent of damage and a list of recommendations for renovating buildings of cultural heritage. The results of the research will be available to the renovation companies and the institutions responsible for the maintenance of cultural heritage.

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# DEGRADATION OF MELANIN AND BIOCIDES BY LIGNINOLYTIC FUNGI

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## Abstract

Molds which produce the black pigment melanin overgrow wooden art objects, especially when the artifacts are stored in high air humidity. To protect wooden object against pests they were often treated with organochlorine biocides such as lindane, sodium pentachlorophenol (PCP) and polychlorinated biphenyl (PCB). Both melanin and biocides have a negative influence on the objects – melanin as aesthetic damage and organochlorine biocides as toxins, which pose a serious threat to humans and the environment. Therefore, there is an increased need for development of methods to remove both from the objects. Melanin and organochlorine biocides are extremely recalcitrant organic compounds and cannot be removed from the art by classical conservation procedures. Since several organic biocides, as well as melanin, are chemically similar to lignin, white rot fungi and their oxidative enzymes are able to degrade them.

Melanin degradation by laccase was studied on artificially molded filter paper samples. The most pronounced bleaching of the molded paper samples was observed in the treatment at heightened air pressure. Mycoremediation potentials of four white rot fungi (*Pleurotus ostreatus*, *Trametes versicolor*, *Chondrostereum purpureum*, *Hypoxylon fragiforme*) and one brown-rot (*Gloeophyllum trabeum*) were evaluated for the degradation rate of lindane, PCP and PCB, respectively. *P. ostreatus* showed the highest efficiency in degrading lindane and PCB, while PCP was most efficiently degraded by *T. versicolor*.

Keywords: molded objects, biocides, mycoremediation, white rot fungi, oxidative enzymes, melanin bleaching, melaninised paper, object detoxification.

## 1. Introduction

Molds often infest wooden art objects, especially when the artifacts are stored in high-humidity environments. Molds produce the black pigment melanin (an aromatic polymer that can be found in spores, embedded within hyphal cell walls, and even found extracellularly), which causes undesirable stains on the surface of the objects [1]. Melanin is an extremely recalcitrant polymer and cannot be removed from the objects by classical restoration procedures. Therefore, melanin traces remain on the surfaces as aesthetic damage. Melanin is chemically very similar to lignin, a major constituent of wood. Therefore white-rot fungi and their oxidative enzymes are capable to degrade melanin, also [2]. These enzymes were tested for their ability to selectively degrade melanin.

Molds are not the only biological threat to objects of cultural heritage; wood decay fungi and insects can also cause severe damage. Therefore several different organic biocides were used to protect the objects. Unfortunately, many of these compounds are nowadays recognized to be harmful for humans and environment [3]. There is an urgent need to remove biocides from the treated objects, which would enable safe research to the curators and safe handling to collection managers and conservators. It is also especially important to enable safe exhibition of the objects to visitors.

Since several different organic biocides, like melanin, are chemically similar to lignin, white rot fungi and their oxidative enzymes were tested for their degradation and thusly removal of the xenobiotics from the objects [4], [5], [6]. Lignin peroxidase catalyzes the oxidation of nonphenolic lignin-like structures to aryl cation radicals and has been shown to oxidize several polycyclic aromatic hydrocarbons (PAHs) by the same mechanism [7]. Manganese peroxidase also promotes the peroxidation of unsaturated lipids, and under these conditions nonphenolic lignin-like structures are oxidized [8], [9]. The activity of laccase is restricted to compounds with low ionization potentials such as phenols. However, it has been shown that the substrate range for laccase extends to nonphenolic

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lignin-like structures when so-called mediator compounds such as 2,2'-Azinobis-(3-ethylbenzthiazolin-6-sulfonate (ABTS) or hydroxyanthranilic acid (HAA) are present [10], [11]. Free radicals, appearing as products of these reactions, perpetuate oxidation of the organic pollutants [12]. Once the peroxidases have opened the aromatic ring structures by introduction of oxygen, other more common species of fungi and bacteria can intracellularly degrade the products into CO<sub>2</sub> and other benign compounds.

The aim of the investigation was to estimate the bioremediation potential of the selected wood decaying fungi and their enzymes to degrade melanin, as well as polychlorinated organic biocides lindane, sodium PCP and PCB.

## 2. Materials and Methods

### 2.1.

#### Organisms:

Brown-rot basidiomycete *Gleophyllum trabeum* (strain Gt2) and three white-rot fungi: basidiomycetes *Pleurotus ostreatus* (strain Plo5), *Trametes versicolor* (strain Tv6), and ascomycete *Hypoxylon fragiforme* (strain Hf) were obtained from the ZIM collection at the Biotechnical Faculty, Ljubljana.

#### Enzymes:

Laccase (Lac) from *T. versicolor* was from Sigma (Germany) and Fluka (Germany). Manganese peroxidase (MnP) from *Nematoloma frowardii* and Versatile Peroxidase (VP) from *Bjerkandera adusta* were from Jena Bioscience (Germany).

#### Chemicals:

Polychlorinated biphenyl (PCB-153=PCB) was from Dr. Ehrenstofer GmbH (Germany). Sodium pentachlorophenol (NaPCP=PCP) and MnSO<sub>4</sub>×1H<sub>2</sub>O were from Fluka (Germany). Lindane ( $\gamma$ -hexachlorocyclohexane), L-asparagine and were from Sigma-Aldrich (Germany). Veratryl alcohol (3,4-dimethoxybenzyl alcohol) was from Acros Organics (USA). All other chemicals (D-(+)-glucose, KH<sub>2</sub>PO<sub>4</sub>, MgSO<sub>4</sub>×1H<sub>2</sub>O, KCl and n-hexane) were reagent grade and obtained from Merck (Germany). PDA (potato dextrose agar) and yeast extract were from Difco (USA).

#### Culture conditions:

The liquid medium used for fermentation experiments (liquid cultures) contained 110 mM glucose, 5 mM asparagine, 2 mM MgSO<sub>4</sub>, 7 mM KH<sub>2</sub>PO<sub>4</sub>, 7 mM KCl, 2 mM MnSO<sub>4</sub>, 2 mM veratryl alcohol (3,4-dimethoxybenzyl alcohol), and 0.5 g of yeast extract per liter. The initial pH value was adjusted to 4.5.

### 2.2. Preparation of melaninised paper:

Pieces of autoclaved filter paper (Whatman filter paper #2,  $\Phi$  55 mm) were put in Petri dishes, and 5 ml of the above-mentioned suspension containing mould spores was sprayed onto each piece. In approximately one week the paper was overgrown with the mould (with their hyphae entangled in its structure) and pigmented. Blackened filter papers were dried in an oven for 3 days at 60 °C.

### 2.3. Treatment of melaninised paper

Five Petri dishes ( $\Phi$ 120 mm) with 20 ml of different reaction solutions (Na-malonate buffer (0.25 M, pH 4.5) (control); buffer + HBT (5 mM); buffer + laccase (1 EU/ml); buffer + laccase (1 EU/ml) + HBT (1 mM); buffer + laccase (1 EU/ml) + HBT (5 mM)) were prepared for this experiment. Ten melaninised paper samples were put in each of five Petri dishes, and they were placed in a pressure chamber (Kambič, Slovenia) to improve solubility of oxygen in the reaction solutions (72 h, 10 bar, 30 °C). After the treatment, samples were removed from Petri dishes, carefully washed in distilled water and oven-dried (3 days at 60 °C).

### 2.4. Measurements of color changes in melaninised paper samples

Changes in color of melaninised paper samples were investigated with a colorimeter (Microcolor Data Station, Dr. LANGE). Effectiveness of the treatment (bleaching) was determined as a change in lightness ( $\Delta L$ ) in the CIE L\*a\*b system (L\* - lightness values from 0 (absolute black) to 100 (absolute white); a\* - a color value on red-green axis; b\* - a color value on yellow-blue axis). Each melaninised

paper sample was measured for its color before and after the treatment, and the difference in  $\Delta L$  values indicated the effectiveness of the particular treatment. The lightening of the samples results was expressed in percent of change in  $\Delta L$  values.

## 2.5. Biodegradation of biocides

### Liquid fungal cultures:

300 ml Erlenmayer flask containing 50 ml of liquid medium was inoculated with three mycelial particles (8 mm in diameter) obtained from the mycelia cultures of the respective fungi. The inoculated flasks were sealed and kept agitated at 130 rpm and 25 °C in the dark for 22 days. On the day seven, the liquid cultures were respectively supplemented with lindane, PCP or PCB in the final concentrations of 30  $\mu\text{mol}$  per liter and *were inoculated with respective fungal species. The media without biocides were used as controls.* The growth rates were estimated after a 22-day growing, when the mycelia of the respective fungi reached the stationery phase.

### Enzymatic degradation of chlorinated organic biocides in vitro:

Each 25 ml Erlenmayer flask containing 4 ml of enzyme buffer (20 mM Na-acetate, 1,61 mM  $\text{MnSO}_4$ , 4,29 mM  $\text{Na}_2$ -oxalate in 1,07 mM  $\text{H}_2\text{O}_2$ ; pH = 4,5) was supplemented with respective ligninolytic enzyme laccase, manganese peroxidase or lignin peroxidase in the final concentration 1 enzyme unit (EU) /ml (EU was defined as an amount of the enzyme that oxidises 1  $\mu\text{mol}$  of substrate ABTS into radical  $\text{ABTS}^{\bullet+}$  in 1 minute). The enzyme solutions were then supplemented with respective chlorinated phenols (30  $\mu\text{M}$ ) or with lindane (15  $\mu\text{M}$ ) and incubated on a shaker at room temperature in dark for 4 days (96 hours).

### Gas chromatography analyses (GC):

The samples of lindane and PCB were prepared by extraction with hexane, which was added 1:1 volume ratio. GC analyses were performed on a Hewlett Packard 6890 Series chromatograph (Hewlett Packard, USA) equipped with an Electron Capture Detector and HP-1 column (dimensions 30 m  $\times$  250  $\mu\text{m}$ ; 0.25  $\mu\text{m}$  thickness of poly-dimethylsiloxane (PDMS) as stationery phase) (Hewlett Packard, USA). The injector and detector temperatures were 250 and 300 °C, respectively. The initial column temperature was set to 70 °C and programmed to increase to 250 °C (gradient 30 °C  $\text{min}^{-1}$ ), then to 270 °C (5 °C  $\text{min}^{-1}$ ) and finally to 300 °C (15 °C  $\text{min}^{-1}$ ). The  $\text{N}_2$  flow rate was set to 2 ml  $\text{min}^{-1}$  and 1  $\mu\text{l}$  of a sample was loaded each time for the analysis.

### High pressure liquid chromatography analyses (HPLC):

The presence of PCP in samples was analysed with Separation Products Constametric 4100 chromatograph (Thermo Separation Products, USA) equipped with an UV-VIS spectrophotometric detector (LDC Analytical, USA). Separation was performed on ODS 135  $\times$  4 mm 5  $\mu\text{m}$  column (Thermo Scientific, USA) with isocratic elution (60 % acetonitril, pH 2) at flow rate 1.4 ml  $\text{min}^{-1}$  within 15 min of single chromatographic run. Before loading, the samples were filtered through Whatman filter papers.

## 3. Results and Discussion

### 3.1. Color changes of melaninised paper

The treatments showed that laccase/HBT system caused the most prominent changes in melanin composition compared to other treatment procedures in the experiment. As an optimisation, the treatment procedure for lightning of melaninised paper was conducted at elevated air pressure, resulting in higher oxygen concentration in the solution and therefore higher laccase efficiency. Several reaction mixtures were tested for the ability to lighten melaninised paper. As expected from the results on degradation of extracellular DHN melanin in suspensions, laccase/HBT system in 1:5 molar ratio of HBT against laccase activity proved to be the most effective (Fig. 1). Visual inspection of the treated samples indicated lightening, and this indication was confirmed with colorimetry measurements. Therefore it was proven that an isolated laccase (with the mediator HBT) is able to change the appearance of melaninised paper samples in the desired direction via degradation and oxidation of DHN melanin, the responsible agent (pigment) in aesthetic devaluation of paper. The concentration of oxygen diluted in the reaction solution was apparently high enough that laccase was

able to oxidise the majority of the available HBT (inhibitive competition of reduced forms of HBT was prevented), and the oxidation of melanin was more successful than in any of the other combinations of reagents. A thorough visual inspection of treated melaninised paper samples did not reveal any significant damage to the integrity of paper that might have happened as a consequence of the treatment, regardless of the procedure employed.

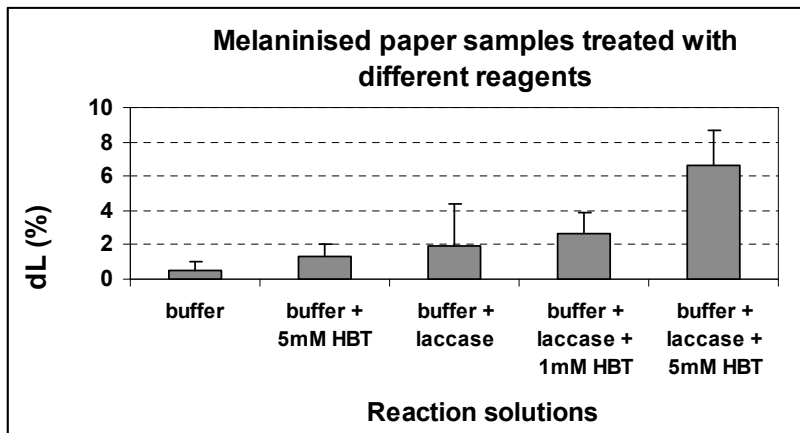


Fig. 1: Treatment of melaninised paper samples with different reaction solutions at elevated air pressure (dL – a change of lightness; error bars indicate standard deviation of ten parallel samples)

#### Fungal growth rates in the presence of biocides

The fungal growth in the presence of biocides was compared to the control media. Lindane showed no statistically significant adverse effect on the fungal growth and in the case of *P. ostreatus* also promoted its growth in comparison to the controls. The same was with PCB, which promoted the growth not only of *P. ostreatus* but also of *T. versicolor*. PCP is reflected as typical fungicide in lower obtained fungal dry masses. They are about 50 % lower in all the tested fungi with an exception of *T. versicolor*, where obtained dry mass yields was surprisingly comparable to the controls (Fig. 2).

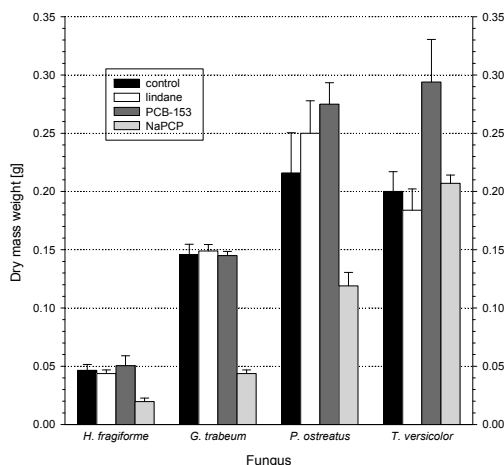


Fig. 2: Dry mass yields of fungal species grown in liquid medium with 30  $\mu\text{M}$  final concentrations of lindane, PCB or PCP, and in control medium without biocide

### 3.2. Biodegradation of biocides

Among selected white-rot fungi, *P. ostreatus* showed the highest efficiency in degrading lindane and PCB. Other two white rot fungus *C. purpureum* and *H. fragiforme*, and a brown rot *G. trabeum* were



considerably less effective in degrading all the tested biocides. *T. versicolor* decomposed PCP totally, while the other tested fungi degraded it to a much lower extent (Fig. 3).

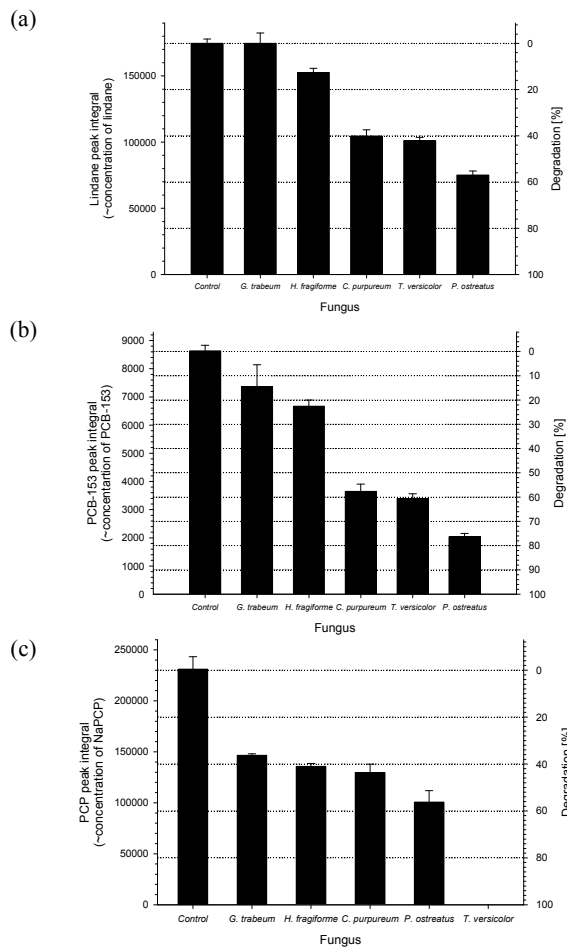


Fig. 3: Fungal degradation of lindane (a), PCB (b), and PCP (c); degradation rates were calculated from peak integrals in chromatograms

In comparison to fungal degradation, an application of respective commercially available ligninolytic enzymes laccase, manganese peroxidase, and versatile peroxidase were less efficient in degradation of lindane or chlorinated phenols. Manganese peroxidase and versatile peroxidase were more applicable enzymes in these cases (Fig 4).

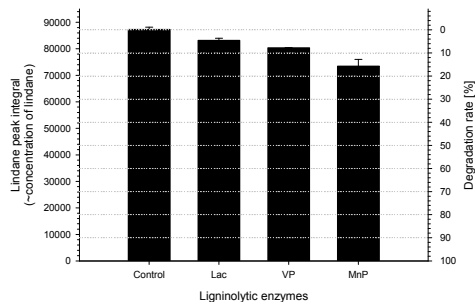


Fig. 4: Enzymatic degradation of lindane degradation rates was calculated from peak integrals in the chromatograms

#### 4. Conclusions

These procedures look very promising, but they need to be optimized and the integrity (or possible damage) of paper samples after the procedure should be carefully evaluated. After scientifically verifying that the melanin degradation procedure is not causing damage to the paper, and that the procedure fulfills paper conservator's requirements, it will be introduced in conservation of invaluable heritage.

Lindane and PCB showed no significant influence on fungal growth, while the growth rate in the presence of PCP was unaffected only in the case of *T. versicolor*. Among tested fungi, *P. ostreatus* most efficiently degraded PCB and lindane. Lindane was degraded in minor extent by this white-rot fungus most likely because of the fact that it does not have the aromatic character. Surprisingly, despite the fact that PCP had been used as a broad spectrum fungicide in many wood preservatives, it has been efficiently degraded by *T. versicolor*. Our results clearly show the promising potential of white rot fungi in bioremediation of organochlorine biocides and melanin in the field of conservation. However, the procedures will require adequate equipment for such restoration treatments.

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# MOULD ON ORGANS AND CULTURAL HERITAGE OBJECTS. INVESTIGATIONS IN EIGHT CHURCHES IN SAXONY

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## Abstract

The decay of wood and wood based materials by mould fungi in new or renovated buildings is – unfortunately – still an ongoing theme for experts like wood preservers or biologists. At both wooden interiors of churches and sacral assets this problem is relevant, too. In several churches of Saxony, where mould decay occurred, complex investigations on mould decay have been carried out. Different materials, typically used by conservators, and fungicides have been tested on their usability under worse conditions and on their effectiveness against mould, respectively. As a result, causes of the mould decay were investigated and recommendations for its prevention and control are given.

## 1. Introduction

For some years, an increasing mould infection is observed in churches and at wooden cultural heritage objects. Often, organs, altars, claddings, paintings, polychrome coatings and wooden sculptures are objected (fig. 1-3). The fact is noticeable that the infection occurs at objects which have been restored shortly before. This concerns wood replacement materials as well as polychrome coatings, although these materials contained fungicidal additives [1-3]. On the one side, this mould attack is a hygienic and aesthetic problem, on the other side, it may damage the precious objects. The objectives of the research project were to clarify the causes of the mould intensified attack and to deduce measures in order to prevent damages in future. A particular problem is the frequent and partly intensive infection of organs, as to be seen at fig. 2. The "normal" visitor of a church often does not know that one third of an organ is made of wood, e.g. body, pipes, chests or actions. The reasons for these problems are complex, since the mould growth depends on specific conditions of temperature, moisture and nutrient availability. If specific climatic conditions are given, mould fungi may infect nearly all materials, except they are treated with a fungicide or have a high pH value. The infection of sacral buildings and objects with mould is a serious problem, both from hygienic aspects and because cultural objects deteriorate. The investigations which are reported were carried out in scope of the project "Development of improved curative and preventive materials and measures for conservation of mould infected churches and cultural heritage objects", funded by the Federal Ministry of Economy and Technology. Partner of the project (worked 2003-2005) was the Heritage Preservation Trust of the Free State of Saxony.



Fig. 1: wooden organ pipes affected by mould (Aspergillus spec.)



Fig. 2: Mould decay on an pipe raster board; board varnished, dry, but with dust depositions

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## 2. Characteristics of Mould fungi

Within the kingdom of fungi, mould fungi are a large group which belong to the classes of Zygomycetes (genus e.g. *Mucor*, *Absidia*), Ascomycetes (genus e.g. *Chaetomium*, *Eurotium*) or Deuteromycetes (Fungi imperfecti; genus e.g. *Aspergillus*, *Penicillium*, *Trichoderma*, *Aureobasidium*, *Cladosporium*) [5].

In the beginning of growth, hyphae originate from germination of spores. These occur in the surrounding air more or less constantly and are – as well as the colourless hyphae – not visible to the naked eyes. Under suitable conditions, the hyphae grow on and branch to a hyphae netting, the so called mycelium.

The reproduction is possible both sexual and asexual. The spores play the key role for the dissemination of mould fungi, since they may be produced in large amounts. Due to the coloured spores, mould may be observed macroscopically as white, green, yellow, brown or black colonies. In case of wood as substrate, mould is accounted to the wood discolouring fungi (together with blue stain fungi).

Moulds usually grow on the surface of a substrate, but the hyphae may grow into porous materials. They may effect discolourations, but also materials damages (e.g. coatings, glues, leather, paper or linen). If hyphae grow between a substrate and its coating, e.g. a paint (polychrome) or a varnish, the coating may leave its adhesion to the substrate and get loose. Additionally, mould may increase the moisture content of a substrate and thus prepare the attack by e.g. wood destroying fungi (basidiomycetes, soft rot).

Independent of materials damages, moulds shall not be tolerated indoor from hygienic aspects, since spores, mycelia fragments, metabolites and mycotoxines as well as microbial volatile organic compounds (MVOC) generally have an allergic potential or may effect diseases. Old infections are to be considered as critically like new ones.

The living conditions are well-known so far. Sufficient moisture is needed, i.e. a relative air humidity above 70 %. An important value for the mould growth is the so called water activity  $a_w$ . It is defined as the quotient of the relative humidity above the substrates surface and those above water at same temperature (usually,  $a_w$  value may be calculated by dividing relative humidity by 100). This value describes the amount of free water of a substrate which is available for micro-organisms at the surface.

Most mould fungi need an  $a_w$  value between 0.80 and 0.85. Some species, e.g. representatives of the *Aspergillus-glaucus* group, may germinate already at values from 0.70. Moulds prefer slightly acid substrates with a pH value of 4.5 up to 6.5; values up to 10.5 at most are tolerated. Some species tolerate extreme acid conditions, e.g. *Aspergillus niger* grows even at pH 2.0.

Moulds have a wide temperature range of tolerance: mycelia of some species may grow at minimum temperatures from  $-2\text{ }^{\circ}\text{C}$  up to  $+5\text{ }^{\circ}\text{C}$  (e.g. *Alternaria alternata*, *Cladosporium herbarum*); the temperature optimum is approximately at  $25\text{ }^{\circ}\text{C}$ , and the maximum temperatures are at  $30\text{ }^{\circ}\text{C}$  up to  $40\text{ }^{\circ}\text{C}$ . In particular, the durable forms of moulds, the spores, may survive at their inactive status very high temperatures, but also other extreme conditions like dryness and lack of nutrients.

From the described living conditions follows that moulds are very modest. They live on easy usable matters like sugars, proteins and fats. As a source of nutrients, deposits on surfaces, like dust, soap, soot or pollen, may be used. Thus, even almost inert materials may be grown on, as Fig. 2 shows. Also thermoplastics, which often contain plasticisers or additives, may be infected or damaged.

## 3. Investigations

At first, eight churches with mould damages from different regions of Saxony were investigated. The extend of mould attack, the conditions of location, outdoor and indoor climate, specialties of the building, including heating, ventilation and use, and last but not least the microbial load of the indoor air were taken into consideration.

Two of those eight churches, the Peter and Paul Church in Reichenbach/Vogtland and the St. Mary Cathedral of Zwickau were selected for more detailed investigations. These two were chosen as

objects of interest, since no obvious building damages, but mould infection at several areas were found.



Fig. 3: painted wooden sculpture affected by mould (*Aspergillus spec.*)

Criteria for the selection were no obvious structural damages, but some relevant mould damages. Within the period from March 2004 until August 2005, air temperature and relative humidity close to at the affected surfaces were recorded hourly with mini data loggers. During several inspections in situ within that period, additional data have been obtained, like water activity, moisture content and surface temperature. From critical areas, infrared thermographs were taken. To determine the mould species, samples from infected areas were taken for laboratory investigations. To assess the hygienic status of the indoor air, airborne spore concentration [5] was determined with a sampler (Holbach LKS 130; fig. 9).

Additionally to that work, the susceptibility of restoring materials within laboratory and field tests was investigated and opportunities for preventive and cleaning measures have been checked. Different paints, solidification materials, glues, lutes and wood species were selected for the investigations, in agreement with the Heritage Preservation Trust.

As an example, Fig. 4 shows the typical curve of the indoor climate of Peter and Paul Church Reichenbach (altar) between March 2004 and June 2005. It is conspicuous that the relative humidity always was below 70 %. Due to the intensive infections (see fig. 1 and 3), much more humidity values were been expected. Obviously, wood interiors may take up moisture peaks rapidly and thus buffer relative humidity. But, this may lead to a higher moisture content of the material and thus to an increased mould risk.

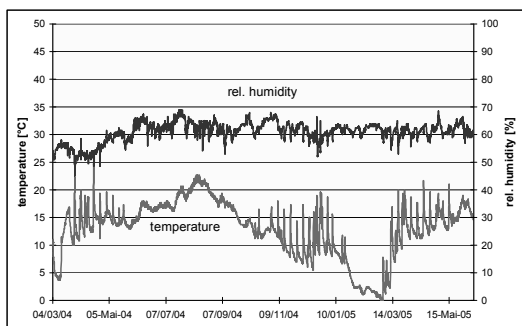


Fig. 4: Run of the interior climate at Peter and Paul,

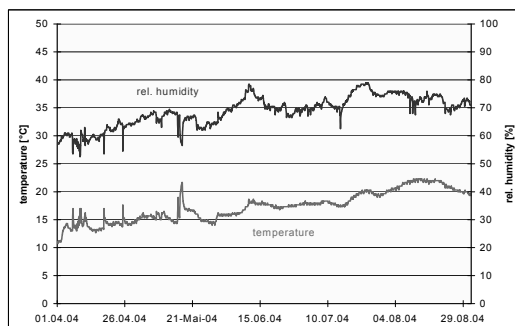


Fig. 5: Run of the interior climate at St. Marien

Reichenbach, between March 2004 and June 2005  
(measuring point: altar)

Cathedral, Zwickau, between April and August 2004  
(measuring point: kneeler)

A more detailed climate curve of St. Marien Cathedral Zwickau from April to August 2004 is shown in Fig. 5 (logger located behind a kneeler/prie-dieu, close to a wet exterior wall). A remarkable higher humidity was determined comparing to the other hall of the church. The figure shows that relative humidity changes more during the year than air temperature (despite of daily changes). During heating phases, the humidity decreases typically with increasing temperature, but the general course of humidity and temperature, influenced by seasons, follows a similar course (effect of outdoor climate). The highest humidity of above 75% occurred at summer time, but 80% have never been reached.

If mould already exists, obviously short-time increases of humidity up to 65...70...75% are sufficient to keep the mould alive. Possibly, the spore production is limited, but hyphae and mycelia are growing. More dry periods – between that phases with higher humidity – may be survived easily.

In addition to the climate records, thermographs from critical areas were taken. Fig. 6 and 7 show those areas: the organ (remarkable infected) is located above a cool, unheated room. Due to the resulting higher humidity (up to 76%) over longer periods, the wooden flooring was getting wet; in March 2004, the moisture content of the wood was between 17% and 19%.

To assess the microbial load, the content of airborne spores was determined in both objects. Surprisingly, the – partly intensive – mould infection was reflected by an adequate spore concentration of the air only in one case. Possibly, due to the very high room volume in relation to the moulded areas the spores concentration in the air is low (thinning effect). The assessment of the airborne spore sampling according to [8] resulted in a normal microbial load. From these results may be deduced no increased health hazard for church servants, organists, visitors etc. in general. Instead, the special hazard can only estimated by investigation of each individual case.



fig. 6: Interior view of the organ of Peter and Paul Reichenbach



fig. 7: Infrared thermograph of the area from fig. 6

The reasons for mould infection which were found out may be allocated to the following categories:

### Causes of structure and construction climate

The investigations have shown that the main reason obviously is to find in the temporal and local differences of temperature and humidity within the church. Critical conditions always come up when warmer air gets to colder surfaces and cools down, followed by an increase of humidity.

Within a church, cold and warm areas are permanent existing, due to frequent heating and cooling cycles during the heating period, seasonal differences between outdoor and indoor climate (also in summer time), the large inner volume of a church and the thermal inertia of the building as well.

The organs mostly are separated from the other room of a church, but an air exchange is possible over lots of active (wind system) and passive (ventilation holes, prospect) openings. Often, the organ wind is taken from outside or from the roof area

The above described effect may occur even here: warm air from the hall may meet colder areas of the organ. Additionally, the installation of organs or other objects made of wood close to often not insulated walls and ceilings may lead to an increased wood moisture.

### Use-related causes

Depositions of dust and dirt, resulting from reconstruction work or from a bad cleaning status, may favour mould infection.

As reported above, a discontinuous heating with short heating and cooling phases, may lead to a wetting of surfaces and materials.

Also a short increase of the humidity may result in a new infection or a growth phase of an older one. Thus, at days with higher amount of visitors, additional moisture input is to be expected. As measurements show, this did not lead stringently to an increase of humidity, caused by the buffering effect described before.

The churches can be ventilated mostly by opening the doors. To open the windows and cross-ventilation is recommended, but often not possible. If a church is ventilated at all, this is done often at unsuitable climate conditions. This may be the case at summer time, when warm air from outside is ventilated into the church and meets there cold areas, with risk of condensation (similar mechanism is known for cellars). With it, the targeted removal of moisture is changed into the opposite. Thus, a ventilation during summer time should be done only in the cooler morning or evening hours. Furthermore, during periods of high relative humidity (foggy weather) in spring or autumn, an intensive ventilation should be avoided.

### Use of susceptible materials

Not all materials used for conservation may be infected in the same extend. Some contain nutrients and thus make mould growth possible even at a lower humidity. Obviously, already some hours with 70 % relative humidity are sufficient to allow mould growth. So, a very strong infection was found at pre-glued wood surfaces, which were coated with a black casein-tempera, at wooden organ pipes which were poured with bone glue, as well as at wood, preserved with the oil-based preservative Hylotox 59 (today forbidden).

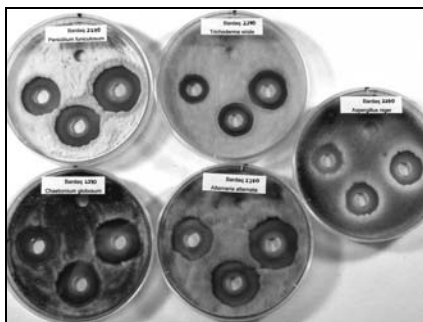


Fig. 8: test of biocides for restoring materials (agar diffusion tests)

In the laboratory investigations, following materials used for conservation were found to be susceptible:

Paints: (ground prepared with glue or lime-glue): distemper, black pigmented egg tempera, ammonium casein tempera;

Glues: leather glue, fish blister glue, technical and ammonium casein, lime casein;

Fillers: glue with clubmoss (*Lycopodium*) seed, cellulose glue;

Wood: Obeche, Linden, Pear, Pine (sapwood)

For the individual case should be proved, if a conservation material is applicable, or if specific measures are necessary, i.e. use of less susceptible materials, addition of fungicides, or storage of single objects in closed containers (e.g. cabinet).

The tests of effectiveness of fungicides e.g. by agar diffusion tests (fig. 8) showed those depends on kind of substrate. Substances like Thymole or clove oil, which are used in conservation to preserve paints, glues etc. are not strictly protecting against mould. Fungicides basing on quarternary ammonium compounds were found to be long-term effective at wood, in particular when combined with octylisothiacolinone. The way to clean infected objects depends on the kind of substrate as well, and is to be chosen for the individual case.

#### 4. Conclusions

The investigations confirmed that mould attack in churches and at wooden cultural heritage objects is a current problem which has to be solved urgently.

The mould decay at the investigated objects was expressed variably; mostly only some areas within the churches were moulded, e.g. organs or altars. However, at one of the objects, an intensive decay on large areas was found.

Unfortunately, the beginning of the attack can mostly not be determined. Possibly, factors which lead to a mould attack years ago are no more existing today, e.g. in result of conservation measures. If there is some mould, obviously a periodic increase of the relative humidity up to 65...70...75 % is sufficient to keep the mould alive. Eventually, the spore production is limited, but hyphae and mycelia are growing. Dry periods may be survived.

The causes for the mould attack were various. In general, a strong correlation between building situation and indoor climate, respectively, and mould decay could not be simply determined. Despite of a noticeable mould decay, the relative humidity of the indoor air did not exceed 80 % within the investigation period. Obviously, a short-term wetting of the surface – which does not lead stringently to a substrate wetting – is sufficient for mould growth.



fig. 9: Airborne spore sampling

#### 5. Recommendations

Mould decay in churches and at cultural heritage objects must not be tolerated and should be investigated, concerning the degree of decay and causes. Suitable measures of clean-up are to be determined rapidly, considering the specialties of the sensitive subject of churches and heritage



objects. A bioremediation has to go in one hand with the elimination of the causes of the mould infection. Good advises for bioremediation and conservation of mould affected objects are given in [6] and [7] (available in German; see [www.uba.de](http://www.uba.de)).

The interior climatic conditions should be recorded regularly. Due to the high volume, at least three devices at critical areas should be placed.

If a heating system is available, a gradually heating-up and cooling-down is recommended. A short-term, discontinuous heating shall be avoided to prevent a wetting of the surfaces. Since differences of temperature and moisture between indoor air and objects and materials are the main cause for condensation and wetting, the interiors shall be given sufficient time to adapt to the changed climatic conditions.

The times for ventilation are to be determined according to exterior and interior climate. For instance, during summer time a ventilation by opening windows in the daytime is to avoid to prevent condensation of warm and wet outdoor air on cooler surfaces within the church. Instead, only during the morning or late evening hours should be ventilated.

After events like worships and concerts, with lots of visitors, the building should be well ventilated, preferably by cross-ventilation.

Restoring materials should be checked for their susceptibility before application if there is a risk of mould because of specific disadvantageous climatic conditions.

Since the reasons for mould decay are various, each object is to be investigated and assessed separately. A lasting bioremediation is only possible if the causes – sources of moisture - are determined and eliminated. Hereby, a close cooperation between public and church administrations, conservators, building biologists and construction experts is to be aspired, taking into consideration the specialties of churches and sacral objects.

The results of the investigations have been summarised in a leaflet (in German).

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# BIOLOGICAL DETERIORATION OF HISTORICAL WOODEN ROOF AND FLOOR STRUCTURES AND THEIR RENOVATION

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## **Abstract**

In the paper a presentation of the most desirable task for the conservation expert is described: how to preserve the old – even severely damaged – wooden structure to be incorporated into the contemporary one. Two major steps are presented: the thorough inspection of wooden load bearing roof and floor structures with a description of deteriorated parts, and the proposal for the further construction measures which have to take into account also the possibility for the replacement or renovation of elements. At the end the architectural solution for incorporation of old structures into a renewed attic is presented (at the time being the building is still under reconstruction).

## **1. Introduction**

The origins of a building of Minorite monastery in Maribor was dated from the 13<sup>th</sup> century whereas the majority of building was renovated in the baroque style in the 18<sup>th</sup> century. The three storey building was firstly used as a monastery, then as barracks (1784 – 1927) and at the end as an apartment building. Due to poor maintenance, especially when the building was used as barracks and for housing, it was in a quite bad shape. Several “ad hoc” renovations have been performed in last 50 years and after a very long period of discussions it has been decided that the building has to be preserved as a historical monument. Also the purpose of the building was defined: after a complete renovation it will be a puppet theatre. Although the degradation of major wooden parts has been noticed, the wish of the investor and – above all – of the architect, was to maintain at least some parts and to include them into the modern structure.

## **2. Description of the building and wooden structures**

The building with a floor area of ca 700 m<sup>2</sup> has a ground floor, two storeys and a high attic which has not been in use. The building has a shape of a horseshoe, the south wing spreads along the river Drava and the north wing is connected to the old church. The length of the north and south wings were ca 28 m and their widths 6.7 m and 9.4 m, respectively. The eastern wing consists of a longitudinally shaped part with dimensions ca 18 x 11 m and a squared shaped SE part, called “tower” with dimensions 15 x 12 – 13 m.



Fig. 1. Views to the east wing (tower) and the western façade of the monastery during a reconstruction.

The walls of the building are made from stone and masonry. The floor structures above the cellar and ground floor are stone and mason arches, and the floors above upper storeys are made from timber. The timber roof is, according to the data obtained, over 200 years old. The roof structures in all wings are symmetric and non-symmetric trapezoid suspended frames lying on the outer walls. The main

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structural frames on the distances 4 – 5 m, carry ridge poles, rafters, and purloins. The building is covered with clay roof tiles. There are two types of floor structures: above the first floor mainly massive timber floors are installed with beams adjacent one to another. Above the second floor some of the floors are massive and some are hollow (beams on the distances ca 0.8 – 1.0 m, closed at the bottom and the top with planks).

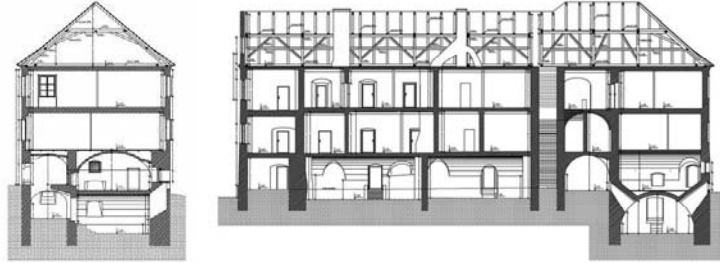


Fig. 2. Typical cross sections of the building [4].

### 3. Assessment of timber structures

Before we were involved into the project, there have been several assessments of timber structures already performed – some of them found installed timber structures damaged in such an extent that complete replacement has been suggested. The renovation procedure of walls and the floors in lower storey have been proposed and performed already but the renovation of timber elements, especially of the roof structure, has been found too complicated. Luckily, the architects wanted to preserve at least some parts of the timber roof because it is a cultural heritage which can not be replaced and finally he got the confirmation that this was possible.

#### 3.1. Inspection procedure

When starting the inspection procedure, the overall impression was that the roof structures are quite in good shape and worth to be preserved; only some structures were at first glance substantially deformed and one major frame has already been re-strengthened due to partial failure.



Fig. 3. Typical timber roof beams and massive floors in the attics.

But after a first assessment we began with the thorough visual inspection and our impression changed. When checking structural elements, we concentrated to the points where deterioration is expected: supports, roof-valley elements, gutters, etc. We paid attention also to signs of biological attack (presence of mould or wooden dust, which indicates an attack of wood fungi and insects). All findings were documented with photos and the problematic spots were marked also on drawings. The text with a description of findings, drawings with elaborated suggestions for replacement/strengthening of particular elements and comprehensive photo-documentation were assembled into the final report. The same procedure was performed also for floor structures.



Fig. 4. Typical floor structures: massive and hollow timber floors.

### 3.2. Degrading biological agents

According to [1] there are two types of biological agents degrading timber: wood destroying fungi and insects. Fungi cause two major kinds of decay – “brown rot” and “white rot”. With brown rot only the cellulose is extensively removed, the wood takes a brownish colour and it tends to crack across the grain. With white rot both lignin and cellulose are removed, the wood appears whiter but doesn't crack. Until it is severely degraded, it retains its dimensions and the deterioration is hardly visible. But both kinds considerably lower the weight and consequently the wood strength.

The second degrading agents are insects. In this part of Europe wood is mostly attacked by powder-post beetles. They lay the eggs in the wood pores and their larvae are feeding with cellulose, starch, sugar, and albumin from the wood, mostly the sapwood. They make tunnels and when grown up leave the wood packed with fine powder. The winged adults leave the wood through the surface which was until then untouched, and we can notice their presence too late. They leave the wooden elements bored through with substantial loss of mass and strength.

They are two major types of insects that attack the installed wood in our climatic conditions: one from Lyctus family called “house capricorn” or “old house borer” (*Hylotrupes bajulus* L.), and another from the Anobiid family (*Annobium punctatum* De Geer). The Lyctus larvae need for their growth higher temperatures (28–30°C) whereas Anobiid beetle can live also on lower temperatures (22–23°C). Both prefer high moisture (30%) but Anobiid beetle can leave also at lower moisture (15%). Climatic conditions favoured by Lyctus beetles are exact the same as those in wet roof structures, so it is very important to prevent leaking or condensation on the closed timber roof structures.

### 3.3. Roof structure - findings

A thorough inspection of practically all roof elements has been performed and a lot of damaged parts of the structure have been found [3]. The most problematic parts of the structure were found on spots where long term wetting was present. In these parts combined attack of fungi and wood insects (*Hylotrupes bajulus* L.) caused substantial deterioration of wood, sometimes even total loss of strength. Wetting was caused primarily by leaking at bad connections of roof planes (especially in valleys) and the bad details at the gutters. (The same findings were reported before [2].)

In our case we found the most damaged parts in the above mentioned bottom of the gutter valleys. These parts were practically completely destroyed by the attack of fungi causing brown rot: the wood became brownish, cracked across the grains and totally lost its strength.

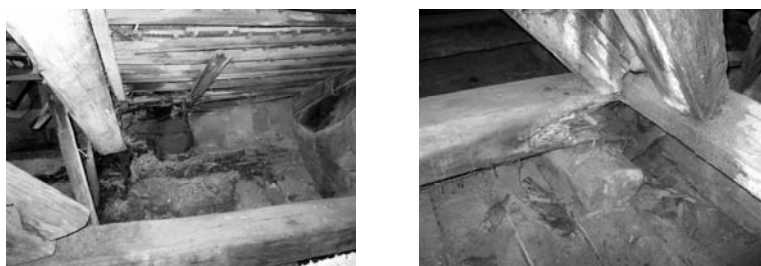


Fig. 5. Destroyed parts in the bottom of gutter valleys due to fungi attack.

Heavy damage has been noticed on the edge of the roof slope in the SE tower: although lower parts of the roof have been replaced by new cover, the supporting elements are still deteriorated and closing elements are missing. The combined attack of fungi and insects was spotted.



Fig. 6. Restoration of the roof cover without replacing damaged supporting elements.

In the tower the most problematic element was one main beam (span 12 m) with completely destroyed lower chord due to the attack of wood destroying fungi. The part was temporary replaced by the new elements but a frame was unable to carry the load and it deformed substantially. The deformation of structure has been visible also from the outside on the roof planes. A reason of destruction was probably a constant leaking of the roof at the ridge (the adjacent longitudinal tie element was destroyed, too).



Fig. 7. Wood in the lower chord of the main frame and in the longitudinal tie beam completely destroyed.

Because of these damaged elements the rearrangement of loads occurred. The load from the roof was taken by the floor structures which have not been designed for that. In addition, some parts of the floors were completely destroyed on the supports and needed to be replaced. Heavy damage, the complicated replacements of the floors and deformed roof structure were the reason why the complete replacement of all timber structures in the tower was proposed.

In the eastern wing the damage due to insect attack was spotted, too. It was the part constructed later (and probably with the lowest quality wood). Not only elements at roof edges and in connecting gutter valleys, but also elements in the dry surroundings were heavily damaged by Lyctus beetle (*Hylotrupes bajulus* L.)



Fig. 8. Attack of the insects (*Hylotrupes bajulus* L.)

### 3.4. Floor structures - findings

A control of wooden floor structures in the second floor showed that degradation of wood due to fungi is presented in several parts of massive floors. The stages of destruction were different: in some places

only surface was affected, in others brown rot was spotted also in the middle, whereas in one corner practically the total height of beams was damaged.

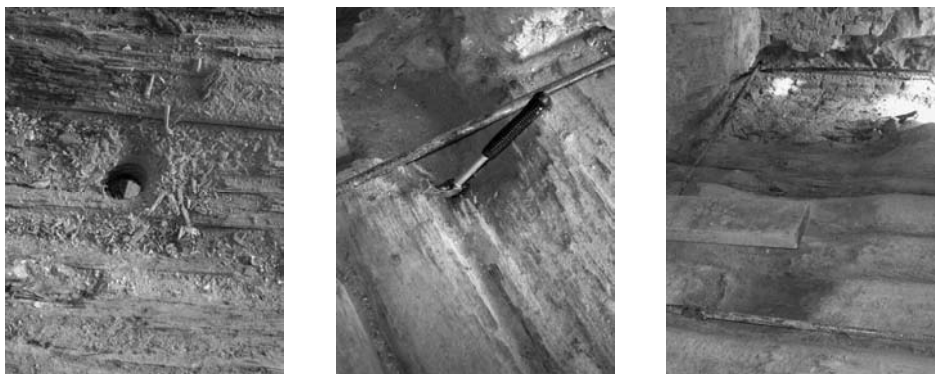


Fig. 9. Massive timber floors with different stages of wood destruction.

In the attics massive floor structures were in some parts completely destroyed (damaged wood on the supports was seen also in the rooms below). On the other hand some parts are assessed to be strong enough to remain installed. The hollow floors are most problematic in the SE tower: beams are completely destroyed and temporary supported; as we already mentioned, they have to be replaced.



Fig. 10. Floors in the attics: brown rot on the supports, complete destruction of beams.

### 3.5. Proposed measures

After inspecting the whole roof structure we came to the conclusions that – in spite the fact that the building is a historical monument – due to heavy damage all timber structures can not be preserved. Also for the parts which can be retained, replacement of several parts is demanded and expert restoration measures will be needed. The general estimation is that structures in two wings are proposed to be preserved, whereas in the most damaged central part (the tower) they should be replaced. We still expect problems in the contact zones between old and new structures where very careful replacements of damaged parts should be performed. For the further use the massive floors in the second floor should be re-strengthened, and combined wood-concrete floors can be installed. But before the installation of connectors, the damaged wood layer should be treated by special restoration resins. In the attics the destroyed wooden floor structures in the tower are proposed to be replaced with concrete ones.

## 4. New design of the attics

### 4.1. General

In the upper storey of a new puppet theatre several activities are foreseen: it will be used for schooling puppet performers, for workshops and also for designing stage scenery for puppet shows. Because in these premises young artistic people will be working, the solution to show them very old structures

which survived all occupants and their actions seems very good. Therefore smaller spaces, required by the users, are accommodated within the existing structure, and the larger volumes, intended for the main purpose, the theatre, such as the main auditorium, are placed within new structures. In the atrium of the monastery there will be the open-air auditorium, intended for open-air events.

#### 4.2. Combination of the old and new structures

The aim of the architect to include old roof structures into new spaces finally got the confirmation with the described inspection and assessment procedure. With this it was proved that some parts of the structure could be preserved and used in the new attics. The final proposal of the outlook is presented below.

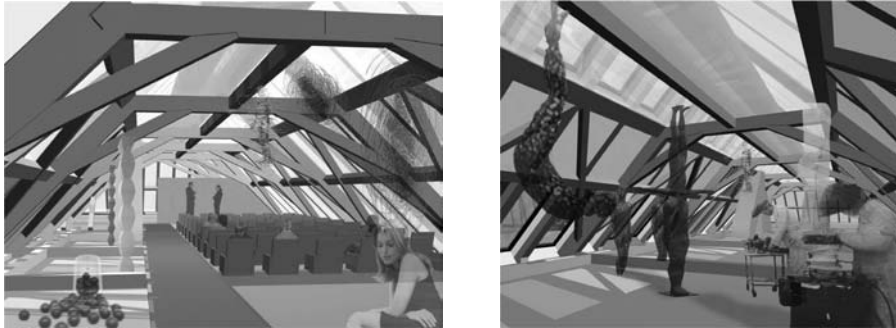


Fig. 10. The old roof beams will be installed in the lecture room and the scenery preparation room [4].

#### 5. Conclusion

After the inspection and assessment performed we can say that the major task was to choose which parts should be preserved and which replaced. This assessment included not only the selection of damaged parts but also recognizing the possibilities of partly replacements of elements. The experienced specialist who is able to recognize wood degrading agents and the level of wood degradation, but also has the knowledge of construction measures, is very valuable. For the replacement of the parts of load bearing structures, the possibility of the supporting the remaining parts should be taken into account.

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# ORIENTATED INVESTIGATION TO KILL THE MYCELIA OF THE DRY ROT FUNGUS, *SERPULA LACRYMANS*, WITH MICROWAVES

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## Abstract

The *Serpula lacrymans* is one of the most important wood decay fungi in buildings. Chemical and physical methods of treatment have been used in the past. In a Masters thesis at the University of Hildesheim the possibility of a thermal treatment using microwaves was investigated. Infected wet and dry pinewood samples were exposed in an open system to microwave fields (at a frequency of 2.45 GHz). Here different kinds of radiations were used for various lengths of time up to temperatures of 50°C. It was concluded that the tested methods are not suitable for the treatment of sensitive wooden artefacts.

## 1. Introduction

The dry rot fungus *Serpula lacrymans* (Wulfen: Fr.) Schroeter holds a special notoriety in the built environment due to its vitality, its very destructive potential and the huge costs involved in rectifying the damage caused.

The German Industrial Standards, under DIN 68800-4, recommend infected buildings are treated by eliminating the source of moisture, generously removing all visibly affected areas, and applying a chemical protecting agent. In the case of historic buildings with valuable works of art, like painted wooden beams, polychrome panelling or carvings, and furnishings, such a form of intervention would be very harmful and certainly contradict the ethical demand to preserve as much as possible of the original substance. For infected timber that has to remain in the installation position, or can not be removed due to its heritage value, the German DIN 68800-4 gives the option to treat the fungi with special physical technology, provided that you can guarantee that you kill all the wood-destroying organisms.

The existing physical possibilities of killing the dry rot fungus were all been based on heat, particularly treatment with warm air. Treatment with electromagnetic waves has been established so far only in Denmark. The reasons for the comparatively limited application in Germany are due to the little knowledge about usage, physical effects, advantages and also disadvantages. Furthermore the little scientific research that exists does not provide a sufficient basis to make an assessment

From a technical viewpoint the treatment of brickwork, cement, concrete or clay with microwaves is unproblematic, as long as this does not involve walls that are several metres thick and which can not be heated up due to the limited depth of microwave penetration. Usually it is possible, depending on the dielectric permittivity, to heat the materials up to 60°C relatively quickly. However, with wood there is not much experience and most companies do not even treat it. To great is the danger of damage in the form of cracks, leaking resin, or damage to the transparent coating or paint. Furthermore, the heating with microwaves is due to the electromagnetic field distribution very inhomogeneous and that is why you have to anticipate high differences in temperatures.

Concerning disinfection, the literature indicates various lethal temperatures for the mycelia of *Serpula lacrymans*, depending on the experimental set-ups [1]. Within the scope of experiments with heat coming from outside, the temperature and duration time goes from 17 hours at 35 - 37°C until 15 minutes at 40 - 60°C. According to the latest insights by HUCKFELDT [2] at least 65 - 70°C are needed to kill the mycelia effectively. Conspicuously lower temperatures were reported from experiments with microwave treatments. BECH-ANDERSEN & ANDERSEN [3] needed only 10 min at 37°C to kill surface mycelia in small diameter glass tubes and KJERULF-JENSEN & KOCH [4] reported that 40 - 50°C was appropriate for infected sawdust. In both cases the temperature had to be

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reached but not to be maintained. Smaller test series in the scope of a research project at the University of Applied Sciences and Arts in Hildesheim achieved the result of a lethal temperature around 35 - 40°C [5]. The treatment with a pulsed microwave system could reduce the lethal temperature even further down to 25 - 27°C. During both methods of treatment the microwave exposition time was not longer than 20 mins.

So far the results gained through microwave treatments have shown the possibility to deal with a method which offers a low lethal temperature, despite known difficulties and risks. According to this it seemed to be reasonable to confirm these results in the scope of a Master thesis at the University of Applied Sciences and Arts in Hildesheim [6], readapted to more practical demands with a changed experimental set-up.

#### Aim of the experiments

The aim of the experiments was to find a cautious method of treatment with a low lethal temperature. Low temperatures result in a homogeneous heat distribution, on the surface as in the cross-section, alter the wood moisture less, cause low vapour pressure, reduce stress for the object and cause less danger of damage. Furthermore special attention was paid to the following questions:

- How long must the object be treated and how high must the temperature be raised to kill the mycelia?
- Do different kinds of radiations have different effects, like permanent treatment, interval treatment or pulsed treatment?
- Are there differences in the lethal temperature depending on the activity of the fungus?
- Does the humidity of the wood have an influence?

## 2. Experiment

About 75 cultures were cultivated in advance, with mycelia of the dry rot fungus. When they had grown 1250 small sterilised wooden coniferous blocks were put into the Petri dishes and on the mycelia. For each type of radiation, test and activity three parallels were prepared. Every parallel attempt consisted of four samples to assess the disinfection, two samples to check the vitality with BacLight™ and growth, and two samples for microscopy and determination of the wood humidity before and after treatment.

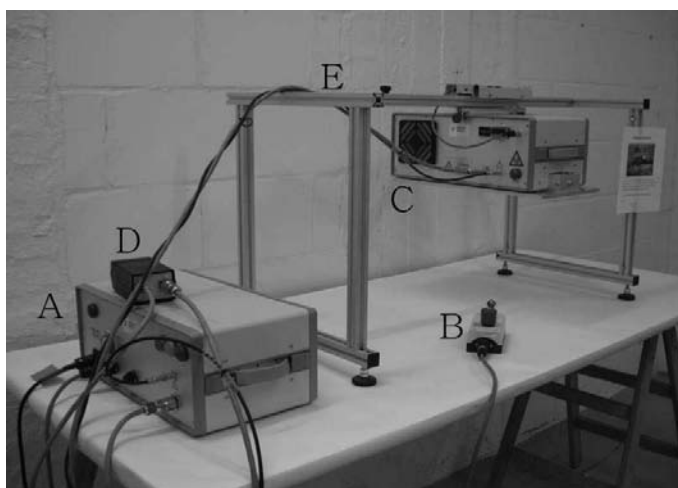


Fig. 1. Microwave system from the Swiss company Service-Partner with all components used in the experiments: Energy supply unit (A), remote control (B), microwave radiator (C), clock timer (D), frame (E)

When the blocks were colonised and penetrated with mycelia, half of the samples were dried to a wood humidity around 18% above salt solutions, to get living but inactive mycelia into a passive state. The second half of the samples with active mycelia and a wood humidity around 80% was directly

treated with microwaves. Therefore an open microwave system from a Swiss company was used that worked with a frequency of 2.45 GHz and 1000 W output power [7].

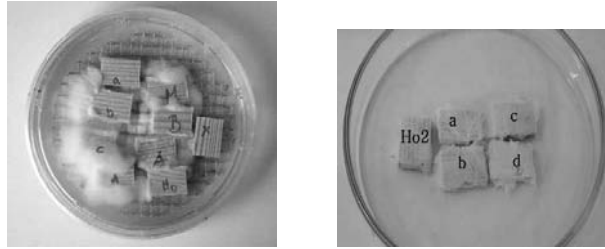


Fig. 2. Infected wooden samples during the incubation time and microwave treatment

The different tested radiations were:

1. **Permanent treatment**, where microwave treatment was held throughout the whole test until temperatures of up 25, 30, 40 and 50°C were reached.
2. **Interval treatment**, where the temperatures of around 25, 30 and 40°C were hold over a time limit from 10 to 30 mins through a manually operated system. Behind this treatment was the idea that the lethal temperature can be reduced by extending the duration time.
3. **Pulsed treatment**. In this case the microwave system automatically pulsed every 5, 10 and 15 seconds. The treatment duration was 15, 30 and 60 mins. In these test series we wanted to stress the cells and to see if killing of the mycelia was based on athermic effects, or not.

The control of the temperatures and the assessment of the heat distribution were carried out with an IR-camera. Right after the microwave treatment the blocks were transferred to fresh malt nutrient agar. After a following incubation time of 6 to 8 weeks the assessment of the disinfection took place on the basis of new mycelia growth.

### 3. Results

The permanent microwave treatment of the active mycelia had neither at 25°C nor at 30°C a disinfecting effect. The first inhibiting temperatures which caused a retardant growth after three or four weeks were around 40°C. But to kill substrate mycelia, temperatures of 50°C and higher were needed.

Both holding a temperature up to 40°C for 30 mins, and the pulsed treatment of active mycelia caused only an inhibited reaction but no disinfection.

Concerning the testing of dry samples with inactive mycelia, it was not clear-cut as almost all of the samples were contaminated with mould. Nevertheless, it was clear that the substrate mycelium of dry samples was more resistant to the higher temperatures. Even temperatures of up to 55°C, which had been high enough to kill active mycelia, were in this case ineffective. Cells performing active metabolism reacted more on lower microwave treatments than those organisms in a passive state.

The humidity of the wood plays – regarding the heat distribution and warming efficiency – an ancillary role. Dry samples were heated up as fast as wet samples were, and had comparably high differences in their temperature distribution. With the help of an IR camera it was possible to observe that every test had a different heat distribution. As the experimental set-ups, like the position of the samples under the sender, the distance between samples and sender, or the distribution of the samples in the petri dish, were always the same, predictions about the heat distribution were not possible.

It is worth noting that the differences in the temperature distribution increased with higher temperatures. If you treat an object with microwaves and you want to have a minimum temperature (set-point-temperature) of around 60°C you have to expect temperatures around 80°C or more in some areas.

The observed low variations of temperature during these experiments around maximal  $\pm 5^\circ$  are caused by the ideal experimental set-ups and the individual on and off turning of the microwave system. But in practice the microwave systems run automatically, which means that the temperature is adjusted by

speed and length of the frame. In these cases you have expect much higher differences of temperatures.

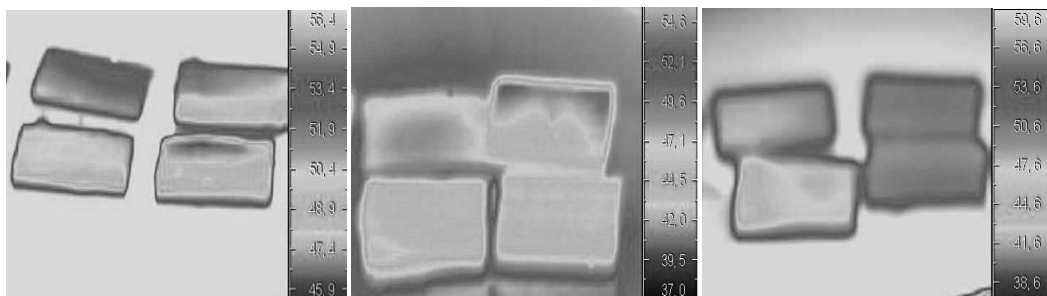


Fig. 3. IR- image of test DA 4 I (left), DA 4 II (middle) and DA 4 III (right) after microwave treatment of up to  $50 \pm 5^\circ\text{C}$ . The heat distribution is inhomogeneous and unpredictable, despite comparable experimental set-ups.

It was also observed that the wood loses increasing humidity with longer periods of treatment. The determination of the moisture content of the samples sometimes showed massive losses of moisture. The diagram 1 below shows the wood moisture before and after interval treatment. You can see that the wood moisture decreases with rising temperature and duration. Already after 20 minutes of heating at  $30^\circ\text{C}$  the moisture fell to half. Extreme moisture loss was caused with 30 mins at  $40^\circ\text{C}$ . After treatment the samples in these series possessed only a moisture content of 2%, which is almost kiln-dried.

Comparable drying tendencies showed the tests with permanent and pulsed microwave treatment. In these test series the wood moisture clearly fell with increasing time duration, even when the surface temperature rise was marginal. Noticeable was, that the duration of the pulse (5, 10 or 15 seconds) had no influence on the moisture change.

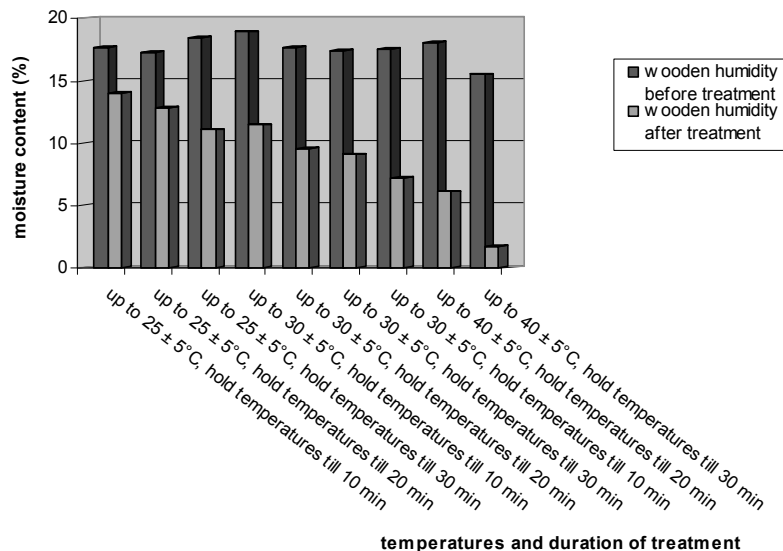


Fig. 4. Change of wood moisture of dry samples with mycelia in a passive state before and after interval treatment

#### 4. Conclusion

To summarize, it seems possible to kill active biological structures with microwaves. But due to the inhomogeneous heat distribution, the resulting high temperatures and the abrupt change of wood moisture, incalculable risks of object damage can result. Possible damage could be cracks, leaking resin and damage to the transparent coating or paint. For this reason the microwave treatment on objects with historical value cannot be recommended.

Nevertheless, the use of microwaves would make sense when they are seen not as a combat method, but rather as a supplementary method to dry affected parts of buildings relatively quickly and in an environmentally friendly way and to stop the infestation this way – of course always apart from the elimination of the humidity source. Application fields could be contaminated profane buildings, brickwork or on localised, restricted areas which have been affected and do not have historical value. The requirements are the inspection of the building, a conducted treatment by a professional and experienced company and the monitoring control with the help of an IR-camera. Last but not least it is still necessary to improve the assessment of this technology with further tests and parameters. For this reason it would be desirable if other institutions take up this theme and continue, complete and improve these results.

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# RESEARCH STUDY ON THE EFFECTS OF THE THERMO LIGNUM® WARMAIR TREATMENT ON ART OBJECTS WITH PAINT AND GILT FINISHES

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## Abstract

The present research study investigated the effects of a humidity controlled warm air treatment with the Thermo Lignum Process on objects which are painted or gilded. The Thermo Lignum Process is an ecological and object sensitive technique to combat insect infestations which applies only humidity controlled warm air. It is founded on the established principle that most insects are reliably killed in all their life cycle stages at a temperature, depending on species, of up to 55°C. The infested objects are heated in a chamber to a maximum temperature of 58°C. Throughout this process the air humidity is controlled in such a way that no drying of or changes in the objects can occur. The objects prepared for the trial were examined before and after the Thermo Lignum treatment by means of light microscopy and scanning electron microscopy. In addition, colour measurements and investigations on potential distortion and adhesiveness to the substrates were carried out. In order to achieve the most comprehensive and meaningful result, the trial was carried out with three different groups of objects. Object Group One consisted exclusively of samples of the binding agents most commonly used in historic finishes. Object Group Two included several newly applied finish layers, whilst Object Group Three comprised historical objects with different finishes belonging to a variety of style periods.

## 1. Introduction

Art objects made of wood often suffer infestations by wood destroying insects (in particular by the anobiidae species). The larvae of these woodborers eat through the timber frequently over several years, and the adult beetles deposit their eggs preferably on the same objects, so that several generations of insect can cause substantial and serious damage to objects. In order to preserve art objects, a form of insect pest control is needed which has no negative effects on the objects requiring treatment nor their constituent materials.

The Thermo Lignum® Warmair treatment offers an ecological alternative to the known conventional eradication methods which count the fumigation of objects with toxic or inert gasses and the treatment with liquid chemicals amongst them.

Applications for the Thermo Lignum® Warmair method to combat an insect attack in objects made of materials as diverse as leather [1], paper [2], textiles [3], photographs [4] and so far as to include insect collections [5], have already been sufficiently scientifically examined.

However, the behaviour of objects with surface finishes during a treatment with the humidity-controlled warm air method had not been sufficiently researched and documented to date, and this is therefore the subject of this study.

## 2. Objectives

The main objective was to investigate the potential effects of a Thermo Lignum® Warmair treatment on objects with paint and gilt finishes. The intention was, on the one hand, to examine, by means of modern investigative methods, historical objects from a variety of style periods with varying finish assemblies for potential changes caused by the Thermo Lignum® Warmair treatment. On the other hand, the current study centred on potential modifications from such a treatment on the most commonly used resins and glues (both individually as well as complete finishes) in the shape of newly manufactured samples.

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### 3. Samples

The examination was carried out on 11 historical objects with a variety of finish assemblies as well as on newly prepared samples. In the case of the latter, resins and glues were applied individually and in combination as complete finish assemblies to Swiss pine or arolla (*Pinus cembra*). In some instances, glass and canvas were also used as substrate material. Given the local geographical importance of pine as work material in the Alpine region, and in order to create a “worst case” scenario (high resin content in this type of timber), *Pinus cembra* was chosen as substrate timber for the purpose of this investigation. Table 1 provides a description of the historical objects which were examined. Table 2 provides an overview of the coatings applied to the newly made samples.

Table 1: Description of the historical objects examined

Object No.	Date/Period	Description	Assembly
A1	1900	Square altar section	Chalk (Black Chalk) ground (Steinkreide grundierung) + gilding
A2	1680	Blossom	Gold on varnish, glue + chalk (Black Chalk), several thick layers of gesso ground + gilding
A3	1770	Ornament	Gesso + gilding
A4	1670	Acanthus leaf	Glue foundation + gesso + gilding
A5	1756	3 Altar sections	Glue foundation + chalk (Black Chalk) ground (Steinkreide grundierung + Polonaise gilding
A6	18 <sup>th</sup> + 19 <sup>th</sup> C.	Finger	2 finishes, original 18 <sup>th</sup> C layer, repainted in 19 <sup>th</sup> C
A7	18th C.	Large top section of an altar with marbling	Gesso + marbling with casein method
A8	2 nd half 19 <sup>th</sup> C.	Casing fragment	White and gold
A9	2 nd half 19 <sup>th</sup> C.	Foot fragment	White and gold
A10	2 nd half 19 <sup>th</sup> C.	Rosetta fragment	Gold on pressed cardboard
A11	2 nd half 19 <sup>th</sup> C.	Snail fragment	White and gold

Table 2: Description of the coating variants

	Type	Variant No.	Description
On pine	Resins	R1	Shellac
		R2	Mastic
		R3	Dammar
		R4	Copal
		R5	Sandarac
	Glues	G1	Bone glue
		G2	Rabbit skin glue
	Combinations	C1	Gesso (Bologna chalk/Rabbit skin glue) + gilding
		C2	Gesso (Bologna chalk/Rabbit skin glue) + oil finish
On	Glues	G3	Carp glue
		G4	Sturgeon glue
On canvas	"Combination aged and consolidated "	C3	Oil paint on primed canvas (with zink oxide white) – consolidated with carp glue
		C4	Oil paint on primed canvas (with zink oxide white) – consolidated with sturgeon glue

#### 4. Methodology

The Thermo Lignum® method is a humidity-controlled warm air treatment and was developed as an ecological alternative in the combat against wood-destroying insects in sensitive objects. In contrast with known conventional treatment methods, the Thermo Lignum® method works exclusively with warm air and humidity, offering the advantage of treating the most diverse materials and objects within a short space of time, and without the use of chemicals and toxins.

The treatment is based on the principle that animal protein is damaged by the effect of elevated temperature, resulting in the death of all insect lifecycle stages (egg, larva, pupa, adult). The exact kill temperature and exposure time depend on the insect species. 55°C, held over one hour, are considered a reliable kill temperature for wood boring insects.

A further critical concept is that the object moisture content is kept constant during the heating and cooling phases. This is achieved by controlling the relative humidity in a treatment chamber in accordance with the Keylwerth Diagram (Fig. 1). During the warming-up phase it is ensured, apart from the regulation of the humidity, that the temperature differential ( $\Delta t$ ) between the object surface temperature and the core temperature never exceeds a set limit. Equally, the maximum room temperature (max  $T_R$ ) is not allowed to exceed a preset limit during the treatment. Both  $\Delta t$  and max  $T_R$  are object and material related and are based on empirical data.

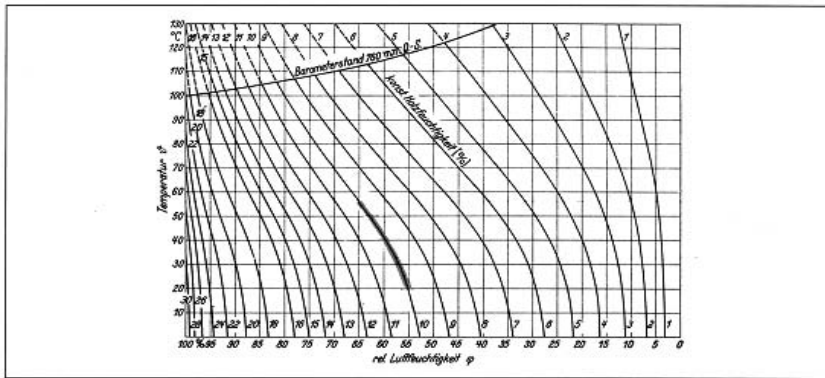


Fig. 1: The Keylwerth Diagram

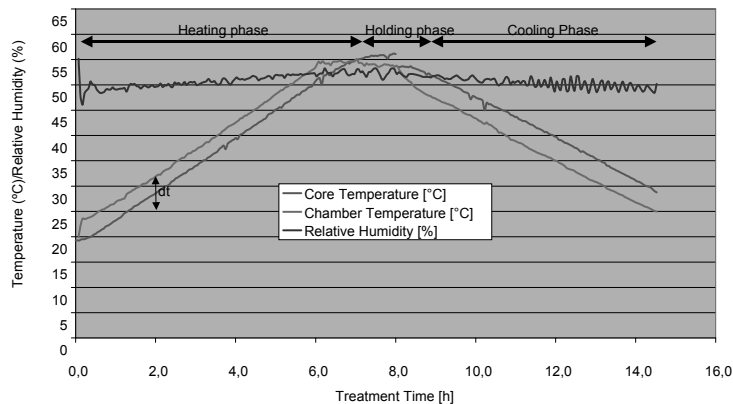


Fig. 2: Diagram of a Thermo Lignum® treatment

## 5. Examination

Apart from the precise analysis of the coating assembly of the historical objects, the investigation centred also on the layer thickness of the resins and glues as well as other finishes applied to the pine wood samples.

The examination of the samples in the laboratory of Holzforschung Austria was based on the following parameters:

Changes in the surfaces of all treated samples by means of microscopic assessment using optical microscopy (Olympus SZH) and scanning electron microscopy (JEOL JSM-6100)

Cross sectional changes in the finishes or coatings (both optical microscopy and scanning electron microscopy)

Changes in elasticity of the finishes or coatings applied to the newly prepared pine wood samples (examination to Austrian Standard ÖNORM C 2350).

## 6. Results

No changes could be found as a consequence of the Thermo Lignum® Warmair method, neither in the historical objects, nor in the newly made-up samples, with the exception of the shellac-polished pine wood samples.

The change in the shellac-polished pine wood samples consisted in the diffusion of resin droplets from the underresinated pine wood samples into the polish. However, this phenomenon of resin diffusion can also be observed under normal ambient environmental conditions, particularly with underresinated pine wood.

No changes could be established through optical microscopy and scanning electron microscopy examination in the remaining, newly made-up samples. More specifically, the humidity-controlled warm air treatment did not result in any blistering, flaking, cracking or splitting in the coatings or polishes.

Particular attention was focussed on potential changes in the cracks, evident already prior to the treatment, in the case of the canvas samples which were aged and consolidated with carp and sturgeon glue respectively (variants C3 and C4). Again, no changes could be found during microscopy examination. Fig. 3 exemplifies a typical result before and after the treatment of sample C4 (see table 2).

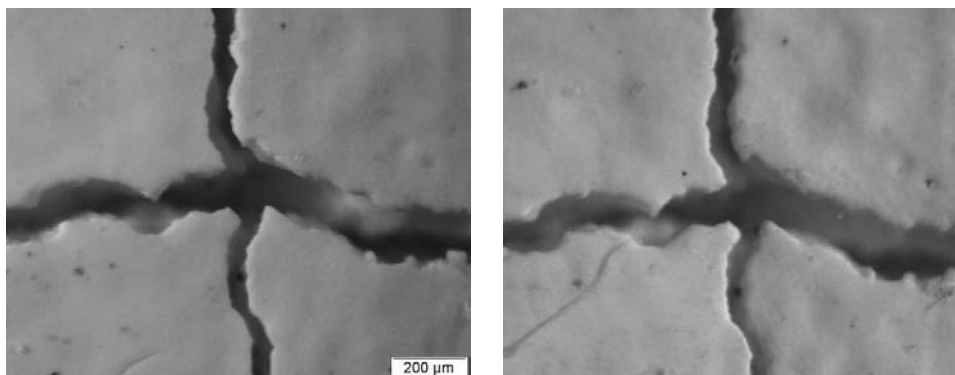


Fig. 3: Microscopic photograph of sample C4 (aged and consolidated with sturgeon glue, on canvas) before and after the Thermo Lignum® Warmair treatment (magnification approx. 55 times)

No changes as a consequence of the treatment were found in the historical objects with optical microscopy and scanning electron microscopy examination, nor was there any reduction in adhesion strength or increased cracking in both the prepared coating samples and cross-sectional samples.

Fig. 4, which shows a photographic image of Sample A5, serves as an example of the macroscopic comparison.



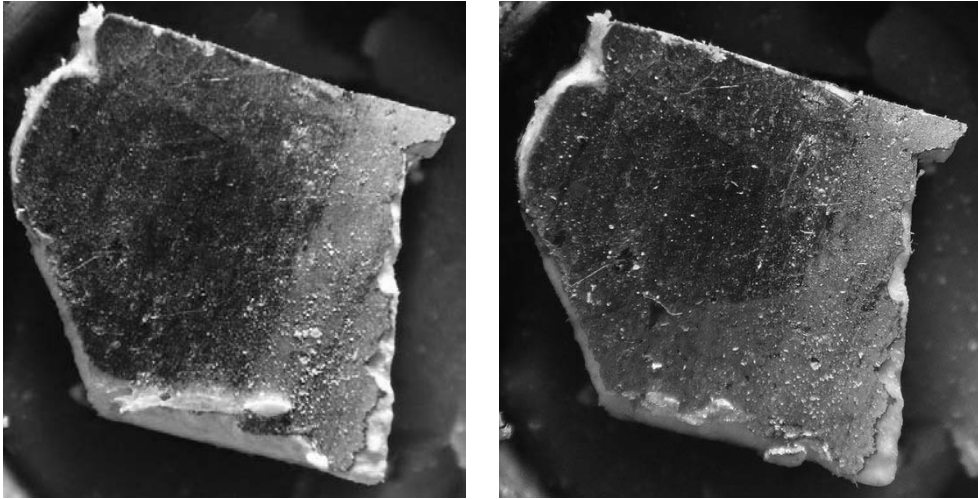


Fig. 4: Macroscopic comparison of sample A5 - approximately 9 mm long (pre-treatment on the left, post-treatment on the right)

The Deformation Test to Austrian Standard ÖNORM C 2350 was unable to establish any change as a consequence of the Thermo Lignum® treatment in the deformation characteristics in the newly made-up pine wood samples.

## 7. Conclusions

Apart from the resin diffusion observed in the shellac samples, the present examinations did not find any changes, neither in the historical samples nor the new samples.

This scientific research also confirms the long-standing practical experiences which underpin the use of the Thermo Lignum® Warmair method as a means to eradicate destructive insects in historical objects of art with paint and gilt finishes without negative consequences for the objects.

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# (C) CHARACTERIZATION AND MEASUREMENT TECHNIQUES



# NON DESTRUCTIVE IMAGING FOR WOOD IDENTIFICATION

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## Abstract

Wood identification of Sesin-bosatsu-rituzou, registered national treasure, curved by UNKEI in Kamakura period, has been performed by means of micro-CT imaging at SPring-8, Japan. The small fragment less than 1mm was investigated and 3-D information at 0.7  $\mu\text{m}$  special resolution was obtained. Although the samples was compressed in tangential direction and partly deteriorated, we could identify as Katsura, *Cercidiphyllum japonicum*

## 1. Introduction

Wood identification based of anatomical features requires the observation of microstructure from 3 directions, axial, radial, and tangential. A way to do is to use a razor blade for making a thin slice from wood blocks, and prepare the microscopy preparation of corresponding 3 directions. Nevertheless this method becomes simple after training and experience, it is not applicable for example when only too small sample is given as is always the case for national heritage class wood works or artifacts. Therefore, we investigated the use of the synchrotron radiation facility(1) to shed a light on the anatomy of national and world heritage wooden objects. X-ray micro-CT imaging at BL20XU was carried out on specimens including cultural assets with a resolution of 0.7 micrometer. Sample twig was trimmed less than 1 mm diameter (long along longitudinal axis), and 11 softwood species has been carefully investigated with a series of programs developed by BL20XU, such as SLICE(2). The resolution is good enough to visualize most of the species-specific anatomical features necessary for the identification

## 2. Material and methods

The wooden statue Sesin-bosatsu-rituzou, registered national treasure, curved by UNKEI in Kamakura period. was investigated. The sample was collected during its repair, a small piece the detached small fragments with a dimension of 0.4 mm (R), 0.2 mm (T) 1.5 mm (L) as shown in Fig1. The sample was so brittle to be sectioned without embedding. Furthermore, the sample was compressed in tangential direction so that the surface was not clear enough to be explored by a scanning electron microscope. As such, the sample was most suitable for X-ray CT imaging to be tested. X-ray micro-CT imaging was done at BL20XU, SPring-8, Harima, Japan(1) and the data were analysed by a series of software developed by BL20XU, such as SLICE(2)



Fig 1 Seshin Bosatsu Ritsuzo, foot parts (R and L) and samples corrected.

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### 3. Results

From visual, tactile, mechanical inspection by a professional craftsman, the wood where the sample was collected was expected to be either Hoonoki (*Magnolia obovata*) or Katsura (*Cercidiphyllum japonicum*). Special colors of these woods are not conclusive because of the change in color by aging. Anatomically, both are diffuse-porous woods and vessel diameters are around 50-80 micrometer. Rays are multiseriate, consisting of procumbent and upright cells. The vessel perforation, however, shows distinct differences: simple perforation is dominant in *M. obovata*, whereas scalariform perforation with many bars is in *C. japonicum*. Fig. 2 is a reconstituted and computer sliced section of “inside” wood. Scalariform perforation is clearly visualized and thus wood is identified to be *C. japonicum*.

### 4. Conclusion

In conclusion, we confirmed that SR X-ray tomography would open new field in wood anatomy. As discussed here, it is indeed good for visualizing national heritage class wooden specimen. In addition, detailed analysis of tomography data may provide 3-dimensional quantitative structural data, that accounts for many complex physico-mechanical behavior of wooden materials.

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3. The investigation is in collaboration with Bijutsuin (Japanese Academy of Fine Arts), Kokuhou Shurisho (Institute of Conservation of National Treasures) under official agreement between Research Institute for Sustainable Humanosphere, Kyoto University.



Fig. 2. A typical 3D view of the sample from Sesin-bosatsu-rituzou, registered national treasure, curved by UNKEI in Kamakura period, obtained at BL20XU, SPring-8. Wood is identified as Katsura (*Cercidiphyllum japonicum*)

# METHODS OF NON-DESTRUCTIVE WOOD TESTING

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## Abstract

Methods of non-destructive wood testing are getting more and more important. Online tools, for example to control the production, are effectually in use for years. Based on a measuring systematic (physically active principle and important influencing factors), a summary of methods to assess cultural goods is given in the following. To adopt the methods based on physical effects, essential knowledge of wood physics is inevitable (knowledge of interdependencies, in particular).

## 1. Introduction

Methods of non-destructive wood testing are getting more and more important. Online tools, for example to control the production, have been in use for years. These tools can detect cracks in boards and their size (measured via ultrasound), density perpendicular to the board plane and surface irregularities. Chromatometers, gauges for thickness measurement, ultrasonic tests (including computed tomography), X-ray equipment, equipment to analyse chemical structures as well as NIR spectroscopy equipment are available. Optical systems can be used to detect strain concentrations that occur due to humidity changes. Based on a systematic of physically active principles and important influencing factors, a summary of methods to assess cultural goods is given in the following. To adopt methods based on the consideration of physical effects, an essential knowledge of the underlying wood physics is required, in particular knowledge of interdependencies.

## 2. Short overview of commonly applied methods

Table 1 (at the end of the paper) gives an overview of available methods for non-destructive wood testing.

### 2.1. X-ray tomography (detection of internal defects):

Fig.1 and 2 show an X-ray tomography from spruce with knots at different distance from the surface.

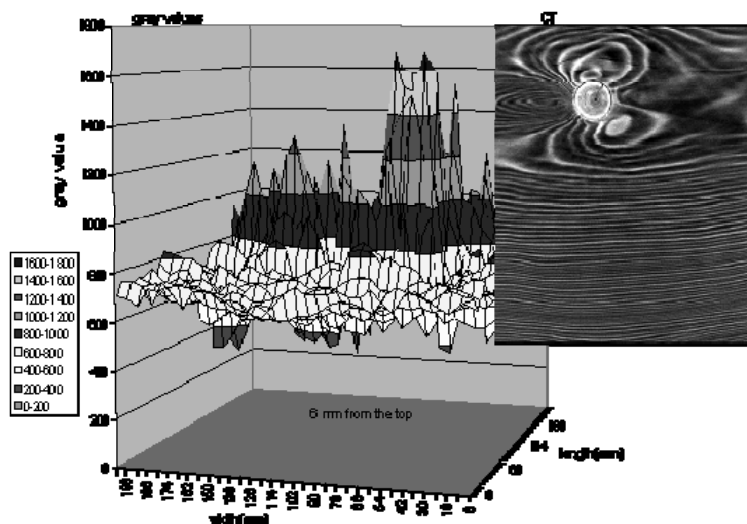


Fig. 1. X-ray tomography of spruce with knots

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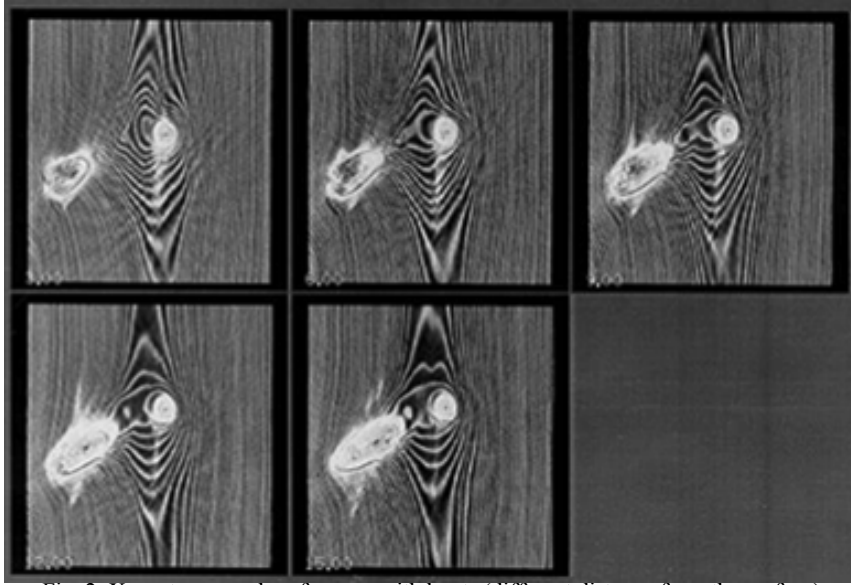


Fig. 2. X-ray tomography of spruce with knots (different distance from the surface)

## 2.2. Thermography:

The thermography allows the detection of defects near the surface (approx. 1mm). The delamination of surface material is detectable (Fig. 3).

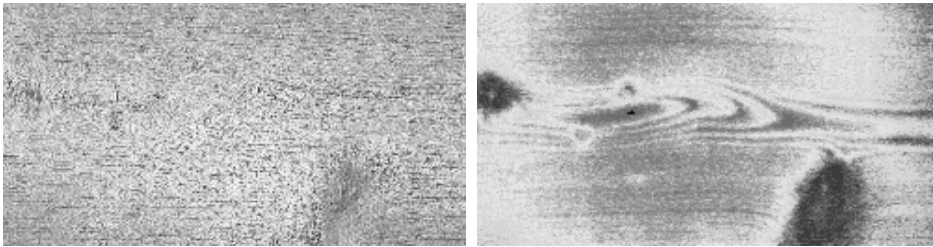


Fig. 3. Thermography (left) and photography (right) of spruce with knots

## 2.3. Strain distribution measured with Video Image Correlation

With Video Image Correlation, a visualisation of the strain field at the surface (produced by swelling, shrinking, mechanical stress) is possible (Fig. 4, 5).

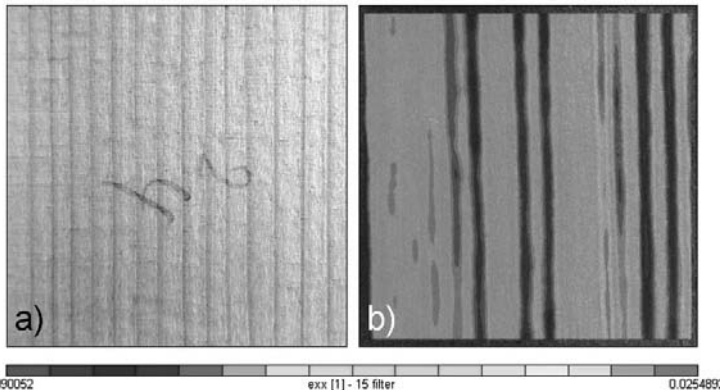


Fig 4. Dimensional deformation due to swelling on the longitudinal-radial surface of a spruce disk (L: 5 cm, R: 5cm, T: 5 mm). (a) The spruce sample without paint coating. (b) The same sample with a grey speckle pattern applied to the surface by airbrush.



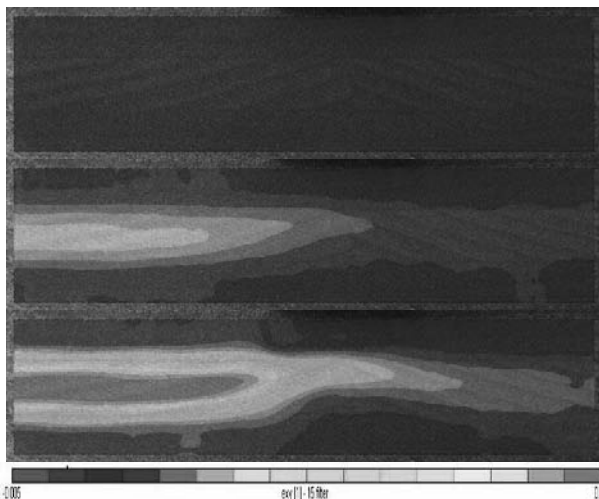


Fig. 5. Shear strain distribution in a cross laminated solid wood panel under mechanical load

#### 2.4. Sound velocity measurements

Measuring the ultrasound velocity make calculating elastic constants (MOE; G) possible. Detecting knots and small defects, however, is difficult in most cases (Fig. 6, 7, 8).

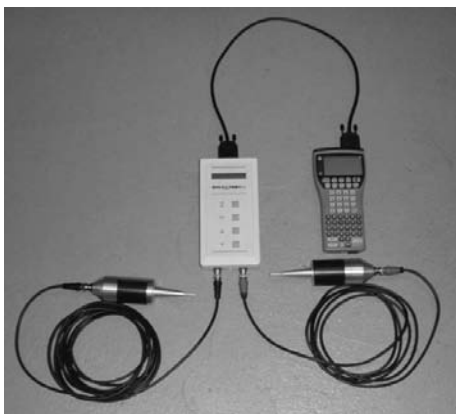


Fig. 6: Ultrasound testing equipment (Sylvatest 2)

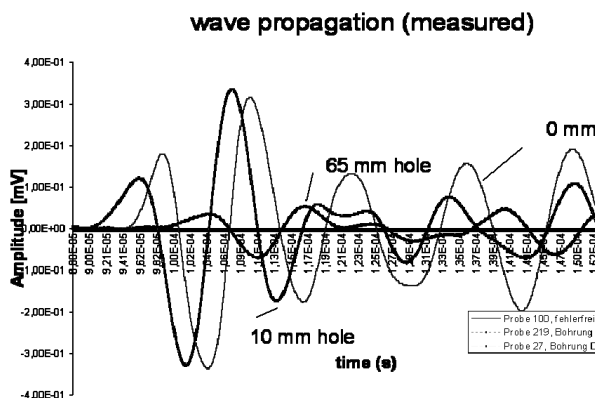


Fig. 7: Sound propagation in a spruce sample with various holes

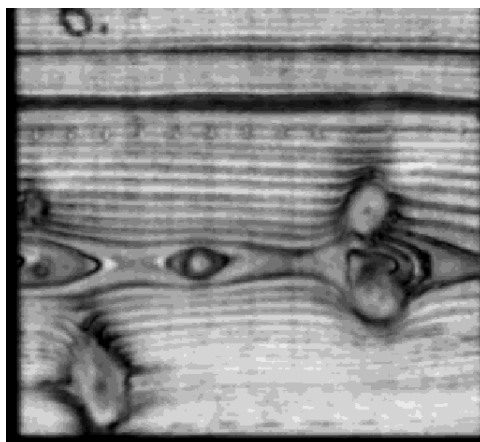


Fig. 8 Acoustic tomography of spruce with knots

#### 2.4. Neutron radiography/tomography and synchrotron light

A visualisation of water in wood is detectable with neutrons. The dimension is in the cm range (neutrons), mm range (synchrotron); with synchrotron light, a higher resolution is possible. The maximum sample dimension is in the mm range (Fig. 9, 11).

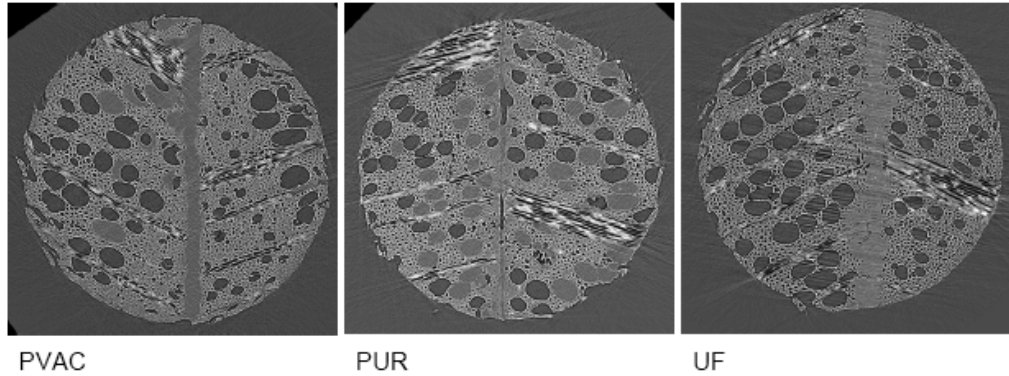


Fig. 9: Penetration of different adhesives into beech (analysed with synchrotron light)

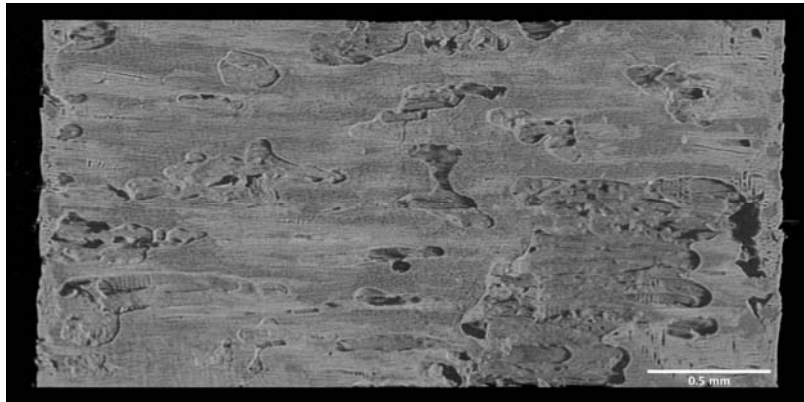


Fig. 10: Tomography of a glueline (synchrotron light)

#### 2.5. Mechanical properties

Fig. 11 and 12 shows tools than can measure the penetration depth (to detect defects on the surface area) and the drilling resistance (to detect fungal decay).

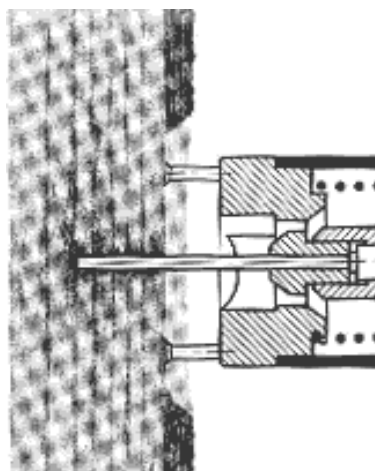


Fig. 11: Pilodyn tester (penetration depth)

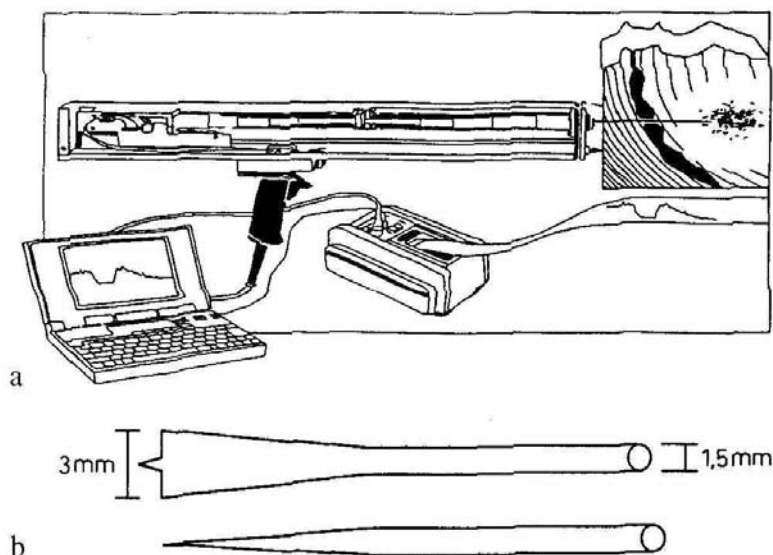


Fig. 12: Resistograph (drilling resistance tester)

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Table 1: Summary of methods of non-destructive wood testing

Property	Basic physical principles	Measurable properties	Suitability for investigating cultural heritage
Mechanical properties	Drilling resistance, hardness, intrusion behaviour	Detection of fungal decay, density	x
Electrical properties	Electrical resistance		x
	Correlation between electrical resistance and moisture content	Moisture content	x
	Correlation between electrical resistance and fungal decay	Detection of fungal decay	x
	Dielectrical properties	Moisture content	
Acoustical properties	Sound velocity; sound reflection; sound attenuation	Elastic constants (E,G) Defect detection	
	Acoustic emission	Micro cracks, eating noise of insects	(x)
	Eigenfrequency	Elastic constants (E,G) Delamination in glued wood joints	x
Thermal properties	Heat radiation (thermography)	Defects on near-surface areas (error adhesion of inlay, opened fugues)	x
Particles	Neutron radiation	Allocation of humidity	for laboratory testing only
Electro-magnetic waves	Visible light (ageing)	Colour measuring (CI-Lab), aging, colour differences	x
		Video Image Correlation (cross correlation, strain distribution)	x
	IR/NIR radiation	humidity, chemical analysis (impurities), partly mechanical attributes	(x)
	X-ray (absorption/diffusion)	density, local density allocation, annual grow ring profiles, angle in S2 (Sylviscan)	(x), complex equipment  3 constructions worldwide only
		Synchrotron radiation	micro structure analysis

# MEASUREMENT AND SIMULATION OF DIMENSIONAL CHANGES DUE TO FLOWS OF HEAT AND MOISTURE IN WOOD AND WOOD-BASED MATERIALS: HOW OBJECTS OF CULTURAL HERITAGE CAN BE STUDIED BY METHODS DEVELOPED FOR INDUSTRIAL APPLICATIONS

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## Abstract

The response of wood and wood based materials to environmental factors such as temperature and moisture is significant for technical applications as well as for works of art. Whereas climatic conditions for works of art are generally less severe and more stable than in technical applications, damages to objects of cultural heritage would imply more than financial loss. Consequently, the study of dimensional stability with respect to flows of heat and moisture is equally important in both fields. The aim of this contribution is to outline the state of the art from a technical point of view and to illustrate applications to culture heritage.

## 1. Techniques for the measurement of dimensional changes

A variety of techniques to measure dimensional changes due to climatic variations has been developed over the years. Besides conventional strain gauges, optical methods are often preferred since they are strictly non-contact and can cover large areas in short time. One has to differentiate between techniques which measure relative and absolute dimensional changes, respectively. Due to space restrictions, this paper will only discuss relative techniques. Relative techniques are generally more sensitive than absolute techniques and include sophisticated laser methods such as Electronic Speckle Pattern Interferometry (ESPI) as well as the rather simple but powerful Digital Image Correlation Technique (DICT). Both ESPI and DICT can measure displacements in all three spatial directions if the right set-up is used.

### 1.1. ESPI

If a rough surface is illuminated with a coherent laser beam, the reflected light rays can interfere and form a so-called speckle pattern which looks like an arrangement of dark and bright dots (Fig. left). Speckle patterns are fingerprints for the surface under study. In ESPI, the speckle pattern is superimposed with a reference beam derived from the same laser [1-3]. The result is recorded with a camera and taken as the reference image (Fig.1 right).

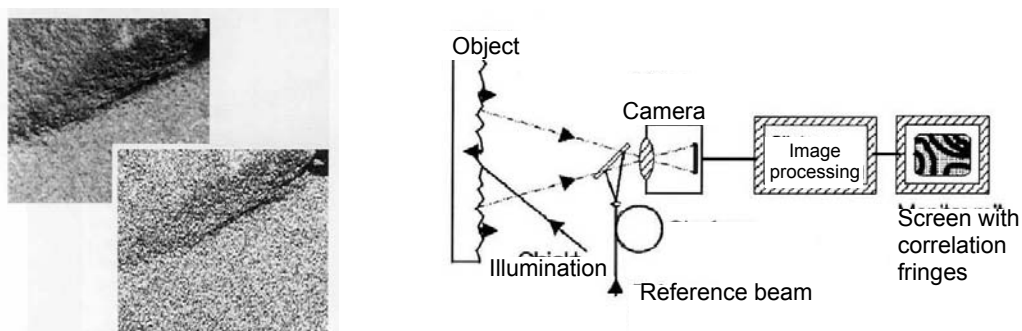


Fig. 1: Surface of a sandstone piece and its speckle pattern (left) and set-up for ESPI (right)

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Any physical stimulation leading to a surface deformation (for example vibrations or temperature changes) changes the speckle pattern and consequently the superimposed image. By subtracting the reference image from the active image, so-called correlation fringes are obtained which are lines of equal displacement (Fig 2 left). A displacement of half the laser wavelength is sufficient to generate a clear signal. Thus, the resolution of this technique is in the sub-micrometre range.

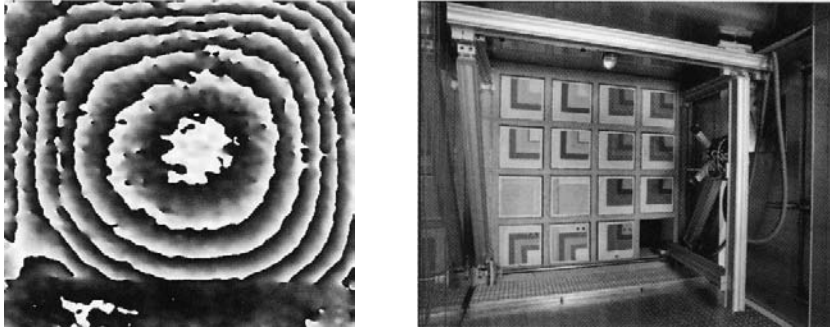


Fig. 2: Correlation fringes (left) and test frescoes mounted in a double climate chamber (right)

The setup shown in Fig.1 (right) allows the measurement of displacements perpendicular to the surface only (z direction). Two similar set-ups are necessary to measure displacements parallel to the surface (x and y direction). Fig. 2 (right) shows an application to the study of frescoes: A number of test frescoes is mounted between the two chambers of a double climate chamber in the WKI.

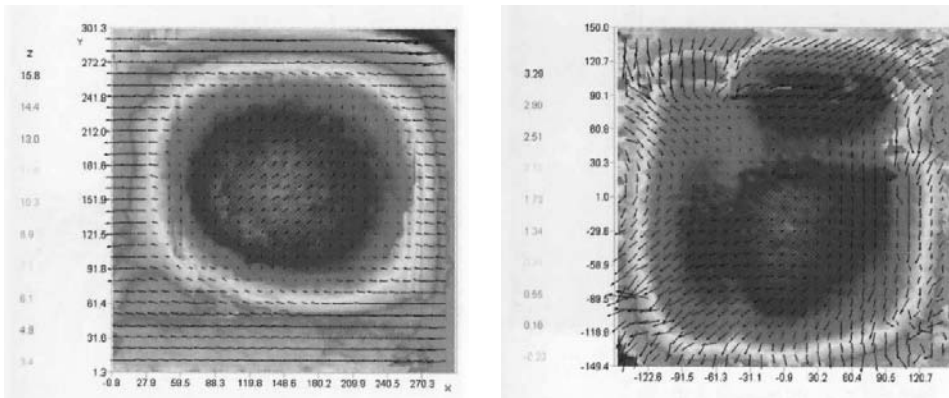


Fig. 3: Displacement plots for a good (left) and a defective (right) test frescoes

Increasing the surface temperature by 5 K on one side resulted in a deformation of the frescoes. The resulting displacements are plotted in Fig.3 for two different pieces measuring 30 cm x 30 cm. The displacements in z direction is colour coded and given in  $\mu\text{m}$ . The in-plane displacement is depicted by arrows. In the left part of the figure, the maximum z displacement is around 12  $\mu\text{m}$ , whereas the maximum in-plane deformation is lower than 2  $\mu\text{m}$ . Both the displacements perpendicular to the plane and in the plane vary little over the surface. This is typical for intact frescoes. In contrast, the fresco shown in the right part of Fig. 3 shows deformations varying strongly over the surface, and the in-plane displacement even changes its direction over a short distance. Although the maximum displacements are in the normal range (x: 5  $\mu\text{m}$ , y: 6.5  $\mu\text{m}$ , z: 3.5  $\mu\text{m}$ ) this is a clear evidence for the beginning destruction of the fresco.

## 1.2. DICT

The digital image correlation technique is a photogrammetric method. In contrast to conventional photogrammetry no externally applied markers are need. These are replaced by the surface features of the object. Consequently, DICT will not work for extremely smooth, featureless objects. In cultural heritage, however, this situation is encountered very rarely.

The hardware needed for DICT is simply a digital camera suited for photogrammetry and having a reasonably high resolution and a homogenous illumination (Fig.4 left). Until recently resolutions exceeding 10 Megapixel could only be achieved by mechanical scanning. For example, the results presented below were obtained using a Rollei SX6006 having a CCD line detector with 5000 pixels (size:  $15\ \mu\text{m} \times 15\ \mu\text{m}$ ). The detector was scanned mechanically in 5048 steps over the image plane resulting in a resolution of more than 25 Megapixel. Even today the so-called micro-scan principle is frequently used to increase the resolution of matrix detectors. In this type of camera, the detector can be shifted  $1/2$  pixel (or some other fraction of a pixel) in both directions of the detector plane. In such a way four images with different detector positions can be taken and combined into one high-resolution image. A detector having  $2048 \times 2048$  pixels makes thus images having an effective resolution of  $4096 \times 4096$  pixels.

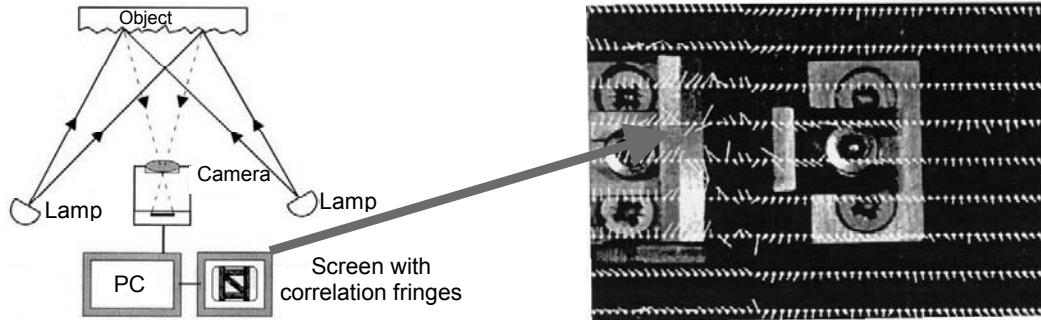


Fig. 4: Set-up for DICT measurement on beams of half-timbered houses (left) and a typical result (right)

By acquiring a reference image before and a final image after applying a load to an object (e. g., a climatic change) the displacement can be calculated by dedicated algorithms. These compare a given pixel in the reference image with every pixel in the final image in the following way: A subset of  $n \times n$  pixels centred around each of the pixels to be compared is defined. The cross correlation function between these two subsets is calculated. The pixel in the final image giving the largest correlation coefficient is identified with the pixel in the reference image so that the displacement can be calculated (Fig. 4 right). The procedure is repeated for every pixel in the reference image. This is obviously a time-consuming procedure. For practical reasons it is often necessary to limit the number of final pixels to be compared with a given reference pixel. This is normally done by defining a search region of size  $N \times N$  around the reference pixel with  $n < N \leq 100$ .

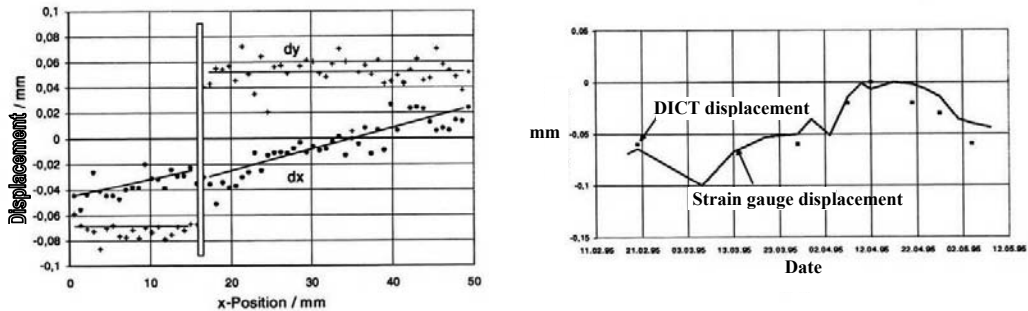


Fig. 5: Displacements of a beam of a half-timbered house in x- and y-direction along a line parallel to the beam (left) and comparison of DICT and strain gauge data (right)

The displacements obtained in such a way are often displayed by vectors as shown in the right part of Fig.4 for the example of a beam of a half-timbered house. The image shows also a conventional strain gauge which is mounted in order to compare their data with DICT measurements. The displacements along a line parallel to beam are plotted in the left part of Fig.5 separately for the x- and the y-

direction. The drastic change of  $dy$  at position 16 mm indicates a crack. The right part of Fig.5 shows a comparison of DICT and strain gauge data which correlate quite well [4].

### 1.3. Other experimental details

Since the measurement generates point clouds which are subjected to errors, sophisticated algorithms are needed in order to reconstruct the surface form the measurement. Dimensional changes can of course be calculated by comparing the object's data for different climatic situation.

In order to measure the object's response to climatic variations one can just utilize natural variations, or one can simulate them in climate chambers. Double climate chambers are a very convenient way to study the results of differential climates. WKI has a double climate chamber which can accommodate wall elements as big as 8 metres long and 2.3 metres high.

## 2. Numerical simulations

Experimental studies of dimensional changes are time consuming and expensive. Furthermore, experiments with objects of the cultural heritage are often not possible since the objects are unique, and possible damages cannot be accepted. For these reasons, numerical simulations are very important in the study of climate effects. They can also reveal insight into stresses and strain in the interior of the object, which are difficult to measure. Many authors have worked on the differential equations describing the flow of heat in moisture in materials, especially with respect to their application in civil engineering. Wood and wood based panels are often used as walls for buildings, and their insulation properties against heat and moisture are consequently important. Furthermore, heat and moisture exchange affects dimensional stability. Cracks and warping can be the consequence of improper design.

The more advanced equations are based on the principles of conservation of enthalpy and mass and consider effects such as phase transitions and the coupling of heat flow and moisture flow [5]. Numerical simulations of heat flow concentrate on heat conduction and vapour flow (associated with a phase transition) because the other mechanisms (thermal radiation and flow of air or liquids) are not relevant in practice or too difficult to calculate. These two effects are described by the following equations:

$$q = -\lambda \vec{\nabla} \theta \quad (1)$$

and

$$S_h = -h_v \vec{\nabla} \bar{g}_v \quad (2)$$

where  $q$  [ $W/m^2$ ] is heat flow density,  $\lambda$  [ $W/mK$ ] the thermal conductivity,  $\theta$  [ $K$ ] the temperature,  $S_h$  [ $W/m^3$ ] the heat generation associated with a phase transition from vapour to liquid or vice versa,  $h_v$  [ $J/kg$ ] the latent heat of water and  $\bar{g}_v$  [ $kg/m^2s$ ] the vapour flow density.

Possible mechanisms for the transport of liquid water are capillary transport, surface diffusion, percolation, hydraulic flow, electric fields, and osmosis, among which the first is by far the most important. Using the Kelvin formula which relates capillary pressure to the relative moisture of air  $\phi$  one obtains

$$\bar{g}_w = -D_\phi \vec{\nabla} \phi \quad (3).$$

In this equation  $D_\phi$  [ $kg/m \cdot s$ ] is the transport coefficient.

Finally, the transport of water vapour has to be considered which is possible by gas diffusion, molecular transport, diffusion by solution in other materials (e. g., polymers), and convection. Convection is normally not considered because it is not often relevant and difficult to calculate. The other three mechanisms are summarized by

$$\bar{g}_v = -\delta_p \vec{\nabla} \phi p_{sat} \quad (4)$$

where  $\delta_p$  [ $kg/m \cdot s \cdot Pa$ ] is the vapour permeability and  $p_{sat}$  [ $Pa$ ] the saturation pressure of vapour.



In order to link all these equations, conservation laws can be used. Since phase transitions are involved, conservation of enthalpy gives

$$\frac{\partial H}{\partial t} = -\vec{\nabla} \cdot \vec{q} + S_h \quad (5).$$

Inserting terms results in

$$\frac{\partial H}{\partial t} = \vec{\nabla} \cdot (\lambda \vec{\nabla} \vartheta) + h_v \vec{\nabla} \cdot (\delta_p \vec{\nabla} (\varphi p_{sat})) \quad (6),$$

$$\frac{dH}{d\vartheta} \frac{\partial \vartheta}{\partial t} = \vec{\nabla} \cdot (\lambda \vec{\nabla} \vartheta) + h_v \vec{\nabla} \cdot (\delta_p \vec{\nabla} (\varphi p_{sat})) \quad (7),$$

and

$$\frac{\partial \vartheta}{\partial t} = \frac{1}{C\rho} \left[ \vec{\nabla} \cdot (\lambda \vec{\nabla} \vartheta) + h_v \vec{\nabla} \cdot (\delta_p \vec{\nabla} (\varphi p_{sat})) \right] \quad (8)$$

using the definitions of specific heat  $C$  [J/kg·K] and density  $\rho$  [kg/m<sup>3</sup>]. On the other hand, using conservation of mass one can write

$$\frac{\partial w}{\partial t} = -\vec{\nabla} \cdot (\vec{g}_w + \vec{g}_v) + S_w \quad (9)$$

where  $S_w$  [kg/m<sup>3</sup>·s] describes a possible mass source (e. g., a water leak). Inserting (3) and (4) one obtains

$$\frac{\partial w}{\partial t} = \vec{\nabla} \cdot (D_\varphi \vec{\nabla} \varphi + \delta_p \vec{\nabla} (\varphi p_{sat})) \quad (10).$$

Observing that  $dw/d\varphi$  is a material property describing the so-called sorption isothermal lines we obtain

$$\frac{\partial \varphi}{\partial t} = \left( \frac{dw}{d\varphi} \right)^{-1} \left[ \vec{\nabla} \cdot (D_\varphi \vec{\nabla} \varphi + \delta_p \vec{\nabla} (\varphi p_{sat})) \right] \quad (11).$$

With equations (8) and (11) we now have two equations for temperature and humidity, respectively, which are coupled by the last term in (10) and also by the fact that many of the material properties depend on temperature or moisture. Consequently, the equations have to be solved numerically. The WKI has developed a software called TUN which can solve the equations in two dimensions by the finite difference method. Work is under way to include the equations in a commercial finite element code.

A typical application is the analysis of the moisture behaviour of a wood panel wall with brickwork curtain as shown in Fig. 6. The question was whether the water repellent layer is really necessary. A typical summer climate for central Europe was taken as the boundary conditions (Fig. 7 left). The resulting moistures for selected parts of a construction without water repellent layer are displayed in the right part of Fig. 7. The result is that the moisture at the front side of the first particle board (approximately 20%) is too high, and that the moisture gradient over the first particle board is too high as well leading to the risk of unacceptable distortion. Consequently it is indeed necessary to keep the water repellent layer or to change the properties of the particle board.

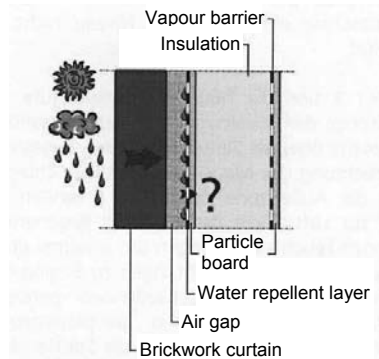


Fig. 6: Wood panel wall with brickwork curtain

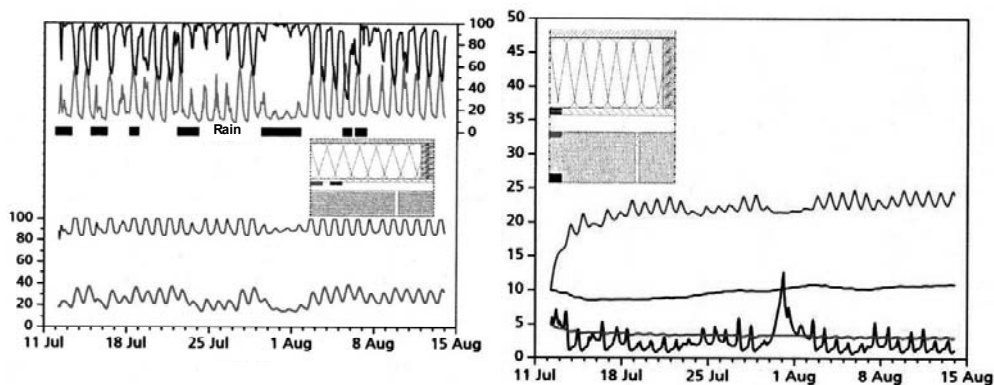


Fig. 7: Right: Central European summer climate as the boundary condition for the simulation of a wood panel wall. Orange: radiation temperature, blue: outdoor humidity, green: air gap humidity, red: air gap temperature. Left: Resulting moisture for selected parts of the construction as a function of time. Black: front side of brick wall, red: back side of brick wall, green: front side of first particle board, blue: back side of first particle board

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# IN THE HEART OF THE LIMBA TREE (*TERMINALIA SUPERBA* ENGL. & DIELS): DETECTION METHODS FOR HEART ROT AND FALSE HEARTWOOD

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## Abstract

Both resistance and velocity measurements were performed on planted and natural limba trees in the Democratic Republic of Congo. While 2 velocity measurements per tree are sufficient for the quick diagnosis of rotten or hollow trees, a compilation of 16 resistance profiles is necessary to distinguish the so-called false heartwood (limba noir). On those two-dimensional reconstructions, small peaks in resistance correspond roughly with the boundaries of limba noir. Detection methods are influenced by diameter, wood density and the presence of buttresses. Further research on density profiles is recommended to analyse these influences and the link with both detection methods.

## 1. Introduction

Limba (*Terminalia superba* Engl. & Diels) has a very large distribution: from Sierra Leone until the north of the DRC from the east and from the south, until northwestern Angola [1]. This heliophilous species - often with large buttresses - is typically found in secondary forests and fallows [7] but also in plantations in Bas Congo (Democratic Republic of Congo). The presence of heart rot in older trees and the formation of a so-called limba noir or false heartwood [2] alters the popularity of this species.

The incidence of these two wood anomalies makes limba a very suitable species for the study of detection methods for as well heart rot as wood discolorations. This study is set up within the framework of sustainable forest management of tropical forests: detection of anomalies before exploitation prevents useless cutting and the loss of trees with an ecological function as e.g. seed trees. This implies certain conditions for detection methods: in situ applicable, not too expensive, fairly quick and easy to interpret. Two methods that meet all conditions are resistance [5, 6] and acoustic detection methods [9], mostly used as control methods for more specialised detection techniques. This case study wants to answer the following questions: (a) Are resistance and acoustic methods able to detect rot and/or wood discolorations like limba noir? (b) Which factors influence the measurements (density, buttresses, diameter)?

## 2. Material and methods

### 2.1. Site description

In the present case study, we focus on a 58 year old plantation of *Terminalia superba* at the southern border of the Mayumbe Forest and a secondary forest with natural limba trees more eastward, near Tshela. The limba plantation lies within the transition area of the Man and Biosphere Reserve of Luki, about 400 km coastward from Kinshasa, and surrounds a core area of drier semi-evergreen Guineo-Congolian rain forest with large parts of secondary forests where cultivation took place [8]. The forest near Tshela is also characterized by this alternation of secondary forest and agricultural lands.

### 2.2. General and detailed study

The detection methods were applied in two ways: 2 perpendicular measurements per tree (general study) and 16 measurements per tree at equal distances (detailed study).

The general study is meant to evaluate the quick diagnosis of wood anomalies but also the relation between the two detection methods and the influence of diameter and buttresses. Next to the detection measurements, two wood cores per tree were taken to control the state of the wood just above or

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below the measuring point. Notes on the health status of trees were written down. The general study took place on 87 trees in the plantation. None of them showed signs of limba noir. Next to limba trees, other tree species were analysed with the acoustic method to investigate the influence of density and diameter ( $n = 10$  trees per species).

The detailed study is reconstructing the inside of the tree, providing two-dimensional visualisations. This more intensive case study was performed on freshly cut stem disks, where disk surface and detection measurements could be compared. In total, 8 disks from 5 trees were analysed.

### 2.3. Detection methods

Both detection methods are standard methods for the detection of cavities and wood anomalies. However, these two methods are not often used on tropical tree species.

All resistance measurements were performed with a resistograph of the type IML-Resi B400. This instrument has a needle that penetrates the wood and has a precision of 0.04 mm. Sound wood has a higher resistance than infected wood. The measurement is semi-destructive, leaving only a small channel where the needle passed. The result is a resistance profile from the outside of the tree (without bark for the general study, with bark for the detailed study) to the pith.

A cheaper method consists of the determination of the velocity of sound within stems. Two sensors were placed upon the tree on opposite sides. With a small hammer, one of the sensors was hit, transmitting a sound pulse to the opposite sensor. The result has a precision of 1  $\mu$ s. Every travel time is the result of five repetitions that were recorded with the Fakopp Microsecond Timer. This measuring device only leaves two small holes in the tree where the sensors were placed and is less destructive than the resistograph. When the distance between the two ends of the sensors is measured with a calliper (precision of 0.5 cm), the velocity can be calculated. The result is one velocity value per measurement.

### 2.4. Data analysis

Comparing one velocity value with a complete resistance profile is complicated. After the first trials, a transformation of the resistance profiles into variables that are comparable with velocity values, took place. These variables are all values instead of profiles which makes comparison easier. Every resistance curve shows a similar pattern (Fig. 1): an increasing trend as the needle enters the stem (zone A) followed by a more stable trend within the main stem (zone B). Using a double linear regression, we converted the resistance profiles into point values: the breakpoint or C (distance from the start of the profile to the intersection of the two linear regressions with the best cumulated determination coefficient,  $R^2$ ), the slope of the first (zone A) and second linear regression (zone B), the maximum, minimum and range of resistance values after the breakpoint (within zone B, zone A is not taken into account since resistance values are still in the transition stage after entering the stem). All these values were compared with velocity and diameter values with special attention for the outliers of velocity (wood anomalies!).

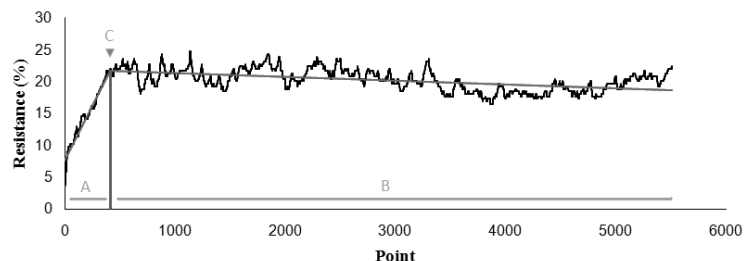


Fig. 1 – Example of the transformation of resistance profiles to point values. The result is a breakpoint at point 407, corresponding with a distance of 16.28 mm.

During the detailed study, interpolation of the resistance profiles in order to visualize the internal distribution of resistances is based on the principle of inverse distance weighing. For every unknown

pixel, a value is estimated based on the known resistances within a certain radius of the unknown pixel taking into account that the further from the unknown pixel, the less influence it has in the estimation. The acoustic reconstruction is based on an existing algorithm [4]. The current images are based on the travel times (10-5 s).

### 3. Results

#### 3.1. Diagnosis of wood rot

Variation in resistance and velocity within and between sample trees is moderate to large. For instance, the differences in velocity within a tree are considered not significant (ANOVA,  $p = 0.72$ ). However, it is important to keep the original values since averaging would only permit to detect rot in the central part of the tree. According to the boxplot, normal velocity values are situated between 1000 and 1650  $\text{m s}^{-1}$ . When rejecting the velocity outliers, the mean velocity of mature limba trees is  $1288 \pm 120 \text{ m s}^{-1}$  ( $n = 174$ ). This can be considered as the reference velocity ( $V_{\text{ref}}$ ) within a 95% confidence interval from 1270 to 1306  $\text{m s}^{-1}$ . The relative decrease in velocity can be calculated like this:

$$\text{Relative decrease of sound velocity} = (V_{\text{ref}} - V_{\text{mes}}) / V_{\text{ref}} * 100 \quad (1)$$

On a total of 184 velocity measurements, only 7 measurements or less than 4 % appear unusually low, ranging from 298 to 917  $\text{m s}^{-1}$ . The corresponding relative decrease of sound velocity ranges from 29 to 77 %. The lowest record of velocity (298  $\text{m s}^{-1}$ ) is excluded for further analysis because the sensor was placed in the rotten area, a position that causes diverging values. Compared to the table on the distributor's website ([www.fakopp.org](http://www.fakopp.org)), this decrease should equal a decayed area of 75 % and more (hollow trees). At the other side of the boxplot, 3 measurements are extremely high, reaching velocities of 1676 to 2181  $\text{m s}^{-1}$ . Mostly, those maxima are associated with the presence of buttresses. Before converting the resistance profiles, we compared the outliers of the velocity values with the resistance profiles. Sometimes, the decrease in velocity corresponds with a sudden decrease in resistance that is clearly visible (tree 8) while in other cases, this decrease can also appear to be subtle or even absent (tree 27) (Fig. 2). Looking at the notes on the outer state of the tree and the wood cores, we can see that rot detection methods generally confirm what is suspected. Only little rotten zones near the bark, with velocities from 1050 to almost 1200  $\text{m s}^{-1}$ , are still classified as healthy because they are not considered as outliers.

After transformation (Fig. 1), a significant Pearson correlation of 0.23 ( $p < 0.01$ ) was found between velocity and minimum resistance. Still, this correlation is not sufficient enough to use minimum resistance as a criterion for detection. Correlations do not improve when only outliers are analysed.

#### 3.2. Influence of diameter, buttresses and density

The diameter is most significantly correlated with velocity ( $r = 0.44$ ). Other significant Pearson correlations ( $p < 0.01$ ) include the variance of the velocity measurements, the breakpoint, the maximum resistance and the range of resistance ( $r = 0.20$ - $0.29$ ). When reconstructing these relations graphically, only diameter (D) and velocity (V) result in a satisfactory linear regression model (Adjusted  $R^2 = 0.191$ ,  $p < 0.001$ ):

$$V = 514.16 D + 1030.5 \quad (2)$$

Diameter is also positively related to the velocity of seven other African tree species: *Millettia laurentii*, *Lovoa trichilioides*, *Aucoumea klaineana*, *Nauclea diderrichii*, *Pterocarpus soyauxii*, *Entandrophragma utile* and *angolense* ( $r = 0.53$ - $0.91$ ). Only *Prioria balsamifera* didn't reveal significant correlations with velocity values. Density (literature data) and velocity of the tree species are described by a logarithmic regression model, concluding that a higher density ( $\rho$ ) goes along with a higher velocity (Adjusted  $R^2 = 0.66$ ,  $p < 0.001$ ):

$$V = 866.77 \ln(\rho) + 1941.96 \quad (3)$$

Last but not least, it sometimes was not possible to avoid buttresses during the measurements. Although this is not a general rule, high velocity measurements ( $> 1350 \text{ m s}^{-1}$ ) were often found where measurements were pressed between two buttresses. When the actual measurement occurred on top of the buttress, velocity tends to drop below the mean velocity.

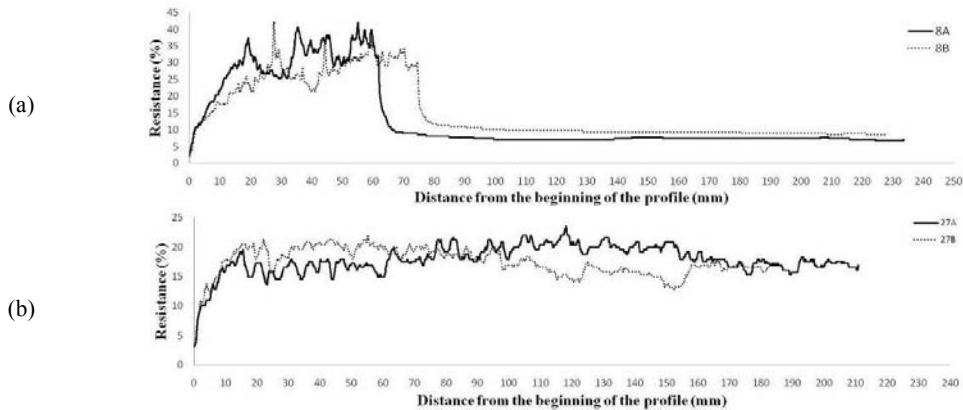


Fig. 2 – Resistance profiles of two infected trees: (a) Tree 8: both resistance profiles visualize a large rotten zone and have low velocities (ca.  $850 \text{ m s}^{-1}$ ). (b) Tree 27: measurement B has a low velocity ( $917 \text{ m s}^{-1}$ ), but the decrease in resistance could be ascribed to natural density variations if velocity measurements were absent.

### 3.3. Qualitative assessment of heart rot and limba noir

Two stem disks from Tshela were selected (Fig. 3): the first stem disk of the third tree (3.1) containing rot and a cavity and the first stem disk of the seventh tree (7.1) with limba noir. When looking at disk 3.1, it is clear that the rotten zone surrounding the cavity is also reflected by a decreased resistance. Resistance is the highest at the outsides, possibly as an indicator of a higher density (caused by e.g. narrow growth rings). Preliminary research on dry stem disks from Ivory Coast showed a peak value in resistance just after entering the darker heart. Passed this point, the resistance decreased quickly. On fresh stem disks like 7.1, this effect is less clear but not absent. A ring with a higher resistance value is noticed but the peak values do not always occur after entering the dark heart; they more or less vary around the border of the dark heart and do not decrease as fast as on dry stem disks. The reconstruction based on travel times is in both cases problematical.

## 4. Discussion

Normally, mature limba trees are sensitive to heart rot. In this case, less than 5 % of the sampled trees was infected, possibly the consequence of good planting material and favourable site conditions. Rotten or hollow trees are mostly found next to little paths that are regularly used by the local population. Using our detection methods, hollow and heavily rotten trees are detectable, where before only hollow trees could be distinguished using velocity measurements [9]. With only two measurements per tree, the resistance profiles are sometimes hard to interpret. Velocity measurements give a more straightforward value that can be immediately compared to reference velocity values. Therefore, velocity measurements are recommended for quick diagnosis. The range of allowable velocity measurements is still large ( $650 \text{ m s}^{-1}$ ) so we propose further research on the diagnosis of small cavities or wood rot within the lower end of this range ( $1050\text{-}1200 \text{ m s}^{-1}$ ).

We confirm earlier results on the influence of density [3]. Higher diameters generate higher velocities in several tropical tree species, while previous research concluded the opposite [3]. We could expect that even-aged trees with a larger diameter are fast-growing so less dense and with a smaller velocity. Still, the trends in several African tree species are far more significant than in the previous study. The reason for this phenomenon is unknown and could be linked to wood anatomical features.

Transforming resistance profiles into point values is not recommended. Resistance profiles are most appropriate for mapping of the impact, shape and location of wood anomalies. When analyzing the reconstructions, small resistance values often indicate rotten areas while peak values can be associated with the boundaries of limba noir. Acoustic measurements do not provide clear-cut reconstructions because they were made on stem disks, ignoring the influence of sound propagation in an axial way. Since stem disks often show cracks, these anomalies are also detected by velocity measurements,

complicating reconstructions. Acoustic reconstructions should be based on standing trees above the buttresses.

Resistance and velocity values are not easy to link since they weren't taken at exactly the same spot but 5 to 10 cm one below the other. The presence of buttresses also influences results but there is no general trend visible in velocity or resistance measurements. Buttresses sound quite hollow but can cause reaction wood once they join the main stem, creating additional density variations. Analysis of density (buttresses, wood cores and stem disks) is recommended to find the missing link between resistance and velocity measurements.

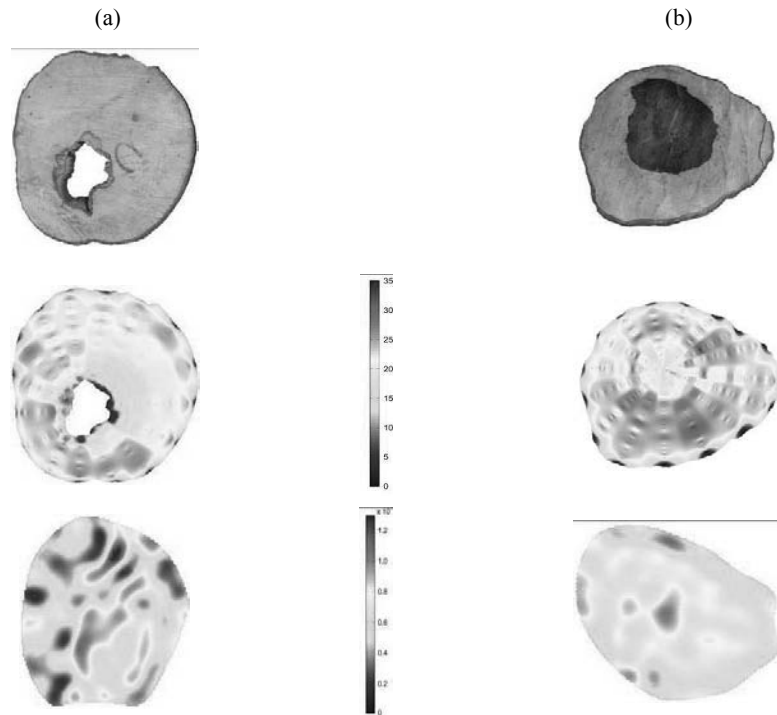


Fig. 3 – Two-dimensional reconstruction of two stem disks. From top to bottom, the stem surface, the resistance (%) and acoustic reconstruction with travel times ( $10^{-5}$  s) is shown.

(a) Stem disk 3.1 with rot and a cavity in the central part of the stem

(b) Stem disk 7.1 with discoloured wood or limba noir

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# LOCAL DEFORMATION REACTIVITY OF PANEL PAINTINGS IN AN ENVIRONMENT WITH RANDOM MICROCLIMATE VARIATIONS: THE MALTESE MAESTRO ALBERTO'S *NATIVITY* CASE-STUDY

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## Abstract

An experience of deformation analysis on a panel painting under uncontrolled exhibiting conditions is here described. The measurement session has been carried out during an STSM in the frame of the COST-IE0601 Action. The measurements were made using two Deformometric Kits, apparatuses that were developed in DISTAF during the last ten years. They allow to obtain useful information about in-plane and out-of-plane variations of wooden objects undergoing microclimatic variations. The two Kits were applied parallel in different parts of a single board panel painting under restoration, in order to evaluate its *reactivity* to the microclimate variations. The measurement evidenced that the painting reacts with significant differences in the different areas. The data were normalized in order to make them comparable. Some analytical tools have been discussed and applied to the data in order to focus on the short term variations.

## 1. Introduction

In this paper, the first experimental results of an STSM performed in Malta are discussed. The analysis of deformative response of a panel painting to microclimate variations, in its exhibiting environment, are shown in order to analyse its complex response, influenced both by the time-dependent parameters, like moisture diffusion, hysteresis and mechano-sorption, and by the mechanical characteristics, like anisotropy and local defects effect.

The concepts of PEMC (Potential Equilibrium Moisture Content) and of *reactivity* as the rate of response of a wooden object to the microclimatic variation are briefly introduced.

The attended results of this analysis will become the basis for choices regarding the need of active or passive climate control aiming to improve the conservation of the painting.

The STSM has been performed during the month of June 2008 and the system is now applied and working on a painting in the Heritage Malta laboratories.

## 2. Materials and methods

The artefact that has been analysed during the STSM and currently under monitoring is a painting undergoing restoration at the laboratories of Heritage Malta, Kalkara, near Valletta. It is a *tempera* painted plank of a minor artist of the XVI century, Maestro Alberto, representing a religious subject, the *Nativity*, normally exhibited in the Museum of Fine Arts in Valletta (Fig. 1).



Fig. 1: the Maestro Alberto painting, with the red circles showing the most damaged parts.

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The painting has many painted layer's stability problems, indicated in red circles in the figure, probably depending on the reactivity of the wooden support to the microclimate variations.

The object is normally exhibited in an uncontrolled environment, where no climate logging system is installed. The reports on the microclimatic conditions are based on synthetic and subjective descriptions needing to be more objectively represented.

The mechanical structure of the painting is very simple, the support is constituted of one single poplar board, horizontally oriented and with the following approximate dimensions:

- length: 1580 mm;
- width: 320 mm;
- thickness: ~24 mm.

It has no stiffening elements, like cross-beams or timberframe, and this makes the deformation data we obtained easier to be interpreted, because of no interactions with external structures.

During the restoration period the painting was placed in the laboratories of Heritage Malta, in Qalqara, and it stood on an easel on the middle of its length (Fig. 2).

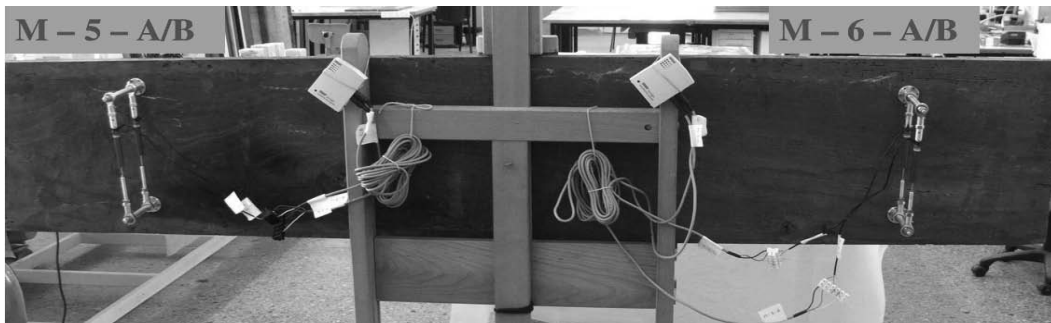


Fig. 2: the two Deformometric Kits applied on the back of the painting, in correspondence to the damaged areas.

### Description of the Deformometric kit (DK)

The measurement system adopted for the monitoring is called Deformometric Kit and it has been developed during some years at the DISTAF at the University of Florence.

The benefits of this system are primarily:

- the low impact on the paintings, usually welcome by the restorers and the conservators of the artifacts;
- the high sensitivity to the dimensional variations;
- the quality of in-plane and out-of-plane variations data it can describe, through combined linear deformations, thanks to the geometry of the system.

In order to evaluate the amount of the reactivity, the monitoring system applied on the painting can give direct local information but, after a geometric and statistical elaboration, it is possible to obtain more general and comparable data.

The system, as already described in previous papers [1,2], is based on two parallel transducers joined by means of rod end bearings (bronze-steel in order to make the rotation friction negligible) to two rods connected with wood screws perpendicularly to the back surface of a wooden panel (Fig. 3); because of the wood cupping deformation, while the two vertical elements remain perpendicular to the local surface, their axes form a variable angle with an infinite radius in case the surface is flat; the radius becomes shorter (and, as a consequence, the included angle gets wider) according to the increase of cupping.

The distance between the two elements is measured and logged by the transducers connected to a data-logging system that, in the DK most advanced configuration, is able to power the sensors and to record the signal they output proportionally to their core length.

The DK is at any rate very flexible in its measuring possibility, allowing to adapt it to different measurement needs [2].

In order to simplify the elaboration the measured raw data by the persons involved in the conservation of the object, a synthetic spreadsheet to be filled with the raw data, allows to obtain different geometric parameters:

- the *base distance* between the connections of the DK to the panel ( $l$ );
- the *cupping angle* of the panel ( $\beta$ );
- the *deflection* of the panel ( $f$ ).

It has been elaborated as a *technical annex* of the system and it can be used after specifying the initial parameters adopted for the positioning of the DK:

- the total length of each system “transducer-connections” when the core is completely inside the transducers;
- the distance of the lower transducer from the back surface of the painting and the distance between the transducers;
- the calibration parameters of each transducer, necessary to convert the Volt values, recorded by the logger, in absolute distances corresponding to the core length.

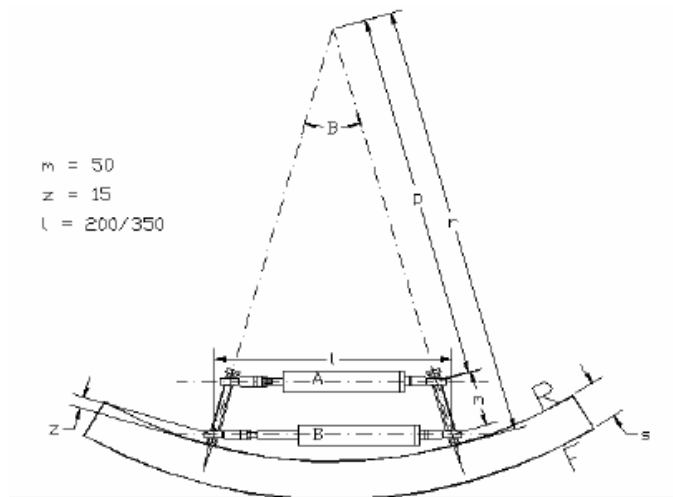


Fig. 3: geometric configuration of the Deformometric Kit for cupping analysis.

### Installation of the system

Due to the previously described simple mechanical structure of the painting, we considered interesting the analysis of the behaviour of the plank in different parts of its length, reasonably influenced by local irregularities of its physical structure. It was so decided to install two parallel systems placed in a similar way (almost the same distance between the transducers, according to the local structural differences) in different parts close to the more damaged areas (Fig. 1) along the length of the plank (Fig. 2).

### Data analysis criteria

The period examined in this paper is limited to ten days, randomly chosen, between 02/09 and 13/09/2008.

In Fig. 4 it has been chosen to aggregate the Temperature and Relative Humidity in one single value, called Potential Equilibrium Moisture Content (PEMC), obtained using the Hailwood-Horrobin model[3]. It doesn't represent the actual value of wood moisture but the value to which wood would tend to equilibrate in the stable conditions of the given T and RH; it has both the practical utility to be synthetic and to be easily relatable to the mechanical reactivity of the measured objects. The analysed period are evidenced in yellow.

Even though in the restoration laboratories of Kalkara the RH and T are not controlled, the EMC values are in general far from high extreme values (*e.g.* dangerous for possible fungal attacks, over the 20% EMC threshold), despite the *traditional* opinion of Malta as a very humid region.

After the elaboration of the experimental data, it was chosen to display them in two different ways:

- in the first one (Fig. 5) the deformation data are shown as a function of the PEMC variation; in order to compare the different responses deriving from different starting base distances of the two DKs, the variations have been normalized and expressed as a percentage of the initial base distance;
- in the second one (Fig. 6) the short-term *reactivity* of the two systems is displayed in parallel to the PEMC variation curve; also in this case the data have been normalized and expressed as a percentage, divided by the chosen sensitivity parameter  $\Delta_t$ , as displayed in the secondary Y axis.

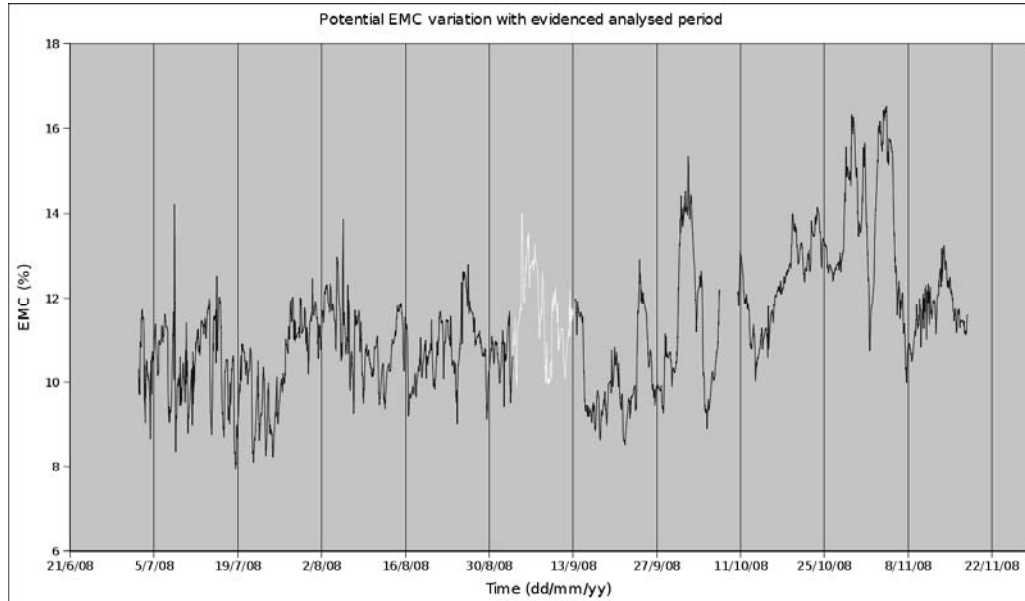


Fig. 4: the PEMC (Potential Equilibrium Moisture Content of wood) values updated till December 2008 and, in yellow, the period specifically analysed in this paper (02/09/2008-13/09/2008).

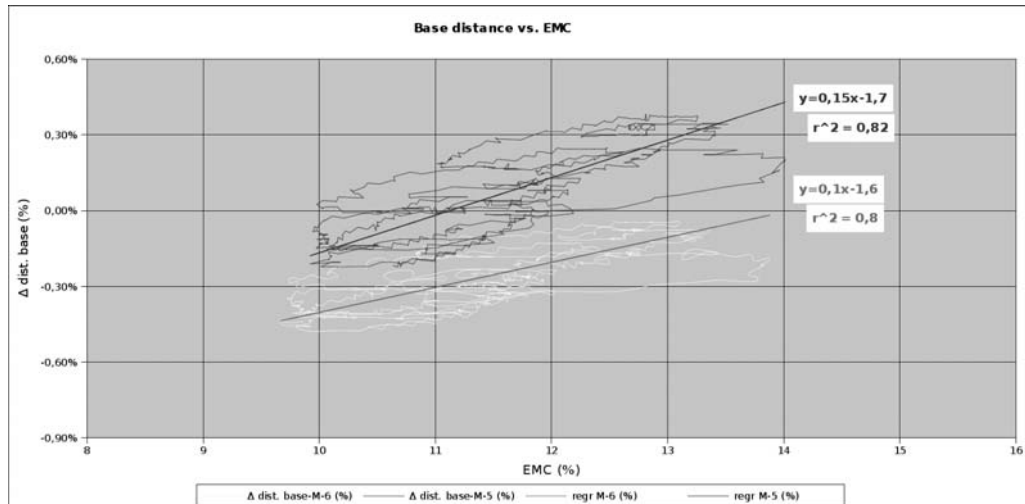


Fig. 5: The comparison between the normalized variation of the base distance (expressed in percentage) of the two systems as a function of the PEMC variation during the analysed period; the linear regressions of the two populations are displayed with the  $r^2$  index.

This approach is proposed as a way to compare the specific sensitivity of a particular object to the variations with other reference testing setups, like mock-panels with an extreme permeability asymmetry on the faces, as in a previous DISTAF experience [5]. In the two different graphs the geometrical parameter shown, according to the following relation, is the *base distance*, (in Fig. 2 indicated as  $l$ ) intended as the distance between the contact points of the two DK's vertical elements with the panel. This parameter has been arbitrarily chosen and in its place other geometrical parameters could be chosen, like the cupping angle ( $\beta$ ) or the deflection ( $f$ ).

The mathematical expression of this approach is described in the following two formulas; the quasi-instantaneous reactivity of the panel is expressed by equation (1):

$$100 \cdot \frac{mm_{t_x} - mm_{t_x - \Delta t}}{mm_{t_0}} \Delta t \quad (1)$$

and the quasi-instantaneous variation of the PEMC is expressed by equation (2):

$$\frac{PEMC_{t_x} - PEMC_{t_x - \Delta t}}{\Delta t} \quad (2)$$

for  $\Delta t = 6$  hours.

The 6 hours time average window range has been chosen as a reasonable compromise between the need of smoothening both the signals (deformation and PEMC) and the need to correlate the response delay of wooden objects to “readable” PEMC variations that were adopted previously [4], but this can be changed according to specific needs.

This visualization describes in a synthetic way the reactivity of the support.

It is important to underline that the wide variation from the linear interpolating regression in the first graph is due to the presence of the humidity gradients in the thickness of the plank that influence very much the time of equilibration of the whole board. Considering the random variation of the microclimate, the board will never describe a closed path because, in front of a new variation, the distribution of moisture in the thickness will induce a different path from the previous one.

As already described in some past studies [5], it is also important to consider the presence of the *flying wood* effect in the resulting induced deformation; this phenomenon is caused by the physical moisture exchange asymmetry, that has a great importance in panel paintings because of the different permeability of the painted layer and of the back surface, usually untreated.

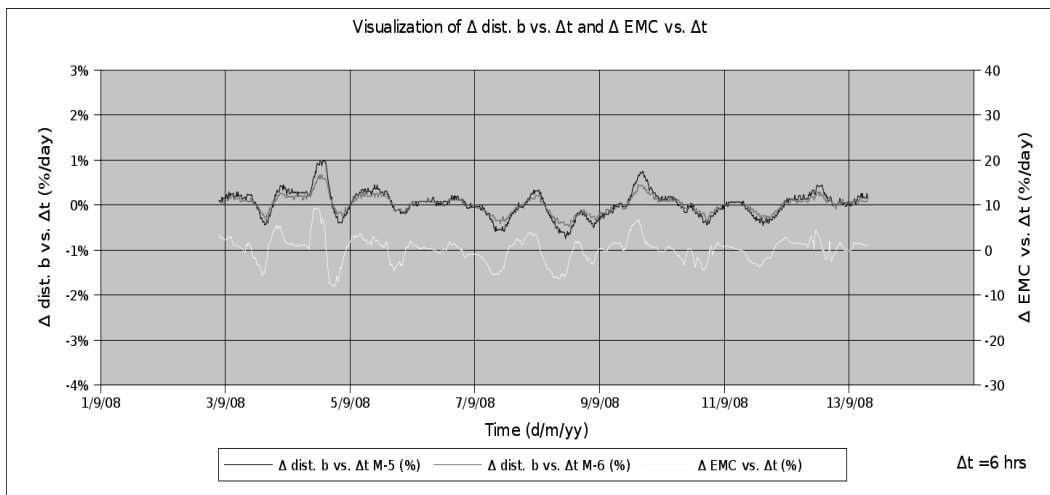


Fig. 6: the comparison between the different reactivities of the two systems applied on *Maestro Alberto* painting compared with the quasi-instantaneous variation of the PEMC.

### 3. Conclusions

The results obtained during this STSM can be collected in two groups: specific and general results.

#### Specific results:

- a quantitative description of the reactivity of the painting to microclimatic variations;
- the analysis of the eventual different deformation of different parts of the painting.

#### General results:

- the widening of the case studies database, with the aim of considering WCHOs singular entities, to be analyzed with general procedures taking care of their specific reactivity.
- a previously DISTAF implemented visual diagnosis form, to be used in the first phase of analysis, in order to have a standard technological description of the object, was used. The form is at a draft stage, but it has to be considered as a tool of the same importance of the measurement apparatuses.

The experimental results deriving from the analysis of a short period of data show quite clearly the following points of interest:

- the sensitivity of the object to the variations is quite evident, but there are no elements, at this analysis stage, that show if they can be considered harmful for the object;
- the reaction of the two systems placed in different parts of the plank is coherent with the general behaviour but different in the amount. This difference, to be investigated more deeply, can induce on the painted surface non homogeneous local compression and tension stresses during the microclimatic variations.

### Acknowledgments

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# PHYSICAL AND MECHANICAL CHARACTERIZATION OF ANCIENT WOODEN MUSICAL INSTRUMENTS FOR CONSERVATION PURPOSES

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## Abstract

The conservation of ancient wooden musical instruments is a topic of interest both for wood technologists and conservators. In particular, the state of the wooden sound box is investigated. The climatic conditions of exhibition and performance rooms are monitored. Moreover, the state of tension when tuning is analyzed and both forces and deformations are measured. The results obtained constitute the first step to acquiring the know-how to further the mechanical behavior of instruments subjected to long term loading tests, such as creep and mechanosorption.

## 1. Introduction

Ancient musical instruments are objects of great value both as historical relics and as real musical instruments that have been played up to now. In order to preserve these precious objects, it is necessary to monitor and analyze the conditions of conservation that are largely dependent on the conservation of the sound box which, in stringed instruments, is typically made of wood.

The aim of this ongoing study is to understand the effects of the events that may occur during the daily life of musical instruments, which, due to their importance, are considered not only as musical instruments but also as components of cultural heritage.

Besides biological decay, wood ageing is determined by the effects of both moisture variation and mechanical stress, as well as the coupling effects determined by their interaction over time. A cyclic variation of environmental parameters (T, RH%) increases the creep of wood, and it has a direct effect on fracture, time dependent deformation and stress relaxation in wood.

In the wealth of studies on the characterization of the acoustic properties of wood, the effects of material elasticity and viscosity on sound speed transmission (or on damping) of wood have been clearly established. Few studies have focused on the measurements of the load applied on the soundboards as effects of the tensioning of the strings.

All of this research is an important source of knowledge, but it does not enable drawing any conclusions for establishing the real effects of using the instruments on their conservation.

The main issues that this study aims to address are the following:

- mechanosorption: does it actually and significantly occur as a consequence of performance (theoretically load is applied and the moisture content of the wood changes during concerts)?;
- load condition: as consequences of load application two opposite phenomena are theoretically possible: wood relaxation and string creep;
- moisture variation: the actual amount and level of moisture variation inside the wood of instruments during their ordinary life (including performances).

In particular this paper is centered on the analysis of:

- climatic conditions of the exhibition and concert rooms;
- tuning forces;
- elastic deformation due to tuning.

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## 2. Materials and methods

Measurements have been performed on several instruments, but the main source of data comes from a conservation study on Paganini's violin "Cannone" by G. Guarneri del Gesù, which is now property of the city of Genoa in Italy (fig. 1).

### 2.1. Monitoring temperature and relative humidity of the air

The first step to guarantee optimal conservation of the instrument is to monitor the climatic conditions to which the violin is subjected, both during the exhibition and the performance.

The violin is conserved inside a glass case (fig. 1) in which the temperature and the relative humidity of the air are monitored by two probes (Rotronic, Hygroclip): the first one is set on top of the case and the other one is on the bottom, in order to measure a possible vertical gradient.

A special device was designed to measure the relative humidity of the air and the temperature of the violin during the performance. A miniaturized probe (Sensirion SHT75) is set in the hole that fits the end button, too. An electronic board (tmote sky, Moteiv), set between the top and the chin rest, sends the collected data to a laptop by radio wave system.



Fig. 1: the violin, mounted on the frame in the exhibition case.

### 2.2. Monitoring the weight variation

Climatic variations, particularly RH variation, inside the case are responsible for the violin weight variation. In order to monitor it, the instrument is hung on a frame which is placed on the scale (*ohaus, AV2102C*) that is connected to a PC in order to continuously collect the weight data. The data are saved in a txt file according to an acquisition rate of one reading every five minutes.

### 2.3. Measuring tuning forces

During the tuning, the strings produce a force that depends on the frequency, the length and the unit mass of the string itself. In order to measure force, a specific load cell was designed at DISTAF in cooperation with Deltatech. It is an exact copy of the bridge mounted on the "Cannone". It is made of iron and is equipped with extensometers (fig. 2). This measuring bridge is able to measure the force produced by the strings according to two perpendicular axes, named Z and X. Along the X axis we measure the transversal force, that is the force that develops along the perpendicular directions to the main axes of the violin itself, and along the Z axes we measure the force orthogonal to the top. The

accuracy is 1mV that is the equivalent of 0,018N. The data are collected by an electronic board, NI USB621, and sent to the PC to guarantee continuous acquisition.



Fig. 2: the equipped bridge mounted on the violin.

#### 2.4. Measuring elastic deformation

The force produced during tuning induces a deformation of the violin. The immediate deformation, that is the elastic deformation, is measured by an optical 3D digitizing system. The measure is based on the interferometric principle, that is the 3D scanning of the instrument due to the intersection of two different light beams. It enables generating a perfect reproduction of the object and the measure of its deformation according to an accuracy of 20  $\mu\text{m}$ . The scanning is made by ATOS III scanner and Geomagic Studio carries out the image elaboration, in cooperation with DigiLab (fig. 3).



Fig. 3: the 3D scanning of the violin.

### 3. Results and discussion

#### 3.1. Monitoring climatic conditions and weight variations

Inside the conservation case the climatic conditions can be considered stable during the whole of 2007. The temperature varied from 21°C to 25°C during the whole year. The RH% usually moved about 57.5% for the whole year, except for the autumn when the RH decreased to 51%. As a consequence,



the weight variations are small, approximately 0,5 g that is 0,1% of the violin weight. An interesting thing happened after concerts. Usually the performance room is drier than the exhibition case, that is why the violin loses weight, but, after being in the case again, it continued to lose weight notwithstanding the increased relative humidity of the air. It has been assumed that the influence of the climate inside the case was great. That is why it is necessary to measure the climatic conditions inside the sound chamber (the empty space between the two boards) during performances (fig.4).

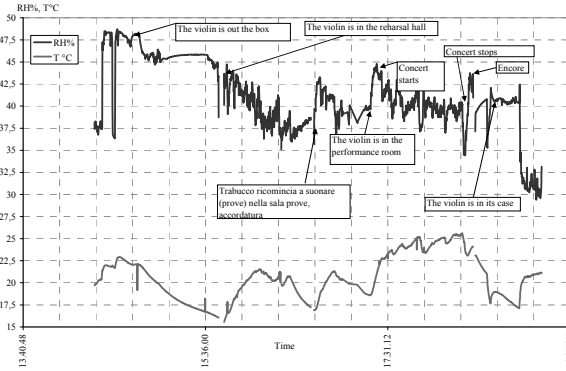


Fig. 4: climatic conditions inside the violin's sound chamber during a performance.

The analysis of weight variation during a performance is shown in Fig. 5; the violin loses about 1 g during the concert itself because of the different climatic conditions (fig 4). After this extraordinary event, the required time to regain the original weight, which is considered the equilibrium weight, is about one month notwithstanding that the climatic conditions in the conservation case are constant (fig 5).

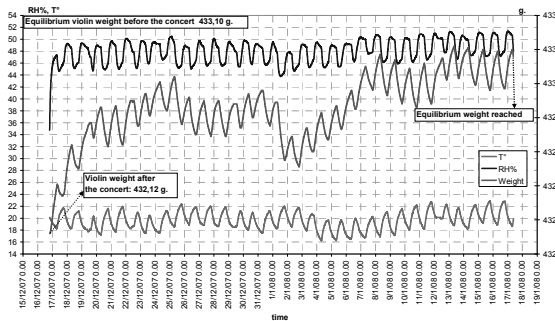


Fig. 5: time to regain the equilibrium weight after the concert.

### 3.2. Measuring tuning forces

The equipped bridge gives useful data on the forces produced during tuning (fig 6).

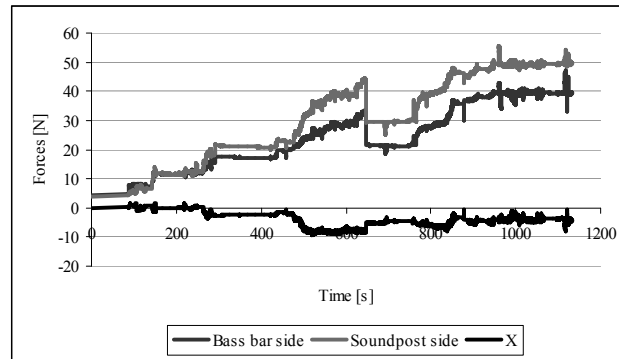


Fig. 6: forces shown during the tuning.

Looking at the Z axes we can see that on the soundpost side the forces, amounting to 50 N, are higher than the bass bar side (40 N). This is due to the different longitudinal tension necessary to tune the strings: the MI string is on the soundpost side and it is the one that needs greater force to be tuned. The gap between the two sides is 10 N, that is equivalent to 20% of the maximum force.

Analyzing the forces parallel to the top of the violin (X axes), we observe low values, around 4 N, when the violin is tuned. It means that the tuning stress according to that direction is negligible compared to the orthogonal forces (4,5% of the orthogonal forces).

### 3.3. Measuring elastic deformation

The results of the 3D scanning of the tuned violin (fig. 6 for the forces shown during measurement) show the deformation of the sound box, c cupid arch, and the high movement of the neck. Tuning produces a tension of the strings that is balanced by a deformation of the violin, that in simple terms consists of a compression of the top and a tension of the back. The neck is pulled up 1,335 mm by the action of the strings (the red zones in fig. 7a), and at the same time the fingerboard is pushed down 0,55 mm, which produces a rotation of the neck around its insertion point in the sound box.

The sound box is not deformed on the edges (the green zones in fig. 7a), but it shows a clear cupid arch deformation at the centre. In the bridge area the top is pushed down by the direct orthogonal forces expressed by the bridge itself (blue zone in fig. 7a). Near the end button and the fingerboard the top is obviously pulled up by the action of the tailpiece on one side, and the neck on the other, while the bridge acts as if it was a separating line.

The deformations are asymmetrical on the top, because of the presence of the soundpost and the bass bar on the opposite sides. Where the soundpost is present the deformation produced by the bridge stress is rather small (0,117 mm), because the soundpost itself produces a restraining force. As a consequence of this mechanism, the deformation of this side of the top, shown as convexities, is more evident far from the bridge (0,222 mm near the fingerboard and 0,257 mm near the tailpiece).

On the opposite side, the bass bar, set along the top, does not contrast the bridge stress (the deformation amounts to 0,457mm), but it makes the violin stiffer far from the bridge (0,181 mm near the fingerboard and 0,029 mm near the tailpiece).

Therefore the deformations are greater in the bridge area and smaller far from it.

On the back (fig. 7b), the neck and the end button area are pushed up by the strings (blue and light blue zones, fig. 7b). The sound box shows no deformation, or very small deformations (green zones, fig. 7b) limited between +0,05 and -0,05 mm, except for the soundpost area (yellow and orange areas, fig. 7b) and the deformation amounts to 0,13 mm. In fact, the soundpost produces a localized transversal compression on the back. Whereas on the other side the presence of the bass bar, which is not attached to the back, does not transmit deformations to it.

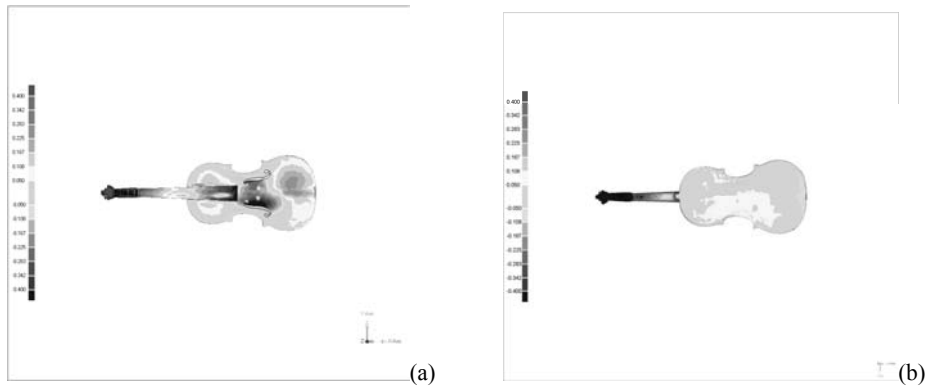


Fig. 7: elastic deformation on the top (a) and the back (b) of the violin tuned up with a string set mounted on during the performances.

#### 4. Conclusions

The work described herein is part of a more complex study on improving the conservation conditions of wooden musical instruments, particularly violins. Results obtained thus far relate both to the climatic conditions and mechanical stresses to which the violins are subjected during exhibition and performances.

##### 4.1. Variation in climatic conditions related to weight variations

The climatic conditions inside the exhibition case are stable and the weight variation of the violin is limited.

The main variations, both in relation to environmental conditions and weight, relate to the performance.

Moving the instrument to a usually drier room for concerts, results in loss of weight (1g), which needs about a month to be regained. This weight variation suggests that the violin is not perfectly insulated because of the imperfect waterproofing of the varnish and the contribution of the inside of the sound box, which is raw.

##### 4.2. Mechanical behaviour

The study of the elastic deformation shows the deformative behaviour of the violin which can be generalized as follows. The violin can be divided in two parts: the neck and the sound box. The neck is subjected to a rotation around its insertion point on the sound box. The sound box is subjected to compression of the top and to tension of the back, in simple terms. The marginal areas are not deformed, while the central part shows a cupid arch deformation.

This knowledge of the elastic deformation of the violin represents the first step towards furthering the mechanical behaviour of the instrument subjected to long term loading such as creep and mechanosorption.

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# NIR ARCHAEOMETRY AS A POWERFUL TOOL FOR INVESTIGATING THE ARCHAEOLOGICAL WOOD - INVESTIGATION OF THERMAL DEGRADATION MECHANISM OF SOFTWOOD AND HARDWOOD-

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## Abstract

Degradation mechanism of wood in terms of the variation of strength was analyzed, where the sample was thermally treated in steam atmosphere. Near infrared (NIR) spectra and compressive Young's modulus of the artificially degraded wood as an analogue of archaeological object were systematically measured. Chemometric analysis was employed to predict compressive Young's modulus. It was suggested that the variation of compressive Young's modulus with hydrothermal treatment was governed by two main processes, the depolymerization of polysaccharide and the variation of the cellulose crystallinity [1].

## 1. Introduction

As the thermal treatment is one of "degradation" processes, this technique is sometimes applied in archaeology, when artificially degraded wood is utilized for repair of historical wooden artifact. Nondestructive evaluation of hydrothermally treated wood is also important from the view point of wood archaeology [2]. Near infrared (NIR) spectroscopy has shown significant potential for obtaining physical, chemical and mechanical information from wood while retaining its anatomical, or biological structure [3]. Comparative studies on modern and archaeological wood using NIR technique demonstrated that the ageing degradation was mainly dominated by decrease of hydrogen bonding in the amorphous region of polysaccharides in wood [4].

In this study, degradation mechanism of wood due to the variation of strength was analyzed in conjunction with NIR spectroscopy and chemometrics, where the sample was thermally treated in steam atmosphere. NIR spectra and compressive Young's moduli of the artificially degraded hinoki cypresses and beech as analogues of archaeological objects were systematically measured. Partial least square (PLS) regression analysis was employed to predict compressive Young's modulus using NIR spectra as independent variables. PLS analysis was applied to interpret the mechanical change of wood sample taking into consideration its molecular structure and interaction between wood properties and mechanical properties..

## 2. Experimental

We prepared the hinoki cypress (*Chamaecyparis obtusa*) and beech (*Fagus crenata*) which had almost the same oven-dried density of 0.45 g/cm<sup>3</sup> and 0.65 g/cm<sup>3</sup>, respectively. The sample dimensions were 20×20×50 mm in tangential, radial and longitudinal directions. Stepwise hydrothermal treatment was performed at 140°C under steam atmosphere condition to make artificially degraded wood as an analogue of archaeological objects. The hydrothermal treatment times ( $t_{ht}$ ) were set up to 100 hours for hinoki cypress and 50 hours for beech, respectively.

NIR diffuse-reflectance spectra were obtained from tangential face of each sample using a FT-NIR spectrophotometer (Bruker MATRIX-F). 128 scans were co-added at a spectral resolution of 8 cm<sup>-1</sup> over the wavenumber range 10000-4000 cm<sup>-1</sup>. After the NIR measurements the static compressive tests parallel to the grain were performed on the basis of the Japan Industrial Standards Z 2101.

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### 3. Chemometrics

The spectral, chemical and mechanical data were imported into the Unscrambler (v. 9.6; CAMO software AS.) for the data analysis. Calibrations were developed for compressive Young's modulus by partial least squares (PLS) regression analysis.<sup>5</sup>

Two calibration models were constructed, labeled as Model-I and Model-II, respectively. In the case of Model-I, NIR spectra of air-dried hinoki wood were used for the prediction of compressive Young's modulus of hinoki wood. In the case of Model-II, NIR spectra of air-dried beech wood were employed as independent variables for the prediction of compressive Young's modulus of beech wood. Methods used for preliminary examination of the data included multiplicative scatter correction and the Savitzky-Golay smoothing of 21 points. The optimum number of PLS components was determined by full inner cross-validation method (leave on out). Calibration and validation were evaluated by correlation coefficient ( $R$ ) between measured and predicted values, and root mean square error of prediction ( $RMSEP$ ), respectively.

## 4. Results and discussion

### 4.1. Variation of NIR spectra with hydrothermal treatment time

Fig. 1 shows the variation of original and second derivative NIR spectra of oven-dried hinoki wood samples with  $t_{ht}$ . We can find clear decrease of absorption band due to the degradation of amorphous region in cellulose, while there is little difference in the absorption bands assigned to the crystalline regions in cellulose.

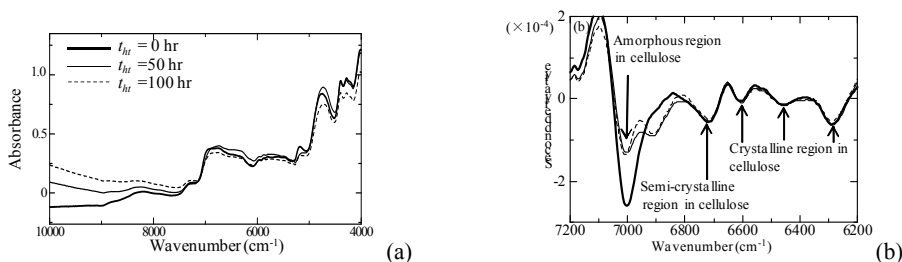


Fig. 1. Variation of NIR (a)original and (b) second derivative spectra of Hinoki wood after hydrothermal treatment.

### 4.2. Variation of compressive Young's modulus with hydrothermal treatment

Fig. 2 shows the variation of mechanical, physical and chemical properties of hinoki cypress with  $t_{ht}$ . Compressive Young's modulus of thermally treated hinoki (Fig. 2), continuously decreased as  $t_{ht}$  increased. On the other hand, compressive Young's modulus of beech showed characteristic variation with  $t_{ht}$ .

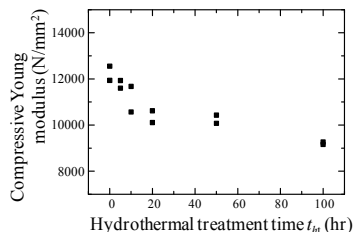


Fig. 2. The variation of compressive Young's modulus of hinoki cypress.

### 4.3. PLS analysis for compressive Young's modulus

Two PLS regression models (Model-I and Model-II) were developed for the determination of compressive Young's moduli of hydrothermally treated hinoki and beech wood. In the case of Model-I, NIR spectra of air-dried hinoki wood properties were used for the prediction of compressive Young's modulus of hinoki wood. In the case of Model-II, NIR spectra of air-dried beech wood were

employed as independent variables for the prediction of compressive Young’s modulus of beech wood.

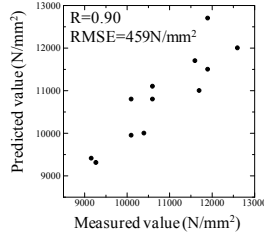


Fig. 3. Relationship between measured and predicted values from NIR spectra by PLS regression analysis for compressive Young’s modulus of thermally treated hinoki cypress.

Relationships between measured and predicted values for compressive Young’s modulus of Model-I is shown in Fig. 3 and statistical results are summarized in Table 1. In both cases of Model-I and Model-II, the prediction gave strong relationships between measured and predicted value with correlation coefficients of 0.90 and 0.91, respectively.

Table 1: PLS regression analysis for compressive Young’s modulus

Independent variables	Pre transform	Number of optimal factor	R	RMSEP(N/mm <sup>2</sup> )
Hinoki: NIR spectra (Model-I)	MSC, Smoothing	2	0.90	459
Beech: NIR spectra (Model-II)	MSC, Smoothing	6	0.91	996

Table 2: X-explain and Y-explain value for PLS component

Independent variables	X-Explain	Y-Explain
Hinoki: NIR spectra (Model-III)		
1st component	86	80
2nd component	11	11
Beech: NIR spectra (Model-II)		
1st component	98	30
2nd component	0	49

#### 4.4. Loading plots and score plots derived from PLS analysis

The spectral loading plots  $\mathbf{p}_1$  and  $\mathbf{p}_2$  derived from Model-I are shown in Fig. 4. The loading plots and the score plots give significant information about data set. PLS score of  $a$ -th component as latent variable is

$$\mathbf{t}_a = \mathbf{p}_a \mathbf{X} \tag{1}$$

where  $\mathbf{X}$  block is the variance in independent parameters.

PLS component score of  $\mathbf{t}_1$  and  $\mathbf{t}_2$  were calculated from the linear combination of absorbance at all measured wave numbers.  $\mathbf{t}_1$  might be regarded as an emphasized parameter of the degree for hydrothermal degradation (depolymerization of polysaccharide). In the case of  $\mathbf{p}_2$ , characteristic positive peaks could be observed around  $7000\text{cm}^{-1}$  due to amorphous region in cellulose and  $5200\text{cm}^{-1}$  due to water.  $\mathbf{p}_2$  around  $7000\text{-}5300\text{cm}^{-1}$  and  $5000\text{-}4300\text{cm}^{-1}$ , which are due to crystalline region in cellulose and lignin showed negative values. Therefore,  $\mathbf{t}_2$  might express the amorphous degradation, which is inversed parameter of the degree of crystallinity.

Fig. 5 depicts the score plots derived from PLS analysis for Model-I. Numbers in the figure indicates  $t_{ht}$ .  $t_1$  shows gradual decrease with  $t_{ht}$ . Decreasing of  $t_2$  explains the decreasing of amorphous region, or increasing of crystallinity.

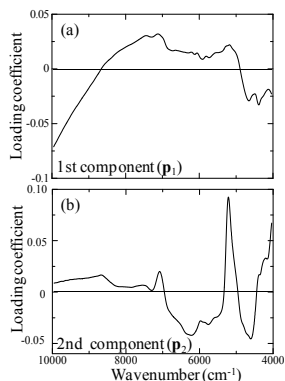


Fig. 4. Loading plots of PLS components of hinoki cypress: (a) 1st component (b) 2nd component

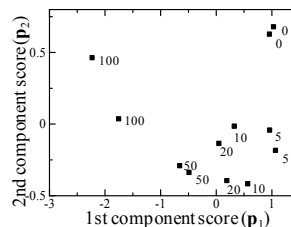


Fig. 5. Score plots derived from PLS analysis of hinoki cypress.

## 5. Conclusion

In this study, degradation mechanism of hydrothermally treated hinoki cypress and beech were investigated in conjunction with NIR spectroscopy and PLS regression analysis where the sample was regarded as an analogue of archaeological objects. Two PLS regression models were developed for the determination of compressive Young's modulus of hydrothermally treated wood. The prediction gave strong relationships between measured and predicted value both in Model-I and Model-II. It was suggested that the variation of compressive Young's modulus with hydrothermal treatment was governed by two main processes, that is, the depolymerization of polysaccharide and the variation of the cellulose crystallinity both in hinoki wood and beech wood.

It was concluded that the NIR spectroscopy with aid of chemometrics is useful to understand the degradation mechanism of archaeological hardwood taking into consideration its molecular structure and interaction between wood properties and mechanical properties.

## Acknowledgement

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# NIR ARCHAEOMETRY AS A POWERFUL TOOL FOR INVESTIGATING THE ARCHAEOLOGICAL WOOD - SPECTROSCOPIC OBSERVATION OF THE DEGRADATION PROCESS IN THERMALLY TREATED WOOD USING A DEUTERIUM EXCHANGE METHOD

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## Abstract

The NIR diffuse-reflectance spectroscopy was applied to monitor the diffusion process of deuterium-labeled molecules in hinoki wood, where the sample was thermally treated for increasing periods of time. The saturation accessibility varied characteristically with thermal treatment time reflecting the OH groups in different states of order in the wood substance. The variations of saturation accessibility with hydrothermal treatment time were applied to reveal the hydrothermal change of crystalline and amorphous regions in cellulose.

## 1. Introduction

Near-infrared (NIR) spectroscopy has shown its significant potential in analyzing changes due to ageing in mechanical, physical and chemical properties of wood based materials [1]. Tsuchikawa *et al* applied NIR spectroscopy for monitoring the diffusion process of deuterium-labeled molecules in archeological wood [2]. It was revealed that, for polymers with labile hydrogen atoms in OH functionalities, H/D isotope exchange (deuteration) in combination with FT-NIR spectroscopy is a powerful technique to determine the accessibility. In this study, degradation mechanism of wood due to the variation of accessibility was analyzed in conjunction with NIR spectroscopy and deuterium exchange, where the sample was thermally treated in steam atmosphere. The accessibility of the artificially degraded hinoki cypresses as an analogue of archaeological object was systematically measured.

## 2. Experimental

### 2.1. Sample preparation and hydrothermal treatment

We prepared hinoki cypress (*Chamaecyparis obtusa*) which had almost the same oven-dried density. The number of sample is 16. The sample dimensions were 20×20×2 mm in tangential, longitudinal and radial directions, respectively. Gradual hydrothermal treatment was performed at 140 °C under steam atmosphere condition to make artificially degraded wood as an analogue of archaeological objects. The hydrothermal treatment times ( $t_{ht}$ ) were set up to 150 hours (0, 2, 5, 10, 20 50, 100, 150 hours). Two wood samples were prepared at same thermal treatment time to check the reproducibility.

### 2.2. Deuterium exchange and NIR measurements

After the treatment, wood samples were immersed in 5 ml of the deuteration agent at 40 °C and NIR diffuse-reflectance spectra of oven-dried wood were recorded before and after 24 h immersion. NIR diffuse-reflectance spectra were obtained from tangential face of each sample using a FT-NIR spectrophotometer (Bruker MATRIX-F) with a fiber optic probe with a diameter of 7 mm and a measurement area of 49 mm<sup>2</sup>. This measurement area contains three or four year tree rings, where averaged spectral information of early-wood and late-wood were acquired. In order to improve the signal-to-noise ratio, 128 scans were coadded at a spectral resolution of 8 cm<sup>-1</sup> over the wavenumber range 10000-4000 cm<sup>-1</sup>. A good reproducibility was observed for the spectroscopic data for the investigated samples.

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### 3. Results and discussion

#### 3.1. Variation of NIR spectra with hydrothermal treatment time

Fig. 1 shows the variation of original and second derivative NIR spectra of oven-dried hinoki wood samples with  $t_{ht}$ . The absorption bands in the NIR region are conclusively associated with three polymers in wood; cellulose, hemicellulose and lignin. The absorbance between 10000-7500  $\text{cm}^{-1}$  increased with  $t_{ht}$ . This has originated from the remarkable darkening of wood by hydrothermal treatment. In the expanded view of second derivative spectra of hinoki wood sample (see Fig. 1-(b)), we can find clear decrease of absorption band due to the degradation of amorphous region in cellulose, while there is little difference in the absorption bands assigned to the semi-crystalline and crystalline regions in cellulose.

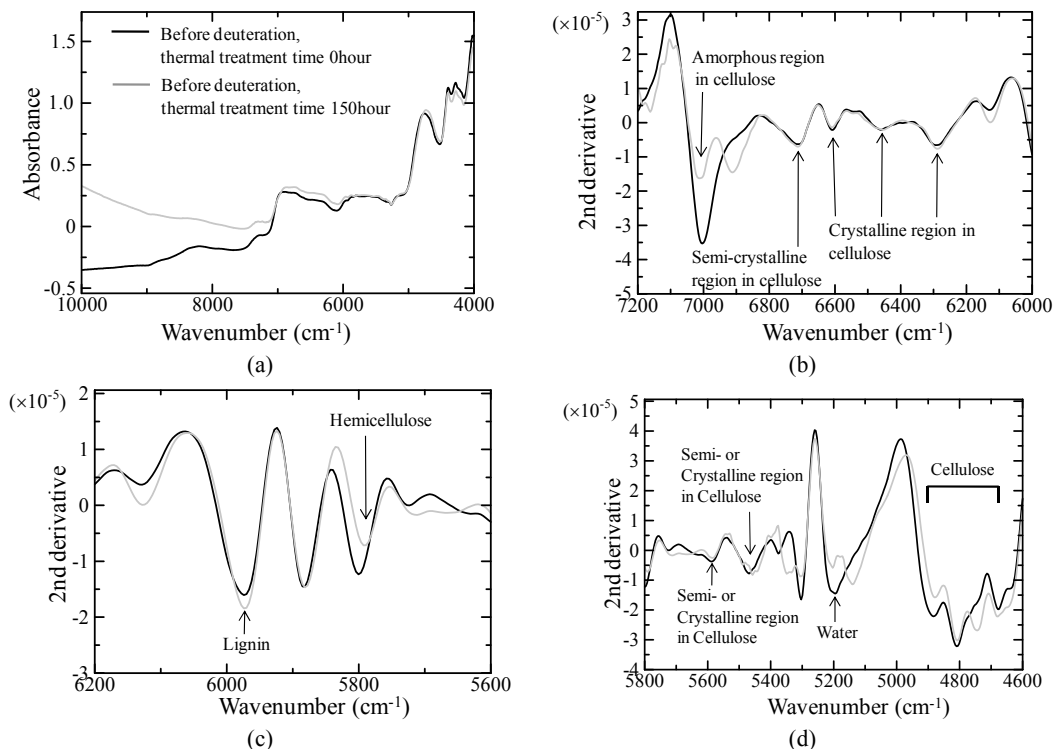


Fig. 1. The variation of (a) NIR original spectra and (b)-(d) second derivative spectra (7200-6000 $\text{cm}^{-1}$ ) (6200-5600 $\text{cm}^{-1}$ ), (5800-4600 $\text{cm}^{-1}$ ) with thermal treatment time.

#### 3.2. Variation of NIR spectra with deuterium exchange

The NIR diffuse reflected spectra (10000-4000 $\text{cm}^{-1}$ ) of the thermal hinoki cypress before and after deuteration with  $\text{D}_2\text{O}$  are shown in Fig. 2-(a). Significant reduction of intensity at 7200-6000  $\text{cm}^{-1}$  and 5000-4500  $\text{cm}^{-1}$  could be observed. These intensity reductions suggest that the hydrogen atoms of OH groups in wood substance were replaced by deuterium atoms of the diffusants. Except of slight frequency shifts, there are no remarkable variations in the other wavenumber regions. We focused in detail on the analysis of the wavenumber range 7200-6100  $\text{cm}^{-1}$ . The spectra are subjected to baseline correction in this range.  $A_m$ ,  $S_c$ ,  $C_I$ ,  $C_{II}$  and  $C_{III}$  were due to OH groups in the amorphous ( $A_m$ ), semi-crystalline ( $S_c$ ), and the intramolecularly hydrogen-bonded crystalline regions ( $C_I$ ,  $C_{II}$  and  $C_{III}$ ), respectively. The accessibility of the thermal treated wood samples in the deuteration process was calculated for each diffusion agent. The absorbance is proportional to the concentration of the absorbing material according to Beer's law. The accessibility of the diffusion agent can be calculated from the integrated absorbance of the OH-related absorption band as follows:

$$Z(\%) = \frac{A_{t=0}(\text{OH}) - A_t(\text{OH})}{A_{t=0}(\text{OH})} \times 100(\%) \quad (1)$$

where  $A_t$  (OH) and  $A_{t=0}$  (OH) are the integrated absorbances of the OH-related band measured at saturated diffusion condition and before the start of the deuteration, respectively. The isolated left wing  $-7000\text{ cm}^{-1}$ , and the integrated absorbance of  $\pm 20\text{ cm}^{-1}$  at  $6718$ ,  $6600$ ,  $6450$ , and  $6287\text{ cm}^{-1}$  were employed for the calculation of the accessibilities of the  $A_m$ ,  $S_c$ ,  $C_I$ ,  $C_{II}$  and  $C_{III}$  regions, respectively.

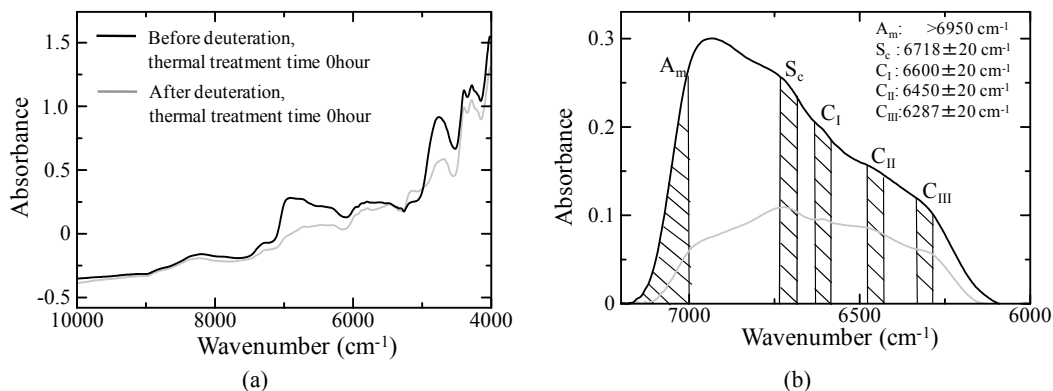


Fig. 2; The variation of (a) NIR original spectra and (b) baseline corrected NIR spectra ( $7200\text{-}6000\text{cm}^{-1}$ ) with deuterium exchange.

### 3.3. Variation of accessibility at OH-related absorption with thermal treatment time

Thermal changes of the saturation accessibilities in thermally-treated wood samples were observed. During the initial stage of hydrothermal treatment up to  $t_{ht} = 10\text{ hr}$ , the saturation accessibilities of  $A_m$  (i.e. the amorphous region) rapidly increased with increment of  $t_{ht}$ . The increases of the saturation accessibility suggest that the inter-microfibril space has expanded causing disintegration in wood cell wall, and that, consequently, the region itself disappears locally. In the case of  $S_c$  originating from the semi-crystalline regions, the saturation accessibility for  $\text{D}_2\text{O}$  also increased. Also in the cases of  $C_I$ ,  $C_{II}$  and  $C_{III}$ , the saturation accessibilities for  $\text{D}_2\text{O}$  increased. From the observation of variation of accessibility with the thermal treatment time, respectively, we could find the supermolecular difference of the fine structure in the microfibrils in the cell walls.

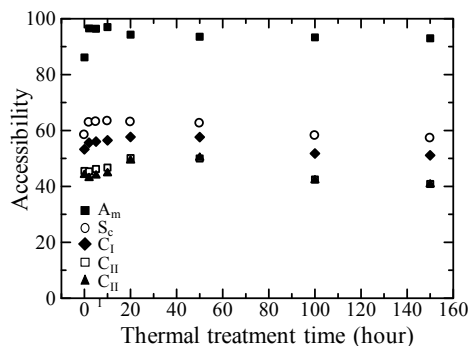


Fig. 3- The variation of accessibility of amorphous ( $A_m$ ), semi-crystalline ( $S_c$ ), crystalline regions ( $C_I$ ,  $C_{II}$ ,  $C_{III}$ ) in cellulose with thermal treatment time.

### Acknowledgement

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# NIR SPECTROSCOPIC MONITORING OF WATER ADSORPTION/DESORPTION PROCESS IN MODERN AND ARCHAEOLOGICAL WOOD

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## Abstract

We investigated the adsorption/desorption mechanism of water for modern and archaeological wood using near-infrared (NIR) spectroscopy. A mixture model of water was used to decompose the near-infrared difference spectra into three components (free water molecules ( $S_0$ ), those with one OH group engaged in hydrogen bonding ( $S_1$ ) and those with two OH groups engaged ( $S_2$ )). The variations of each water component with relative humidity could be explained by proposing a model that describes water absorption in three stages. It was concluded that the ageing phenomenon in wood leads to the decrease of adsorption sites on hemicellulose and amorphous cellulose [1]

## 1. Introduction

The physicochemical condition of the hydroxyl groups plays a key role in water adsorption/desorption process in wood. However, for lack of an effective analytical technique, the behavior of water in wood is not well understood at a molecular level. Near-infrared (NIR) spectroscopy is useful in a comprehensive material assessment as a means of nondestructive measurement for biological materials (e.g. forest and agricultural products, textiles and so on) [2]. NIR spectroscopic information on biological materials is mostly related to water, which has specific absorption bands at  $5200\text{cm}^{-1}$  (combination of stretching and deformation vibrations for OH) and  $6900\text{cm}^{-1}$  (first overtone of the OH stretching vibration) [3].

In this study, we report on the changes of water condition in modern and archaeological wood as inferred from NIR spectra. The objective was to investigate the water adsorption mechanism in wood using a structure model of water. NIR spectra were decomposed into three components of water. The change in water adsorption mechanism with ageing was examined by comparing the analytical results between modern and archaeological wood.

## 2. Experimental Section

We used modern and archaeological wood samples of Hinoki cypress (*Chamaecyparis obtusa*). The modern wood sample was taken from a living tree. The archaeological sample was collected from an upright pillar of a historical building in Japan of which construction date was estimated to be the early 7th century by the documentary analysis. Archaeological wood and modern wood plates were  $10\times 20\times 0.5$  mm and  $50\times 50\times 2$  mm in tangential, radial and longitudinal directions of the samples, respectively.

The wood plates were humidified gradually from an oven-dried to the fiber saturation state in a sealed desiccator, in which the internal temperature was maintained at  $20^\circ\text{C}$ . Subsequently, the plates were dehumidified to an oven-dried state.

Diffuse reflection NIR spectra were measured on a FT-NIR spectrophotometer (Bruker MATRIX-F) with a fiber optic probe. 128 scans were signal-averaged at a spectral resolution of  $8\text{ cm}^{-1}$ .

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### 3. Result and Discussion

#### 3.1. Decomposition of NIR spectra

Variation of the NIR spectra with water adsorption onto wood was analyzed basing on three structural forms of water molecules: free water molecules ( $S_0$ ), molecules with one OH group engaged in hydrogen bonding ( $S_1$ ) and molecules with two OH groups engaged in hydrogen bonding ( $S_2$ ) [4].

Curve fitting was undertaken to separate the NIR difference spectra using OPUS (ver. 4.0, Bruker Optik GmbH). Fig. 1 shows the difference and the decomposed spectra of water in the modern sample. An areal integral for each component,  $AI(S_n)$ , was calculated using Eq. (1), where  $S_n$  is one of the structural form of water molecule ( $n=0,1,2$ ), and  $DA(\nu)$  is the difference absorbance at a wavenumber  $\nu$ .

$$AI(S_n) = \int DA(\nu) d\nu \quad (1)$$

#### 3.2. Adsorption/desorption isotherm for the modern and archaeological wood

Fig. 2 shows the adsorption/desorption isotherms of the modern and archaeological wood samples. Both samples show hysteresis loops. The equilibrium moisture content of the archaeological samples is reduced compared to modern samples at each  $RH$  level. This is due to the decrease with ageing of hemicellulose of which OH groups form hydrogen bonding with ambient water molecules.

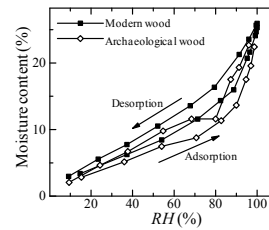
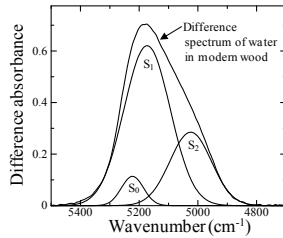


Fig. 1. Difference and decomposed spectra of water in the modern hinoki wood sample.

Fig. 2. Adsorption/desorption isotherm for the modern and archaeological hinoki wood samples.

#### 3.3. Spectroscopic interpretation of the mechanism of water adsorption by wood

Fig. 3 plots the variation of the areal integral ( $AI(S_n)$ ) (a) and the peak wavenumber ( $\nu_p(S_n)$ ) (b) for three water components in the NIR difference spectra of the modern sample versus  $RH$ . The archaeological sample shows the same tendencies. For the sake of simplicity, we assumed that an areal integral derived from the spectral decomposition is proportional to the amount of the adsorbed water molecules.  $S_1$  and  $S_2$  components show the hysteresis loop, which is not evident for the  $S_0$  component. The variation of  $AI(S_n)$  and  $\nu_p(S_n)$  can be explained by classifying the  $RH$  range into following three stages: Stage I ( $RH=0-40\%$ ), Stage II ( $RH=40-90\%$ ) and Stage III ( $RH=90-100\%$ ). Table 1 summarizes the spectroscopic characteristics.

In Stage I, the water molecules interact with wood substance more strongly than in the other stages since a monomolecular layer of water is formed. It is therefore suggested that most of monomolecular layer is composed of the  $S_2$  component. The wavenumbers,  $\nu_p(S_0)$  and  $\nu_p(S_1)$  showed almost the same value both in the adsorption and desorption processes at Stage I. This suggests that the water molecules adsorbed in wood substance consist predominantly of two of the structural forms, namely,  $S_2$  and  $S_0$  (or  $S_1$ ) components. The  $S_0$  component is very likely to exist when adjacent water molecules are sparse so that the  $S_0$  component increased with an increase of  $RH$  at this stage. On the other hand, the  $S_0$  component decreased gradually at the  $RH$  more than 40% possibly because of an expansion of the upper layers.

In Stage II, the water molecules interact with adjacent water molecules, because two or more layers (multilayers) are formed on wood surface. The areal integral,  $AI(S_0)$  decreased with an increase of  $RH$ . This might be due to the three-dimensional (inter-layer) expansion of water molecules and the increase of bonding force within water molecules. The shift of  $\nu_p(S_0)$  and  $\nu_p(S_2)$  to higher and lower wavenumbers, respectively, may also be caused by the inter-layer expansion of water molecules.

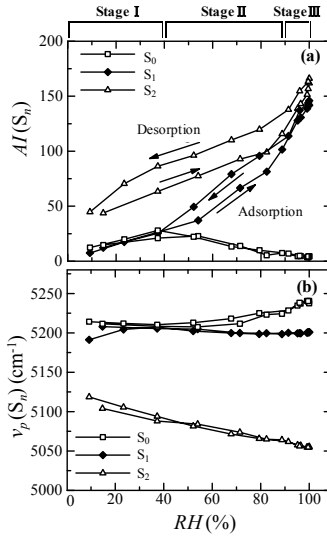


Fig. 3. Variations of the S<sub>0</sub>, S<sub>1</sub> and S<sub>2</sub> components with RH for the modern hinoki wood sample.

Bulk water (or capillary condensed water) is observed at Stage III, where the water molecules exist in not only adsorbed but also bulk states. The increase of the S<sub>1</sub> and S<sub>2</sub> components in this RH range is directly associated with the increase of bulk water.

Table 1: Adsorbed water condition in wood derived from NIR spectra.

		Areal integral	Water condition in wood
Stage I	0-40 %	AI(S <sub>0</sub> ) = AI(S <sub>1</sub> ) < AI(S <sub>2</sub> )	Monomolecular layer
Stage II	40-90 %	AI(S <sub>0</sub> ) < AI(S <sub>1</sub> ) < AI(S <sub>2</sub> )	Multimolecular layer + Minor bulk water
Stage III	90-100 %	AI(S <sub>0</sub> ) << AI(S <sub>1</sub> ) = AI(S <sub>2</sub> )	Multimolecular layer +Major bulk water

### 3.4. Changes of water adsorption mechanism by wood with ageing

The areal integral ratio of each water component to the total integral ( $PI(S_n)$ ) was calculated to compensate for the difference of absorbance due to the sample differences:

$$PI(S_n) = \frac{AI(S_n)}{(AI(S_0) + AI(S_1) + AI(S_2))} \times 100\% \quad (2)$$

Fig. 4 shows the variations of  $PI(S_n)$  and  $v_p(S_n)$  for the modern and archaeological hinoki wood samples with RH. In Stage III (almost close to the fiber saturation point),  $PI(S_n)$  and  $v_p(S_n)$  depend on the porous structure in wood, converging on the respective values for both the modern and archaeological samples. In the case of Stage I and Stage II, relatively large differences in  $PI(S_n)$  were found between the modern and the archaeological samples. It can therefore be concluded that modern wood adsorbs water molecules more strongly than archaeological (i.e. degraded with time) wood, since the S<sub>2</sub> component interacts more strongly with wood than the other components. It was suggested that the above-mentioned difference of water condition between the modern and archaeological wood occurs due to the decrease of adsorption sites in hemicellulose or amorphous cellulose.

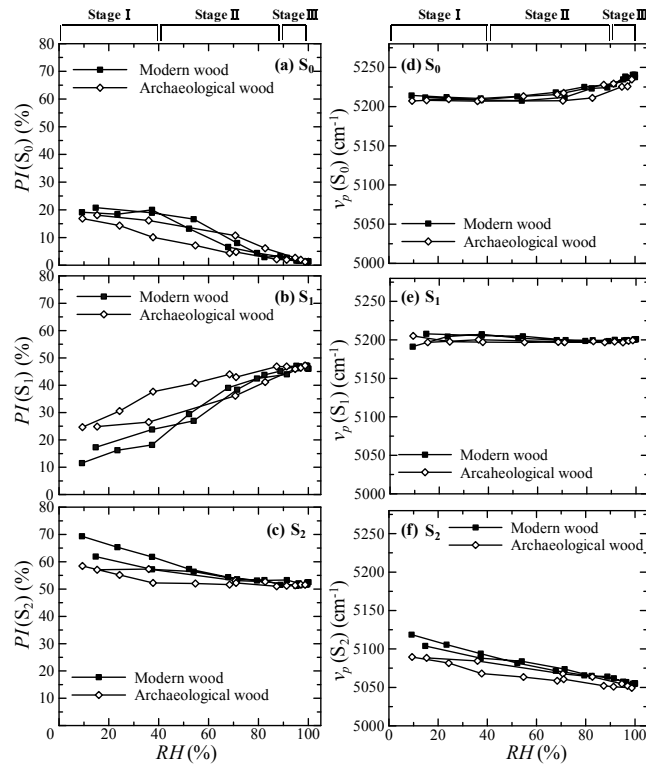


Fig. 4. Variations of the proportional areal integral  $PI(S_n)$  and the peak position  $v_p(S_n)$  of the three water components ( $S_0, S_1$  and  $S_2$ ) with  $RH$ .

#### 4. Conclusion

We investigated the adsorption/desorption mechanism of water in wood and its change with ageing in archaeological wood, using NIR spectroscopy. The mixture model of water was adapted to explain the wood-water interaction, where the difference spectra were decomposed into three components. The variation of the areal integral and the peak wavenumber for each component with relative humidity could be explained by proposing a three stage model for water adsorption.

#### Acknowledgement

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# NUMERICAL SIMULATION OF THE STRENGTH OF WOODEN STRUCTURES: APPLICATION TO HISTORIC PIANOFORTE

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## **Abstract**

Goal of the project to be presented is to introduce computer oriented simulation techniques and methods into structural investigations and restoration tasks of historic pianofortes. In order to achieve a realistic description of wooden structures at ultimate loadings, modelling of ductile behaviour under compression and brittle behaviour at tension or shear loading is required. The underlying nonlinear framework for the simulation of the local and global behaviour is based on the finite element method. The material formulations, which represent the state of the art in modelling of wooden structures, will be adapted to the requirements of historic pianofortes.

## **1. Motivation**

Historic instruments are part of our cultural heritage. The importance of old instruments is not only restricted to aesthetical aspects, in fact, these instruments provide an opportunity to experience, understand and interpret old music. Starting in the 1960s, a historically oriented movement has been developing that tries to perform old music composed in the period from 1550 to 1850 on historic instruments. Therefore, many musical instruments museums would like to investigate, conserve and restore old instruments, bring them into a state so that they can be played. However, the steps necessary for restoration may only be taken when the original substance can be preserved. Historic pianofortes are endangered, particularly because of the lack of knowledge in the fields of structural mechanics and construction. The project described below pays attention to this need of research.

## **2. Project**

The goal of the project “Static structural analysis of historic pianofortes”, which is supported by the Federal Foundation of Culture in Germany, is the restoration of the pianoforte constructed by Franz Jakob Späth and Christoph Friedrich Schmahl (Inv. MS-30, Fig. 1) shown at the Händel-Haus-Halle. The important aspects of restoration can be pointed out by looking at two other instruments of the museum, the pianofortes built by Johann Schmidt in 1786 (MS-28) and Conrad Graf in 1835 (MS-44). Both instruments are playable for the time being and will act as reference objects.

These two pianofortes are characterised by substantial distortions, cracks in components and connections caused by stringing (see Figs. 2 and 3). Therefore, an emphasis of the project is the basic understanding and analysis of these damages by investigating the static and structural behaviour of the instruments. The investigations will be carried out by using computer-aided simulations. The obtained results will serve as a basis for developing restoration measures for the pianoforte. Simultaneously, a research study to answer the following questions is being developed:

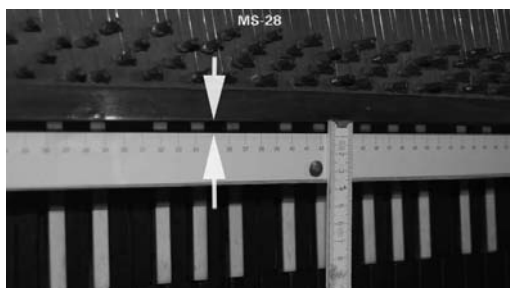
- assessment of the basic static construction with respect to stability in initial state,
- determination and assessment of the damage potential and sensitivity of certain construction details,
- consequences of existing damages for the behaviour of the construction and
- long-term behaviour of the construction with respect to climate fluctuations.

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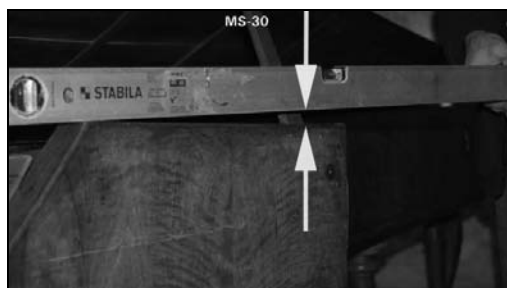
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Fig. 1. Historic pianofortes at the museum of Händel-Haus-Halle

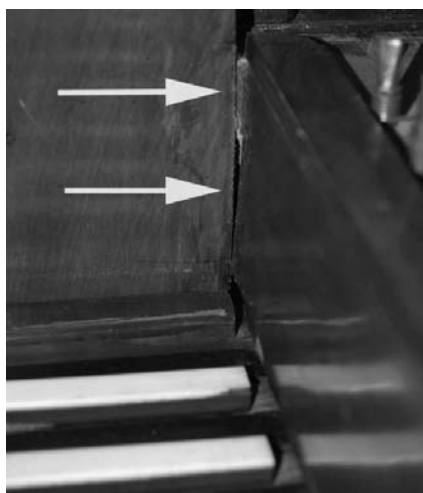


(a)

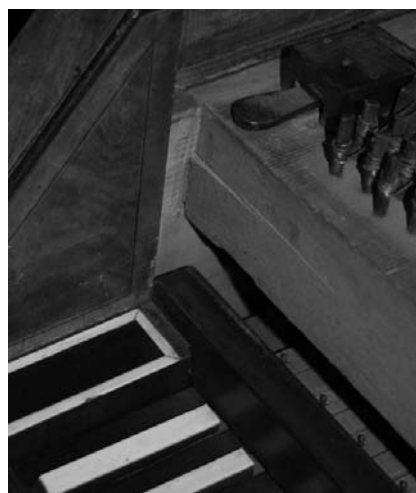


(b)

Fig. 2. Displacements of the pin block of the pianoforte built by Johann Schmidt (a) and deformations of the pianoforte corpus (Franz Jakob Späth and Christoph Friedrich Schmahl) (b)



(a)



(b)

Fig. 3. Cracks at the connection between pin block and corpus (a) and within the pin block (b)



### 3. Methods and models

For the comprehensive analysis of the construction of historic pianofortes, the determination of the strength properties of the component, the loading due to stringing as well as the distribution of forces in the structure are necessary regarding climate and humidity fluctuations plus known damages. Due to the geometrically complex structure and the nonlinear material behaviour, numerical methods of structural analysis are required.

One common approach of numerical simulation is the Finite-Element-Method (FEM) used in many fields in engineering and material science. The FEM is the main simulation-tool of modern structural analysis. Using the FEM, profound knowledge on the mechanical properties and predictions of the structural behaviour can be obtained without destroying the construction by experiments.

The basic idea of the FEM is the division of a complex not analytically calculable structure into discrete finite elements. The load-deformation-dependency of element-types like solids, plates and bars can be described easily basing on analytical relations, energy principles and suitable material models. By assembling the finite elements, the numerical calculation of stresses, strains and deformations of the whole structure are possible, considering boundary conditions (forces and constraints) and continuity conditions on the element border. In the case of a load-dependent force-displacement-relationship due to e.g. nonlinear material behaviour of creeping and cracking, an iterative-incremental solution algorithm (e.g. Newton-Raphson scheme) is carried out. All mathematical functions of the FEM are formulated consistently in vector and matrix notation.

With respect to the constitutive description of wooden material, models are introduced, which represent the moisture and temperature-depending anisotropic elastic material behaviour, ductile failure due to compression loading and brittle failure and cracking due to tension and shear loading. These models are already used for applications like simulation of the load-bearing behaviour of wood connections and components. An extension of the material formulations will be obtained by finding and modifying the sets of engineering input parameters for the wood species in construction of historic pianofortes. The material models are classified by the elastic, the ductile and the brittle formulation. A tensor notation is used for the following mathematical formulas to support more ready understanding in combination with a short syntax.

#### 3.1. Elasticity

The cylindrical anisotropic elastic behaviour of wood is based on the strain-energy-density function:

$$\Psi = \frac{1}{2} \underline{\underline{E}} : \underline{\underline{C}} : \underline{\underline{E}} \quad (1)$$

In this formulation, the elasticity tensor  $\underline{\underline{C}}$  includes the Young's moduli  $E_r$ ,  $E_t$ ,  $E_l$ , the shear moduli  $G_{rt}$ ,  $G_{tl}$ ,  $G_{rl}$  and the Poisson's ratios  $\nu_{rt}$ ,  $\nu_{tl}$  and  $\nu_{rl}$ , where the indices  $r$ ,  $t$  and  $l$  stand for the cylindrical material directions of wood (see Fig. 4). With the relation  $E_i/E_j = \nu_{ji}/\nu_{ij}$ , the stress tensor:

$$\underline{\underline{S}} = 2 \frac{\partial \Psi}{\partial \underline{\underline{E}}} = \underline{\underline{C}} : \underline{\underline{E}} \quad (2)$$

is determined.

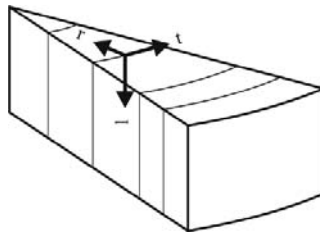


Fig. 4. Material directions of wood

### 3.2. Ductile Behaviour

Primarily at compression loading, wood is characterised by ductile behaviour and failure with irreversible deformations. To simulate this material behaviour, a three-dimensional multi-surface plasticity model is developed, which allows a coupled consideration of the elastic-plastic failure behaviour of wood as a result of compression, tension or shear loading. In addition, material direction dependent post-failure behaviour is taken into account by defining different softening and hardening relationships. The multi-surface-plasticity is validated using experimental data from [1].

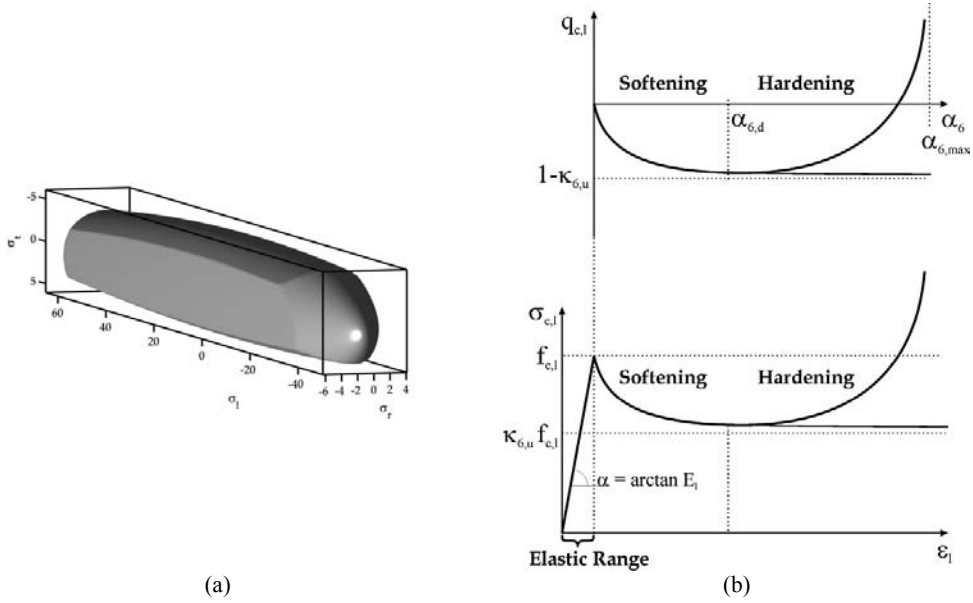


Fig. 5. Yield surfaces in r-, t- and l-direction for compression and tension (a) and damage function  $q$  and stress-strain-relationship (b)

The limit between the elastic and plastic zone is defined by the flow rule including yield conditions for each surface in form of:

$$f_m = \underline{a}_m : \underline{\sigma} + \underline{\sigma} : \underline{b}_m - q_m - 1 \leq 0 \quad (3)$$

where  $\underline{a}_m$  and  $\underline{b}_m$  are strength tensors and  $q_m$  is a function of softening and hardening. Seven possible failure modes need to be considered. Therefore, seven yield conditions  $m$  are defined. Fig. 5a shows the yield surfaces of Norway Spruce due to tension and compression in every material direction.

The damage function:

$$q_{m,u} = (1 - \kappa_{m,u}) \cdot \left( 1 - e^{-\eta_{m,u} \cdot \alpha_m} \right) - \zeta_{m,u} \frac{(\alpha_m - \alpha_{m,d})^2}{\alpha_{m,max} - \alpha_m} \quad (4)$$

allows the simulation of softening in the post-failure behaviour. In case of compression loading, an additional term defines hardening. Both are shown exemplary in Fig. 5b. A stress-strain function for compression is given in Fig. 5b, too. Further information about the multi-surface plasticity model with regard to material parameters, implementation and verification as well as applications is given in [2] and [3]. Fig. 6 shows deformations of the pin block at compression loading, which can be simulated using the multi-surface plasticity model.



Fig. 6. Plastic deformations in pin block.

### 3.3. Brittle Behaviour

Wood loaded in shear and tension perpendicular to fibres fails markedly brittle. To simulate this behaviour using FEM, a special element formulation as well as a suitable material model are needed.

In a 3-D structure, cracks will develop in a plane. This fracture surface can be suitably modelled using so-called cohesive elements in a FE-model. These interface-elements are defined by the nodes of two surfaces, which are congruent in the initial configuration (see Fig. 7a). Corresponding to the loading and the interface-material law, the two planes of the element can move e.g. tangentially in the directions 1 and 2. In this case, a shear loading yields a shear crack. Analogously, the planes shift apart in direction 3 due to a loading normal to the surface.

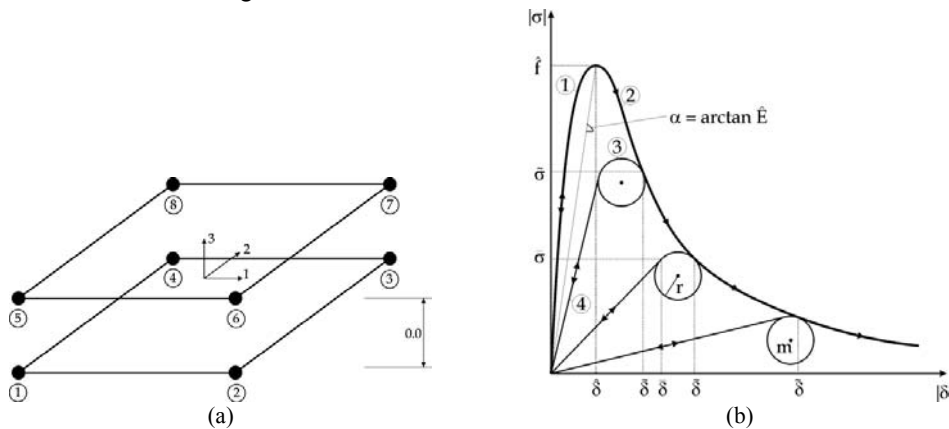


Fig. 7. Geometry of the interface element and definition of the local coordinate system (a) and stress-displacement relationship for the interface-material model with status 1 to 4 (b)

To simulate brittle failure of wood and aligned to the interface-element formulation, a coupled material-model is introduced. Basically, this model is characterised by a stress-deformation-relation. Using the input parameters, the stress-deformation function (see Fig. 7b) is composed of a sinusoidal-function in the elastic range (status 1) and the function, which describes the softening behaviour in the damage area (status 2). In case of unloading after softening or hardening, the path is defined by a spherical surface (status 3) and linear function to the starting point (status 4). The material formulation is continuously differentiable in the domain of definition due to the fact that all transitions are  $C_1$ -continuous. The material formulation takes into consideration the anisotropy and, therefore, the not coaxial orientation of the displacement and stress-vector. For further information about the material law and its numerical implementation see [3] and [4].

As an example, Fig. 9 shows a beam with an eccentric hole. The centric loading of the beam causes brittle failure. The FE-Model of Fig. 9 shows the expected crack propagation along the beam, starting at the cutout. Using the interface-elements in combination with the material model, the correct type of failure is predicted. Analogously to the example of Fig. 8, the introduced cohesive elements and material formulation can be used to simulate cracks in pianofortes as shown in Fig. 8.

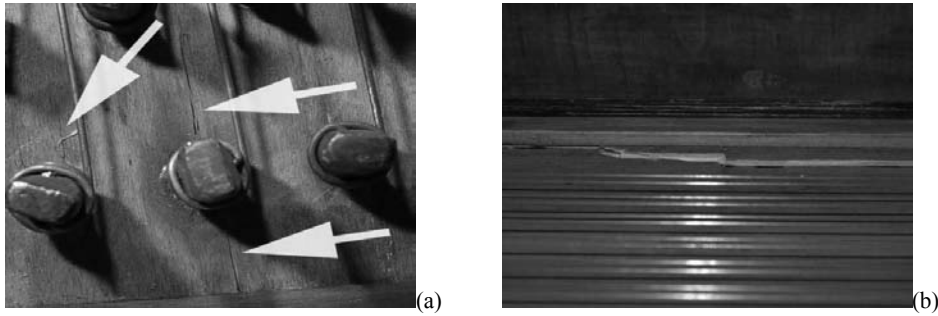


Fig. 8. Cracks in pin block (a) and at the border of the sounding board (b)

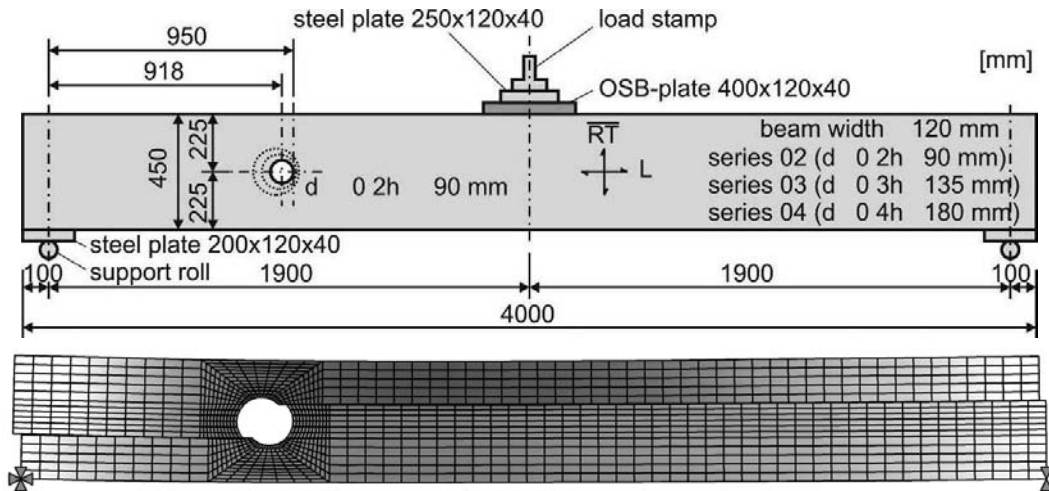


Fig. 9. Deformed system after completed crack propagation

#### 4. Outlook

Several different preliminary steps are required before numerical simulations of the historic pianofortes can be performed. Firstly, a digital mapping of the geometry of the construction is carried out. A 3-D volume model based on measured data will be constructed in a CAD-working environment. The digital geometry can be transferred to different FE-software platforms. Secondly, after identifying the wood species (e.g. Norway Spruce, Fir, Oak and European Beech) used for constructions, the determination of suitable material parameters is needed. Finally, climate measurements have to be analysed to assess the need for multi-physics formulations in numerical simulations.

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# SIMPLE ELECTRONIC SPECKLE PATTERN INTERFEROMETER (ESPI) FOR THE INVESTIGATION OF WOODEN ART OBJECTS

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## **Abstract**

The development of a simple electronic speckle pattern interferometer (ESPI) for the non-invasive, non-contact detection and characterization of early-stage damage of painted surfaces, like fracturing and layer separation, is reported. The ESPI system developed relies on the analysis of sound-induced vibration of the investigated surfaces. A simplified optical system as well as special algorithms for the analysis of interferograms were developed for the purpose of gaining precise information on the different indicators of the destruction process: the characteristic resonance frequency for the damaged area, its size and the spatial distribution of the vibration amplitude. The technique was applied to trace damage in specimens of painted wood subjected to damaging cycles of dimensional change.

## **1. Introduction**

The detection, characterization and tracing of the development of early-stage damage of painted surfaces using non-invasive (non-sampling, non-contact) techniques is a very important task for conservation science, as works of art are usually both fragile and extremely valuable. In particular, preventive conservation in museums and historic sites needs scientific tools capable of detecting cracks, delaminated areas, the fracturing of decorated surfaces at the micro-level well before damage becomes visible. They can serve as ‘early warning’ systems and help to establish an acceptable range of environmental conditions in the micro-environment of art objects on display, while in storage or during transportation.

The past ten years have seen much interest in the application of deformation/strain measurement techniques for the inspection and monitoring of works of art, covering point-strain measurements using resistance-strain gauges and fibre-optic sensors, as well as full-field optical measurement approaches such as holography, electronic speckle pattern interferometry, photoelastic stress analysis and photogrammetry. Dulieu-Barton et al. reviewed the relevance of each technique from a conservation perspective through accounts of usage on a wide range of artworks, including panel paintings, statues, wall-paintings and mosaics [1]. An earlier review by Ambrosini and Paleotti [2] critically discussed the developments in the use of holography and related techniques in the cultural heritage field since the first application of holographic interferometry to diagnose paintings in 1974 [3, 4].

Electronic speckle pattern interferometry (ESPI) has proven an especially attractive optical-based non-contact tool for the investigation of artworks made of diverse materials [5-9]. The technique is very accurate, being capable of mapping out-of-plane displacements to a fraction of a micrometer. It also has the capability to detect fractures, micro-cracks and surface flaws. However, despite its unique characteristics and established diagnosing capacity, ESPI has not yet been applied on a wider scale in day-to-day museum and conservation practice. There are several reasons that prevent the use of ESPI by conservation practitioners. Commercially available ESPI systems are expensive and require trained researchers to carry out the measurements and interpret the results. In particular, defect identification and the analysis of defect development is not simple or unambiguous, as it requires relating the registered deformation of the object’s surface to the change of its mechanical characteristics.

The principal aim of this study was to design, build and implement a simple and inexpensive electronic speckle pattern interferometer which could be used as a standard diagnostic tool in museums and field work, capable of making measurements at the micro-level scale. An optimisation of the apparatus, measuring protocols and analysis algorithms is proposed based on inexpensive and

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high-quality optical components available on the market and also the sufficient computing capacity of portable computers.

## 2. Features of the espi instrument

The diagram of the optical setup of the instrument is presented in Fig.1. A laser beam from a continuous-wave diode-pumped Nd:Yag laser (100 mW) is divided into two beams, called object and reference beams, using a glass plate. The object beam illuminates the investigated surface while the reference beam passes through the ground glass and is merged with the beam reflected from the investigated object using a beamsplitter cube. The object and reference beams superimpose coherently producing an interferogram recorded by a BCi4-6600 CCD camera. The interference of light is possible only when the difference between the optical paths of the object and reference beams does not exceed the coherent length of the light source. In the developed system, a laser light source with the coherence length of 30 meters (the distance at which interference of the laser light is possible) is used, which exceeds all dimensional scales involved in the measurement. The parameters of the CCD camera - 6.6 MPixels with 3.5 x 3.5 pixel pitch - together with the long coherence length of the laser also make possible measurements for large-scale objects like wall-paintings or other architectural surfaces. An important feature of the camera is its ability to increase the frame-recording rate with the simultaneous reduction of spatial resolution. This feature makes it possible to trace processes in small areas at a reduced resolution and high frame-recording rate, or trace long-term changes for large-scale objects at a high resolution and slow frame-recording rate. It should be stressed at this point that both the light source (laser) and the detector (CCD camera) are crucial for the measuring system to perform well, but the system can be made less expensive by the careful selection of their operational parameters.

The simplification of the design and operation of the system without compromising its sensitivity was achieved by simplifying the optical system, in particular by reducing the optics that form the reference beam. A ground glass was used in the reference beam path before the imaging optics [10]. As a result, the reference beam is a speckle beam but the performance of the system is unchanged from that with a smooth reference beam with the exception that the irradiance of the reference beam contains a position dependent phase. This fact

must be taken into consideration when choosing a proper algorithm for the analysis of the interferograms. The adopted solution has two advantages. Firstly, it avoids a complicated and expensive optical system for the reference beam. Secondly, the reference beam does not need to be realigned when the distance from the object, or size of the analyzed area, is changed, which is very convenient for an operator. Realignment of the instrument, while recording data, is very simple, as it is carried out by focusing imaging optics - using a commercial camera lens - and choosing the intensity of the reference beam by rotating one of the linear polarizers in the reference beam path.

Sound- or thermal-induced deformation of the object's surfaces can be recorded and analyzed to provide information about the preservation state of the surface as well as the development of damage. However, to obtain quantitative information about damaged areas, it is crucial to know the exact values of the dimensional response of damaged areas to stimulation. Since the interpretation of

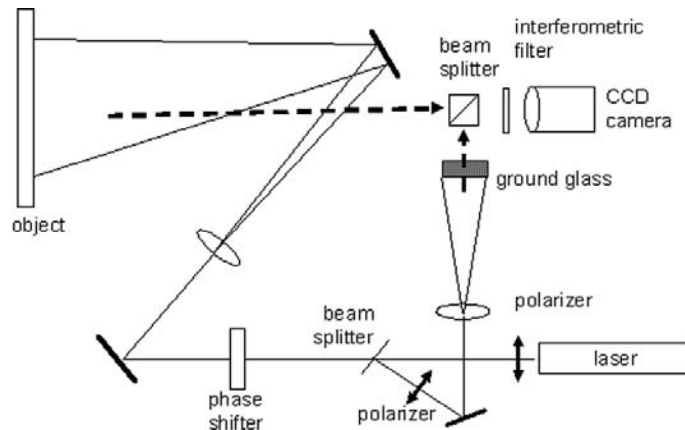


Fig. 1. Experimental setup for ESPI

recorded interferometric patterns is difficult and even ambiguous, a method for the determination of absolute values of phase at every point is needed. This is achieved by using a liquid crystal ARCOptix phase shifter with 10nm adjustment precision for recording several images with well-defined phase shifts between the object and reference beams. These images are used for phase unwrapping, and provide straightforward deformation data.

The last element of the optical system is a narrow band interferometric filter placed right before the CCD matrix. The filter is chosen to block all wavelengths different from  $532 \pm 1\text{nm}$  which is the output wavelength from the Nd:Yag laser used in the instrument. Therefore, any ambient light is blocked and the interferometer can be used for the investigation of objects illuminated by sunlight or artificial light.

The ESPI method proposed in this study has been based on the analysis of sound-induced vibration of the investigated surface. A signal with controlled frequency and amplitude is generated by a National Instrument NI PCI 6221 computer card, and further transformed by a Monacor Img Stage Line STA-302 amplifier. To generate a sound wave, a Monacor MPT – 177 loudspeaker with a frequency range from 3.5 to 20 kHz is utilized. The sound wave can induce vibrations of delaminated parts of a decorative layer when the generated wave is close enough to the resonant frequency. When the time of measurement is much longer than the vibration period, the intensity of light in the plane of the interferogram can be represented as a two dimensional fringe pattern [11] and provides information on the amplitude of the object vibration. However the information is still relative because the intensity of light as well as its initial random phase in plane of interferogram are unknown. One may assume that the intensity of light is constant during the time of the measurement so its value is irrelevant for the final result of the analysis, but the map of the initial phase values is a key piece of information which must be found for an unambiguous analysis of the deformation of the surface.

### 3. Data analysis

In general, there are two types of phase-unwrapping procedures: temporal and spatial [12]. In the presented system, the first type of procedure is applied. In order to simplify the calibration of the instrument and the measuring procedure, a version of the normalized max-min phase shift scanning routine proposed by Vikhagen [13] was utilized. The process of fringe identification in the obtained interferogram is semiautomatic. A preliminary identification of fringes is done manually but the procedure of the assignment of particular pixels on the image to consecutive minima and maxima of the Bessel function is automatic – based on an iterative algorithm.

An exemplary analysis of the damaged specimen of painted wood is presented in Fig. 2. Gesso was composed of rabbit-skin glue and ground chalk; the ratio of the inert solid to glue expressed as the pigment-volume concentration (PVC) was 92%. The PVC value was practically selected by a participating restorer as being that which has been commonly used in the restoration of panel paintings. Six coatings of gesso were laid and the thickness of dried gesso layer was approximately 1 mm. A crack and a loss in the

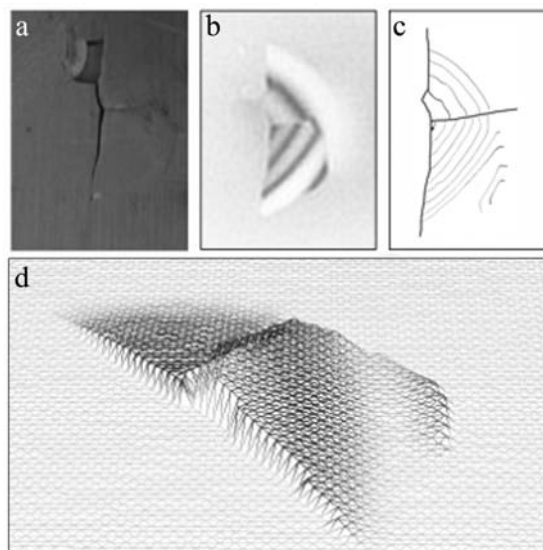


Fig. 2. Consecutive steps of the analysis of sound-induced vibration of the damaged gesso layer on wood. (a) photograph (2.5 x 4cm) of a crack in the gesso layer (b) interferogram after the phase-unwrapping procedure, (c) map of the delamination (isolines every 40nm) (d) 3D visualisation of the spatial distribution of the vibration amplitude for the delaminated areas.

decorative layer are visible in the upper part of the specimen (Part (a) of the figure). A series of measurements were performed at increasing frequency of the sound enforcing vibration of the surface, which enabled the detection of the delamination on the right side of the vertical crack. Furthermore, the measurements revealed the existence of two mechanically independent delaminated areas divided by another, horizontal crack visible on the photograph. For the best visualisation of the complexity of the damaged area, a sound frequency of 3400Hz was chosen to induce vibration of the surface. This frequency is intermediate between the resonant frequencies of the two delaminated areas, thus allowing the vibration of both areas to be simultaneously observed. The difference in response to sound vibration for both areas is clearly visible in Fig. 2b and a further analysis of the interferometric fringes gives precise information about the spatial distribution of the vibration amplitude for the investigated area. The results are presented as a 2D map of isolevels drawn every 40nm (Fig. 2c) and a 3D map (Fig. 2d).

#### 4. Monitoring the expansion of damage

An exemplary result is shown in Fig. 3. The measurement was performed on a wooden board coated with a gesso layer. In the central part of the specimen, a circular area of delaminated gesso about 1 cm in diameter was introduced. To record differences in the state of preservation of the object during the damage development process, two measurements were made. The first, the reference measurement, was made before the application of any external forces on the object while the second was performed after the object had been subjected to damaging cycles of stretching and compressing. Temperature and relative humidity in the laboratory were kept constant during the measurements to eliminate their influence on the surface shape. The amplitude of the stretching/compressing cycles was increased until some additional damage was detectable. An interferogram for the damaged specimen obtained by the thermally induced ESPI is shown in Fig. 3a: three microcracks are visible on the surface of the decorative layer. Sound-induced ESPI measurements conducted prior to and after damaging cycles and the respective interferograms reveal the vibrating delaminated areas, and are shown in Figs. 3b1 and b2 (the respective resonant frequencies were 11.5 and 11.8 kHz). The comparison of the two images allows the shape and size of the delaminated areas to be determined. A detailed calculation gives a 6% increase of the delaminated area as a result of the damaging process. The spatial distribution of vibration presented in Figs. 3c1 and 3c2 also differs for both cases, but the difference is not very pronounced. On the other hand, an easily detectable change appears in the resonant frequencies of the delaminated areas (Fig. 3d). The resonant frequency shift of about 300Hz reflects a change in the mechanical properties of the defect with a high sensitivity, and can serve as a quantitative measure of the development of the damage.

In conclusion, it can be stated that rough changes of size and shape of the delaminated area can be followed relatively easily by recording the vibrating areas after each consecutive damaging event (the analysis is fast and direct). Though the distribution of the vibration amplitude changes when the delaminated area increases, the parameter is difficult to measure since it depends on the relative position of the loudspeaker and the investigated surface. The resonant frequency of delamination is the most sensitive parameter determined by the developed system. Moreover, it is independent of the relative position of the camera, the loudspeaker and the object, or the amplitude of the sound.

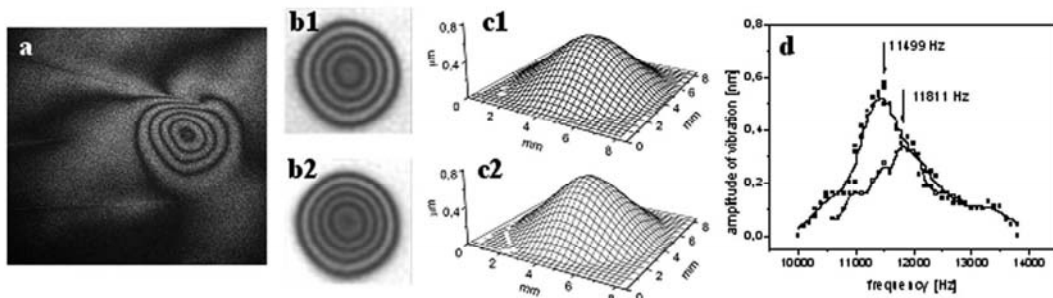


Fig. 3. Different indicators of the development of damage recorded using sound-induced ESPI.  
See text for explanation.



## 5. Conclusions

A simple ESPI system, capable of measuring sub-micrometer, out-of-plane surface deformations and thus able to detect early-stage development of damage in decorated surfaces of works of art, was developed. By an appropriate design of the instrument and selection of the measuring protocols and analysis algorithms, it was possible to significantly reduce the costs, to make the device simple without losing its sensitivity, and to meet the requirements of practical use in the cultural heritage field also by non-research personnel with backgrounds in conservation or heritage management. The results presented have demonstrated that the relatively simple system developed is adequate in terms of accuracy and repeatability for any practical requirements of the conservation field. The multilevel analysis of the data proved capable of providing information on a desired level of complexity and accuracy.

The speckle interferometer described is still a laboratory device. However, the results presented establish firm foundations for the development of a fully portable instrument which can be taken to museums and historic buildings and can operate on site.

## Acknowledgments

This research was supported by the Financial Mechanism of the European Economic Area, grant PL0086 ‘Establishing standards for allowable microclimatic variations for polychrome wood’. Further support from the Polish Ministry of Science and Higher Education, grant POL-POSTDOC II PBZ/MEiN/01/2006/10 is acknowledged.

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# STRUCTURAL DEFECT DETECTION USING ACOUSTIC HOLOGRAPHY IN THE CULTURAL HERITAGE FIELD – A PRELIMINARY STUDY

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## Abstract

We are now able to measure a dynamic property of the wood [1], the loss angle through the whole audio frequency range (20 Hz- 20 kHz), when the wood sample is excited with a continue vibration (simulation of the playing effect). But its disadvantage is that it's a destructive technique because we need a wood sample. So, in addition, to know better the dynamical behaviour of a whole wooden structure we use holographic acoustic.

After preliminary acoustical holography experiments realized on a 17<sup>th</sup> century harpsichord, a detailed study has been carried out to measure the wooden structure ageing using this technique. The main objective of this measurement is to control the stress on a soundboard before the rupture due to string tension, due to vibration when the musical instrument is played, or due to low hygrometric variations. Indeed, all these modifications in the structure will modify dynamical response of this structure. We show that this technique is able to detect a simulated structural defect in the soundboard, by measuring a shift for a few resonance frequencies.

## 1. Introduction

The Musée de la musique laboratory is carrying research to improve the knowledge of the mechanical phenomena related to conservation and restoration of the soundboards of musical instruments. Indeed, the soundboard is one of the most important elements in the sound production of stringed instruments.

Musical instruments preserved in the museum are cultural heritage objects, and not only their material integrity, but also their sound production function should be preserved, when possible. This goes with the conservation of the vibrational properties of the wood of the soundboard (Young's modulus, loss angle and resonance frequencies). Particularly, it is interesting to understand the mechanical effects due to string tension and/or to hygrometric variations on load structure as the soundboard. How is it possible to detect cracks, or loading variations on the soundboard through vibrational analysis without contact? Any structural modification of the vibrating element induces a variation in its acoustic response.

The playing conditions for a musical instrument induce quick hygrometric variations for the wood. In fact, the proximity of the musician generates an increase of temperature and humidity in the musical instrument's environment. Preliminary viscoelasticity measurements of the variation of the loss angle with hygrometry changes have shown that this property is very sensitive to water content in wood (Fig. 1) ([2]).

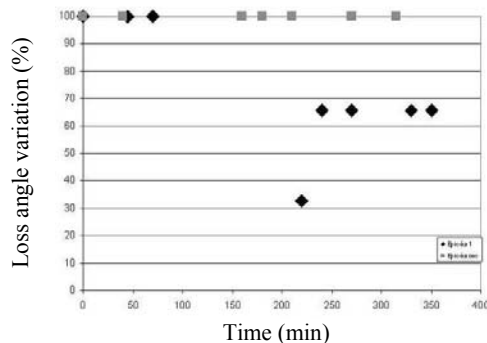


Fig. 1 Water content effect on the spruce loss angle at 300 Hz. The grey squares are for a dried sample, and the black ones for a sample conditioned at 12 %

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## 2. Humidity gradient effect on dynamic properties – simulation

A finite element model using Cast3m of a bi-clamped wooden beam is used to study the effect of an hygrometric gradient on its resonance frequencies. The hygrometric gradient is calculated according a Fick's law. The stress due to this gradient is modelled as a linear function that modify the mass and the elasticity of the material.

We have calculated the modal frequencies to evaluate the modification in the vibrational properties of the beam (Fig. 2).

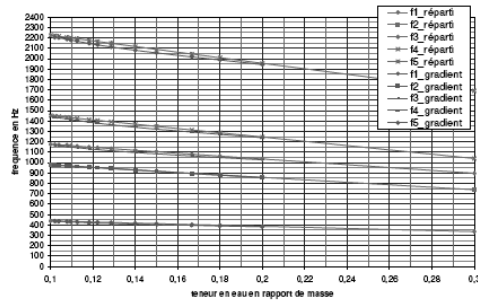


Fig. 2 Eigen frequencies variation with hygrometry (gradient or uniform repartition)

Considering the hygrometric stress as a structural defect on the vibrational properties of our objects, which contactless technique can we use to evaluate the structure damages?

## 3. Impulse Nearfield Acoustical Holography principle

The complete methodology will be published elsewhere in a near future. Here we briefly describe the set up and have just a look on the results.

### 3.1. Principle

The acoustical holography technique is here used in the aim of achieving a structural modal analysis. The Nearfield Acoustical Holography (NAH) process of planar harmonic pressure fields is exhaustively described in [3]

The impulse response of the vibrating source is measured in term of radiating acoustic field with a microphones array. The impulse response to a punctual impact excitation on the structure is measured with a microphones array, as radiated acoustic field.

The vibrational behavior of the source is then deduced, in terms of normal vibration velocity, with the help of an inverse calculation method based on spatial 2D Fourier transforms. Following the measurement stage, a classical frequency analysis provides a set  $Ph(\omega, x, y, z_n)$  of harmonic hologram pressure fields over the desired frequency band.

Contrary to more classical experimental modal analysis techniques (laser vibrometry, piezoelectric accelerometers) that measure directly the vibrational behavior of the structure, this technique implies the following approximations :

- the vibration source is supposed to be planar
- the vibration source is reconstructed on a virtual rectangle of the same size as the microphones array.
- The source distribution is supposed to be continuous
- Since the measurements are performed in near acoustic field, evanescent components are partially covered by noise and the information they contain is then filtered during NAH operation.

From the last two points imply a lack of precision in the reconstruction of the vibrational field at the vicinity of edges areas, where sharp discontinuities are present. However, these limitations were shown to be minor in a recent study ([4])

IPNAH exhibits however valuable advantages compared to classical methods, especially in the study of fragile structures like ancient musical instruments:

- except for the excitation process, it is a non contact method
- since an important number of grid points (120) can be measured at the same time, the number of excitation impacts on the structure is strongly limited.
- The measurement time is comparatively very short.

In the present study it took less than 120 mn for recording the set of 15360 point impulse responses to be processed.

### 3.2. Experimental set-up

The impulse response of the harpsichord soundboard is measured in the semi-anechoic chamber at the Musée de la Musique (Fig. 4 (a)). This experiment is described in [5]. In such a room noise level is seriously attenuated. This condition allows minimizing the soundboard excitation level and also optimizing the signal to noise ratio for evanescent waves.

Since the instrument has to be kept under tension, the strings are muffled in order to eliminate their sound production (Fig. 4(b)).

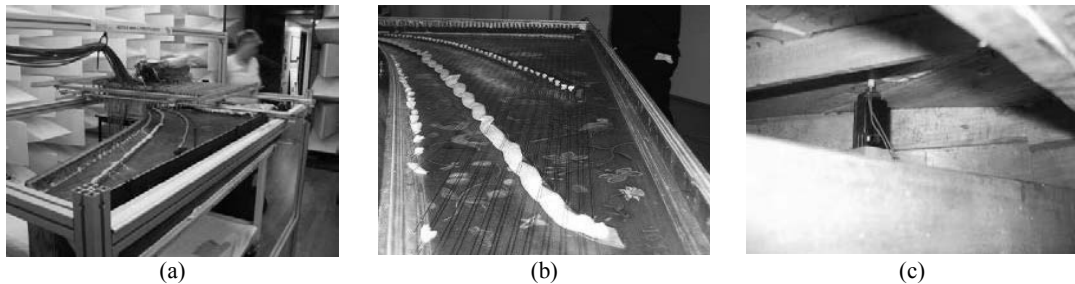


Fig. 3 NAH experimental set up: (a) holographic microphone array, (b) string muffling (c) impact hammer

The impulse response is calculated from the measured harmonic acoustic nearfield using a NAH reconstruction process. A point impulse excitation of the soundboard is provided by an automated hammer driven by an electromagnet that produces a reproducible impact (Fig. 4(c)). The excitation position is chosen on the underside of the soundboard. The keyboard is therefore removed. The position of the impact is chosen so as to mobilize significant flexural vibration modes of the soundboard.

A 12 by 10 electret microphones array, with a 50 mm step, has been used to collect the pressure field. So as to fit the measurement grid, the array is moved into 8 positions. For each of these positions the array is also moved according to 16 interleaved positions so as to refine the measurement step grid to 12.5 mm. The 120 impulse pressure responses for each position of the array are collected using a home-made 128 channels synchronous digital recorder. Each measurement associated to one impact on the soundboard has to be phase referenced by systematically recording, along with the acoustic signals, the constant impulse response of an accelerometer positioned on the soundboard. The resulting acoustic impulse response field is measured over a parallel plane at a distance  $z_h = 72$  mm. This unusually large distance for NAH has been imposed by technical reasons of accessibility. The field is finally sampled according to a 1162.5 by 1762.5 mm rectangle grid with a 12.5 mm step. The different sets of measurement finally count 13348 point acoustic impulse responses (Fig. 4).

### 3.3. Radiated sound pressure

On Fig. 5, we present the pressure level, averaged on the whole measurement plane, from 0 to 2000Hz. This graph shows a significant radiation level for frequencies between 140 Hz and 2000 Hz. An important modal density in the usable [0 2000Hz] bandwidth can be observed.

The frequency response depends on the geometry, the material, the limits condition, and on the environmental conditions during the experiments. This frequency spectrum is the response of the structure for a mechanical state: it is a sort of the identity signal of the harpsichord.

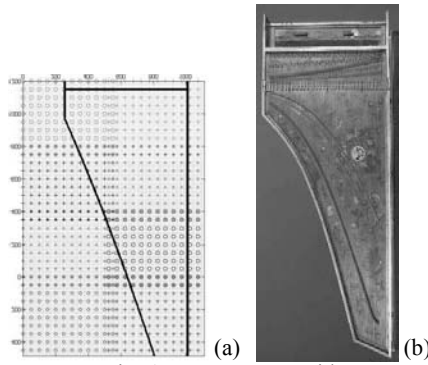


Fig. 4 Measurement grid

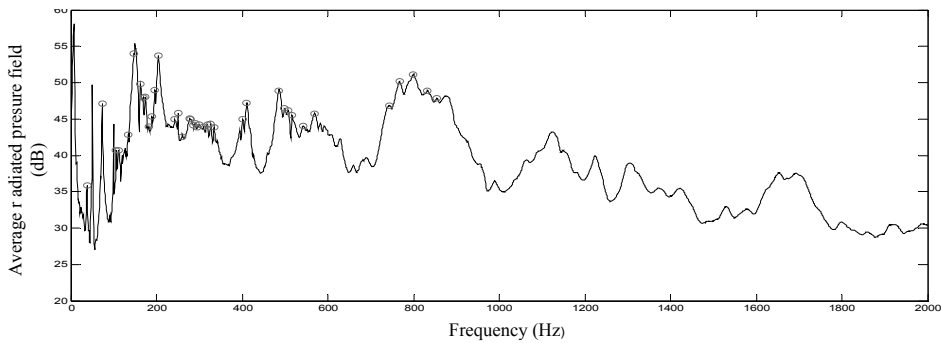


Fig. 5 Averaged pressure level from 0 to 2000 Hz with modal frequencies retained

It is difficult to compare this spectrum to the one obtained on another harpsichord because each musical instrument is unique and we do not know another harpsichord made by Couchet kept in the same state. Moreover, it is impossible to realize another modal analysis on this instrument because it will imply contact. Nevertheless it is possible to compare this spectrum with theoretical results obtained from a finite element model (comparison will be analyzed in our afoot article). From this spectrum, the mathematical process consists in selecting peak one by one to reconstruct the mode (resonance frequency and modal shape) of the structure. An example is given on the Fig. 6.

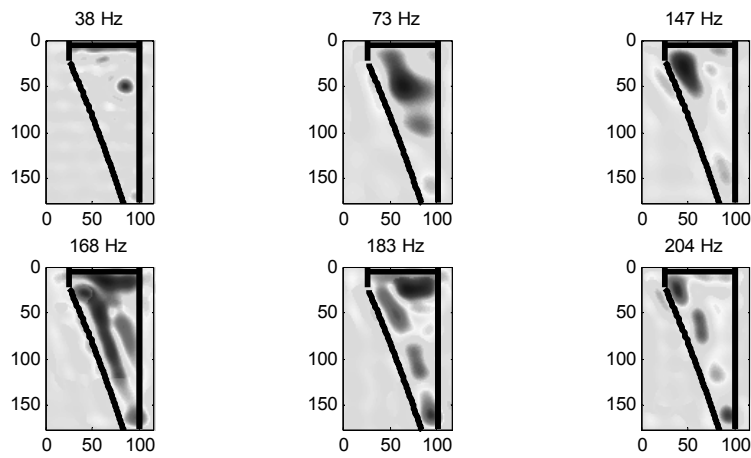


Fig. 6 First modal shape for the harpsichord Couchet

The modal decomposition for a structure is the resolution of this equation:  $[M]\ddot{x} + [K]x = 0$  where  $[M]$  and  $[K]$  are the mass and stiffness matrices, and  $x$  the displacement. For a given structure, any

modification due to a default or a load will appear in  $[K]$  or in  $[M]$  and the solution  $x$  to the problem will be different. For this reason, we have simulated a default in the harpsichord by adding a mass on the bridge. As a preliminary experiment to simulate a structural defect, a mass of 110g was cautiously deposited on one bridge (Fig. 7). The impact acoustic response of the harpsichord was measured in the same conditions (hygrometry, temperature and string tension) as before in the limited field near the keyboard (Fig. 7).



Fig. 7 Artificial added mass on the soundboard

Fig. 8 shows the frequency variations on the average level measured after the added mass. We are able to detect variations induced by the mass addition. Indeed, the second mode around 80 Hz is lower in the case of the mass addition than without. And we can see that a lot of resonance frequencies (marked on the spectrum from the Fig. 6) are shifted of a few Hertz.

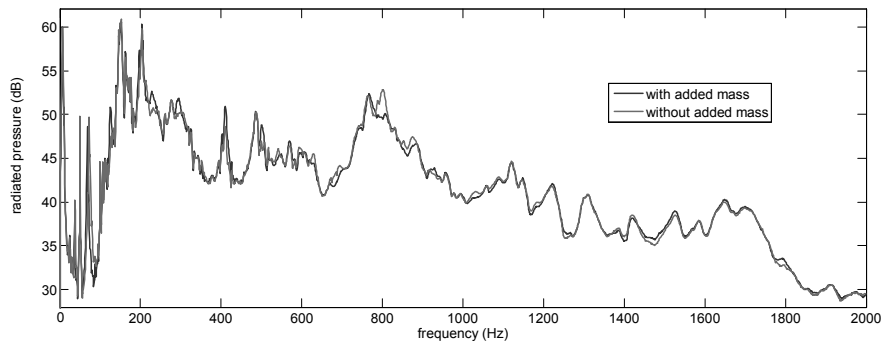


Fig. 8 Added mass effect on the radiated pressure spectra

#### 4. Conclusion

These first experiments have shown the reliability of the acoustical holography in the cultural heritage survey. The main advantage of this technique is the non contact aspect of the measurement. The preliminary experiment of the structural defect simulation indicates that acoustical holography is a very promising technique for the structural defect detection in vibrating structure. Future work will include the evaluation of the detection limit.

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# EXPERIMENTAL AND NUMERICAL MECHANICAL STUDY OF A FRAMING TECHNIQUE FOR CUPPING CONTROL OF PAINTED PANELS COMBINING CROSSBARS AND SPRINGS

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## Abstract

This communication deals with theoretical and experimental researches being carried out by the authors in order to model an actual wooden support, and its deformational behaviour, after a back frame has been applied by means of springs, in a Florentine restoration laboratory. Such a device is aimed to serve as a framing technique useful for conservation of one-sided painted boards of wooden artworks. The main outcome of such a research, still ongoing, is a calibrated mathematical and numerical model, which allows one to choose the most appropriate mechanical parameters for springs, according to expected environmental conditions, in order to achieve a balance between deformation control and stress control.

**Keywords:** finite elements; wood structures; hygromechanical coupling; conservation; cultural heritage

## 1. Introduction

A challenge for curators and restorers of panel paintings of cultural heritage is to perform restoration interventions which will contribute to the long time conservation in the best possible state. One of the main causes of damage is due to the environmental microclimatic variations, which may induce moisture changes, moisture gradients, transient and permanent deformations, stresses and damage to the pictorial layers up to their decohesion from the wooden support. Wood science, specifically with the modelling of phenomena like shrinking/swelling, creep, relaxation, and mechano-sorptive effects, may help to analyze and calibrate restoration interventions, taking into account the individual painted panels and the conservation environment. The wooden supports of many Italian panel paintings, especially in Central and Southern Italy, during late-thirteenth, fourteenth and early-fifteenth century, were typically made with boards of Poplar (*Populus alba L.*) glued along their edges, to form a planking. Ground layers, paint layers and protective varnishes were applied on the support face. Frames and cross-beams were often applied on the rear face, having – among others – the function of controlling the deformations of the support due to the humidity changes. Connections between the boards and the cross-beams were of various types, including nails (clined back into the wood to prevent their extraction), metal or wooden bridges or other devices allowing for some sliding, dovetailed joints (trapezoidal shape of the cross-beam cross-section, inserted in corresponding grooves made in the planking; sliding was not so easy, due to the large friction forces acting on the slanted edges). Although the above connections were conceived and executed to allow for some movements and to minimize damage, they could not cope with large environmental variations, taking place – just to make a few examples – when paintings were moved from original locations to museums where heating systems were installed, or when a large number of visitors severely modified the environment. Many interventions have been devised and performed in order to modify the mechanical characteristics of the connection between planking and cross-beams, and hence to reduce damage. Although based on good intentions, many of such interventions resulted in negative consequences. At present time, the soundest approach appears to try to solve the problems by controlling the environmental conditions rather than modifying the physical structure of the wooden support. However in several cases there is a need for redesign of the system of cross-beams; e.g. when it has been destroyed by previous interventions, or when serious accidents have severely damaged the support.

In these cases a compromise needs to be reached: a stiff connection will prevent permanent deformations (caused by complex rheological phenomena in wood) and damages to paint layers (unable to follow the wood movements) but it may produce cracks and ruptures in case of large environmental variations; whereas a too yielding connection will be useless, being at all unable to control deformations.

An interesting new framing technique was developed at OPD (*Opificio delle Pietere Dure* – A state restoration laboratory located in Florence – Italy) consisting of a back-frame, connected to the rear of the panel with springs (fig. 1).

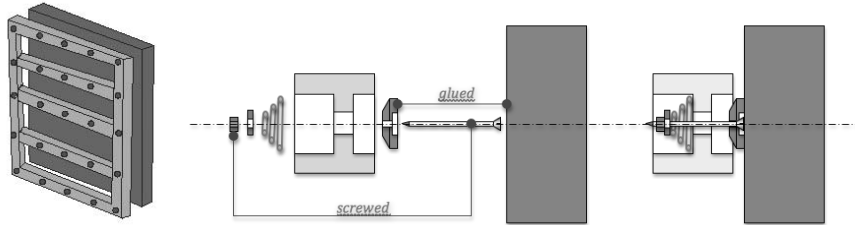


Fig. 1. Back-framing technique

The practical implementation of such a technique is quite satisfactory, and has now been adopted by several restoration laboratories; however one of the main problems, still unsolved, is the definition of the most desirable mechanical parameters of the springs, *i.e.* their stiffness and the pre-load to be used when installing them. Of course such parameters cannot be the same in any case, but need to be calibrated for each situation.

## 2. Modelling the frame action

A numerical one-dimensional beam model, based on finite elements, is used to simulate the frame action on panel movements (fig. 1). The panel movements are predicted by the model, with the assumption that the front (painted) face is totally impervious to water vapour, which reflects an ideal situation, where protective varnish is still an efficient continuous layer. This situation will emphasize the transient deformations caused by the presence and the evolution in time of non-symmetric moisture gradients.

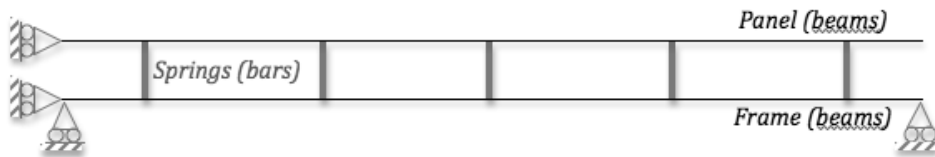


Fig. 2. Simple FE model of wooden panel with frame linked with springs

The model takes into consideration the unilateral contact conditions, the stiffness of springs and their pre-load. When the contact between frame and panel is active the contact force equals the initial pre-load. On the other case, the spring force is larger than the initial pre-load and tends to reactivate the contact. We assume here a linear relationship between the spring force and its compression (fig. 3).

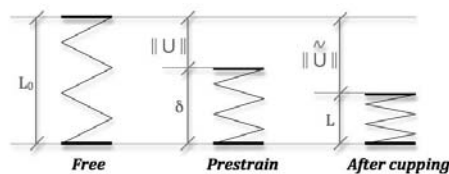


Fig. 3. Spring Force definition

$$F = (\|U\| + \delta - L).k \quad (1)$$

$$v = (\delta - L) \quad (2)$$

$L_0$  : free spring length / mm

$\delta$  : initial spring compression distance (prestrain) / mm

$v$  : spring compression distance due to the panel cupping / mm

$k$  : spring rigidity / N.mm<sup>-1</sup>



The moisture distribution to the thickness is here assumed to be linear (fig. 4), though experimental results of Kollmann [4] can be used as an alternative. This assumption on the moisture variation in the thickness will lead to an overestimation of the deflection. A linear model for the swelling/shrinkage behaviour is selected.

$$\varepsilon_T = \alpha_T w \left(1 - \frac{y}{h}\right) \quad (3)$$

$$\sigma_T = E_T \varepsilon_T \quad (4)$$

$w$  : change in moisture content / %

$\varepsilon_T$  and  $\sigma_T$ : strain field, and stress field / Pa

$\alpha_T$  : shrinkage parameter / %/%

$h$  : board thickness / mm

$E_T$  : transverse Young modulus of wood / Pa

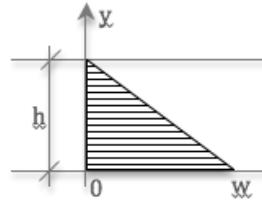


Fig. 4. Humidity gradient in the thickness

The torque  $Mz$  due to moisture content gradient in the thickness can be expressed as:

$$Mz = b \int_0^h \sigma_T \left(\frac{h}{2} - y\right) dy \quad (5)$$

$$Mz = \frac{1}{12} bh^2 E_T \alpha_T w \quad (6)$$

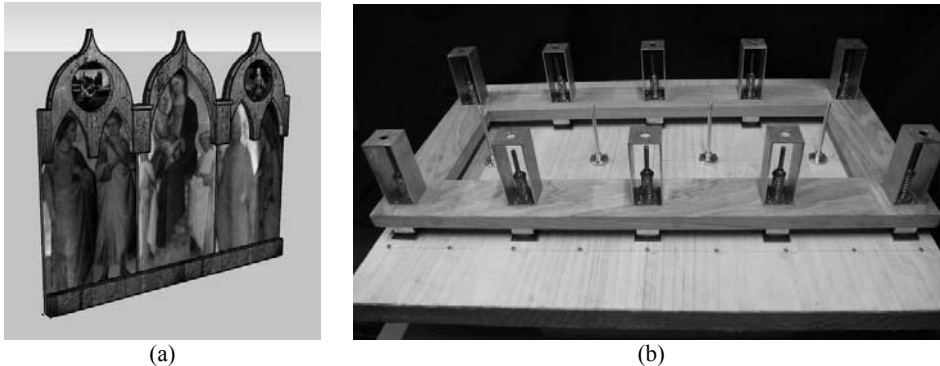
where  $b$  is the width of the wood piece.

The model is parameterized in order to be of practical use for curators (initial compression distance of springs, number of springs, panel dimension, wood mechanical characteristics of panel and frame...)

Note that, in this simplified model, the mechanical stiffness of the painting layer has not been taken into account. A crude homogenization estimation with two layers of different stiffness in bending leads to the following values: If the gesso is not damaged (no micro-cracks), with an elasticity modulus ten times higher than  $E_T$ , and with a thickness of 0.5 mm for 38 mm of wood thickness, the overall stiffness in bending is increased by 36 %. Taking into account this additional stiffness is not an issue, but renders the model more difficult to manage (with the requirement for a new constitutive parameter to be estimated).

### 3. Experimental device

In order to check and calibrate the finite element model, we designed a mock-up panel, reproducing the physical and geometrical characteristics of a real panel, which is part of a Florentine triptych presently being restored, and of the frame with springs purposely built by a restorer (fig. 5, 6).



(a)

(b)

Fig. 5. Florentine triptych (a) - Triptych mock-up with frame (b)

An identical mock-up panel without frame has been built as well, to provide a reference. The mock-up panels have been vapour-proofed with rubber latex on the front face (Rewultex®), simulating a protective varnish, as well as on the four lateral edges to eliminate edge effects.

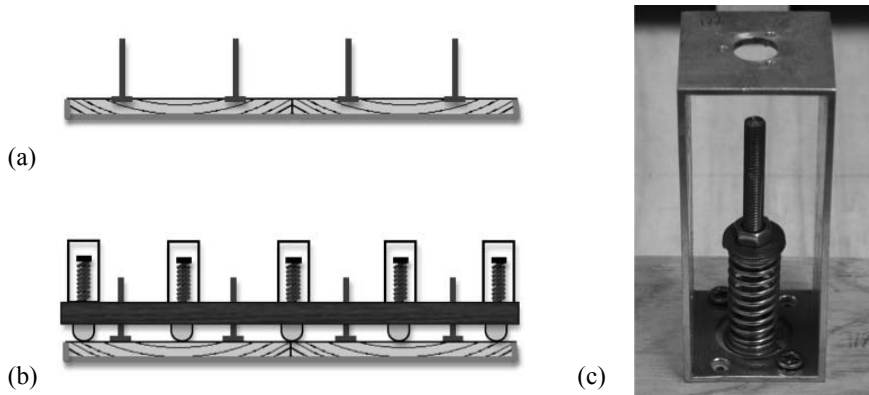


Fig. 6. Reference mock-up without frame (a) – Mock-up framed (b) – Spring and displacement measuring reference (c)

22 displacement sensors can be also integrated for accurate monitoring in time of the deformations and forces in the springs, though up to the moment, the monitoring is performed manually. Moreover we measure the shrinkage/swelling at several points of both panels.

#### 4. Results

##### 4.1. Numerical results

The simulation concerns a panel initially at a stable humidity state which is dried by reducing the ambient humidity by 20 % (= 3 % of moisture content). The parameters used are detailed in Table 1.

Table 1- FE model characteristics

Panel (poplar)	Frame (oak)	Springs
Thickness “h” = 38 mm	Thickness = 22 mm	Number of springs = 5
Width = 768 mm	Width = 768 mm	$k = 2.4 \text{ N.mm}^{-1}$
Height “b” = 600 mm	Equivalent Height = 160 mm	Pre-load = 24 N
Weight = 8 363 g	Weight = 3 602 g	$\Delta w = -3 \%$
$E_T = 576 \text{ MPa}$	$E_T = 1027 \text{ MPa}$	
$\alpha_T = 30 \%$		

The model response of such loading is showed in fig. 7:

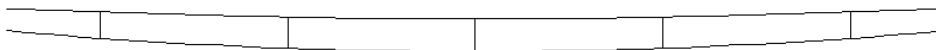


Fig. 7. Numerical solution for the drying of the panel

The maximum obtained deflection is about 16.7 mm. The maximum tension in springs is located at the middle of the panel with a value of 66 N. If the frame does not exist (frame with  $E_T \approx 0$  or  $k \approx 0$ ) the deflection is about 17.46 mm. It proves the effectiveness of the frame that has reduced the deflection by 5 %. To check the model results, we can compare with the analytical solution for the deflection (maximum value, obtained at the middle of the structure) with the following equation:

$$f = \frac{L^2 \cdot \Delta \varepsilon}{8 \cdot h} = \frac{L^2 \cdot \alpha \cdot w}{8 \cdot h} \quad (7)$$

The analytical solution for such conditions is a deflection of 17.55 mm which is very close to the numerical result. The table 2 gives any results of the model in different conditions.

Table 2- Numerical results for various springs configuration: deflection in mm

v / mm	k / N.mm-1				
	≈ 0	2.4	10	24	240
5	17.461	16.853	16.152	15.987	15.987
10	“	16.693	15.987	15.987	15.987
15	“	16.532	15.987	15.987	15.987
20	“	16.372	15.987	15.987	15.987

We can notice that the frame/spring device is not very effective here. The frame structure is not so rigid and the selected springs have a low rigidity. This tends to consider the frame structure underestimation because with such apparatus we obtain a maximum reduction of the deflection by 2 mm.

#### 4.2. Experimental results

For the experimental test we choose to stabilize the panels at the relative humidity of 65 % and at the temperature of 25 °C during one month to get a constant weight. The panels are then placed in another climatic chamber (45 % of relative humidity, 35 °C). To measure the curvature, a device using a differential length measurement ( $l_2 - l_1$ ) is depicted on Fig. 8.

Up to now, the measurements are hand-made, so the curvature of the panels is not estimated very well. An error of  $\Delta l_1 = \Delta l_2 = 5/100$  mm causes an error on the curvature of  $\Delta R/R \approx 100$  % for the present device, this is why the experimental study is not yet presented here.

We have therefore planned to make an automatic measuring of every distance to increase the data precision. The error on the curvature determination is reduced to less than 10 % when using sensors with an accuracy of 1/100 mm.

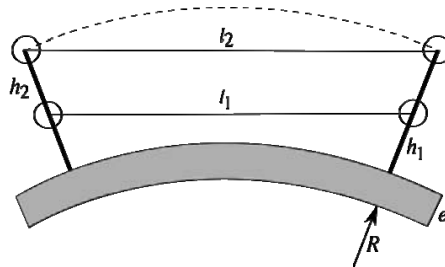


Fig. 8. Sketch of the experimental device to measure curvature of the panel

#### 5. Conclusion

Regarding to the numerical results, we can assume that the frame has to be more rigid to be efficient. Up to now, the maximum effect (with stiff springs and a consequent pre-load) is a reduction of 12 % of the deflection. With an even more rigid frame, we expect to obtain a higher influence.

On one hand this affirmation has to be tempered by the fact that the model has not yet been checked against experimental results.

On the other hand we still have to discuss the proposed changes with curators and conservators because a too rigid frame/spring system could damage the panel. A study of forces and stresses in the panel has to be carried out to get more relevant answers.

### **Acknowledgements**

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# EFFECT OF THERMAL TREATMENT ON STRUCTURAL DEFECTS OF MODEL PANEL PAINTINGS

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## Abstract

The European research project “MultiEncode” (Multifunctional Encoding System for the Assessment of Movable Cultural Heritage) involves the use of holography for the “encoding” of structural defects of a painting and consequently the development of a tool to confirm the originality and to diagnose the state of preservation of the object, before and after exhibition or loan. The National Gallery’s participation includes the construction of model panel paintings, which were holographically examined before and after thermal treatment. This paper describes the thermal program followed and some preliminary results of the samples examination by means of Dynamic Holographic Interferometry (DyHI).

## 1. Introduction

The *MultiEncode* European research project aims to use the advantages of holographic technology in order to develop an innovative *Impact Assessment Procedure*. The proposed methodology relies on the idea of holographic “encoding” of structural defects of an artwork that will be transported or loaned. The selected defects are considered to form the “signature” of the object. The same defects are to be detected again after the return of the artwork to the museum. By this way, it is possible to confirm the originality and to diagnose the state of preservation of the object [1].

National Gallery’s participation in the project as an end-user comprises the definition of “signature defects” of panel paintings, the construction of artificial samples that imitate original panel paintings and the evaluation of the holographic experimental results [2]. For the construction of the model panel paintings, an integral protocol has been developed. The protocol comprised the design, construction and documentation of the samples, as well as the thermal treatment before and after conservation interventions.

## 2. Materials and methods

A series of artificial samples was prepared, taking into consideration the typical structure of a panel painting (for example, a post-Byzantine icon), the factors of deterioration and the type of defects that usually occur (see Fig. 1). The main target was the simulation of all possible structures of a panel painting (use of textile, nails, etc.), and the reproduction of all the types of defects, such as knots, cracked wood, loss of ground and paint layers, etc. The samples were divided into 6 groups; each group contained 3 identical samples. Thus each of the three technical partners (Centre Spatial de Liège - CSL, Institute of Electronic Structure and Laser, FORTH Crete - IESL and Institut fuer Technische Optik, Stuttgart – ITO) acquired all six types of samples. Pine wood was selected, according to traditional handbooks that suggest softwoods as substrates for byzantine and post-byzantine icons. At the beginning, the samples were sent to the three technical partners for holographic examination and then they were sent back to the National Gallery for thermal treatment.

Referring to thermal treatment, it has to be noticed that the present experimental procedure aimed to provoke defects to the wooden substrates, in order to evaluate a novel structural diagnostic methodology, without taking into account the alterations of the painted surface. For this, extended literature research has been performed. Most of the international standards (ISO, ASTM, etc.) referring to artificial ageing of wood, aim to test products that prevent wood degradation. Furthermore,

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the authors of the NG have not come across any published scientific work focused on artificially aged wooden samples that imitate old artistic substrates.

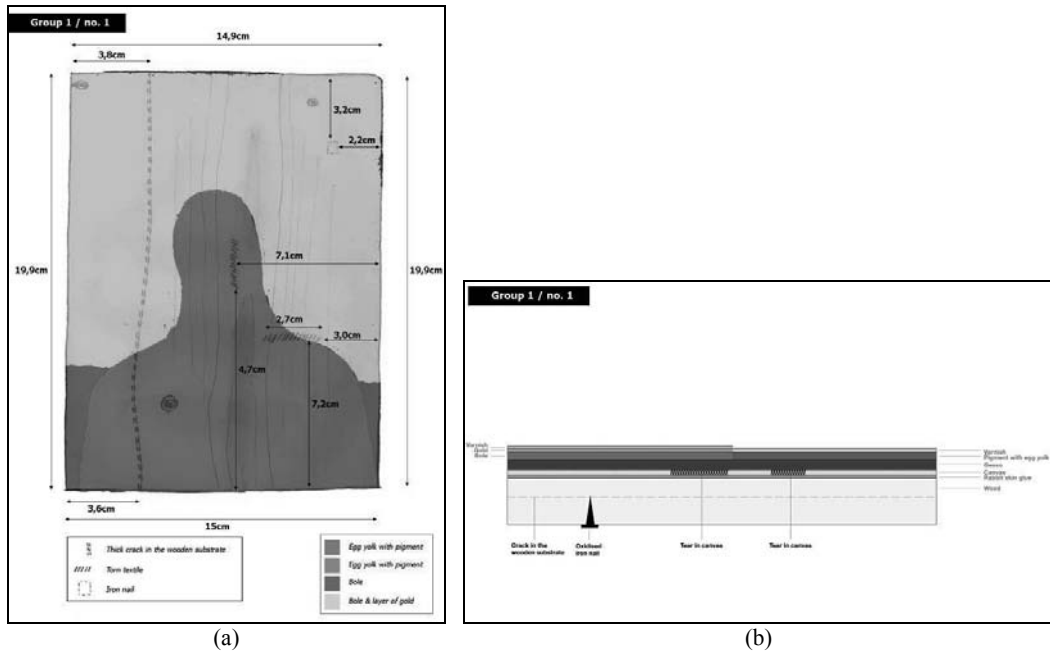


Fig. 1. (a) An artificially-made sample (Sample 1-Group 1); (b) Schematic cross-section of the sample.

The protocol followed at the first stage of thermal treatment of the model panel paintings is presented in table 1. The procedure was performed in a thermal chamber (Memmert Company).

The samples were examined by the technical partners by means of Digital Speckle Holographic Interferometry (IESL), Shearography (ITO) and Dynamic Holographic Interferometry (CSL). At a second stage, some of the samples were further treated whereas others were restored and then treated again.

The preliminary results that are presented in this paper have been aroused from the samples' examination performed by the co-authors of CSL, by means of DyHI [3]. Two standard setups have been used, i.e. 2 front illuminations or 1 back and 1 front illumination with 175IR lamps.

Table 1: The protocol followed at the first stage of the thermal treatment of the samples

Group	Sample	Thermal protocol
1	All	Continuous heating at 102°C for 480 hrs
2	All	Heating at intervals at 102°C for totally 66,5 hrs
3	1 (IESL)	Continuous heating at 102°C for 17,5 hrs
	2 (CSL)	Continuous heating at 102°C for 46hrs
	3 (ITO)	Continuous heating at 102°C for 141 hrs
4	All	Continuous heating at 85 °C for 247 hrs
5	All	1. Continuous heating at 60°C for 66,5 hrs 2. Continuous heating at 90 °C for 148 hrs
6	All	Same as GROUP 2

### 3. Results

Some representative results of the numerous experiments that have been performed by CSL are presented in Fig. 2 – Fig. 6. The presented interferograms have been processed so that sheared images and diagrams are acquired. Nevertheless, only some examples that may substantiate the effect of

thermal treatment are presented in the current paper. The red marked areas on the interferograms (b) show defects caused deliberately to the samples during the construction. The red marked areas at the interferograms (c) are indicating the defects detected after the thermal treatment.

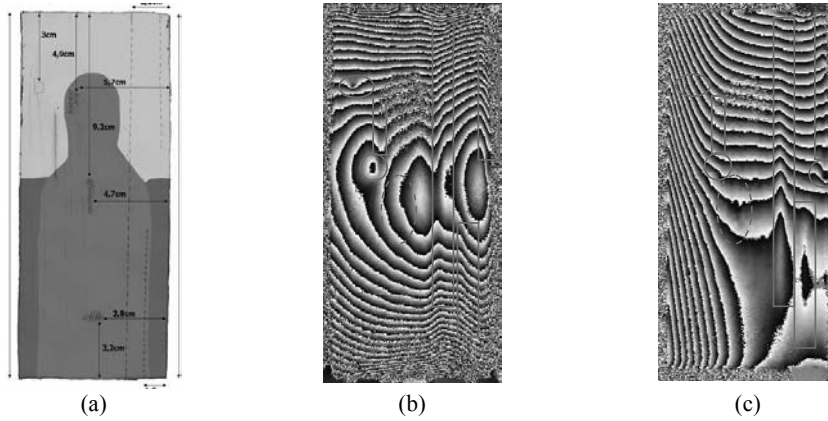


Fig. 2. (a) Visible image of the sample Group 1 N° 2; (b) Untreated sample – 9 min 25 s after back/front heating; (c) Thermally treated sample – 1 min 30 s after back/front heating.

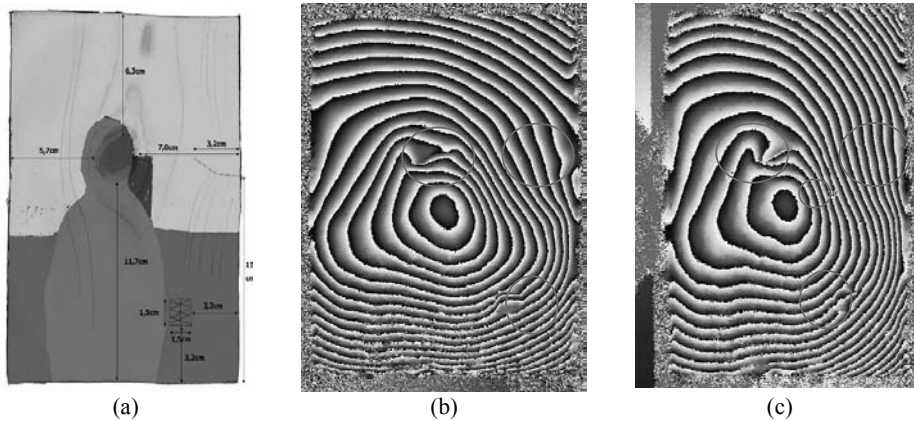


Fig. 3. (a) Visible image of the sample Group 2 N° 2; (b) Untreated sample – 2 min after front heating; (c) Thermally treated sample – 2 min after front heating.

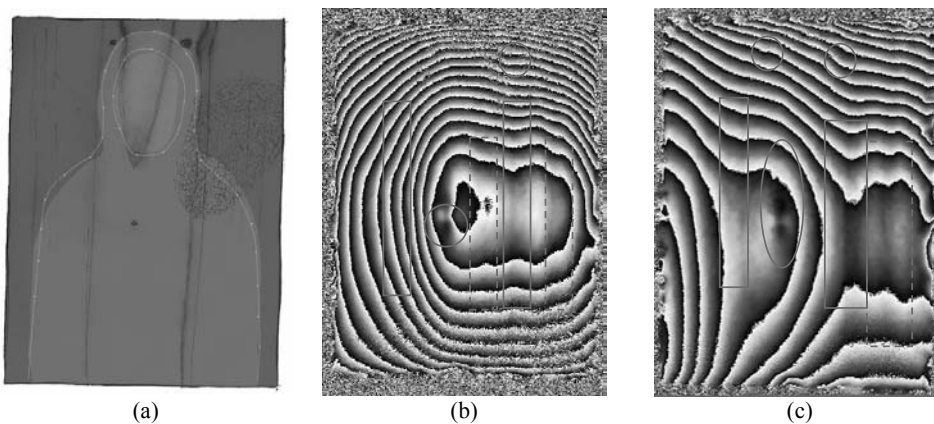


Fig. 4. (a) Visible image of the sample Group 3 N° 2; (b) Untreated sample – 3 min after front heating; (c) Thermally treated sample – 3 min after front heating

All the defects that have been suggested by NG as signature defects have been detected both on the untreated and the thermally treated samples. Yet only a few defects attributed to thermal ageing can be related to a specific thermal protocol or a particular type of sample. For example, a signature defect has appeared on sample 2.2 after thermal treatment, but this was not detected on sample 6.1 that has undergone the same thermal procedure. Concerning the sample 5.1, the fringe pattern looks more or less similar before and after treatment. For samples 3.2 and 6.1, the surface texture looks altered after treatment. Concerning the sample 1.2 no conclusions can be extracted as the test conditions were different before and after thermal treatment.

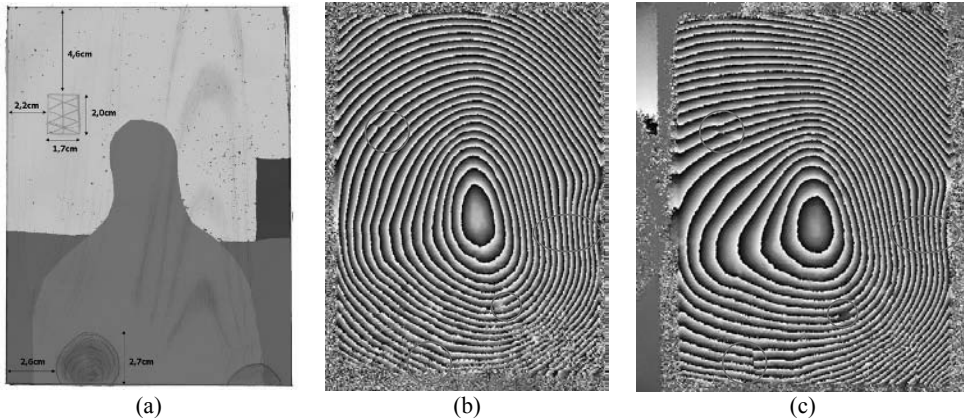


Fig. 5. (a) Visible image of the sample Group 5 N° 1; (b) Untreated sample – 1 min 30 sec after front heating; (c) Thermally treated sample – 1 min 30 sec after front heating.

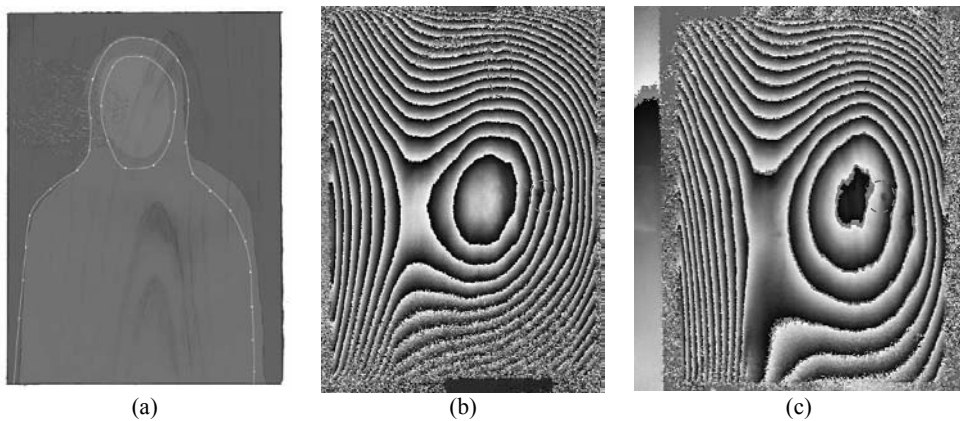


Fig. 6. (a) Visible image of the sample Group 6 N° 1; (b) Untreated sample – 1 min after back/front heating; (c) Thermally treated sample – 1 min after back/front heating.

#### 4. Conclusions

The thermal treatment of model panel paintings performed at the framework of *MultiEncode* European Project successfully served the aims of the holographic experiments referring to repeatability. Nevertheless, the several thermal programs that have been investigated by NG may be considered only as preliminary results of the integral study of artificial ageing. Such protocols should be based on a complete series of experiments involving humidity, UV-radiation and their combinations. Additionally, it has to be examined whether the structural diagnosis, the chemical analysis or the measurement of mechanical properties is the optimum method for studying the effects of such



treatment on wood. Complete protocols for imitating old wood for artistic and conservation purposes would be an ulterior expectation of the authors.

### **Acknowledgements**

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# INTER-COMPARISON OF COLOUR MEASUREMENTS OF POLY-CHROMED WOODEN OBJECTS WHICH WERE IRRADIATED FOR DISINFECTION REASONS

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## Abstract

Gamma irradiation treatment is an efficient mean of mass disinfection of wooden objects contaminated by fungi and insects. Among the more or less significant changes in the physical-chemical properties of materials which could be induced by gamma irradiation, colour changes of poly-chromed wood are often feared by the conservators in charge of such collections and have to be evaluated objectively and precisely using colour measuring instruments. These instruments come in many configurations, so the inter-instrument agreement is very important. The aims of this work are to check the inter-instrument agreement between two reflectance spectrophotometers and evaluate the colour stability over time for a set of un- and irradiated painted wooden panels.

## 1. Introduction

The most important species involved in the biodeterioration of wooden objects are fungi and insects. Gamma irradiation is a treatment aiming in prevention from these biodeteriorating organisms and remediation (curing) of infected objects. Applied absorbed doses are chosen according to radio-resistance of the species implied in biodeterioration, ranging from 500 Gy (enough to kill larvae and prevent the emergence of adult insects) to about 10 kGy (at which most of the fungi are killed) [1]. The effects on wooden material at these doses are known to be acceptable for the community involved in conservation of cultural heritage artefacts.

The case of poly-chromed wooden objects (that is, painted wooden objects like wooden paintings or painted wooden statues) has to be studied separately. Polychrome layers are subdivided in a preparation layer and a paint layer, the chemical composition of which varies according to the mode and the period of painting. For the historical period in European regions, the preparation layer is usually made with lime or gypsum with addition of animal or vegetal glue. On this smooth surface several layers of colour are present, which consist of pigments mixed with binders of oil or distemper (egg or glue). The surfaces could also be covered with a thin, translucent protective varnish.

Because of more or less significant changes in the physical and chemical properties of materials which could be induced by gamma irradiation, the unlikely but still possible colour changes of poly-chromed wood have to be evaluated. Perceptions of colour being subjective and physiologically dependant, colour measuring instruments (colorimeters and spectrophotometers) must be used to quantify these potential colour differences. There are six parameters that affect the colour measurement: colour scale, illuminant, standard observer, the instrument itself (type, geometry, and standardization mode), the way of presenting the sample to the instrument, and finally the method of preparing the samples themselves. If one or more of these parameters is changed, the colour values are also changed. The first three of these criteria have to be chosen according standardization. Spectrophotometers come in many sizes, shapes, geometries, and configurations, so inter-instrument agreement is a complex topic, but it theoretically belongs to standardization. As a matter of fact, to obtain the best inter-instrument agreement in terms of absolute colour values, one has to use the same model of instrument from the same manufacturer with all the six parameters matching. However, instruments of the same geometry should agree on difference values from a physical standard, even if they do not agree exactly on absolute colour values [2].

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The aims of this work are to:

- check the inter-instrument agreement between two reflectance spectrophotometers: HunterLab Miniscan XE Plus (HL) and Konica Minolta CM-508i (CM);
- evaluate the colour stability over 1 year for a set of un- and irradiated painted wooden panels.

## 2. Materials and methods

Two sets of samples were used in this work: (A) for the inter-comparison of the two spectrophotometers and (B) for checking the colour stability over time for un- and irradiated painted wooden samples.

Samples (A), prepared at ARC-Nucléart (France), are small pieces of wood covered by a preparation layer (chalk in mixture with rabbit skin glue) on which binders (3, 6, 9, and 12 layers) or pigments in mixture with poppy seed oil are painted. These samples were prepared in two series: one unirradiated, used as a reference and the other was gamma irradiated at 200 kGy (approximately twenty times the usual dose for eradication of fungi). As the uniformity of the samples is very important in colour measurement comparisons, samples (A) were visually inspected (for cracks, traces of brush strokes etc.) and arbitrarily grouped in three classes according to their uniformity: good, medium and bad.

Samples (B), prepared by Conservation-Restoration Department of National University of Arts (Romania), consist of small pieces of wood covered by a ground layer (chalk in mixture with rabbit skin glue) and pigments mixed with tempera (aqueous solution of yolk egg and water). Half of every sample was additionally varnished with Dammar- this is indicated by "+v". Samples (B) were prepared in three series: one unirradiated, used as a reference, and two irradiated at 11 kGy but using two dose ratios: 35 Gy/min (indicated by "d") and 245 Gy/min ("D").

Reflectance spectrometry was used for colour measurements of these opaque samples. The results of measurements are reported in both CIE L\*a\*b\* and CIE L\*C\*h and presented as differences between sample and its corresponding reference [3]. Total colour differences  $dE^*$  being small,  $dE$  CMC formula was also used with a lightness to chroma ratio of 2:1. In Table 1 are presented the characteristics of the two colour measuring instruments used in the inter-comparison. The two spectrophotometers differ in: spectral resolution, reflectance range and diameter of measuring beam / view area; they should agree on difference values.

Table 1: Characteristics of reflectance spectrophotometers

Instrument	Miniscan XE Plus (HL)	CM-508i (CM)
Manufacturer	Hunter Associates Laboratory, Inc.	Konica Minolta Sensing, Inc.
Colour scale	CIE L*a*b* and CIE L*C*h	CIE L*a*b* and CIE L*C*h
Illuminant	D <sub>65</sub>	D <sub>65</sub>
Standard observer	10°	10°
Geometry of measurement	d/8°	d/8°
Spectral range	400 – 700 nm	400 – 700 nm
Spectral resolution	10 nm	20 nm
Reflectance range	0 – 150 %	0 – 175 %
Port diameter / view diameter	6.0 / 4.0 mm	11.0 / 8.0 mm

## 3. Results and discussion

### 3.1. Intercomparison

Samples (A) were measured with CM at ARC-Nucléart and with HL at IFIN-HH; time interval between the two series of measurements is two weeks so the chemical changes (due to chemical reactions between compounds of the painting layers or influence of atmosphere on the surface of samples) that can affect the colour of the samples could not be significant. The results are presented in

table 2 as differences between colour values obtained from irradiated samples (200 kGy) and their corresponding unirradiated references. Colour differences are given with their corresponding uncertainties, expressed as one standard deviation, calculated by applying the law of propagation of uncertainties on colour difference formulae. Standard deviation of the mean was used for every colour parameter. The CIELAB total colour differences with their corresponding standard deviations are presented in Fig. 1.

Table 2: Colour differences for samples (A) measured with HL and CM

sample [uniformity]	instrument	dL* (1 s.d.)	da* (1 s.d.)	db* (1 s.d.)	dC* (1 s.d.)	dh (1 s.d.)	dE* (1 s.d.)	dE CMC
rabbit skin glue (12 layers) [good]	HL	-1.18 (0.15)	-0.19 (0.02)	6.35 (0.07)	6.33 (0.07)	1.10 (0.04)	6.46 (0.07)	3.61
	CM	-2.60 (0.51)	-0.10 (0.05)	6.05 (0.23)	6.02 (0.23)	1.15 (0.10)	6.59 (0.44)	3.39
linseed oil (12 layers) [good]	HL	-0.56 (0.29)	0.40 (0.05)	0.62 (0.87)	0.66 (0.87)	0.82 (0.30)	0.92 (0.61)	0.52
	CM	-2.54 (0.72)	0.47 (0.04)	0.61 (0.17)	0.66 (0.17)	-1.09 (0.49)	2.65 (0.36)	1.06
poppy seed oil (12 layers) [good]	HL	-0.17 (0.22)	-0.46 (0.04)	1.57 (0.22)	1.62 (0.22)	1.14 (0.14)	1.64 (0.21)	1.42
	CM	-1.33 (0.42)	-0.48 (0.03)	2.36 (0.11)	2.41 (0.11)	0.64 (0.36)	2.75 (0.38)	2.14
ivory black [medium]	HL	-1.51 (0.45)	-0.03 (0.05)	-0.07 (0.08)	0.08 (0.07)	-0.53 (4.20)	1.51 (0.45)	1.33
	CM	-2.01 (0.31)	-0.19 (0.40)	-1.16 (1.11)	0.20 (1.18)	192.44 (0.10)	2.33 (1.02)	2.37
silver white [medium]	HL	-1.85 (0.32)	-0.36 (0.11)	1.97 (0.20)	1.98 (0.21)	1.76 (0.70)	2.73 (0.26)	1.99
	CM	0.35 (0.32)	-0.55 (0.04)	2.74 (0.06)	2.75 (0.06)	3.86 (0.73)	2.82 (0.21)	2.88
Prussian blue [medium]	HL	-0.09 (0.41)	-0.54 (0.10)	4.39 (0.78)	-4.43 (0.77)	0.05 (0.74)	4.43 (0.77)	3.16
	CM	-1.51 (0.15)	-0.93 (0.07)	4.23 (0.38)	-4.32 (0.37)	-1.52 (0.54)	4.59 (0.36)	3.23
Brunswick blue [medium]	HL	-0.75 (0.36)	0.84 (0.23)	2.01 (0.40)	-2.13 (0.43)	2.35 (1.05)	2.30 (0.38)	1.83
	CM	-0.92 (0.38)	0.84 (0.14)	2.99 (0.68)	-3.10 (0.68)	1.04 (0.93)	3.24 (0.64)	2.44
raw umber [good]	HL	0.73 (0.44)	-0.33 (0.24)	-0.34 (0.68)	-0.41 (0.65)	1.19 (1.68)	0.87 (0.46)	0.66
	CM	0.44 (0.72)	-0.05 (0.07)	-0.33 (0.47)	-0.30 (0.47)	0.78 (0.50)	0.55 (0.57)	0.43
cadmium red [bad]	HL	-0.02 (0.54)	0.31 (0.47)	1.02 (0.58)	0.83 (0.69)	0.62 (0.25)	1.06 (0.57)	0.56
	CM	-0.26 (0.10)	0.20 (0.17)	-0.64 (0.15)	-0.19 (0.09)	-0.59 (0.19)	0.72 (0.15)	0.47
Solferino lake [bad]	HL	-2.10 (0.62)	-1.71 (0.59)	0.35 (0.70)	-1.50 (0.44)	0.76 (0.67)	2.73 (0.61)	1.24
	CM	1.81 (0.74)	0.86 (0.93)	-1.02 (1.55)	0.45 (0.69)	-1.05 (1.50)	2.25 (1.54)	1.17
sample [uniformity]	instrument	dL* (1 s.d.)	da* (1 s.d.)	db* (1 s.d.)	dC* (1 s.d.)	dh (1 s.d.)	dE* (1 s.d.)	dE CMC
cobalt blue [bad]	HL	-2.10 (0.64)	3.22 (0.55)	-4.07 (0.79)	3.68 (0.73)	4.46 (0.79)	5.60 (0.70)	2.98
	CM	-4.92 (0.31)	7.18 (0.28)	-3.49 (0.08)	2.68 (0.08)	8.96 (0.32)	9.38 (0.29)	5.37

ultramarine [bad]	HL	0.90 (0.79)	2.00 (0.94)	-3.00 (1.17)	3.32 (1.32)	1.37 (0.72)	3.72 (1.09)	1.60
	CM	0.39 (0.29)	5.45 (0.41)	-6.05 (0.35)	7.05 (0.43)	3.83 (0.28)	8.15 (0.36)	3.69
titanium white [medium]	HL	-1.60 (0.62)	0.26 (0.07)	0.76 (0.20)	0.76 (0.21)	-2.26 (0.55)	1.80 (0.56)	1.00
	CM	-1.31 (0.46)	-0.12 (0.02)	1.45 (0.10)	1.45 (0.10)	0.77 (0.43)	1.96 (0.17)	1.51
napples yellow [medium]	HL	0.82 (0.33)	-0.18 (0.06)	-1.57 (0.55)	-1.58 (0.55)	0.18 (0.08)	1.79 (0.51)	0.82
	CM	-0.66 (0.08)	-0.51 (0.06)	-3.70 (0.53)	-3.72 (0.53)	0.59 (0.08)	3.79 (0.34)	1.79
cadmium yellow (citron) [bad]	HL	-1.39 (0.25)	1.03 (0.15)	-1.92 (0.47)	-1.96 (0.47)	-0.61 (0.10)	2.58 (0.38)	0.91
	CM	-0.20 (0.18)	0.23 (0.17)	-0.20 (0.30)	-0.21 (0.30)	-0.14 (0.11)	0.36 (0.29)	0.15
cadmium yellow [bad]	HL	0.54 (0.59)	0.31 (0.37)	2.85 (1.29)	2.70 (1.30)	0.67 (0.24)	2.92 (1.27)	1.14
	CM	2.01 (0.48)	0.75 (0.45)	5.09 (1.69)	4.91 (1.70)	1.05 (0.28)	5.52 (1.53)	2.10
ochre yellow [bad]	HL	4.29 (0.75)	-0.86 (0.33)	4.30 (0.66)	3.55 (0.66)	2.61 (0.33)	6.14 (0.70)	3.19
	CM	3.71 (0.65)	0.50 (0.21)	5.34 (0.38)	5.06 (0.28)	1.81 (0.34)	6.52 (0.51)	2.91

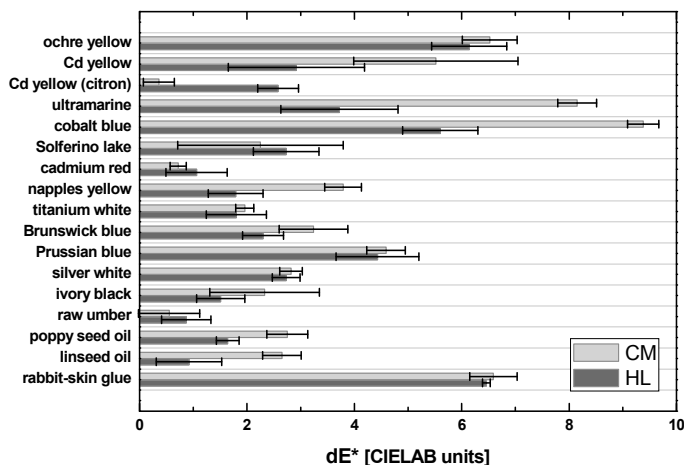


Fig. 1. Total colour differences between unirradiated and irradiated samples (A)

For samples prepared with binders (*rabbit skin glue*, *linseed oil* and *poppy seed oil*), without pigments, the agreement was good. In this paper are presented only the samples with 12 layers of binders; for samples with a lower number of layers the agreement is much better. The highest disagreement, only quantitative, is for lightness ( $L^*$ ): CM indicates a higher difference in  $L^*$ .

Regarding the effect of gamma irradiation, there are small colour differences; even in the case of *rabbit skin glue*, the main component of colour difference is from chroma- considering 12 layers of binder and 200 kGy, this difference is still acceptable, although this dose is 20 times the reference dose for disinfections.

In the case of samples prepared with different pigments in mixture with *poppy seed oil*, the agreement between the two spectrophotometers is depending on the uniformity of the sample. One can observe

that even for the samples of bad uniformity (*Solferino lake* and *cadmium red*) for which the disagreement (considering the colour differences on components) is not only quantitative, but also qualitative, the total colour differences ( $dE^*$  and  $dE$  CMC) are very close.

### 3.2. Colour stability over time

Colour stability over time for samples (B) was evaluated using HL. The interval of time between the two series of measurements is one year. The results are presented in table 3; values obtained in the last year are used as references.

Table 3: Colour shift over time for samples (B)

sample	dL*	da*	db*	dC*	dh	dE*	dE CMC
raw Sienna	-1.32	-0.77	-3.30	-3.32	-0.87	3.64	1.59
raw Sienna (d)	0.47	-0.34	0.18	0.03	0.47	0.61	0.41
raw Sienna (D)	0.71	-0.76	-0.88	-1.11	0.42	1.36	0.63
raw Sienna v	3.04	1.13	3.56	3.65	1.30	4.81	2.51
raw Sienna v (d)	6.28	1.83	7.24	7.25	2.78	9.76	5.12
raw Sienna v (D)	7.18	1.33	8.37	8.01	4.22	11.11	6.19
minium	-0.29	-1.49	-1.28	-1.95	0.17	1.98	0.67
minium (d)	-1.81	-3.27	-4.84	-5.78	-0.67	6.12	2.16
minium (D)	-0.84	-3.00	-7.20	-7.31	-2.21	7.85	3.27
minium v	-0.48	-1.96	-3.09	-3.61	-0.52	3.69	1.32
minium v (d)	-1.91	-2.49	-4.74	-5.18	-1.08	5.68	2.19
minium v (D)	-2.17	-3.45	-5.91	-6.69	-1.18	7.18	2.67
mars red	0.25	-0.81	-0.69	-1.06	-0.21	1.09	0.55
mars red (d)	-0.28	-1.30	-0.82	-1.54	0.07	1.56	0.77
mars red (D)	-1.06	-1.56	-0.59	-1.62	0.74	1.98	1.07
mars red v	-0.44	-1.25	-0.60	-1.37	0.43	1.46	0.74
mars red v (d)	-0.13	-1.40	-0.82	-1.61	0.23	1.62	0.80
mars red v (D)	-0.57	-1.18	-0.57	-1.30	0.39	1.43	0.73
chrome yellow	-1.14	0.41	-1.68	-1.67	-0.27	2.07	0.69
chrome yellow (d)	-0.47	0.54	1.15	1.14	-0.37	1.35	0.49
chrome yellow (D)	-2.30	-0.80	-4.25	-4.23	0.61	4.89	1.63
chrome yellow v	-0.90	0.59	-0.21	-0.19	-0.39	1.09	0.45
chrome yellow v (d)	-1.13	-0.14	-2.27	-2.27	0.12	2.54	0.83
chrome yellow v (D)	-1.64	-1.95	-2.75	-2.70	1.42	3.74	1.49

Colour shift over time is showed as  $dE$  CMC total colour difference in Fig. 2 where a vertical line at one  $dE$  CMC unit marks the limit up to which a normal standard observer could not detect any colour change. In this respect colour shift over time is insignificant for samples prepared with *mars red* and *chrome yellow* (except *chrome yellow* samples irradiated at a higher dose ratio). The colour of varnished samples prepared with *raw Sienna* is the most unstable, even for the unirradiated ones. Regarding the influence of irradiation on the stability of colour over time one cannot draw a general rule. For example, in the case of *minium* irradiated samples are less stable than unirradiated ones but for *raw Sienna* this effect can be seen only on varnished samples. The influence of irradiation is only quantitative on lightness, chroma and hue. Where the colour shift is significant (i.e. more than one  $dE$  CMC unit) it increases with the dose ratio. Varnish does not influence the colour stability over time except samples prepared with *raw Sienna* where the colour shift is higher for varnished samples.

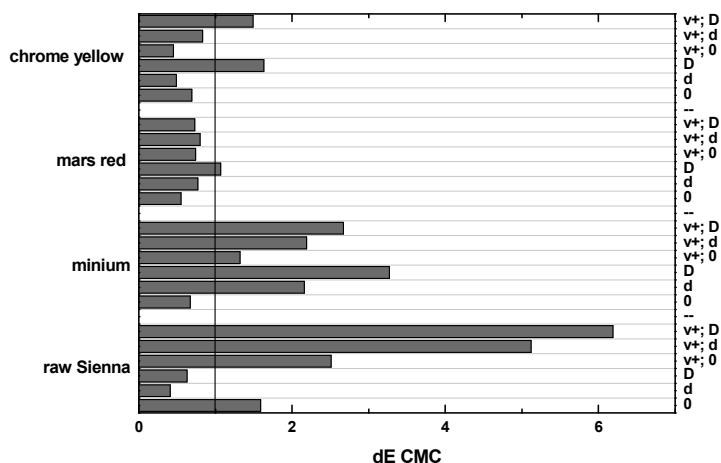


Fig. 2. Colour shift over time (1 year) for samples (B)

#### 4. Conclusions

The agreement of the two inter-compared spectrophotometers is depending on the uniformity of the samples, but even for some samples of bad uniformity as *Solferino lake* the agreement is good in total colour differences values. Regarding the effect of irradiation on the colour of samples (A), total colour differences are lower than 5 dE\* units (i.e. small colour differences) except *cobalt blue*, *ochre yellow*, and *rabbit-skin glue*.

The influence of irradiation on the colour shift over time of samples (B) is specific for the type of sample and it is only quantitative in terms of lightness, chroma and hue. The colour shift increases with the dose ratio and it is not influenced by varnish except samples prepared with *raw Sienna*.

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# CHARACTERIZATION OF WATERLOGGED WOOD BY INFRARED SPECTROSCOPY

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## Abstract

The work concerns the characterisation of waterlogged archaeological wood samples from different excavation sites in Italy by means of infrared spectroscopy. It describes some preliminary evaluations of the analyses carried out, including a quantitative estimation of the holocellulose, one of the structural chemical components constituting the cell walls.

## 1. Introduction

The structural modifications which wood undergoes during burial in waterlogged conditions [1-5] imply the need for an effective treatment for a safe conservation after discovering. Till today such treatments are in most of cases carried out without a complete diagnostic evaluation, except for a few quick measurements of selected physical characteristics of the archaeological material [6]. This fact is basically due to two series of reasons: the first one is imputable to the real usefulness of the diagnostic analyses, actually not well connected to a practical use of the obtainable information; the second reason is associated with the limited accuracy of the current methodologies and to the great effort required in terms of time for their complete execution.

However, a phase of diagnostic evaluation preliminary to any kind of treatments is an essential step to be accomplished because it allows for the possibility of checking, after consolidation, the presence of eventual changes within the treated object, both in the short and the long term, when maintained either in service conditions or during exposure at a museum. From this point of view, the well-known case of Vasa is very representative [7]. Moreover, the evaluation of the extent of the decay allows for its grading and therefore, potentially, for a treatment tailored to the specific state of preservation of a finding. Within this framework, a quantitative, reliable and quick measurement of the structural chemical components constituting the residual cell walls of the fossil material represents an important step toward the broad applicability of a systematic approach intended to evaluate the state of preservation of archaeological wooden finds at the time of their discovering. Among other techniques, those related to infrared analysis seem to be the most promising, due to the fastness of execution, the limited amount of sample required and to the relatively long experience in their application [8]. However, considering the high variability (mainly in terms of chemical structure) of the decayed fossil material [9, 10] and the presence of various other factors potentially disturbing the interpretation of analyses, like for example the ever considerable content of inorganic components, a research effort is required before proposing this technique as a routinely protocol for diagnostic evaluations in the archaeological field.

Aim of this work is describing a series of preliminary evaluations, performed on samples coming from different sites of excavation in Italy, finalised to carry out a quantitative estimation of the chemical structural components constituting the cell walls of archaeological wood, by mean of Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (ATR-FTIR).

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Attenuated total reflectance (ATR) is a sampling technique used in conjunction with Fourier transform infrared spectroscopy (FTIR) which enables samples to be examined directly in the solid or liquid state without further preparation. It can be used to identify compounds, investigate sample composition or for quantitative analysis. In this study ATR-FTIR was used to assess the modifications in the chemical composition of woods during burial in waterlogged conditions, for extended periods of time up to two thousand years, as well to assess the presence and identification of mineral compounds whose presence interfere with gravimetric analysis.

## 2. Material and methods

Samples coming from the archaeological excavations of Pisa (VII c. B.C. - II c. A.D.), the three ships found in Naples (I-II c. A.D.) and the ship of Comacchio (I c. A.D., Augusto's Age) were analysed both in terms of conventional and ATR-FTIR analyses. Small blocks (approximately 4 x 2 x 1 cm<sup>3</sup> edges) were taken from the various findings, being careful to remove the material in the centre of the artefact. Considering that the material was milled after drying, no specific orientation was chosen.

Conventional chemical analyses regarded essentially measures of lignin according to the Klason's method (Tappi T222) [11] and of ash content according to Tappi T211 [12], whereas the 'true' holocellulose amount utilised in the calibration curve was estimated by difference, by taking into account the ash content of each sample that reduces the effective mass of wood. The used methodologies are the most diffused in the field of the wood chemical characterisation by conventional methods, because of the good reproducibility of measurements: a precision within 2% is obtainable according to Rodrigues et al. [13] for the Klason's method.

As provided by Tappi T222, wood meals were subsequently extracted before measurements. In particular:

- extractions in organic solvents were carried out in a Soxhlet apparatus by using a toluene/ethanol mixture 2:1 v/v;
- extractions in water were carried out using deionised water, in a Soxhlet apparatus, on the previously organic-extracted powder.

Instead, in the case of IR analysis, meals were extracted firstly in dichloromethane, followed by ethanol and subsequently in deionised water, after verifying that the extracted substances were quantitatively the same as for conventional analyses.

All the measurements were carried out on sieved material (in the range 40-120 mesh, corresponding to 0.12-0.40 mm). The results of conventional analyses referred to the anhydrous weight of the wood flour and gave the relative amount of the residual chemical components.

ATR-FTIR spectra were recorded with a Bruker FT-IR spectrometer (Alpha) with the following settings: 42 scans per sample, spectral resolution: 4 cm<sup>-1</sup>, wavenumber range: 4000 to 400 cm<sup>-1</sup>, using a diamond single reflection attenuated total reflectance (ATR) and a zero filling of 2. Post spectroscopic manipulation was kept to a minimum. Atmospheric compensation and offset-correction to the minimum between 1920 and 1880 cm<sup>-1</sup> was applied to the ATR-FTIR spectra (Software OPUS 6 from Bruker). Simple linear regression (SLR) modelling was performed with OPUS Quant 1 software.

## 3. Results and discussion

### 3.1. Influence of inorganic fillers

The effect of the content of inorganic fillers of wood, estimated in the conventional analysis as ashes, is very relevant in the ATR-FTIR spectra, considering that some of these components evidence absorbing peaks in important regions of the spectrum, thus hiding or strongly influencing the signals coming from structural components of wood (Fig. 1, Table 1).

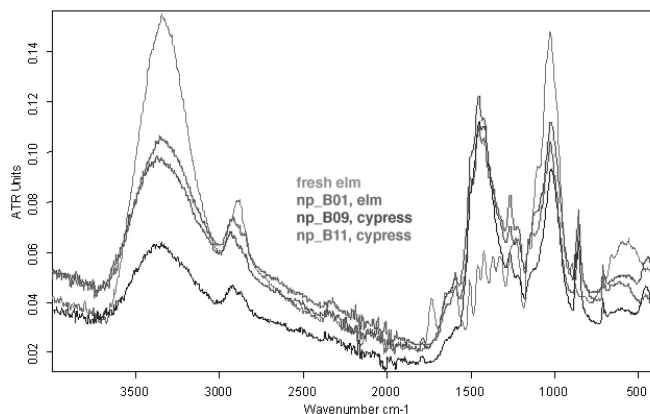


Fig. 1. ATR-FTIR spectra of selected samples from the ship B of Naples, characterised by a high ash content, in comparison with the spectrum of fresh elm (*Ulmus* sp.). Samples were preliminarily extracted with solvents. In the figure, np stands for Naples, B identifies the ship and the number refers to samples collected in different points of the ship)

As an example, the case of sample np\_B01 is reported (Fig. 2). While the spectra of non extracted and extractive free meal evidence minor differences, the treatment of extracted meal by a diluted acetic acid solution evidences that the great part of the signal in the approximately 1300-1450  $\text{cm}^{-1}$  region is due to the contribution of acid-soluble inorganic matter, namely  $\text{CaCO}_3$ , presents as both calcite and aragonite. It is noteworthy that the preliminary extraction of the meal does not remove these substances, due to their scarce solubility. The peak at 1020  $\text{cm}^{-1}$  in the extract is most probably due to low molecular weight water-soluble carbohydrates.

Noticeably, all these signals can strongly affect an eventual quantitative estimation based on the related spectra.

Table 1: The main results obtained for each sample after the chemical conventional analyses. Considering the limited amount of available material, one determination was carried on each sample.

sample	wood species	provenance	lignin (%)	ashes (%)	true holocellulose (%)
cm_c01	elm ( <i>Ulmus</i> sp.)	Comacchio	72.5	10.7	18.8
cm_c02	elm ( <i>Ulmus</i> sp.)	Comacchio	29.4	2.3	69.9
cm_c03	elm ( <i>Ulmus</i> sp.)	Comacchio	30.8	3.6	68.0
cm_c04	elm ( <i>Ulmus</i> sp.)	Comacchio	35.1	3.4	63.7
cm_c05	elm ( <i>Ulmus</i> sp.)	Comacchio	30.6	2.6	68.6
cm_c06	elm ( <i>Ulmus</i> sp.)	Comacchio	35.3	3.7	63.3
cm_c07	elm ( <i>Ulmus</i> sp.)	Comacchio	31.5	2.6	67.7
np_A07	elm ( <i>Ulmus</i> sp.)	Napoli	54.1	14.5	36.7
np_B01	elm ( <i>Ulmus</i> sp.)	Napoli	16.4	54.2	64.3
np_B09	cypress ( <i>Cupressus</i> sp.)	Napoli	13.8	68.9	55.7
np_B11	cypress ( <i>Cupressus</i> sp.)	Napoli	32.9	13.7	61.9
np_C01	oak ( <i>Quercus</i> sp.)	Napoli	64.0	6.9	31.2
np_C02	oak ( <i>Quercus</i> sp.)	Napoli	59.2	11.3	33.3
pp_B02	elm ( <i>Ulmus</i> sp.)	Pisa	76.9	5.4	18.7
pp_B29	elm ( <i>Ulmus</i> sp.)	Pisa	77.4	2.9	20.3
pp_B18	elm ( <i>Ulmus</i> sp.)	Pisa	78.4	6.9	15.8

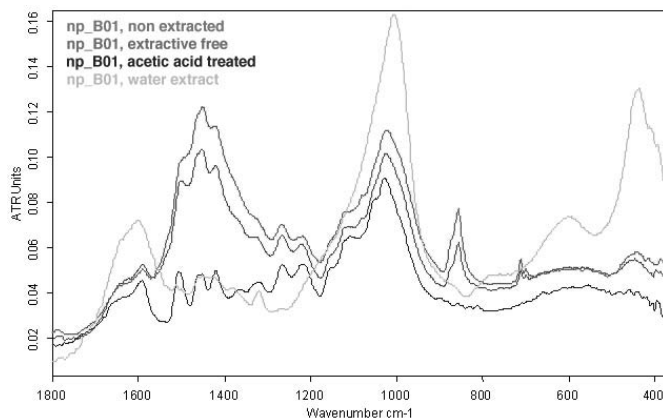


Fig. 2. Spectra of sample B01, from the site of Naples, subjected to several treatments in order to evidence the changes in its spectrum due to the presence of ashes. The spectrum of the water extract is also shown.

### 3.2. Calibration

Calibration is the most important step in the quantitative evaluation of the structural components of wood by means of the ATR-FTIR spectra. In the case of samples having low amount of inorganic components it can potentially give good results, mainly if samples belonging to the same wood species are considered (Fig. 3). The example of elm samples coming from the sites of Pisa and Comacchio shows that only cm\_C1, with an ash content almost tripling (Table 1) the amount of the other samples is an outlier, whereas pp\_B2 and pp\_B29, showing an ash content higher than others but however of the order of 5%, are acceptably aligned along the calibration straightline (Fig. 4). In the same way other samples belonging to other species than elm and having ash content higher than 10% are also not aligned with the identified calibration (Fig. 4), even if a parallel straightline could probably be drawn (not reported in the figure).

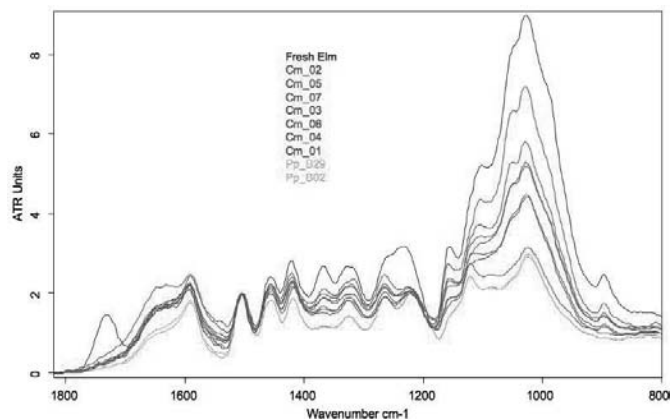


Fig. 3. Min-Max (1800-1507  $\text{cm}^{-1}$ ) normalized spectra of seven extracted elm samples from Comacchio (cm\_C01 to cm\_C07), two from Pisa (pp\_B2 and B29) and the extracted fresh elm spectrum (blue).

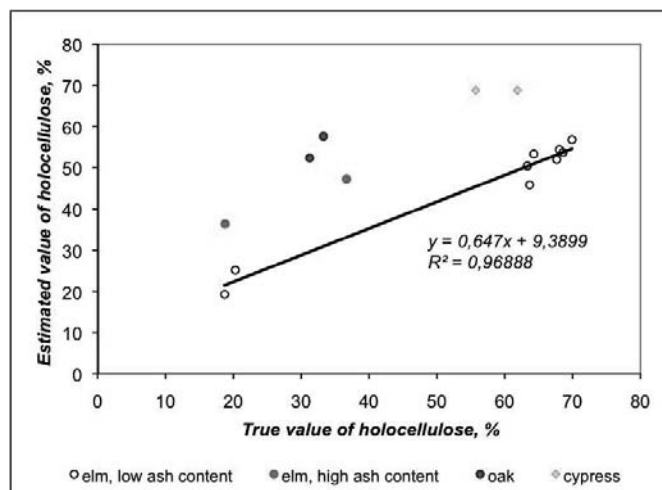


Fig. 4. Linear regression plot of estimated vs. laboratory-evaluated holocellulose content for the analysed samples. The regression model refers only to the model obtained with open black circles, corresponding only to elm samples from Pisa and Comacchio with low ash content (below 6.9% for all of them). The other circles are samples with ash content above 10%, including elm, cypress and oak.

### 3.3. Influence of sample inhomogeneity

Fig. 5 shows two normalized spectra obtained in two different portions of the same fine powder and apparently homogenised meal of the sample np\_A07 (Naples, ship A). As can be observed the two spectra show differences most noticeable at 1454, 1420, and 1024  $\text{cm}^{-1}$ .

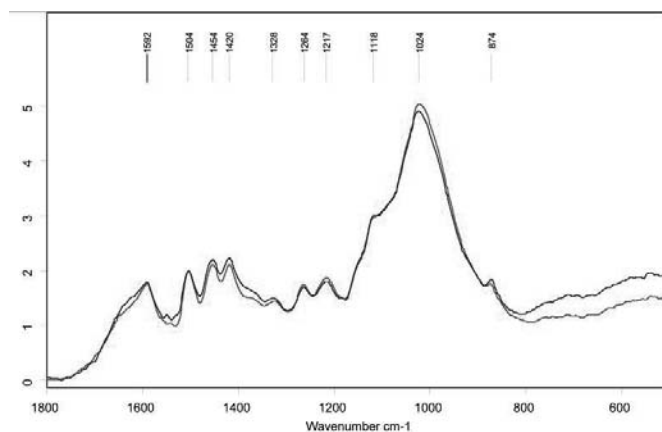


Fig. 5. Min-Max normalized (1800-1480  $\text{cm}^{-1}$ ) ATR-FTIR spectra obtained in two different portions of the extracted meal of the same sample, np\_A07, from ship A of Naples. Main differences can be seen at 1454, 1420 and 1024  $\text{cm}^{-1}$ .

If the two spectra are included in the regression line (Fig. 4), two different values of holocellulose (41% and 55%) are obtained. This addresses the question of the representativeness of the aliquot used to obtain the spectra that could be handled with a careful homogenisation of the samples complemented with an increased number of spectra per sample.

## 4. Conclusions

The results obtained suggest that it would be possible to build models to assess the holocellulose content of waterlogged archaeological wood by means of ATR-FTIR analysis using simple linear

regression analysis. Some precautions have however to be considered in effectively applying it, due for instance to the influence of inorganic fillers, and also the great care in the homogenization of the archaeological material.

Further analysis including more samples and also multivariate regression techniques will be tested to assess holocellulose content and also to build other methods for other structural components of fossil wood.

### Acknowledgements

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# STRUCTURE, MOCK-UP MODEL AND ENVIRONMENT-INDUCED DEFORMATIONS OF ITALIAN LAMINATED WOOD PARADE SHIELDS FROM THE 16TH CENTURY

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## Abstract

Round shields made of laminated wood were widely used in Italy in 15th – 17th centuries for tournaments, parades and exhibitions. The manufacturing techniques originally used are not known from literature nor from any still living tradition. This paper describes: (a) a structural analysis of the shield on which Caravaggio painted his well known *Medusa* (1598); (b) the recently completed construction of a mock-up shield, manufactured with materials and techniques reproducing as much as possible the supposed original ones; (c) the initial results of monitoring performed both on (i) an original shield exhibited in the Museo Bardini in Florence, and (ii) the above mentioned mock-up shield, placed in climatic chambers at DISTAF.

## 1. Introduction

The well known *Medusa* (Fig. 1) exhibited at the Uffizi Gallery in Florence, was painted in 1598 by Caravaggio (Michelangelo Merisi, 1571-1610) on a convex round wooden shield, approximately 580 mm in diameter. Shields of this type were widely used in 15<sup>th</sup> – 17<sup>th</sup> centuries for tournaments, parades and exhibitions; in some cases they were painted with the same techniques as “flat” panel paintings, in other cases they were covered with paper or sheepskin, and decorated with coats of arms or other kinds of drawings or paintings. The manufacturing techniques originally used are not known from literature nor from any still living tradition, but some realistic hypotheses, possibly not far from truth, can be derived from close examination of the existing shields, and from the present knowledge of wood technology and of woodworking techniques. The actual geometrical shape of most existing original shields is not exactly a spherical segment, as supposedly it was at time of their manufacture, but is distorted so it looks similar to a tortoise shell (see below).

As most wooden artworks exposed to environmental variations, these shields uptake/release moisture, and hence their distortion increases/reduces, possibly influencing negatively the well-being and conservation of the artwork; since with time the painted or decorated layers tend to become more fragile, and can not follow without serious risks of damage the contractions/expansions of the wooden support, environmental variations are now especially dangerous. One of the most efficient conservation practices consists therefore in keeping the environment as stable as possible; however scientific knowledge of the climate/deformation relationships can also be very helpful, in order to allow for an evaluation of most favourable climatic environment, based on objective and reliable data. Understanding and describing, both empirically and mathematically, the climate/deformation relationships for this kind of support is even more complicated than for “flat” panels, because of the complex shape of the shields and their laminated structure. As a first step, the authors are carrying out empirical observations of the environment-related deformational behaviour.

This paper has the following main objectives:

- 1) to provide information about the structure of the shield on which Caravaggio's *Medusa* was painted;
- 2) to describe the recently completed construction of a mock-up shield, manufactured with materials and techniques reproducing as much as possible the supposed ones (according to authors' judgement);

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- 3) to report about the initial results of monitoring being performed both (a) on an original shield exhibited in the Museo Bardini in Florence, and (b) on the above mentioned mock-up shield, placed in climatic chambers at DISTAF.

## 2. The medusa shield

### 2.1. The structure of the shield

In 1998, we were asked by the Director of the Uffizi Gallery to analyze the wooden structure of the *Medusa* shield (Fig. 1), at that time under restoration.



Fig. 1 – The Medusa’s truncated head, painted by Caravaggio in 1598 on a wooden shield approximately 580 mm in diameter.

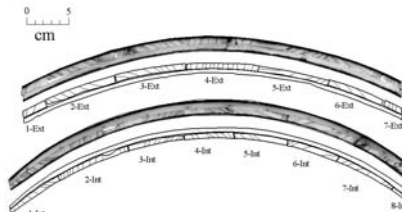


Fig. 2 – Two perpendicular cross-sections of the shield, obtained by Computed Tomography: the upper CT scan is on the smaller (“horizontal”) diameter, whereas the lower CT scan is on the larger (“vertical”) diameter. Drawings placed under CT scans put into evidence the longitudinal and transversal cross-sections of lamellae, and the random orientation of growth rings.

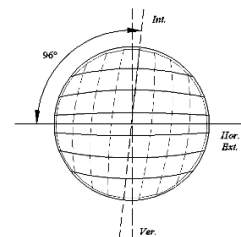


Fig. 3 – The reciprocal orientation of the two approximately perpendicular layers of lamellae.



Fig. 4 - The orientation of the painted image, of the strap-holding nails, and of the directions of the lamination’s layers. The numbered lines are the traces of the planes along which one series of CT scans was taken.



Fig. 5 – An “exploded” view of the shield’s structure.

The growth rings shown are purely fictitious.



Fig. 6 – The wooden mould shaped (by turning) as a spherical section, to be used for assembling the lamellae.

The following main characteristics of the structure have been derived from direct close observation, from other scientific analyses carried out during restoration, and especially with the support of X-ray Computed Tomography [1] [2] [3] (Fig. 2). The over 80 CT-scan images have been digitally analyzed and reassembled by means of a 3-D CAD software. The resulting “exploded” drawing of the structure is shown in Fig. 5.

- The shield is “tortoise shell” shaped and its perimeter, which is supposed to have been originally circular and laying on a plane, is now slightly elliptical and does not lay on a plane any more; its two perpendicular diameters differ in length (570 mm the larger one, and 550 mm the smaller one); its average radius of curvature is 403 mm, and the dome’s height is approximately 115 mm;

- the shield's structure is laminated, made of two layers, glued to each other, of lunule-shaped wooden lamellae (7 in the external layer, 8 in the internal one), approximately 6 mm thick, wide approximately 60 to 92 mm, each layer being approximately perpendicular to the other (Fig. 3, 4)
- the overall thickness of the shield, including the linen and gesso layers, both on the front and on the rear face, is approximately 20 mm, thinned down to approx. 5 mm in the proximity of the perimeter,
- lamellae have straight grain, randomly oriented growth rings, and a constant thickness along both their length and width, indicating that their double curvature derives from a process of deformation, rather than of shaping by means of material removal from a block of thicker wood
- in the normal orientation of the shield, defined both by the painted image, and by the still existing nails which were originally used for fixing the handling straps on the rear, the external layer of the lamellae is oriented "vertically", whereas the internal layer is oriented "horizontally" (Fig. 2, 4)
- anatomical examination carried out by optical microscopy on a wood fragment sampled from the shield during restoration, showed that the lamellae are made of Poplar (*Populus alba* L.) wood ("Gattice" according to Italian standard UNI 2853 [4]).

## 2.2. Hypotheses about the manufacturing process

We were not able to find in the literature any description of the techniques originally used for manufacturing this kind of shields. Based on the evidence obtained from scans, on present knowledge of wood technology, and on a realistic evaluation of the technical means available at that time, we figured out what the manufacturing process could actually have been. Such hypotheses have been practically implemented, and modified as needed, as described in the following paragraph.

## 3. The mock-up shield manufactured at DISTAF

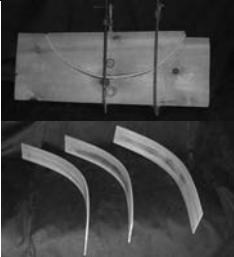





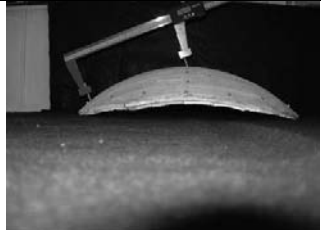


Based on the analyses performed, a mock-up of the *Medusa*'s shield has been constructed at DISTAF, reproducing as near as possible its original structure (amount, sizes, location and shape of the original lamellae), overall size and manufacturing techniques. The shape changes of such mock-up model are being monitored by means of electronic callipers with spherical feelers, and of a reference system of "marks" fixed on the shield's surface. Weight changes have also been monitored.

### 3.1. Manufacturing the mock-up shield

After several tests on the most appropriate procedure, the following practical steps have been chosen, in order to manufacture the mock-shield with simple procedure and equipment.

- individual lamellae, planed at 6 mm thickness from seasoned Poplar (*Populus alba*) modern boards, have been roughly shaped with a band-saw according to drawings made on the basis of the analysis of the original shield
- lamellae have been firstly bent with a "steaming" procedure described below, to a single longitudinal curvature, slightly larger (i.e. with smaller radius) than the final one, to account for a mild post-forming recovery; the mould and counter-mould were easily obtained by sawing a thick wooden board, and then tightened together by means of ordinary carpenter's clamps
- the "steaming" process, carried out on individual lamellae, consisted in (a) soaking the dry lamella in hot water, during approximately 30 minutes, (b) clamping the soaked lamella between mould and counter-mould (Fig. 7), (c) producing quick evaporation of water with a hot air jet (30 minutes at 100 °C was quite effective; in old times, the same result could possibly be obtained by exposing the whole "sandwich" near a fire)
- individual lamellae were then extracted from their "bending" moulds (Fig. 8), and quickly nailed on a wooden "assembling" mould (Fig. 9, 10) turned to the shape of a segment of a sphere, having the "final" desired radius of 403 mm (Fig. 6)
- this nailing process imparted to the lamellae the transverse curvature, needed to fit correctly on to the spherical surface
- the nails (6 for each lamella) were 2 mm in diameter, and were for provisional fixing only, so they were inserted for half of their length, and bent in order to better hold a larger surface of the lamella against the mould (Fig. 10, 11)



		
<p>Fig. 7 – A soaked lamella clamped in the “shaping” mould (above) which imparts them a single curvature, and three shaped lamellae (below)</p>	<p>Fig. 8 – After having been shaped with a hand-held smoothing plane, lamellae are successively nailed on the “assembling” mould with provisional 2 mm nails, half-inserted and bent</p>	<p>Fig. 9 – After extraction of nails from first layer, the second layer of lamellae is also provisionally nailed; the hot animal glue is locally applied, so the two layers adhere thoroughly and permanently to each other</p>
		
<p>Fig. 10 – Shaping and refining by hand the shield, still nailed on the mould.</p>	<p>Fig. 11 – The shield, removed from the mould. The traces of the provisional nails (no more present) are still visible.</p>	<p>Fig. 12 – The rear part of the shield, removed from the mould.</p>
		
<p>Fig. 13 – Measuring the distance between two reference points, with an electronic calliper (accurate at 0,01 mm)</p>	<p>Fig. 14 – The calliper has been fitted with purpose-made feeler pins, ending with a 3 mm diameter steel sphere</p>	<p>Fig. 15 – The spherical end of the feeler pin fits on top of the hexagonal cavity of the Allen screw, hence identifying a reference point in space, no matter the orientation of the reference screw.</p>

- successive lamellae were shaped in-place with a hand-held smoothing plane, in order to give them the exact taper need to fit against the adjacent ones
- after having completed the first layer, the second layer was applied with the same procedure (Fig. 9), the only differences being that before applying the individual lamellae (a) the nails fixing the previous layer are extracted, and (b) the animal glue is locally applied, so that the two layers adhere thoroughly to each other (gluing along the lamellae's edges only would give no strength to the assembly)
- after completing the assembly the shield, still nailed on the mould, was left for approximately on month in a climatic room (20 °C, 65% RH) to reach Equilibrium Moisture Content with the surrounding air; then its periphery was trimmed to a circumference with a hand-held saw, and surface unevenness were smoothed away with a hand-held plane and/or a chisel (Fig. 10)
- (at this point, after removal from the mould, an original shield would have been ready to receive ground-layers and final decorations)

- several stainless steel screws, 3 mm diameter, with cylindrical Allen head, were then applied along “meridians” and “parallels” of the shield, to be used as reference points for successive dimensional monitoring (Fig. 13, 14, 15).
- After approximately one month, the shield was removed from the mould, after having carefully extracted the provisional fixing nails (Fig. 11, 12).

### 3.2. Monitoring weight and shape variations of the mock-up shield

Monitoring of variations of shape and weight of the mock-up shield was carried out during several months, from June 2008 to September 2008.

The following significant points in time can be identified:

In climatic chamber “A” (“normal” climate: 20 °C, 65% RH)

- while the shield was still nailed on the mould, and EMC was reached
- immediately after the shield was un-nailed and removed from the mould
- after it reached equilibrium shape
- In climatic chamber “B” (quite “dry” climate: 30 °C, 40% RH)
- when the shield was moved from chamber “A” to chamber “B”
- when it reached equilibrium shape and weight in the new climatic conditions.

Analysis of collected data, here omitted for the sake of brevity, shows that:

- the only significant change in shape during the conservation in chamber “A”, at constant EMC, was observed at removal from mould; the “vertical” diameter (i.e. the one parallel to the external layer of lamellae) increased, whereas the perpendicular diameter decreased → the global shape changed from a spherical segment to a “tortoise shell” shape, also observed in most existing original shields
- a second significant change in shape showed up while reaching EMC in the new climatic conditions: lowering the wood Moisture Content produced an increase of the “vertical” diameter, and a decrease of the “horizontal” diameter (Fig. 16).

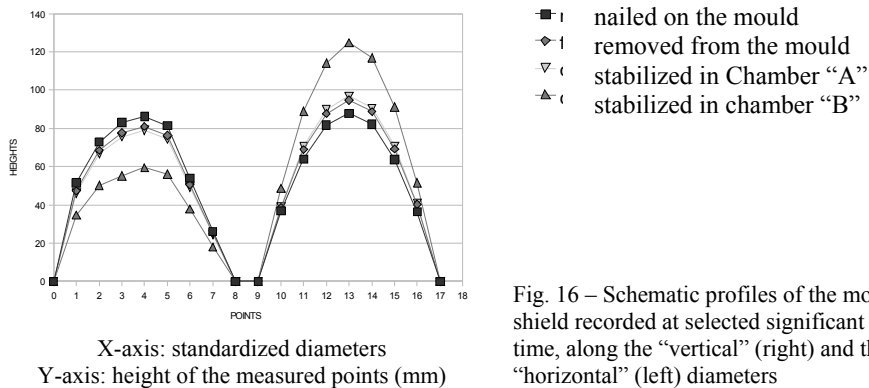


Fig. 16 – Schematic profiles of the mock-up shield recorded at selected significant points in time, along the “vertical” (right) and the “horizontal” (left) diameters

### 4. The monitoring performed on an original shield in “bardini” museum

One of the shields conserved in the Bardini Museum in Florence (inventory n. 408), very similar to *Medusa*’s one, has been monitored during several months by means of a purposely conceived and implemented “deformometric kit”, automatically measuring every 15 minutes the length variations of two perpendicular diameters, and the climatic parameters (temperature and RH) of the surrounding air. The measuring apparatus and some initial results deriving from such monitoring activities are briefly described in Fig. 17, 18, 19, 20. During restoration a very small wooden sample was collected, and the wood species was determined by microscopical examination as Poplar (*Populus alba* L.). The recorded deformations (Fig. 21, 22) show significant variations especially of the smaller diameter (the “horizontal” one, according to the terminology used above), whereas the larger diameter (the “vertical” one) varies only by very small amounts.



Fig. 17 – The “deformometric kit”, purposely designed and built for monitoring this kind of shields. Four Plexiglas feelers slide freely on perpendicular low friction rails.



Fig. 19 – The shield 408 mounted on the apparatus: side view, showing the shorter (“horizontal”) diameter. The 4-channel data-logger has self sufficient batteries, which also supply power to the transducers at moment of measurement, which in this case was set every 15 minutes.



Fig. 18 – The shield 408 mounted on the apparatus, in the Bardini Museum (Florence): top view. Two potentiometric transducers (blue) measure continuously the distance between the opposite feelers.



Fig. 20 – The shield 408 mounted on the apparatus: side view, showing the longer (“vertical”) diameter. The shield lays on a plastic soft cushion, the feelers press very gently on its edge. The data logger’s memory, when measuring rate is set at 15 minutes, has a capacity of about 96 days. The battery can last over 1 year.

## 5. Conclusions

### Wood species used for manufacturing the shields

Some art history literature mentions that these shields were made of “Fig” (*Ficus carica* L.?) wood, but no evidence was found to confirm such assumption; on the contrary, the two wood samples positively identified by us showed that the two examined shields (Uffizi’s *Medusa*, and Bardini’s *408*) were both made of Poplar (*Populus alba* L.) wood. In fact, for its technological characteristics Poplar wood appears much more suitable than Fig wood for manufacturing this kind of shields; and experience in manufacturing the mock-up shield confirms this.

### Deformations of the shields

Considering the three sets of observations reported above, the following qualitative conclusions may be suggested:

- shield were manufactured on spherical moulds, but subsequent “tortoise-shell” deformation was very likely expected
- shields, as any other wooden artefact, deform (or tend to deform) according to variations of climatic conditions in their environment
- however, due to the peculiar structure, similar to a plywood *ante litteram*, deformations tend to affect more the shape of the whole artefact than the linear dimensions of its surface; this might result in favour of conservation of the paint layers, but further analyses are needed.

More detailed conclusions will hopefully be obtained by structural analysis of the artefacts, carried out by FEM method.

## Acknowledgements

The Authors gratefully acknowledge the support received from Dr. Antonella Nesi, Director of the Bardini Museum (Florence), who authorized the monitoring activities, the taking of a wood sample for anatomical examination, and the publication of the collected data.

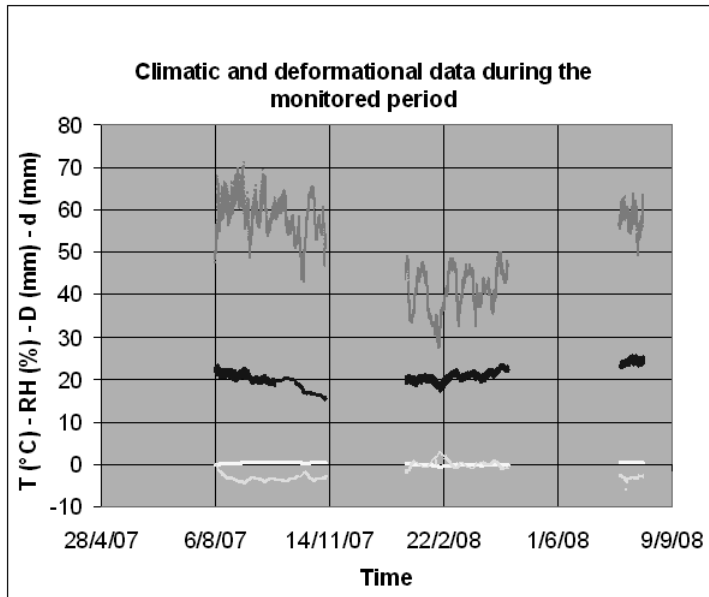


Fig. 21 – Shield 408 in Bardini Museum (Florence) – Climatic and deformational data recorded by means of the “deformometric kit” from June 2007 to September 2008.

Violet = RH (%); Blue = Temperature (°C); Yellow (D) = Variations of “vertical” diameter (mm); Blue (d) = Variations of “horizontal” diameter (mm)

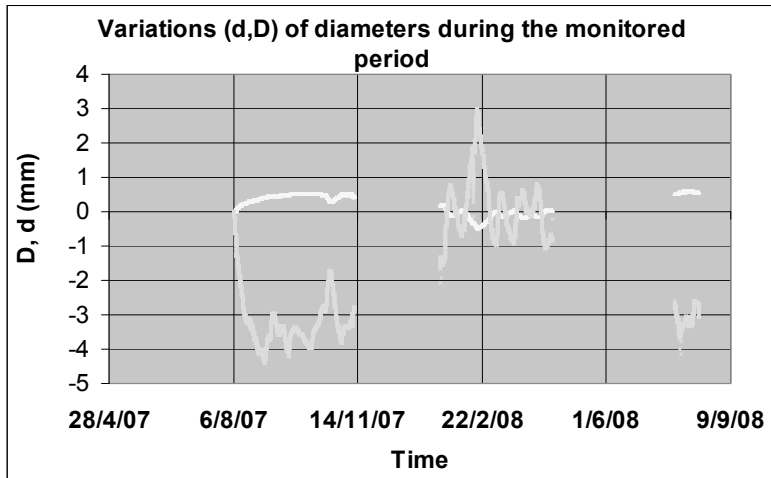


Fig. 22 – Same diagram as Fig. 21, but only diameter variations shown (vertical scale magnified).

“Horizontal” diameter shows larger variations (d): it increases when air RH decreases, and vice-versa.

“Vertical” diameter shows opposite variations (D), but quite smaller. (Unfortunately, due to technical malfunctions, data from two time intervals were not recorded)

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## (D) CONSERVATION



# EUROPEAN TECHNICAL COMMITTEE 346 - CONSERVATION OF CULTURAL PROPERTY - UPDATING OF THE ACTIVITY AFTER A THREE YEAR PERIOD

*Vasco Fassina\**

Soprintendenza per il Patrimonio Storico Artistico ed Etnoantropologico per le province di Ve, Pd, Bl e Tv,  
Venice, Italy

## 1. Introduction

The foremost aim of European standardization is to facilitate the exchange of goods and services through the elimination of technical barriers to trade [1].

Only thanks to a sound scientific knowledge of the materials constituting the cultural property, of its environmental and conservation conditions, the conservation/restoration works can be successfully carried out.

Unfortunately the great experience developed in this field by the different European countries, for the time being can not constitute a common background because there are too many differences not only in the methods of analysis, but also in the terminology used.

A specific European standardisation activity in the field of conservation of Cultural Heritage is essential to acquire a common unified scientific approach to the problems relevant to the preservation/conservation of the Cultural Heritage. Moreover, this common approach and the use of standardised methodologies and procedures would promote the exchange of information, would avoid the risk of duplication and foster synergy between the European experts and specialists involved in the preservation activity.

In particular, the standardisation activity on the conservation of Cultural Heritage deals with:

- i) terminology relevant to movable and immovable cultural property, and to the conservation of the cultural property and of the material constituting the cultural property themselves, so that a common European terminology can be created;
- ii) guidelines for a methodological approach to the knowledge of the cultural property and of the materials constituting the cultural property, of the deterioration processes, and of conditions of optimum long-term conservation/preservation work;
- iii) test and analysis methods for the diagnosis and for the characterisation of the cultural property and of their state of conservation with regards to outdoor and indoor environmental parameters;
- iv) test and analysis methods (in laboratory and in-situ) for the evaluation of the performance of the products and methodologies to be used in the conservation work (ordinary and/or extraordinary maintenance);
- v) test and analysis methods for the evaluation of conservation conditions of indoor Cultural Heritage.

## 2. Market Situation in relation to the objectives of CEN/TC 346

The market relevant to the conservation of Cultural Heritage is considerable and it involves a great number of small and medium enterprises, such as restoration companies, installation companies (e.g. lighting installation, conditioning and heating systems, air quality control), packing and transportation companies, small and medium companies which produce technological instruments and measurement devices, and control and analysis equipment, test laboratories, producers and manufacturers of various materials: paints, stones, plastics, glass, paper, mortars, cement, wood composites, archaeological excavations companies or institutions, etc.

The development of standardised test and analysis methods will provide the cultural institutions, enterprises and laboratories with correct instruments for carrying out their work, improving, at the same time their proficiency/competencies.

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The standardisation for the conservation of Cultural Heritage may influence, determining also specific requirements, the production and adjusting of the following kind of equipment, products and devices:

- a) scientific equipment for laboratory and in situ chemical, geological, physical, mechanical and biological tests, measurements and analysis, in particular, for what concerns non-destructive ones, and production of standard reference materials whose compositions match those of cultural material (i.e. ancient alloys compositions), and reference data related to compounds found in degraded material for analysis purposes;
- b) products used in the different steps of the conservation work/treatment, such as cleaning agents, biocides, sealing materials and mortar for restoration, surface protective materials, water-repellent materials environmental friendly, varnishes, glues;
- c) equipment and technologies used during the conservation/restoration work (e.g. nebulizers/vaporisers, micro and macro-airbrasive machines, laser equipment), which are respectful of the professionals' health, of the cultural property and are environmental friendly.

The materials/products, the equipment and technologies used nowadays in the conservation and restoration works, or which are used in diagnostics laboratories, are materials and equipment often produced by multinationals industries with great experience, but these products and devices haven't been studied specifically for conservation or restoration purposes and, for this reason, need to be characterised and require a specific standardisation activity.

Finally, the programme of work of this CEN/TC, while defining the requirements and characteristics of the materials, of the equipment and technologies, can contribute to the improvement of the existing materials and equipment, and support the development of new ones for a more competitive European market.

### **3. Economical factors in relation to the objectives of CEN/TC 346**

The increasing atmospheric pollution, of different origins, causes decay of exposed building surfaces and the identification of environmental parameters and assessment of material-environment interactions is a cost effective way to increase longevity and reduce maintenance costs [4].

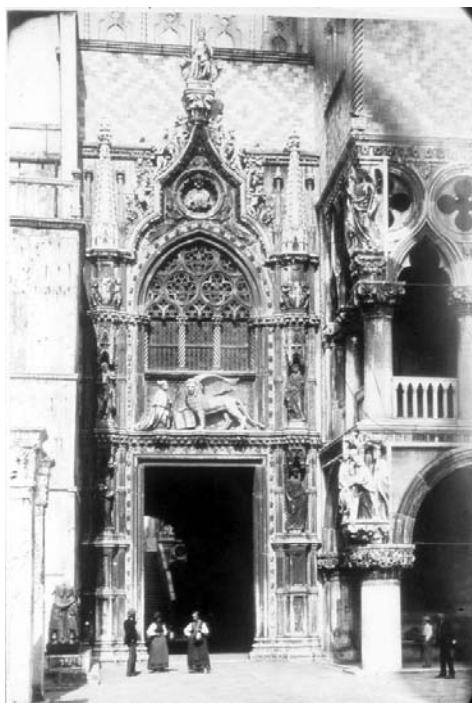
The importance of using correct materials in conservation work is crucial, as experience has shown, for example, that poor quality natural stones used to repair historic structures have in some instances, deteriorated to such an extent that complete restoration has had to be undertaken, costing millions of Euro.

Standardisation in the field of conservation of Cultural Heritage will:

- i) improve the efficiency and pertinence of the diagnosis, reduce their costs, with a subsequent better management of funding for the conservation/restoration works and therefore increasing the number of conservation projects and spin-off economic benefits/opportunities for new investment, and consequent job creation;
- ii) give precise and appropriate indication on the kind of diagnostics studies to be performed, avoiding expensive researches, promoting in this way conservation works on an increasing number of artefacts;
- iii) help to develop and improve products, materials, equipment and technologies to be specifically used for the conservation of Cultural Heritage;
- iv) increase the durability of conservation works therefore reducing costs on a long-term range because conservation operations will be spaced out.

To explain better the advantage of a good standardisation in the field of Cultural Heritage we report an example of a Venetian monument which was restored in 1976 as it was strongly decayed. The Porta della Carta, built in XIV century as the entrance door of Ducal Palace, was preserved in a rather good state for four centuries and the only damage observed were ascribed to the natural weathering agents, as it is clearly visible in the photographic documentation of the last decades of XIX century (figg. 1,2). Comparison between two pictures taken at the beginning of XIX century and after seventy years respectively (figg. 2, 3), shows a sharp increase in the marble decay, ascribed to the contemporary pollution increase occurring in the Venetian district as a result of the industrialization of Porto Marghera, the industrial area, created at the beginning of 1930s [5,6].





*Fig. 1. Porta della Carta in 1875.*

The sharp decay observed in 60'ies induced people in charged in the conservation to recover inside the Ducal Palace museum the four statues to prevent their complete destruction. In 1976 the conservation work of the whole Porta della Carta started, thanks to a financing of the Venice in Peril fund, and it was completed in a three year period. In 2001 in the framework of the National Research Council Project finalized to the Safeguard of Italian Cultural Heritage in cooperation with the Venice Superintendence for Monuments Care a check of the durability of restoration materials used in 1976 restoration work and of the state of conservation was carried out. The results obtained allow us to conclude that the decay processes, which has caused missing of pieces of marbles and disintegration of carved surfaces (as it is possible to observe from the comparison of fig. 2 and 3), since the industrialization until 1976, seems to be arrested or slowed down as it is possible to observe from the comparison of fig. 3 and 4 [7].

We can conclude that the stone treatment is still working after 25 years from the restoration work [8]. We believe that a good standardization process is a useful tool to increase the durability of conservation works therefore reducing costs on a long-term range because conservation operations will be spaced out.

There are also a number of national research programmes funded by EU member states for cultural heritage studies. Italy, for example, has allocated a budget of € 40 million over five years (1997-2002) for a special project on the safeguard of Cultural Heritage.

Since 1986 the European Commission has spent more than 120 million Euros to improve the scientific knowledge in conservation of Cultural Heritage and at the end of 2006 a new Cultural Heritage research programme has been launched: the 7<sup>th</sup> framework Programme. The number of European Universities and research institutions and governmental institutions dealing with basic and innovative research has been increased exponentially in these last two decades.

Let me make some examples: for instances Sweden provides research grants through its Central Board of National Antiquities; the Netherlands announced that cultural heritage will be one of ten research themes for funding by its Organisation for Scientific Research (NWO) over the next three years; the Spanish Ministry of Science and Technology provides funds under the category Civil Construction

and Conservation and Restoration of Cultural Heritage and the UK government funds conservation work undertaken by English Heritage, Historic Scotland and the National Trust.



*Fig. 2. Porta della Carta. Charity statue in 1930.*



*Fig. 3. Porta della Carta. Charity statue in 1978.*



*Fig. 4. Porta della Carta. Charity statue in 2004.*

### 3. Users of CEN/tC 346

Users of the standards developed by CEN/TC 346 are:

- Public and government bodies (e.g. Ministry of Culture and Education, Government Agencies);
- Public national and international non-government bodies (e.g. ICCROM, ICOMOS, IIC, ICOM, Regional administrations, Provincial administrations or local governments);
- Restoration/Conservation schools;
- Ecclesiastical bodies/organisations;
- Public and private analysis laboratories;
- Restoration companies;
- Professionals in the field of conservation and exhibitions planning;
- Distributors and manufacturers of materials used in restoration;
- Companies specialising in the preparation and organisation of exhibitions;
- Transportation and packaging companies;
- Lighting installation companies, air conditioning and heating installation companies, informatics and advanced technology companies;
- Cultural institutions: museums, galleries, libraries, archives;
- Architecture and surveyors;

### 4. Structure of CEN/TC 346 and Work Programme, Chairman Mr. Vasco Fassina (Italy)

The structure of CEN/TC 346 is constituted by 5 Working Groups (WG's), corresponding to the different main areas for which technical development work has to be done. Under each WG the standardisation projects, called Work Items (WI's) will be developed.

#### CEN/TC 346/WG 1 - General guidelines and terminology.

*Convenor: Mr. Lorenzo Appolonia (Italy)*

This WG has the responsibility for the drafting of:

- guidelines on conservation planning, including monitoring;
- standards on terminology dealing with movable and immovable components, with degradation processes and its graphic and symbolic documentation;
- guidelines on security and safety conditions relating to the use of cultural heritage by the public.

The initial programme included the following work items:

WI 001-Terminology

WI 002-Guide to the principles of conservation

WI 003-Condition report of the cultural property

WI 004- Security of cultural property and safety of the public

*Actually the WG1 is discussing the following draft documents:*

*WI 000346002-Main general terms and definitions concerning conservation of cultural property.*

Scope: definitions of general terms used in the field of conservation of cultural property with particular attention to those terms which have wide use or significance.

*WI00346003: Glossary of damage.*

Scope: definition of damage terms determined by macroscopic examination as a first step to diagnosis.

#### CEN/TC 346/WG 2 - Materials constituting cultural property.

*Convenor: Mrs. Vasilike Argyropoulos (Greece)*

This WG has the responsibility to define tests and analyses methods:

- for the characterization of the materials,
- for the evaluation of the state of conservation/preservation of materials

The initial programme included the following work items:

*WI 005-Diagnosis on buildings surfaces and structure.*

*WI 006-Characterization and classification of paint and paintings.*

*WI 007-Characterisation of the state of conservation/preservation of cultural property.*

*WI 008-Characterisation of stone and related building materials.*

Actually the documents in discussion are the following:

- Sampling of cultural property
- Tentative recommendations for the selection of compatible stone for conservation purposes
- Characterisation of stone
- Characterisation of mortars
- Diagnosis of building structures

#### CEN/TC 346/WG 3 – Evaluation of methods and products for conservation works.

*Convenor: Mr. Vasco Fassina (Italy)*

This WG has the responsibility of drafting documents on criteria to select methods and/or products and operating/working conditions in relation to the conservation/restoration, repair, maintenance and preventive conservation works; and of drafting documents on the evaluation of the methodologies to be used.

The initial programme included:

*WI 009-Surface protection for porous inorganic materials-Evaluation of methods and products.*

This document has the scope of giving guidelines on test method and methods of analysis (in laboratory and in-situ) for the evaluation of the performance of the products and of the methodologies to be used in protection of cultural property

*WI 010-Consolidation products-Evaluation of methods and products.*

*WI 011-Cleaning products-Evaluation of methods and products.*

*WI 011bis-Chemical control of bio deterioration-Evaluation of methods and products*

*WI12 Cultural heritage-Conservation of archaeological remains/objects - Evaluation of methods and products.*

*Actually WG3 is discussing the following draft documents:*

*prEN 15801-Determination of water absorption by capillarity (under CEN enquiry).*

Scope : test method to characterise the water absorption by capillarity of porous inorganic materials.

*prEN 15802-Measurement of static contact angle (under CEN enquiry)*

Scope: test method for the measurement of the static contact angle of a water drop of porous inorganic materials.

*prEN 15803-Determination of water vapour permeability ( $\delta$ ) (under CEN enquiry).*

Scope: test method for the calculation of the water vapour permeability (WVP) of porous inorganic materials.

*WI00346007-Colour measurement of surfaces.*

Scope: test method for measuring the surface colour of porous inorganic materials, and their possible chromatic changes due to any conservation work and/or natural ageing or artificial interventions.

#### CEN/TC 346/WG 4 – Environment.

*Convenor Mr. Jesper Stub Johnsen (Denmark)*

WG4 has the responsibility for the drafting of guidelines for the control of environmental variables, and of standards on the measurement of indoor, including exhibition and storage conditions, and outdoor environmental conditions, and on cultural property/environment interaction.

*WI 013-Specifications on environmental conditions*

*WI 014-Guidelines on the measurement of environmental parameters*

Actually WG4 is discussing the following draft documents:

*prEN 15757-Specifications for temperature and relative humidity to limit climate-induced damage in organic hygroscopic materials* (under CEN enquiry)

Scope: general specifications for temperature and relative humidity to limit climate induced mechanical damage of absorbing moisture, organic materials, especially in indoor environments of museums, galleries, archives, libraries, churches and modern or historical buildings

*prEN 15758-Procedures and instruments for measuring temperatures of the air and the surfaces of objects* (under CEN enquiry)

Scope: the procedures for measuring the temperature of the air and of the surfaces of cultural heritage objects in indoor and outdoor environments.

*prEN 15759-Specification and control of indoor environment-heating of historic churches* (under CEN enquiry)

Scope: recommendations for the heating of historic churches in order to prevent damage to the buildings and their contents.

#### CEN/TC 346/WG 5 – Transportation and packing method

*Convenor: Mrs. Anne de Wallens (France)*

This WG has the responsibility for the drafting of standards on methods of packaging and transportation of cultural heritage outside the institutions.

*WI 015 Cultural heritage – Principles of transportation and packing of cultural property*

This standard has the scope of giving a guideline for the choice and the control of the microclimate finalised to the transportation and packing of cultural property

Actually is in preparation an European standard on packing methods

A close co-operation should be established with European Commission Directorate-General for Research as well as European and International non governmental professional organisations dedicated to the conservation of Cultural Heritage, such as International Council on Monuments and Sites (ICOMOS), the European Confederation of Conservator-Restorers' Organisations (E.C.C.O.), or the intergovernmental organization ICCROM (International Centre for the Study of the Preservation and Restoration of Cultural Property).

Recently liaison with the International Council of Museums (ICOM) and the International Institute for Conservation of Historic and Artistic Works (IIC) has been established.

CEN members actually are 30: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Island, Ireland, Italy, Latvia, Lithuania, Luxemburg, Malta, The Netherlands, Norway, Poland, Portugal, Rumania, Slovakia, Slovenia, Spain, Sweden, Switzerland, United Kingdom.

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# THE WRECK OF VROUW MARIA - CURRENT SITUATION AND FUTURE PERSPECTIVES

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## Abstract

The current condition of the almost complete wreck of the 18<sup>th</sup> century merchant ship *Vrouw Maria* lying on the bottom of the Baltic Sea has been investigated and the physical, biological and chemical processes threatening it have been identified. The wood of the wreck appear to be in a good state of preservation and the environment is not immediately threatening to the materials. Due to the good condition of the hull and the artifacts of great cultural historical interest in it, there has been discussion about excavating the wreck or possibly even raising the whole hull.

## 1. Introduction

In 1771 a Dutch merchant ship *Vrouw Maria* was on her way from Amsterdam to St. Petersburg. She passed the Danish straits and according to the entries of the Sound customs house of Denmark she was carrying typical goods for Dutch trade with Russia: sugar, dyestuff, zinc and other metals, cloths, coffee and foodstuffs. There were also art objects and valuable 16<sup>th</sup> and 17<sup>th</sup> century Dutch paintings, which were bought for the Russian Empress Catherine the Great from the auction of the estate of art collector Gerrit Braamcamp. [1]



Fig. 1: Cat-head in the bow. Photo: Jouni Polkko

Unfortunately, in a stormy night *Vrouw Maria* hit rocks in the South-western archipelago of Finland and eventually, after several days of salvage attempts, sunk. There had been time to salvage parts of the cargo, but the majority of it went down, including many of the paintings. Diplomatic correspondence between Russian and Swedish authorities on the lost ship and its cargo started immediately. There were attempts of salvaging the paintings, without success, and eventually the wreck was forgotten. [2]

The ship's story started emerging again in 1970's through archive research. A Finnish researcher of maritime history, Dr. Christian Ahlström, found diplomatic letters concerning *Vrouw Maria*, and brought the story to the wider public. Thus *Vrouw Maria* became a legendary treasure ship and was searched for extensively, in vain for the time being. She was finally found with side scan sonar in 1999

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by the Pro Vrouw Maria association established particularly for the purpose. In authorization and supervision of the National Board of Antiquities of Finland six artifacts were raised for confirming the identification of the vessel. [3]

Since then the wreck has drawn wide attention. Research has been done by the Maritime Archaeology Unit of the National Board of Antiquities of Finland and samples have been taken and analyzed, but excavations have not taken place. Currently there is a lively discussion about the future of the wreck, one of the possible scenarios being to raise and conserve the hull and its contents.

## 2. The wreck and its cultural historical value

The wreck is located in the outer archipelago of South-Western Finland in the waters administered by the Metsähallitus, the Finnish government land authority. It is protected by the Antiquities Act, according to which a circular exclusion zone with a diameter of 1500 meters has been established. In addition and very significantly, the wreck happens to lie within a so-called “strict preserve” part of the Archipelago National Park. This is bound to affect any actions being planned in the wreck-site, possibly even banning any drastic actions. [4]

The wreck lies at a depth of 41 meters on an even depression surrounded by rocky skerries, on a bottom consisting of clay topped by sand and gravel. It lies evenly on its keel, almost completely exposed, listing slightly to starboard. The hull is carvel-built and made of oak, with a length of 26 meters and beam of seven meters. It appears as exceptionally well preserved with about 90 % of the hull preserved and the lower sections of the two masts remaining in place. [5]

Archival sources classify Vrouw Maria as a snow-ship, meaning that it had certain type of rigging. It was a relatively small ship of modest outlook built for practical purposes. Still, there are some deck fixtures adorned with vine patterns and other decorations. Inside the hold, most visible items are hundreds of clay pipes scattered on the rest of the cargo. Zinc ingots, packing crates and barrels can also be seen, but none of the Empress Catherine’s art objects have been seen. [6]

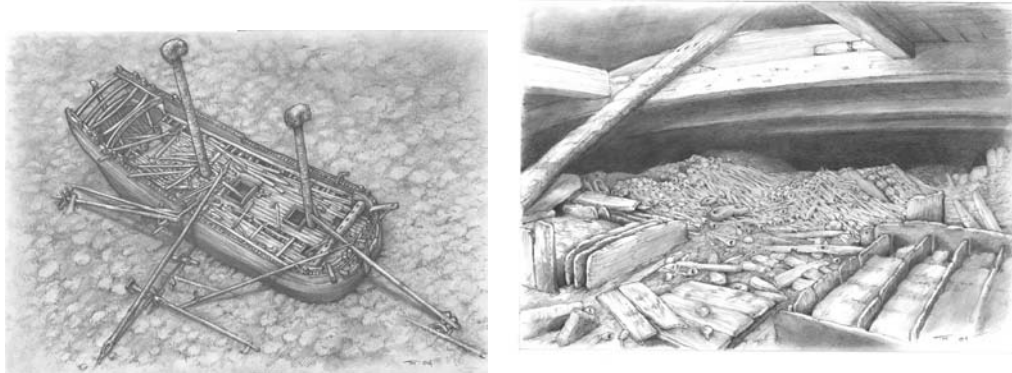


Fig. 2 and 3: Artist interpretations of the wreck and the hold.  
Both drawings: Tiina Miettinen, the National Board of Antiquities of Finland

The Vrouw Maria report published in 2007 by the Maritime Archaeology Unit of the National Board of Antiquities of Finland states that based on the excellent state of preservation, on the wide variety of its contents and on the extensive range of historical data, the wreck is of major cultural historical value on the national and international levels. It is a link in the long history of East-West merchant route in the Baltic Sea. While snow-rigged ships were common in the 18<sup>th</sup> century, this is the only known wreck of such a ship in Finland and the only comparable wreck known elsewhere is the Sjöhästen in Sweden, which is a warship and thus differs from Vrouw Maria. [7]

## 3. Marine archaeological wood

Dead wood in any environment is naturally and unavoidably attacked by biological organisms, and there are also threats of chemical and physical kinds. The actual sinking moment means a very dramatic change of environment and is prone to cause severe reactions in materials. The typical



behavior pattern would be that the degradation reactions accelerate as the materials enter marine environment, but slow down considerably as the materials reach some kind of equilibrium with the new conditions.

As for physical factors, of importance is whether the wood is exposed to seawater or if it is buried in the marine sediment. Often the marine environment is dynamic and physical processes such as scour and sediment movement are potentially threatening wrecks both directly and through destabilizing the environment at the site. [8]

The main causes of deterioration of wood in marine conditions are biological organisms. The wreck will be progressively colonized by a variety of these, the ability of which to survive and destroy wood depends on the environmental conditions such as salinity, temperature and the dissolved oxygen content. The processes start with the attachment of bacteria to the surfaces and from there further inside the wood. In marine conditions these can be erosion-, tunneling- or cavitation-bacteria, erosion bacteria being often the dominant ones due to their ability to survive in conditions of low oxygen levels. Then other micro-organisms, such as fungi, algae, boring crustacean and mollusca can start colonizing on the surfaces. Of these, the marine wood borers, such as the “shipworm” *Teredo navalis*, are by far the most damaging due to their capacity to destroy archaeological evidence, whole wrecks even, in relative short periods of time. Fortunately these can not withstand the conditions of the northern Baltic Sea. Less dramatic but in our case more relevant agents of deterioration are fungi, particularly soft rot fungi. [9]

As for other factors affecting the preservation of marine archaeological wood, it is important to note that different substances can and will accumulate in the wood from the environment and from adjoining materials, iron and sulfur species being perhaps the most significant ones. This is more a post-conservation problem, because basically what happens is that accumulated sulfur and iron compounds, if not removed within conservation, cause chemical reactions which eventually form sulfuric acid on the wood. This endangers long term preservation of conserved wood, as is demonstrated by the reactions reported in the wood of the Vasa in Stockholm, Sweden. This complex issue is certainly a major topic in marine archaeological conservation at the moment. [10]

Research and monitoring of the environment and analysis of artifacts is needed to evaluate the prevailing conditions and to interpret the behavior of materials at the site. To complement the picture, modern reference samples can be placed in-situ and analyzed regularly. Together with the analyses of the actual artifacts and monitoring of the environment this should clarify the deterioration processes and also potentially indicate possible environmental changes. [11]

If marine archaeological wood is to be brought up for conservation, it comes in a variety of states of degradation. It can be very soft, almost bread-like, or sound and apparently well preserved. Often the deterioration leaves wood with a heavily degraded surface layer and a sound well preserved inner core. For this kind of wood a conservation procedure with a suitable mixture of stabilizing agents that stabilizes all states of degradation has to be designed, and then there are iron corrosion products and other alien substances that have to be borne in mind. Iron generally corrodes readily in the Baltic Sea. This distributes troublesome corrosion products all over the place and also weakens structures by dissolving binding bolts and nails away.

#### **4. The surroundings and the condition of the wreck**

Against this background, what is Vrouw Maria’s situation? To start with, it is in the Baltic Sea, which is not a sea but a brackish water basin. It is a mixture of saline ocean water coming through the narrow Danish Straits and fresh water from numerous rivers, so the salinity of water varies considerably depending on the location and season.

The wreck and its surroundings were investigated in 2001-2004 in an EU Culture 2000 programme project called MOSS (Monitoring, Safeguarding and Visualizing North-European Shipwrecks). Data about environmental factors such as pH, dissolved oxygen, temperature and turbidity was monitored by different data-loggers, and a more particular monitoring was done between September 2002 and August 2003 by the Finnish Institute of Marine Research. It turned out that the conditions at the wreck site are typical for the area in terms of salinity, temperature, stratification and dissolved oxygen content. Temperature at the wreck varied between -0,4 and 3 degrees in the winter and even in

summer rarely exceeded six degrees. The salinity at the bottom varied between 5.5 and 6.6 ‰. The pH of the water varied between 7.03 and 7.16. Currents outside the hull were on average weak, less than 4cm/s. There are periodical changes, though. At the start of the period the level of dissolved oxygen was low, even below 1ml/l and the temperature app. 5.3 degrees Celsius, until on 23<sup>rd</sup> October thermocline broke and the water was mixed from surface to bottom. After that the oxygen content jumped to about 7,5ml/l, the temperature increased nearly 3 degrees and the salinity decreased app. 0.9 ‰. The mean current velocity also increased. After that the temperature started decreasing towards winter conditions, but the salinity remained high until the spring, when the water column started stratifying again. [12]

As for biological factors, there is no marine vegetation obscuring the hull. Fungi and bacteria certainly are present, but wood-boring mollusks have not been observed. This was to be expected due to the fortunate fact that wood borers do not survive in such low salinity levels as the site has. One wood sample was recently studied at the University of Helsinki. In his master's thesis to the department of Biological and Environmental Sciences Veijo Kinnunen used light- and scanning electron microscopy to study the condition of the cell walls and to search for signs of microbial attacks. Elemental analyses (SEM-EDS) was used to analyze the sulfur and iron content of wood. DNA isolation and PCR with several primers for bacteria and fungi were also utilized in collaboration with Leone Montonen from the Department of Applied Chemistry and Microbiology.

The species of the sample turned out to be Scots pine (*Pinus sylvestris*), not oak, which should be the principal species used for the hull. The sample appeared to be in a good condition with only a thin surface layer (3-5mm) of very soft degraded wood. Wood residual density was 77%, meaning that the sample was 23% lighter than recent Scots pine on average. As was expected, there were no borers' cavities to be seen and fungi and bacteria turned out to be the principal causes of degradation. At the surface layers soft rot fungi had caused most of the degradation, but also some signs of bacteria were noticed there. Deeper in the wood the degradation was less pronounced and mainly caused by erosion bacteria, which seem to have been able to penetrate into the wood via rays. There were also some signs of fungi spreading deeper in the wood. [13]

According to elemental analyses both sulfur and iron had been accumulated in the wood. It seems that sulfur comes not only from the environment but is enriched in the wood also within anaerobic bacterial activity. This is suggested by the detected DNA of sulfate reducing bacteria at the surface of wood sample. Sulfur concentration at the first 10mm of wood was on average 0,58 mass% with the highest of 1,31 mass%. Iron probably comes mainly from the corrosion of an iron object nearby or within the sample-wood. The concentration of iron was on average 0,54 mass%. [14]

Although this one sample can not be seen as characterizing the whole hull, the study reveals that for now the most efficient wood destroyers – white rot, brown rot or wood borers – are not present in the wreck site. The sample with only a few millimeters of badly degraded wood on the surface and signs of bacterial degradation deeper shows typical deterioration for such wood in this particular Baltic Sea environment. Information of great importance is also the data about accumulation of sulfur and iron inside the wood. While the concentration of sulfur is not nearly as high as it is in the wood of Vasa, it is definitely something to take into consideration, and for sure there is plenty of iron present all over the hull. These are major factors in planning of possible conservation of the hull or individual artifacts. [15]

Analyses should be continued with more samples to determine the overall condition. In addition, analogous samples should be used to monitor the development at the site. During the MOSS-project, such samples were placed in the vicinity of the wreck, both in aerobic and anaerobic conditions. Some of them have been analyzed while many remain in place for future analyses. Samples brought up were studied with light microscopy, scanning electron microscopy and x-ray within intervals of 3 and 12 months. Bacteria and fungi, more particularly soft rot fungi, were found, but the effect was not dramatic after 12 months. As expected, marine borers were not found in these samples either. [16]

## 5. Future perspectives

It seems that the various physical, chemical and biological processes are slow and we have good reasons to believe that the hull is in a good state of preservation. Despite the yearly cycle of condition

changes caused by water stratification breaking periodically, for now the environmental conditions are relatively favorable for long term preservation of the wood of the wreck.

Nevertheless, although the deterioration pattern seen in the actual sample and the analogous samples seem to correlate and there is a good understanding of the environmental conditions, we do not have a complete picture of present state of preservation of the hull, nor of the deterioration patterns. The overall interpretation of all the existing data has not been done properly yet and more samples are needed particularly from the hull. These have to be analyzed together with the analogous samples. Significantly, the overall situation of accumulated iron and sulfur in the wood has to be clarified. And it has to be remembered that there is not only wood down there. The condition evaluation of the iron parts is so far based mostly on hands-on experiences rather than on research of scientific standards. For all we know the iron fastenings might have been rusted largely away leaving the whole structure fragile and unable to withstand stresses such as the possible raising would cause. There is not enough information about the condition of the artifacts either. The hold is largely intact and it is intended to remain so to keep all the artifacts there undisturbed until clear decision are made for excavations or other actions.

Now research and monitoring of the wreck should be continued. There are several ways to proceed. One would be to leave the wreck as it is and monitor it properly and take protective actions should there be reasons for those. Another option would be to carry out proper excavations, raising and conserving the artifacts from the hold. The third option would be to raise the whole wreck and make it a museum exhibit. Although in-situ preservation would be in accordance with the current international conventions and statutes, valid arguments for the other options have been raised too. Therefore I now conclude the article by discussing the possible interventive actions.

If the wreck is to be raised, the fundamental problem is the fact that it is a large composite object made of several contrasting materials. Conservation methods needed for organic materials are basically damaging to metals and vice versa. Conserving and storing wood and metals separately is straightforward enough but together they form a tricky combination. For instance, iron has readily corroded and fused tightly on and in the wood, spreading corrosion products everywhere in the wood matrix. Furthermore, plenty of other substances, such as the mentioned sulfur compounds, have accumulated in the materials. It goes without saying that very dramatic deterioration reactions would take place rapidly if the whole entity is brought up.

How to preserve it then? Mimicking and maintaining the underwater environment already familiar to the wreck would be one option, and stabilizing the wreck to withstand new kind of conditions another. The first option refers to storing and displaying the wreck in some kind of an aquarium – which was actually suggested to us – and the latter option to more conventional interventive conservation actions that would stabilize the wreck and display it dry.

In theory not actively conserving the wreck but keeping it in an aquarium replicating the underwater environment could avoid the sulfur-problem and other difficulties of conserved wrecks, but there are some serious questions marks, at least considering biological problems threatening the materials and ruining the visibility in the aquarium. It is hard to keep the water clear and artifacts stable even in a small tank in conservation laboratory. Many methods have been tried to prevent these biological problems, but none seems to be satisfactory in such a large scale and in a situation where divers should be able to go into the aquarium.

One interesting approach was investigated in a recent article by Charlotte Björdal et al, which discusses the possibility of storing and displaying marine archaeological wood in a sealed aquarium in anoxic conditions. According to this study, the method works and water should remain clear as far as the material inside is solely wood, but the presence of iron brings up preservation and visibility problems. The article concludes that so far wrecks containing iron can not be stored this way. [17]

Another approach after raising the wreck would be to conserve it as has been done with the *Vasa* for instance. This would certainly be a very difficult task, mainly because of the presence of contrasting materials and the mentioned accumulation problems. The sulfur-iron issue is still to be solved, although good progress is being made in the *Vasa* project and elsewhere. It seems established now, that iron has to be extracted from the wood-matrix prior or within conservation, which is a difficult

and time-consuming process no doubt. Research is needed, but even so this option seems more realistic than the aquarium plan.

Not raising the hull but protecting it *in situ* and excavating the artifacts could be initiated by the high historical and artistic values of the contents of the hold, especially the art objects. Starting such a project for the sake of raising something as delicate as the paintings, which most likely have not survived well or at all, would definitely call for a thorough ethical debate. Nevertheless, it has to be done properly fulfilling all the archaeological standards and securing full conservation resources. In practical view, bringing up individual artifacts rather than whole wreck would certainly be easier.

Generally, whatever is decided to be done, the costs will be great, especially if the wreck is raised, in which case they will be enormous, both in the beginning and during the coming decades. The time perspective is very important. Problems will follow if funding and personnel are not secured for many decades. This would also be an international project, and negotiations for collaborations with Russians are currently taking place. What happens now largely depends on resources available, and for now this issue – which in the end is the vital one – unfortunately remains unclear.

### Acknowledgements

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# ROMANIAN ARCHITECTURAL WOODEN CULTURAL HERITAGE – THE PRESENT STATUS - A SURVEY

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## **Abstract**

In Romania there is a great wealth of wooden objects - from village churches in North (Maramuresh), wind- and watermills up to beautiful Greek Orthodox iconostases – wooden religious panel paintings belonging to the Cultural Heritage of our people. A brief description of this architectural wooden cultural heritage, mainly presented in two open-air museums – Muzeul Satului (Village Museum) in Bucharest and Astra Museum near the Transylvanian city of Sibiu - and in Maramures villages is presented. The importance of preservation of traditional techniques for wood “repairs” (carpentry with ancient tools, special timber items) is outlined. A short description of Romanian dendrochronological multidisciplinary research – is also presented.

## **1. The Romanian architectural wooden cultural heritage**

In Romania is a great wealth of wooden objects - from small but tall (bellfries up to 54 m !) village churches in North (Maramuresh), popular technique “machineries” as wind- and watermills up to beautiful Greek Orthodox iconostases – wooden religious panel paintings belonging to the Cultural Heritage of our people [1,2]. A brief characterization of the conservation-restoration specific problems will be given for some of the most representative wooden churches and ancient village houses as presented in two big open-air national popular art museums – Muzeul Satului (Village Museum) in Bucharest and Astra Museum near the Transylvanian city of Sibiu.

Founded by Royal Decree in 1936, and covering some 15 hectares on the shores of Lake Herăstrău, Muzeul Satului [3] is one of the greatest outdoor museums in the Balkans. There are more than 300 original houses, farmsteads, windmills, watermills and churches from all of Romania's historic regions: Transylvania, Oltenia, Dobrogea and Moldavia. Every exhibit has a plaque showing exactly where in Romania it was brought from. Some even now have recorded commentary in four languages. Most of the houses date from the mid 19th-century, but there are some, such as those from Berbești, in the heart of Romania - celebrated for their intricately carved entrances - which date from as early as 1775. The highlight of the museum is probably the steep belfry of the wooden Maramureș church, complete with exquisite but faded icons. It was created in 1936 by Dimitrie Gusti, Victor Ion Popa, and Henri H. Stahl. The museum is organized as a village, with clusters of houses belonging to different areas of the country. They are completely furnished, have gardens and winding paths around so a visit here is like going back in time, visiting real old time rural areas.



Fig. 1 Peasant house – Village Museum, Bucharest

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The "ASTRA" Museum of Traditional Folk Civilization (Romanian: *Muzeul Civilizației Populare Tradiționale "ASTRA"*) is located in the Dumbrava Forest, 3 km south of Sibiu, on the road towards Rășinari, and is easily accessible by car, bus or tramway. Occupying an area of 0.96 square kilometres, it is the largest open air museum in Romania and one of the largest in Central and Eastern Europe. It contains houses and workshops of the traditional Romanian folk culture from the pre-industrial era. Over 300 houses and other buildings are situated in the forest around two artificial lakes with over 10 km of walkways between them. The exhibits are organised into six thematic groups: food production and animal husbandry, production of raw materials, means of transportation, manufacture of household objects, public buildings, an exposition of wooden monumental sculpture. Some of the most spectacular buildings are a group of windmills from the Dobrudja area, a playing area for popice (skittles, an early form of bowling) from the Păltiniș monastery, a small mine from the Apuseni Mountains, a few water-mills, a wooden ferry, and a fishery from the Danube Delta. Also there are houses of shepherds, pottery workshops, iron workshops and others. There is also a working inn, a small pub and a dance pavilion. In the museum there is a wooden church from northern Transylvania brought in 1990-1992 from the village of Bezded in Sălaj County.

The main conservation-restoration specific problems for the wooden churches and ancient village houses presented in these two big open-air national popular art museums are wood preservation, wood moisture relationships and the temperature-moisture and time dependence of wood material properties. The difference between the sub-mountain (forest) humid and cool environment of Astra Museum and the hot and dry plain – urban environment of Romania's Capital is essential, including the influence of anthropic air pollution more dangerous in Bucharest – a big industrial city – than near Sibiu – a historical touristic town. Unfortunately, these specific studies are only started in the two museums, due to their difficult financial problems.



Fig. 2 Watermill, ASTRA Museum

The biggest and most valuable Romanian wooden cultural heritage are the famous wooden churches from Maramures [4]. Maramures is a region situated in the northern range of the Carpathian

Mountains in Northern Romania. The Region is enclosed by high mountains and communicates to the outside world through the narrow Tissa Gate and through several mountain passes. The area is 10,000 sq. km. and it has over 280 villages and towns. In 1900, forest covered 90% of the Maramureș area. The abundance of timber combined with the skill and craftsmanship of the local people brought the area the name of "land of wood". The region is famous for the large number of well preserved wooden churches. Estimated number of wooden churches built before 1850 is 94. There is a strong tradition of building wooden churches right across Eastern Europe, from Karelia and northern Russia all the way to the Adriatic, but in terms of both quality and quantity the richest examples are in Maramureș. From 1278 the Orthodox Romanians were forbidden by their Catholic Hungarian overlords to build churches in stone, and so used wood to ape Gothic developments. In general, the walls are built of blockwork (squared-off logs laid horizontally) with intricate joints, cantilevered out in places to form brackets or consoles supporting the eaves. The tradition of building wooden churches in central and southern Maramureș can be traced from the beginning of the 16th century to the turn of the 18th century. Since the knowledge used to build the local wooden churches circulated throughout Europe, their understanding is of high interest far outside the region. In Maramureș today 42 wooden churches remain, about one third of their total two centuries ago. Besides the extant wooden churches, a major source of knowledge is still saved by a number of practicing senior carpenters with relevant knowledge and skills in traditional carpentry. Maramureș is one of the better-known regions of Romania, with autonomous traditions since the Middle Ages - but still not much visited. Its well-preserved wooden villages and churches, its traditional lifestyle, and the local colourful dresses still in use make Maramureș as near to a living museum as can be found in Europe. The famous wooden churches of the region were built during the 17th and 18th centuries, on the place of older churches.



Fig. 3 Maramureș wooden church during and after repair

They are a response to a prohibition against the erection of stone Romanian Orthodox churches. The churches are made of thick logs, are quite small and dark inside, and painted with rather "naïve" Biblical scenes. The most characteristic features are the tall tower above the entrance and the massive roof that seems to dwarf the main body of the church. Some of them have been listed by the UNESCO as a World Heritage Site in 1999, for their religious architecture and timber construction traditions.

From the Middle Ages until the turn of the 18th century the skills, knowledge and experience to build ample log structures with plane and well sealed walls, as well as with flush joints, were performances out of the ordinary. The craftsmen from Maramureş who were able to reach such levels were not simple peasants but well specialised church carpenters who inherited and maintained this advanced knowledge to exclusively build houses of worship. Since the local tradition to erect wooden churches depended on those who built and used them, it is fundamental to identify the local builders and founders. The earlier blurred distinction between them veiled their separate roles in shaping the wooden churches and hindered us from a clear understanding of the results. The extant wooden churches from Maramureş reveal the existence during the 17th and 18th centuries of at least two main family schools of church carpenters. There are further distinguishable three main itineraries and numerous smaller ones, indicating the work of some of the most important church carpenters ever active in the region and in some cases even shifts among generations. In general, the church carpenters stood for the technical performances, the high quality of the wood work and the artistic refinement. In a long perspective, the true creators of the local wooden churches were actually the commissioning founders. Especially the role of the noble founders of Eastern Christian rite was decisive in the formation of a regional character among the local wooden churches. The wooden churches from Maramureş closely mirror the local society of modest country landlords, manifesting themselves along several centuries in their double condition of Eastern Christians and Western nobles.

The wooden churches from Maramureş open necessary connections with similar performances throughout Europe. Seemingly the local distinction made between sacred and profane rooms was characteristic for many other rural regions on the continent. The highest knowledge in log building seems to have had a sacred purpose with wide continental circulation and therefore in many places requires distinction from the more regionally rooted vernacular one. The most elaborate structures of the Maramures-region are the wooden churches, mostly built during the eighteenth century when this Gothic-inspired architecture reached its height. Originally founded upon huge blocks of wood rather than stone, they rear up into fairytale spires or couch beneath humpbacked roofs, and are generally sited on the highest ground in the village to escape seasonal mud. These churches are outstanding examples of a range of architectural solutions from different periods and areas. They show the variety of designs and craftsmanship adopted in these narrow, high, timber constructions with their characteristic tall, slim clock towers at the western end of the building, either single- or double-roofed and covered by shingles. As such, they are a particular vernacular expression of the cultural landscape of this mountainous area of northern Romania. The wooden churches are a distinct feature of Maramures. Here the natives' woodcarving talent shows up in a display of beauty and creativity. These churches are built in the 17th and 18th centuries without the help of any iron nails, yet reach impressive heights. In fact, here you will find the highest wooden building in Europe (some say in the entire world) - the church of Surdesti, reaching 72 m (236 ft).

The Maramures wooden churches represent the beautiful synthesis of the major architectural elements of Eastern and Western Europe, more precisely a synthesis of the Byzantine plan and the Gothic forms rendered according to an original autochthonous architectural interpretation. The constructions are made out of wood, in conformity with the Blockbau system, according to the traditional technique, which denotes a perfect knowledge of the material. They represent a particular type of construction in the context of the wooden churches of Romania, implicitly of Europe, by:

- the higher naos, and pronaos (and porch, if any), which involved the emergence of the double roof, resulting in a larger church;
- the naos topped with a bell tower; here the Gothic influence is obvious, while technically this original solution is remarkable; the largeness and artistry of this architectural form have given to the churches an unmistakable outline. The intricate structure is remarkable, as every element has a double role, constructive and decorative. The sizing denotes a sure sense of proportions; the inner space is conceived at a human scale, while resting imposing. The interior painting, the cult objects, heighten this effect. The double roof, the horizontal lines of the registers, are new elements that have considerably changed the architecture of the façades. Like the bell tower, that defies the laws of statics, they give to the church a dynamism challenging the robustness and stability of the base.

All these elements converge towards the impressive monumentality of these churches, considered to be true wooden cathedrals.



The preservation of traditional techniques for wood “repairs” (carpentry with ancient tools, special timber items, impregnation – tar-pitch layers) of this extraordinary art, historical and religious monuments is very important.



Fig. 4 Church under repair

## 2. Dendrochronological research

An example of international cooperation in the field of dendrochronology is the project on Maramures wooden churches performed by Architect Alexandru D. Babos, at Lund University, Department of Architecture, Sweden, together with Dr. Ólafur Eggertsson, Senior Lecturer and Head of Laboratory for Wood anatomy and Dendrochronology, Lund University, Department of Quaternary Geology, project performed between 1995 and 1999 [5]. Alexandru Babos has been carrying out fieldwork in the area since 1995 for the architectural studies. The dendrochronological fieldwork was carried out during October 1997, 25 churches were sampled, and an average of 18 samples was collected from each church. Samples from living oak trees were collected from two different stands. Because no tree-ring chronologies are available from the region of Maramures, this project is therefore a pioneer work for the dendrochronologist. Samples had to be collected both from living trees and from younger buildings to be able to date the churches. But surprisingly when the first living tree-ring chronology had been established, covering the period 1997 to 1730, it gave quite high correlation with the South German Chronology and when two churches had been measured and the curves had been averaged they could be dated with the same chronology. The following chronologies have been established:

- A living tree-ring chronology: 1720-1997 (10 samples)
- Chronology from the Churches: 1406-1670 (50 samples)

Preliminary dendrochronological dates of some churches are:

- Calinesti-Caeni: felling of timber: winter: 1628-29
- Breb: felling of timber: winter: 1621-22
- Valea Stejarului: felling of timber: 1657 +/- 5 years
- Barsana Jbar: felling of timber: 1681 +/- 5 years
- Cornesti: felling of timber: 1505 +/- 5 (the old chancel), 1608 +/- 5 (the old church), 1650 +/- 5 (tower and possible reparation)
- Rona de Jos: felling of timber: 1639 +/- 5

- Harnicesti felling of timber: winter 1678-79

In the last two years, in Romania some studies are also started as a cooperation between ICAS - Forest Research and Management Institute, Research Station for Norway Spruce Silviculture Suceava (Ionel Popa and Cristian Sidor – dendrochronology, Olivier BOURIAUD – dendroclimatology) and TDL - The Szekler Museum of Ciuc,

Transylvanian Dendrochronological Laboratory (Botár István). ICAS has expertise in Lintab and Lignovision systems, dendrochronology and wood dating, wood anatomy laboratory and wood identification, and TDL in dendrochronology, techniques to measure tree rings, dendroarcheology – wood dating. They started two projects: Archaeology – medieval settlement history and material culture (dendrochronology – historical roof constructions) and Medieval ecclesiastical architecture. They also have good connections with Art, Archaeological and History Museums. Next year, the first results of the projects will be published.

### 3. Conclusions

Despite the very precious Romanian architectural wooden cultural heritage – especially churches and peasant houses -, due to the financial difficulties, until now the main activity for its preservation was an empirical one: repairs and reconstruction of damaged items. Only in the last years we could speak about a scientific approach of the conservation – preservation problems, mainly through the specialized laboratories belonging to Village Museum in Bucharest and Astra Museum in Sibiu. Also the dendrochronology activity effectively started in our country in the last few years. Next year, we hope to have finalized the first projects in the field and to make public the results acquired.

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# RESSURREIÇÃO DE CRISTO – PANEL FROM THE XVI CENTURY, MUSEU DE ARTE SACRA DA SÉ DE ÉVORA. A REPAINTED PAINTING – DATING AND TREATMENT (1974-2008)

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## Abstract

The damaged painting came to the ex-Instituto José de Figueiredo, in the year of 1974, with extensive problems of lack of adherence of the paint layer as well as lacunae and repaints. Concerning the cleaning of the painting, in the seventies the restorers choose to remove the repaints, except for some areas, where no original paint was kept under the overpaint. The stabilization of the paint layer was a problem through years. Now a compromise solution was undertaken: the masticage, the levelling of the lacuna by adding a filler, and the retouching of the lacunae, the areas where the paint is lost, have not been total (Fig.1). The support panel was identified as oak, and a dendrochronological study, based on tree-ring measuring, was done.

## 1. Introduction

Only few works of art come to us, trough centuries, untouched. So, when they arrive to the conservator-restorer, it has not only the natural alterations of the materials but all the accidents and repairs that have happened to it during the years.

Conservation or restoration? Today's question of deciding between conservation or restoration was not always an issue. Earlier, if a painting was damaged, it was repaired, repainted or restored. When the painting *Ressurreição de Cristo* came to the ex-Instituto José de Figueiredo, in 1974, the painting had extensive problems of lack of adherence of the paint layer as well as lacunae and repainted areas (Fig. 2).

Concerning the cleaning of the painting, in the seventies the restorers choose to remove the repaints, except for some areas, where no original paint was kept under the overpaint.

The stabilization of the paint layer was a problem for many years. The masticage and the retouching of the lacunae have not been total - a compromise solution was chosen in the seventies. An additional problem for the conservator-restorer today, was that the painting was used as an object for practical exercise for students in conservation/restoration with the result that the retouched areas differed over the painting. To finish the work and give it back to the Museum a revision not only of the masticage but also a "uniformization" of the final retouching were necessary .

Dendrochronology is an important method of dating using tree rings measurements. It dates the wood, not the painting, but if the wood is newer than the painting, we can say without doubt that the painting is more recent than its attribution.

## 2. Methodology

### 2.1. Conservation

The painting arrived with many problems: burned by candles (Fig. 3), blistering on the pictorial layer (Fig. 4), wet rot with cuboidal cracking (Fig. 5), fungi on the painted surface (Fig. 6) and damages by woodboring anobiid beetles (Fig. 7). A barb, an accumulation of the ground layer due to an original frame still existed. (Fig.8) The barb has nothing to do with the actual frame.

The support has been consolidated, the masticage and the retouching of the lacunae have been made in some places (Fig. 9), the pictorial layer was fixed and cleaned, and all the painting has been treated with fungicide.

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## 2.2. Dating of the painting

The boards of oak were prepared and marked to see the rings (Fig. 10). Then the rings were measured with a manual magnifying glass with a scale inside (Fig. 11). With the obtained values, graphs are made on paper. The various graphs are compared to see if the boards are from the same tree (Fig. 12). The values are also processed by a computer program (Fig. 13) to match with a master chronology of the same geographic origin. The oak of the Portuguese panels generally came from the Baltic region, so we can date them, matching with the chronologies of this region, using the TsapX program (Fig. 14). The obtained value of the latest ring is an exact year. But to date the panel we must add the value of the sapwood rings (9-36, median 15) and a storage time (2 to 10 years). So the value we add is a statistical figure.

In this painting “Ressurreição de Cristo”, made of six boards, we measured four, the other two where not in good condition. As we can see (Tab. 1), 1551 is the more recent and an exact year. Adding the necessary parameters (sapwood 15 years and storage time 2 years) to this value we obtain the probable year 1568, after which the painting was made.



Fig. 1 – “Ressurreição de Cristo” after treatment.



Fig. 2 – “Ressurreição de Cristo” before treatment.



Fig. 3 – “Ressurreição de Cristo” – Detail from the inferior left corner, burned by candles.



Fig. 4 – Blistering on the pictorial layer.



Fig. 5 – Wet rot with cuboidal cracking.

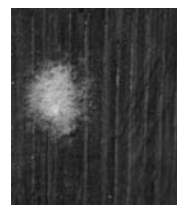


Fig. 6 – Fungus on the surface.



Fig. 7 – Damages by woodboring anobiid beetles.



Fig. 8 – Barb - accumulation of the ground layer due to the original frame.



Fig. 9 – The masticage, as well the retouching, was partial.

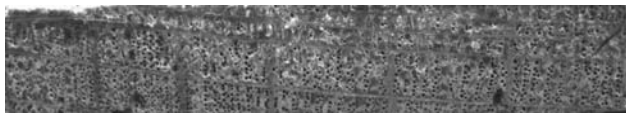


Fig. 10 – Wood of one panel marked to the tree-rings measuring.



Fig. 11 – Measuring consecutive rings using a manual magnifying glass with a scale inside.

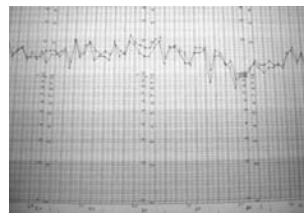


Fig. 12 – Graphic of two panels measures superimposed, where we can observe its analogy and to conclude that the panels are made with wood from the same tree.



(a) (b)

Fig. 13 – Graphics (a) and datings (b) obtained by informatics methods, for one of the panels of this painting.



Fig. 14 – Match of the dates with TsapX program.

Table 1: Dates obtained from measuring the boards.

Boards	Obtained values
Board I	Not measured
Board II	1377-1549
Board III	1454-1546
Board IV	Not measured
Board V	1378-1551
Board VI	1382-1550

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# ON 18<sup>TH</sup>- AND 19<sup>TH</sup>- CENTURY SACRISTY FURNITURE IN THE MALTESE ISLANDS: MATERIALS AND TECHNIQUES

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## **Abstract**

Most of the sacristy furniture in Malta and Gozo dating to the eighteenth and nineteenth centuries has been investigated to evaluate differences and similarities in style, manufacturing techniques as well as types of wood used. The majority of sacristies under study belong to the eighteenth century. Classical decorative elements were more common in the eighteenth century, although some characteristics persisted also in the nineteenth century. Framed door constructions developed and became common during the nineteenth century. Analyses showed that no wood came from local trees. The dominant types of wood used were *Larix decidua* (larch) and *Picea sp.* (spruce). The latter was mostly used for the construction of carcasses, drawers and doors, especially when the furniture was painted utilising a graining technique which was quite popular in eighteenth-century sacristy furniture.

## **1. Introduction**

### **1.1. Aims of the study**

The principal aim of this study is to research and investigate the different styles and manufacturing techniques used in sacristy furniture found on the Maltese Islands. Attention is mainly focussed on wood, being the main constituent material, and the different techniques applied during the eighteenth and nineteenth centuries. Wood analysis has been carried out on a considerable number of sacristies to explore whether wood from local trees was used for construction purposes as well as to outline and highlight the different types of wood used.

### **1.2. General remarks**

Some sacristy furniture presents excellent examples of grand style artefacts. Such furniture has an important role in daily ecclesiastical functions apart from being of utmost historical importance. The furniture that was earmarked for this study came from 33 different churches in total; 44 different pieces of sacristy furniture have been examined. Some churches have more than one sacristy and these are often referred to as the *old* and *new* sacristy. Unfortunately in some locations the original sacristy furniture has been replaced and discarded completely.

## **2. Comparative study of materials and techniques used in the sacristy furniture**

### **2.1. Indigenous and cultivated trees in Malta and their probable use in furniture decoration and construction**

Plants growing on the Maltese Islands adapted to the long dry season and the two most dominant species of trees are the *Quercus ilex* (oak) and *Pinus halepensis* (pine). [1] Lanfranco states that woodland in Malta was exterminated soon after man settled here, about 7000 years ago, as trees started being cut down for firewood and ground was cleared for agriculture.

*Tetraclinis articulata* (Vahl), known as sandarac, was used for decorative tabletops during the Roman period. [9] Sandarac may have also been used for its resin, more specifically for the prized varnish recipes, but it is not known whether this technology was imported to the Maltese Islands by the Romans. This tree, locally referred to as “Gharghar”, is very rare on the Maltese Islands, and in fact it was declared the national tree in 1992 and, a year later, was protected by law. [10]

During the time of the Order of St John in Malta (1530-1798), there was already a shortage of wood, and it was imported at a high cost. [3] The vegetation throughout the eighteenth and nineteenth

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century was most probably as can be found today. [2] Buskett, considered as woodland, has a dense population of oak, pine, olive, ash and carob trees which regenerate naturally.

Luqa, a local town, was named so due to the presence of the considerable number of poplar trees referred to in Maltese as “luq”. Forests of evergreen oaks (*Quercus ilex*) must have been common in Malta. [6] The valley of St Julians, known as *Balluta*, meaning oak in Maltese, must have been highly populated with local oak. [7]

Several archives indicate that wood was imported. Although the probability is small, it is not known whether wood from indigenous and locally cultivated trees was used for furniture construction. Olive wood has been used for the construction of whole pieces of furniture, the wood or furniture having possibly been imported. *Olea europea L.* (olive) and *Ceratonia siliqua* (carob) wood, both indigenous, were used for local marquetry works. Wood from local fruit trees like ‘orange’ and ‘lemon’ was also used for such decoration.

Bonello points out that *Pinus sp.* (pine), *Quercus sp.* (oak) and *Fraxinus sp.* (ash) were types of woods used for local furniture construction during the time of the Knights.[8] One needs to note that *Pinus halepensis* (Jerusalem pine), *Quercus ilex* (holly oak) and *Fraxinus angustifolia* (narrowed leaved ash) are all Maltese indigenous trees. As a result, one should not exclude *a priori* the possibility that local trees might have been used occasionally for local consumption. To this date the author, while carrying out wood analysis, has never found evidence of any indigenous woods in any furniture, panel paintings or canvas stretcher frames.

Further deforestation must have taken place during the First World War for firewood and also when the Luqa airfield was constructed. [4] Few areas of local oak remained, mainly in Wardija. [5] Some of these trees are believed to be 500 to 900 years old.



Fig. 10 – Sacristy of the Parish Church of the Assumption of the Blessed Virgin Mary, Mosta (left), The sacristy of the Conventual Church of St John’s, Valletta (centre) and sacristy of the Parish Church of St Paul’s Shipwreck, Valletta (right)

## 2.2. Wood analysis carried out on the sacristies

Stained furniture renders macroscopic identification of coniferous wood more difficult and therefore microscopic identification is invaluable. Galea-Naudi states that red deal was mentioned to have been used throughout the dark stained vestry at St John’s Conventual Church, Valletta [11] (Fig.1). Scientific investigations carried out by the author indicate that at least three types of wood were used in this furniture. The upper sculptural elements are made of *Pinus sp.* (likely to be *sylvestris*), while the larger part of the pieces of furniture were constructed from a combination of *Larix decidua* (larch) and *Picea sp.* (spruce).

In this study 110 wood analyses were carried out. Six of these tests were performed on indigenous trees for comparative reasons. In some parts of furniture, when possible, two samples were taken from different locations for verification purposes. In some cases, like St Philip’s Sacristy in Żebbuġ, it was found that two types of wood were used in some door constructions (Fig. 2). Fig. 3 shows the list of results obtained. In those cases where more than one sample was taken from the same furniture piece, only one result was reported.

The results show that *Picea sp.* (spruce) and *Larix decidua* (larch) were by far the most commonly used for sacristy furniture throughout the eighteenth and nineteenth century. In cases where the results indicate the possibility of both *Picea sp.* and *Larix decidua*, it means that the wood was

macroscopically *Larix decidua* but microscopically no or very few biseriate bordered pits were observed.



Fig. 11 - Sacristy of the Parish Church of the Assumption of the Blessed Virgin Mary, Victoria Gozo (left), Sacristy of the Parish Church of St Philip's, Żebbuġ (centre and right)

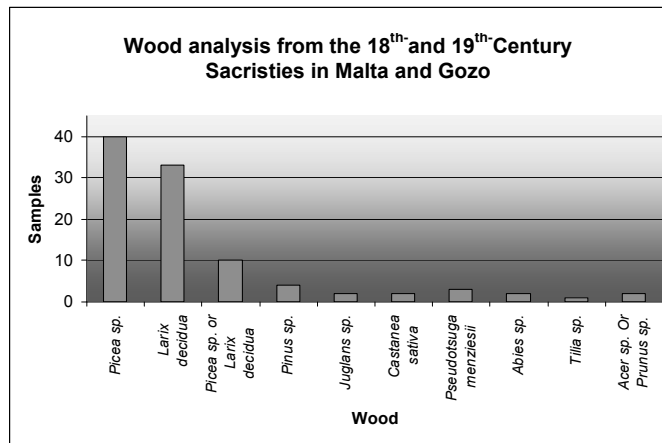


Fig. 12 - 18<sup>th</sup>- and 19<sup>th</sup>-century sacristies – wood analysis [12]

Surprisingly enough, *Abies sp.* (fir) was very rarely observed. During the past 8 years the author encountered several panel paintings of the same period made from this wood. No wood coming from indigenous trees was observed and this indicates that even though, at the time, wood in Malta was very expensive, it was mainly imported from abroad. In addition, this could have been the case since deforestation had taken place centuries before and therefore, like today, there were very few trees for furniture production. Little wood from angiosperm trees was found. This is not surprising since it is more expensive, harder to work with and also less abundant, despite that there were pieces of furniture completely constructed from *Castanea sativa* (chestnut).

*Larix decidua* was mostly popular for cupboard door construction. Comparing it with *Picea sp.*, it was most probably slightly more expensive and also harder to work, but it is more suitable to varnish. The use of *Picea sp.* for door construction, like in the case of the eighteenth-century sacristy furniture at the Church of St Publius and the eighteenth- and nineteenth-century sacristy furniture at St Helen's Parish Church, may have been due to the application of the graining technique requiring the wood surface would not be exposed (Fig. 4). Chairs, needing a delicate but strong construction were carried out using *Juglans sp.* (walnut). As expected, drawer constructions were all done from *Picea sp.*

Wood from eighteenth-century and nineteenth-century pieces of furniture was compared. Samples coming from the eighteenth-century represent 78% of all the samples. Therefore there might be a bias in this sense when it comes to comparisons, but indications are that both *Larix decidua* and *Picea sp.* remained popular even throughout the nineteenth century.





Fig. 13 – Sacristy furniture with graining technique: Parish Church of St Paul, Rabat Malta (left), Parish Church of St Helen, Birkirkara (centre) and detail from sacristy furniture at the Church of St Publius, Rabat Malta (right)

### 2.3. Surface coatings, adhesives and metal furniture fittings

Very little can be said on these materials since little literature is available and unfortunately very little scientific research has been carried out in this regard. The use of stains was mentioned by Galea-Naudi stating that churches and other country houses made use of red deal which they stained in order to make it appear like chestnut. [13] On the other hand, senior restorers state that in older times the main type of surface coating was linseed oil often mixed with earth colours like burnt sienna. The author insists that in cases where more than a single type of wood was used, like in the case of the eighteenth-century sacristy furniture at the Conventual Church of St John's, Valletta where there was a mixture of the dark-coloured *Larix decidua*, *Pinus sp.* and the pale-coloured *Picea sp.*, staining was inevitable. Apart from the application of a graining technique, some antique furniture used to be applied with linseed oil or beeswax. Such polishing would have been continued until the desired lustre was achieved. [14] The use of resin varnish may have been imported like other materials.

The most commonly used type of glue, probably also imported, was the hot animal skin/bone glue which has been used for thousands of years. This glue, which sets on cooling and evaporation of water, was extensively used till the twentieth century until the development of synthetic glues, which then dominated the market. Furniture metal fittings, like hinges, locks, escutcheon plates, keys, bolts and forged nails were locally manufactured but importation was also probable. One characteristic of Maltese furniture is the iron locks which were placed and nailed rather than inserted into the wood. [15] Two types of locks were available, those fixed directly to the back and those recessed at the back mostly for drawer or lid construction. Each lock had its particular key. Metal was probably imported in sheets of different thicknesses. Unfortunately a considerable number of these locks have been replaced.

### 2.4. Development of Maltese furniture

Very little or no furniture survived in Malta from the Middle Ages to the sixteenth century. [16] On the arrival of the Knights of the Order of St John in 1530 there started to be upgrading, embellishing and reconstruction of existing buildings and early auberges, churches and dwellings. Records often mention lists which include furniture which unfortunately rarely describes the pieces. [17] Until the beginning of the eighteenth century, the poorer Maltese families had almost no furniture in their homes. [18]

The element of decoration in Maltese furniture was not expressed before the seventeenth century. [19] During this period, pieces of furniture were mainly constructed from *Juglans sp.* (walnut), referred to as *noce* and *Castanea sp.* (chestnut), referred to as *castanea*. *Fagus sp.* (beech wood), referred to as *fago* and *Diospyros spp.* (ebony), were also in use. The latter was also used in exceptionally high decorations and was imported from outside Europe. [20] Other types of wood were often disguised to look like ebony while cheaper furniture was produced from deal. [21] This wood was considered as being the ordinary type, perhaps due to its abundance. It was not before the British occupation in Malta that mahogany or satinwood started to be imported.

Motifs, mouldings and other decorative features are not only attributable to architecture but also to furniture. The Baroque period had a great influence in Malta and this style was also adapted to less important and ordinary pieces of furniture. [22]

During the two years of French occupation in 1798-1800, new developments in furniture may not have been present due the great instability which existed until the British took over. [23] With the new British occupation of Malta, local craftsmen grasped new methods of furniture production and furniture design was therefore directed towards a British style with some Italian embellishments.

Galea-Naudi states that it was during the eighteenth century that sacristies were furnished with built-in cupboards. [24] The large wall-to-wall cupboards were mainly constructed for sacristies and other important buildings. Their function was basically to store liturgical vestments. In the eighteenth century, the design of such cabinets was mainly geometrical. [25]

Towards the end of the eighteenth century, furniture styles became less elaborate. [26] Galea-Naudi states that during the first half of the nineteenth century, wood was abundant for the building and repair of ships. He also states that in the second half of the century, following the Industrial Revolution, Maltese churches underwent enormous structural alterations as well as redesigning and changing church decorations which originally dated back to the seventeenth and eighteenth century. [27] It was then that Maltese furniture developed a truly independent style. [28] The Maltese cabinet makers, by the nineteenth century, specialised in the craft of finishing. With the introduction of British styles, Maltese craftsmen started copying English furniture and produced it abundantly. [29] Nonetheless the importation of Italian furniture continued. [30] Baroque influences persisted till the nineteenth century and, even during the British period, local craftsmen were still asked to incorporate Baroque elements of design. [31]

## 2.5. Styles and techniques

Following the collection of all data, two comparisons were made: the first delineates the differences between the two centuries under consideration, while the second compares styles and techniques used between Maltese and Gozitan sacristies (Fig. 5).

On analysing results, the following can be hypothesized:

- The central cupboard was perhaps more frequently included in nineteenth-century sacristies. This depended mostly on the size of the room.
- During the nineteenth century, there were more framed door constructions with joints.
- Pilaster decorations as well as the use of platforms remained popular throughout both centuries.
- Eighteenth-century sacristy furniture was richer in decoration.
- The use of the bench, integrated with the wardrobe construction, is missing in nineteenth-century furniture.
- In both centuries, the use of transparent coatings was more popular, yet the graining technique was still present in 26% of the total sacristy cupboards visited.
- The central cabinet was more popular in Maltese sacristy furniture.
- The use of joints in door construction was more evident in Gozitan sacristies.
- The bench was more popular in Malta while the platform dominated more in Gozo.
- Comparing percentages, the graining technique was presumably more popular in Maltese sacristy furniture.

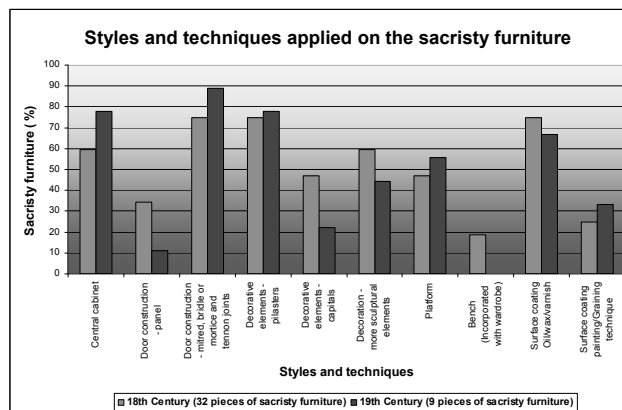


Fig. 14 - 18<sup>th</sup> and 19<sup>th</sup>-century sacristy furniture - styles and techniques [33]

Both nineteenth-century sacristy furniture and that found in Gozo represent a minority and so the above interpretation may be slightly inconclusive. Even though both islands are very small and close to each other, a slight difference in style and technique was noticed. There is no doubt that the boom in sacristy furniture construction occurred in the eighteenth century in Malta and Gozo.

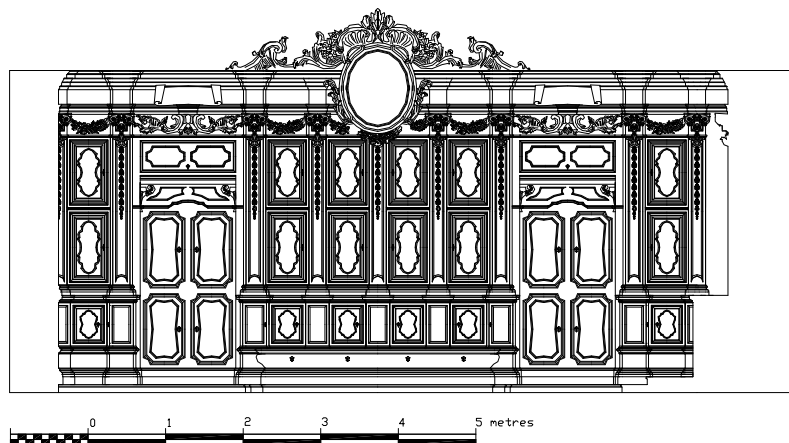


Fig. 15 - Graphic documentation of part of the eighteenth-century sacristy furniture of the Parish Church of St Philip's, Żebbuġ [33]

### 3. Conclusion

Considering all the analyses on the wood samples, the presence of wood from local trees was nil, implying a low probability in the use of indigenous trees for local furniture construction during the eighteenth and nineteenth centuries, *Picea sp.* (spruce) and *Larix decidua* (larch) being the two dominant types of wood used throughout the two-above mentioned centuries. In cases of intricate carving works, a harder and denser type of wood from broad-leaved trees was often used.

A boom in sacristy furniture construction is noticeable during the eighteenth century. Some elements of furniture, such as the sacristy bench, which is incorporated within the main construction, seem to be pertinent to the Maltese Islands. The latter was so popular during the eighteenth century that none were found in the nineteenth-century sacristy furniture considered. Eighteenth-century furniture was richer in decoration with respect to the furniture dating to the nineteenth century, yet, some classical features, such as the pilaster, the capital and the frieze, still remained popular during the latter century. The graining technique was found to be quite popular during the eighteenth century becoming less significant during the following, although the framed door constructions developed with greater frequency in the nineteenth century.

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# DEFIBRING OF HISTORICAL ROOF BEAM CAUSED BY AMMONIUM SULPHATE AND AMMONIUM PHOSPHATES BASED FIRE RETARDANTS

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## Abstract

Some chemicals, which are ingredients present in preservatives, can induce chemical reactions that cause damage to wood polymer. Among such chemicals are ammonium sulphate and ammonium phosphates in fire retardants, which cause damage to historical wood surface, called defibring. Such damaged has been reported to exhibit enhanced acidity and moisture content. Defibred wood was studied by electron microscopy and infrared spectroscopy, by pH value measurement and termogravimetry. Measurements of climatic conditions in the attics and mechanical properties of defibred wood were carried out. It is concluded that the fibres on the wood surface consist mainly of cellulose, which is occasionally partly damaged, and that, contrary to general belief, the observed increase in wood acidity is not due to the previous application of fire retardants and subsequent acid hydrolysis; instead, this effect seems to be due to oxidation of the structural components of wood. Defibring affects only mechanical properties of the wood surface layers.

## 1. Introduction

Some chemicals that are present in preservatives can induce chemical reactions, which cause damage to wood polymers. Among such chemicals are ammonium sulphate and ammonium phosphates that serve as components of fire retardants. Many roof beams in historical buildings in the Czech Republic have been repeatedly treated with these fire retardants. This treatment brought about damage to the historical wood surface consisting in a release of loose wood fibres, named as “defibring”. For instance, the roof beams of the Old Royal Palace within the Prague Castle (Czech Republic) are damaged by defibring. There were subject to extensive investigation roughly in 1997-2001 [1-4]: The wood pH value was 4-5 in dependence on the sampling point. Damaged wood was removed from the beam surface with a brush and the beams were treated with a neutralizing solution containing calcium carbonate and boric acid. The composition of the solution was modified so as to match the observed acidity of the wood. The neutralization process resulted in a pH value increase roughly to 5-6. Subsequently the parts were treated with a boric acid based protective agent. As it appears, however, this procedure is inefficient, and in a few years the process of defibring of the wood surface will set in again.

Such damaged wood has been repeatedly reported to exhibit a higher acidity and increased moisture content. The latter effect is attributed to the hygroscopic nature of the salts. The change in the wood pH value is believed to be a result of salts dissociation due to high air humidity, resulting in the formation of acid or alkaline solutions. Therefore it is assumed that the wood damage mechanism consists in hydrolytic degradation or acid catalyzed dehydration. The extent of this effect depends on the salt concentration and on temperature. In addition, highly hygroscopic salts are assumed to exert a mechanical effect arising from crystallization pressures. [4-7]

The effect of fire retardants based on ammonium sulphate and ammonium phosphates has been examined extensively by USDA Forest Service, Forest Products Laboratory (e.g. by J. E. Winandy and S. L. LeVan), particularly in relation to a decrease in the strength of plywood sheathings and strandboards under conditions of increased temperature and humidity. Knowledge from the previous studies is summarised by Winandy in his work [5], where he states the following:

Mechanism of thermal degradation in fire retardant treated plywood is acid hydrolysis.

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Degree of damage is also influenced by thermal exposure during pretreatment, treatment and post-treatment processing and in-service exposure, hence, by the combination of high temperature, humidity and type and concentration of the present chemical.

Decrease of pH value significantly occurs during increased temperature (66°C) and may be regulated by addition of pH buffers.

In her work, Lebow [7] tested the influence of the composition of the fire retardant based on ammonium dihydrogen phosphate on the pH value of plywood. Based on her results she hypothesises that ammonium dihydrogen phosphate converts into phosphoric acid at increased temperatures.

however, signs of wood damage similar to those occurring after treatment with fire retardants based on ammonium salts were also detected in the wood roof constructions near Sydney in Australia [8]. These roof construction beams were destroyed due to the effect of sea salt. Wood defibring, however, occurred here mainly on the bottom side of the beams and pH value of wood surface was within the normal range but wood moisture content was very high, often more than 35 %. The ash analysis showed high contents of sodium and magnesium cations. Examination of the wood under the electron microscope uncovered the damage of middle lamella as well as the secondary cell walls (mechanical damage and erosion). Wood degradation caused by wood-destroying fungi or bacteria was observed very rarely.

## 2. Experimental part

The results presented in this paper were obtained based on analysis of wood samples taken from damaged roof beams of the Český Krumlov Castle (Czech Republic), and of a house in street U Půjčovny, Prague, which had presumably never been treated with any preservation procedure and damage roof beams of the Old Royal Palace (Prague Castle), which had undergone a preservation procedure, as mentioned above. Wood samples from the roof beam surface comprised loose fibres (defibred wood) as well as wood chips split off from the surface by using a surgical knife.

Changes in the wood structure were examined by optical and electron microscopy. Chemical changes (wood corrosion) occurring in the wood structure were studied by FTIR spectrometer connected to a microscope. The reflectance technique was applied. The spectra were measured over the region of 4000-600  $\text{cm}^{-1}$  at a resolution of 4  $\text{cm}^{-1}$  applying 256 scans, and were normalized with respect to the absorption band at 1032  $\text{cm}^{-1}$ . Infrared spectroscopy was applied to samples of damaged wood (fibres and chips) following their leaching in water.

The wood pH value was measured by the aqueous leachate method. The leachate was prepared from 0.5 g of sample (fibres, chips) in 50 ml of distilled water (previously boiled and cooled down). The sample was allowed to leach for 1 hour with occasional stirring, the leachate was filtered, and its pH value was measured with a combined pH electrode. The pH values of ammonium sulphate, ammonium hydrogen phosphate, and ammonium dihydrogen phosphate solutions in distilled water at different concentrations were measured as well. Each final value was obtained from triplicate measurement.

Furthermore, there were studied mechanical properties of the defibred wood surface layers in the connection with the reference samples made of deeper (undamaged) wood layers that were extracted from the damaged roof frame of the house in Prague, street U půjčovny. The observed properties were compression strength and modulus of elasticity along the fibres as per Czech standard ČSN 49 0111 (size of samples 20×20×30 mm), tensile strength along the fibres (microsamples of triangular cut 5×5×7,5 mm) and wood hardness measured by Janka method (Czech standard ČSN 490136) and by using a Pilodyn 6J Forest resistance indenter.

Climatic conditions in the attic of the Old Royal Palace of the Prague Castle were monitored with sensors providing temperature and relative humidity records.

## 3. Result and discussion

The surface of the damage beams are covered by fibres of different size and colour shade. In some cases, the wood surface freed from the layer of released fibres is brown-red colour. Separation of wood fibres (defibring) in the transverse direction can be seen when observing the damaged wood (chips and fibres) under an optical microscope. In chip samples, the damage is associated with the formation of cracks and

microcracks and fibres being torn from the wood surface. In the samples consisting of loose fibres, individual fibres whose length and thickness decrease gradually even within one sample can be observed (Fig. 1-3).



Fig. 1 – A chip sample of the defibred wood.

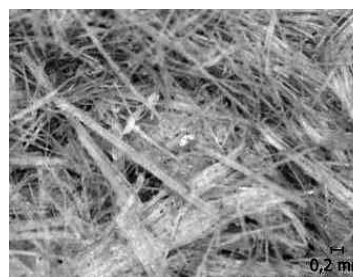


Fig. 2 and 3 - The fibres occurred on the wood surface

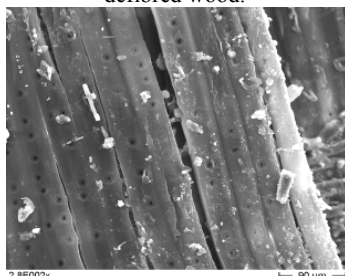


Fig. 4 - Wood cells separation in the chip sample taken from defibred roof beam surface.

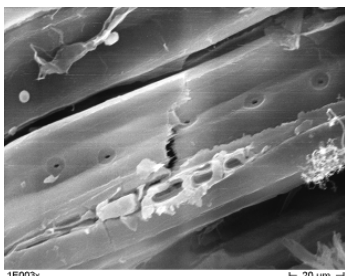


Fig. 5 - Cells separation and cell wall crack in the chip sample taken from defibred surface.

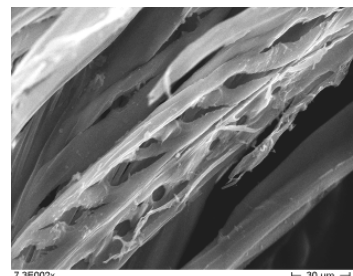


Fig. 6 - Cell wall damage and missing margo strand.

Examination of the anatomical structure of the damaged wood by electron microscopy revealed that both cellulose and lignin are damaged by chemical reactions. In some cases, mechanical damage of the wood structure due to crystallization pressures is also conceivable. Separation of cells, apparently due to a damaged middle lamella, was observed (Fig. 4-5). Damaged of margo strand of bordered pits on the tracheid walls were also apparent (Fig. 6). Both the middle lamella and the margo strand consist mainly of lignin. Some samples have damaged secondary cell walls, which primarily consist of cellulose. This damage is represented by various cracks in the cell walls (Fig. 5-6). Fig. 7-9 show the wood fibers which originated from the beam surface damaged by defibring. Samples consisting of fine fibres did not exhibit any wood structure and this indicates that the cell walls have been disintegrated into the fibrous tangle - presumably supermolecular cellulose formations. Additional experiments will be necessary to gain a deeper insight into this.

The infrared spectra were evaluated in dependence on the degree of wood defibring, i.e. very fine fibres, coarser fibres and chips (Fig. 1-3). The infrared spectra show that with the increasing level of wood defibring decreases relative lignin content in wood which is demonstrated mainly by the decrease of the absorption band with its peak in  $1510\text{ cm}^{-1}$ . This band belongs to the stretching vibrations of the lignin aromatic ring double bond. The decline of this band, indicating the decrease of lignin, was evaluated by means of correlation with the chosen reference absorption bands of cellulose with its peaks in  $1372\text{ cm}^{-1}$  (out-of-plane bending vibrations of CH bonds of cellulose glycoside ring). The intensity rate of the bands  $I_{1510}/I_{1372}$  drops rapidly in the connection with the level of wood defibring (see Table 1). This relative decrease of lignin was observed in all the samples of the damaged wood taken from all of the observed buildings.

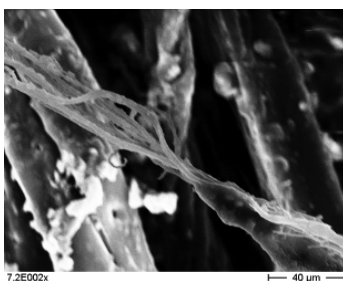


Fig. 7 and 8 - Fine fibres present on the wood surface due to the defibring.

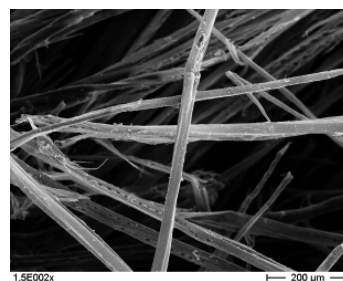


Fig. 9 - Coarser (longer) fibres present on the defibred wood surface.

Table 1: Loss of lignin as determined by FTIR  $I_{1510}/I_{1372}$  band intensity ratios. Each value used is average of triplicate measurements. Band assignment:  $1510\text{ cm}^{-1}$  – stretching vibration of the double bond in aromatic ring of lignin,  $1372\text{ cm}^{-1}$  – out-of-plane bending vibration of CH bond at the cellulose glycoside ring.

	Sample	$I_{1372}$	$I_{1510}$	$I_{1710}$	$I_{1510}/I_{1372}$	$I_{1710}/I_{1510}$	
Undamaged wood	Fir	0,169	0,235	0,036	1,391	0,153	
	Pine	0,086	0,123	0,028	1,434	0,224	
The Český Krumlov Castle	Wood chip	2	0,127	0,083	0,121	0,658	1,449
		8	0,111	0,100	0,088	0,898	0,881
		10	0,061	0,066	0,235	1,089	3,567
	Coarser fibres	1	0,081	0,046	0,151	0,574	3,244
		4	0,079	0,041	0,100	0,516	2,449
		5	0,083	0,027	0,206	0,329	7,534
	Fine fibres	2	0,092	0,023	0,159	0,247	6,997
		7	0,133	0,015	0,124	0,114	8,136
		13	0,125	0,036	0,088	0,285	2,462
The Old Royal Palace, Prague Castle	Wood chip	10	0,111	0,220	0,072	1,985	0,327
		11	0,089	0,134	0,147	1,510	1,097
		13	0,112	0,139	0,213	1,241	1,528
	Coarser fibres	8	0,246	0,432	0,229	1,759	0,530
		10	0,062	0,091	0,189	1,482	2,073
		11	0,114	0,060	0,125	0,522	2,101
	Fine fibres	9	0,155	0,046	0,138	0,295	3,007
		12	0,261	0,149	0,162	0,569	1,091
		21	0,288	0,087	0,378	0,303	4,338
House in street U Půjčovny	Wood chip	5	0,132	0,324	0,135	2,453	0,417
		7	0,049	0,142	0,112	2,894	0,790
		11	0,058	0,136	0,141	2,338	1,032
	Coarser fibres	1	0,080	0,058	0,188	0,727	3,241
		4	0,063	0,081	0,412	1,286	5,122
		8	0,077	0,107	0,147	1,386	1,374
	Fine fibres	2	0,070	0,045	0,060	0,641	1,330
		3	0,129	0,105	0,124	0,813	1,177
		6	0,076	0,096	0,198	1,262	2,052

The relative increase in the carbonyl group content of the wood matter was evaluated based in the  $I_{1710}/I_{1510}$  band intensity ratio where the band at  $1710\text{ cm}^{-1}$  is due to stretching vibrations of the carboxyl C=O bond and the band at  $1510\text{ cm}^{-1}$  is due to stretching vibrations of the double bond in the lignin aromatic ring (Table 1). This relative carboxyl group content in the wood was found to increase with



increasing degree of defibring, apparently as a result of oxidation of hydroxyl groups both in cellulose and in lignin.

Furthermore, increasing extent of wood damage is accompanied by a shift of the absorption band at 1420-1430  $\text{cm}^{-1}$  towards higher wavelengths, suggesting that the crystalline fraction of cellulose increases in relative terms due to a loss of the amorphous fraction.

The pH values of sound wood taken from a roof beam roughly 150 years old and of defibred wood are given in Table 2. Samples taken from roof beams at the Český Krumlov Castle exhibit pH values both lying within the region of natural wood acidity and corresponding to elevated acidity. For the roof beams of the Old Royal Palace, the pH values occupy a wide range, due to the previous conservation treatment procedure. Although treated with a neutralizing solution, some beams exhibit elevated acidity (pH value lower than normal). This can be explained in terms of a poor preservation procedure and/or formation of new acid groups in the wood polymers due to their oxidation. On the contrary, one sample taken from a beam in the attic above the Vladislav Hall of the Old Royal Palace was strongly alkaline, attaining pH value 9.2. Such extreme pH value would be a result of an alkaline chemical having been spilt on it.

Table 2: pH values of damaged and sound wood and chemicals.

Sound wood samples (undamaged, approx. 150 year old roof timber)	pH value	Samples from the Český Krumlov Castle (non-treated)	pH value
Pine	4.4	Fibres	3.9- 4.4
Fir	5.0	Wood chip	3.3-3.4
Spruce	4.4	<b>Samples from the attic in street U půjčovny (non-treated)</b>	
<b>Samples from the Old Royal Palace – attics after conservation treatment</b>		Soft fibres (dust), wood chip	4.2
Attic above the House	3.1-3.2; 5.9-6.0	<b>Chemicals</b>	
Attic above the All Saint Church	4.2; 4.8; 5.3; 6.5	$(\text{NH}_4)_2\text{HPO}_4$ , 10% (w/w) concentration	8.4
Attic above the Vladislav hall - a highly damaged site of the beam	<b>9.2</b>	$\text{NH}_4\text{H}_2\text{PO}_4$ , 10% (w/w) concentration	4.2
Attic above the Vladislav hall	4.9; 5.6; 6.3	$(\text{NH}_4)_2\text{SO}_4$ , 10% (w/w) concentration	5.3

In some cases, the observed pH values of damaged wood suggest that the wood may have been damaged as a result of hydrolysis. For pH values lying below natural wood acidity levels (i.e. below pH value 4.0), acid hydrolysis of polysaccharides (first of all hemicellulose) can be assumed, whereas for pH value 9.2, alkaline hydrolysis of the structural components of wood is presumably involved. Oxidation of lignin is also conceivable in alkaline environments in some circumstances [9]. Ammonium hydrogen phosphate and ammonium sulphate solutions do not increase the acidity of the treated wood whereas as regards ammonium dihydrogen phosphate, its effect on wood acidity is not impossible. Overall, the wood acidity increase cannot be ascribed unambiguously to the application of those salts as shows Table 2.

Compression strength and modulus of elasticity along the fibres were tested on the samples that were prepared both from the defibred surface layer and the following layer in the direction to the centre of the beam (layers 0-20 mm and 25-45 mm). The measured values of the observed mechanical properties were a little higher for the samples from the surface layer. This fact is probably due to the higher density of the surface layers that contained very thin annual rings in contrast with the inner parts of the tested wooden element where annual ring were wider. Tensile strength along the fibres was tested on specially made microsamples with a triangular cut ( $5 \times 5 \times 7.5$  mm), which enabled more accurate determination of the observed property in different depths under the surface of the damaged wood. The samples were prepared from the defibred surface layer (0-5 mm) and the wood layer from the depth of 25 mm (25-30 mm). The results of tensile strength of the defibred surface layers were

much lower, sometimes even by half, when compared to the test results of the undamaged wood from the depth of 25 mm. Wood hardness measured by Janka method and by using a Pilodyn 6J Forest resistance indenter was performed in a total of five layers of the tested wood which was gradually milled out by 5 mm for each layer from the surface of the beam to its centre. Both of the used hardness tests mutually corresponded and proved the influence of wood damage for the first and the second surface layers only. Other tested layers manifested no changes in hardness.

#### 4. Conclusion

The results of this investigation give evidence that the fibres on the wood surface mainly consist of cellulose, which is occasionally partly damaged, and that, contrary to general belief, the observed increase in wood acidity is not due to the previous application of fire retardants and subsequent acid hydrolysis; instead, this effect seems to be due to oxidation of the structural components of wood. Oxidation of lignin appears to be the major mechanism of wood defibring. This fact also explains why the current remedial action consisting in the application of a neutralizing solution is inefficient. Additional measurements on model wood samples will be necessary to prove this hypothesis unambiguously. The decrease of mechanical properties only concerned the surface layers of beams affected by defibring (no deeper than 5 mm in our tests). The reduction of mechanical parameters of the damaged surface layer of the wood was appreciable, sometimes making over 50% in comparison to the non-damaged wood sections (tensile strength along the fibres, hardness).

#### Acknowledgement

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# **NOSSA SENHORA DO ROSÁRIO AND ANJO CEROFERÁRIO. EXAMINATION AND CONSERVATION OF TWO DIFFERENT WOODEN POLYCHROME SCULPTURES, AT THE IMC'S DEPARTMENT OF CONSERVATION**

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## **Abstract**

This paper presents two cases of Portuguese sculptures that will be object of a future investigation project at the Departamento de Conservação - I. M. C., Portugal. The motivation will be to create a *corpus*<sup>1</sup> with the most common construction techniques which were employed when carving these great scale works of art, during the 17<sup>th</sup> and 18<sup>th</sup> centuries. Direct observation and X-ray exams revealed that one of the sculptures was composed of several boards of different wood species, while the other was made of a section of a single tree trunk. Optical microscopy was used to characterize wood species found in each sculpture. Both construction techniques showed signs of physical instability which were responsible for the appearance of distinct irreversible damages of the polychrome surface.

## **1. Introduction**

Since pre-historical times, due mainly to its workable characteristics [1], wood has been preferably chosen as support of several utility objects, as well as a mean for artists expressions when creating works of art. However, limitations and susceptibility to adverse environmental conditions of this material are well known. Consequently, both in form of plane shape panels or carved sculptures, the work of art tend to suffer physical alterations due to the different tension rate in each transformed material.

During 17<sup>th</sup> and 18<sup>th</sup> centuries, the artist's knowledge, skills and quality of work were highly controlled [2] which resulted in a great number of very good polychrome sculptures, both regarding the carved work and the paintwork.

Like it happens in many other cases, it is very difficult to understand an artist's choice when selecting the material or technique to construct a sculpture. The order documentation, which could have revealed important aspects of the client's demands regarding materials or techniques is most times lost in consequence of a fire or a flood. [3],

## **2. Characterization and conservation condition**

The first case is a 160 cm high whole-volume sculpture, belonging since 1950 [4] to the Museu de Santa Cruz, Vila Viçosa. The sculpture represents the Virgin *Nossa Senhora do Rosário* (Fig.1). It was produced around 1751-1800 by an unknown artist for the church of São Bartolomeu at Vila Viçosa, and was located standing in a niche, in the left side of the main altarpiece. Its conservation condition demanded an urgent intervention due to poor adhesion between structural wooden pieces which caused polychrome detachment. Fissures in the paint layer were originated not only because of the natural expansion and retraction movements of wood, but also because of the weak binding between the glue and the wood with diverse grain orientations (Fig. 2).

The second case is also a whole volume sculpture, 136 cm high, belonging to the Igreja de Nossa Senhora do Carmo in Beja, representing an angel, *Anjo Ceroferario* (Fig. 3). The sculpture was produced late in the 18<sup>th</sup> century by an unknown artist. These sculptures were very commonly found in

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<sup>1</sup> *Corpus* is the Latin name to body. In this context it relates to the study of a collection with the same typology of works of art.

Portuguese churches, usually placed near the altarpieces (Fig. 4), and used as candlesticks for lighting, during 17<sup>th</sup> and 18<sup>th</sup> centuries [5].



Fig. 1 – *Nossa Senhora do Rosário*.

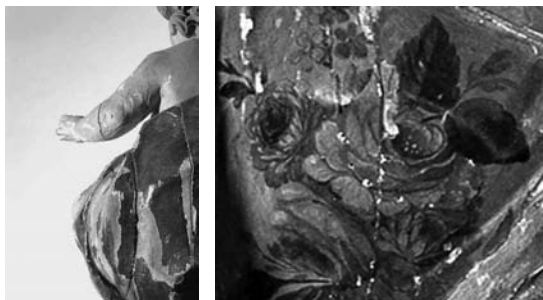


Fig. 2 – *Nossa Senhora do Rosário*. Areas where longitudinal fissures can be seen on the painted layer. They correspond to the assembling joints of the pieces of wood.



Fig. 3 – *Anjo Ceroférario*.

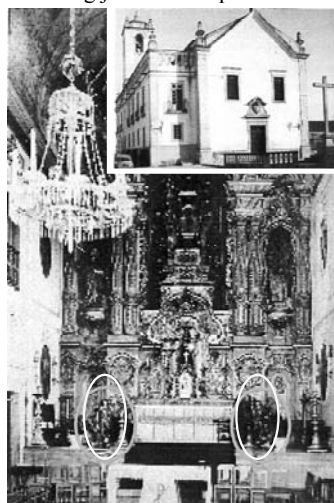


Fig. 4 – Interior of the Church of *Nossa Senhora do Carmo* in Beja. Original flanking position of the sculptures on the main altar (yellow).

### 3. Methods of analysis and construction techniques

#### 3.1. *Nossa Senhora do Rosario*

To characterize the sculpture's support, small samples of wood from the base were collected for optical microscopy (OM) analyses which, within reasonable limitations, confirmed the existence of cedar (*Cedrus* sp.) and mahogany (*Kaya* sp.) (Fig. 5). Although those wood species were very commonly used in Portugal by that time [2], are easily workable and allow good finishing, they were not so often used for sculpture making [6]. Characterization of the wooden support identified a multiple number of cedar and mahogany pieces, combined side-by-side, building up the sculptures

volume, with a hollow space in the centre. This can be seen in a X-ray image (Fig. 6). In addition to these large scale wood pieces, smaller and more irregular cedar pieces were put to fill spaces, gaps and edges, especially in the Jesus' head. With the X-ray we could also identify two different kinds of nails, some large hand-made nails, used inside the structure to build up the sculpture, and other smaller industrial-like nails, used in a recent intervention, over the paint layer, to prevent the detachment of the multiple wood pieces.

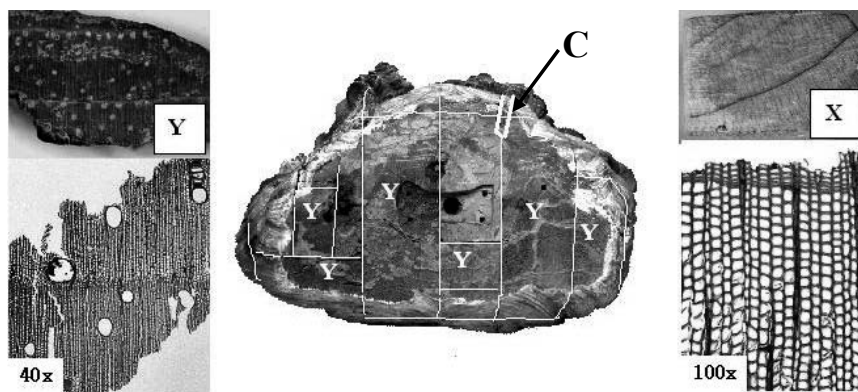


Fig. 5 – *Nossa Senhora do Rosário*. Base of the sculpture and correspondent cut sections of the two different species: (Y) *Khaya* sp. (mahogany) and (X) *Cedrus* sp. (cedar); (C) wood wedge. The lines show the distribution of the joint lines.

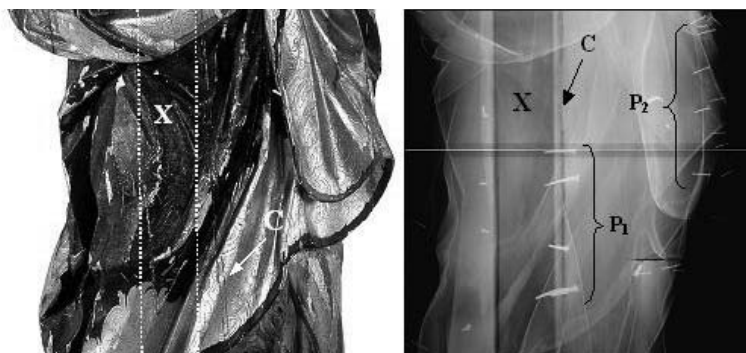


Fig. 6 – *Nossa Senhora do Rosário*. Left: detail photograph of the sculpture's waist; Right: X-Ray of the same detail. (X) board of cedar; (C) wood wedge, (P<sub>1</sub>) handmade nails, (P<sub>2</sub>) industrial nails.

Several experimental studies of the physical behaviour of the wood and the ground layer were made, based on the original construction techniques<sup>2</sup>. We wanted to understand if the combination of a coniferous (cedar) with a deciduous (mahogany) wood, and the interaction between the wood and the ground layer materials, might have influenced the sculptures' conservation condition, leading to its actual critical state of conservation. By gathering records of the different reactions of wood to various climatic conditions of temperature (T) and relative humidity (RH) our studies confirmed that hypothesis [7].

<sup>2</sup> During the year 2007 Diogo Sanchez completed his studies of conservation and restoration at the Universidade Nova de Lisboa, Faculdade de Ciências e Tecnologia, Lisboa, comparing *Nossa Senhora do Rosário's* wood behavior and its effects on the ground layers, first with diagnosis and later with experimental work with the same kind of materials. The physical behavior of the ground layer materials and its interaction with the wooden support was evaluated. Physical parameters as hardness, break force, flexibility and color alteration were analyzed as well the different reactions of the wood to various climatic conditions of temperature and relative humidity. The conservation work on the *Nossa Senhora do Rosário* is still being done.

### 3.2. Anjo Ceroferario

This sculpture was made of a section of an oak tree (*Quercus* sp.), characterized by OM identification and a sample both taken from the sculpture (Fig. 7). The X-ray examination and the diagnosis of the conservation condition revealed extensive internal damage caused by one longitudinal split. This was only slightly visible on the outer surface with an unaided eye, but it could endanger the sculpture's stability and its vertical standing (Fig. 8). From the outside we could only see the paint layer fissured, not an extensive crack. Only at the bottom area of the sculpture was it possible to see a large crack right in the centre (Fig. 9).

After having studied the composition of the paint layer and the causes of material alterations, the treatment was done by filling the crack, with stable pieces of old oak wood and PVA (polyvinylacetate based glue), to prevent the sculpture to break and split apart (Fig. 10).

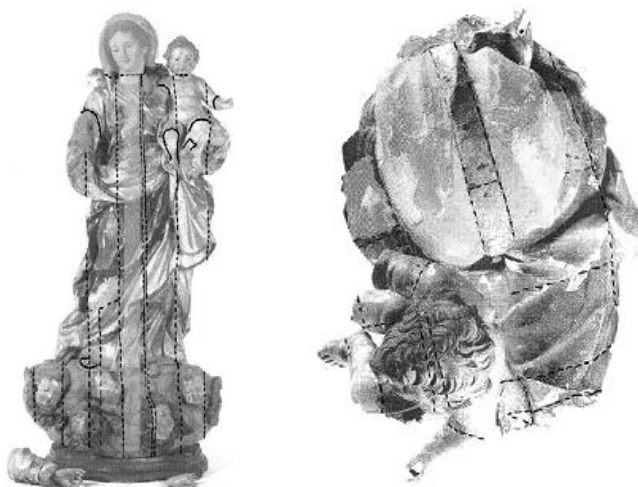


Fig. 7 – *Nossa Senhora do Rosário*. Indication of the assembly lines and the location of the cedar parts (yellow). Left: front view; Right: upper view.



Fig. 8 – *Anjo Ceroferário*. X-ray examination of whole sculpture, where we can see an internal long longitudinal crack.



Fig. 9 – *Anjo Ceroferário*. Areas where the longitudinal crack inside the support has also fissured the painted layer.

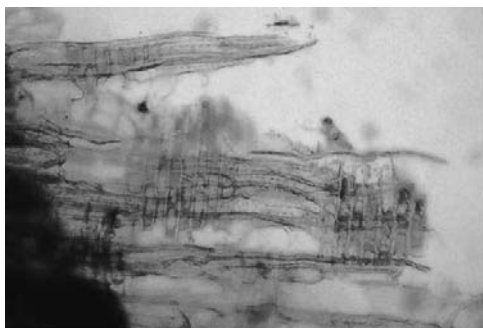


Fig. 10 – *Anjo Ceroferário*. Dissociated elements of a wood sample collected from the sculpture’s support characterized as *Quercus* sp. (oak).

#### 4. Observations

There are two important aspects that the future investigation following the examination of these two sculptures may involve. In one hand we are able to gather considerable information about the single objects and to compare compiled results in order to establish distinctive groups of artistic production centers, not only in Portugal but possibly also in other European countries.

On the other hand, the concern for the preservation of these objects is of high importance, as the techniques used to produce them do not guarantee them “a long life”. Preliminary diagnoses may help to establish the most adequate conservation conditions, which in itself is a challenge when dealing with composite objects [8]. Material alterations demands tailored treatment procedures, as well as suitable exposure conditions.

During the work with the two sculptures there were questions raised about the woodworking knowledge of the sculptor. Some parts of the sculptures were a combination of unreasonable choices of materials and/or techniques for the task, combined with high quality carving and paintwork. Some cases suggested that in particular situations artisans could also have the concern of sparing good quality wood available in the place at that time, but we are still chasing for proofs.

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# THE CONSEQUENCES OF WOODEN STRUCTURES CONSOLIDATION THROUGH TRADITIONAL TECHNIQUES ON THE RESISTANCE OF SUPPORT PANELS AND PAINT LAYERS (SOME CASE STUDY - STELEA MONASTERY TARGOVISTE, HUMOR MONASTERY AND ARBORE MONASTERY)

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The majority of Romanian icons, especially those from XVI-XIX centuries, are painted on panels of soft wood, like lime – the most widespread, poplar or coniferous trees. Icons on hard wood panels, as oak or beach, are rarely found. For this reason the major part of the icons preserved onto this day has suffered a xylophages insects attack, sometimes resulting in a considerable loss of mechanical strength of the panel. In time, various methods aiming to restore the panel resistance were tested, as impregnation with consolidant substances and inserting wedges of different materials, hoping in an improvement of the icons state of conservation. In most cases the results were far from those foreseen by restorers.

As an exemplification is presented the case of icons from the church and museum of Stelea Monastery - Targoviste, as well as the icons and iconostasis of Humor and Arbore Monasteries, displaying a large range of deteriorations, frequently found in the majority of the orthodox icons collections from Romania.

The icons of Stelea Monastery were reinforced by two traditional methods: impregnation and inserting of some new wood fragments on the icon back. Most of the interventions implied both treatments. In most cases, the impregnation was carried out using a wax-colophony mixture, imparting a greater mechanical resistance to the wooden panel. However the results were not quite satisfactory for many reasons.

- the impregnation is not uniform, as the liquid penetrates through the exit holes of xylophages insects, randomly distributed;
- in some cases, due to the correspondence between the insect channels and because the consolidant is poured in a liquid form the wax-colophony mixture reaches the painted surface, deteriorating it;
- the increase of material density in the impregnated areas (about 5 times greater, in our estimation) creates additional strains at the level of already existent cracks, resulting in a fracture along the incipient fissure;
- the most destructive consequence is the formation, on the icon back, of a barrier for water vapors as well as for the xylophages insects resistant to disinfection (as a rule, this is the case of heavily contaminated icons, the biocide treatment being carried out by brushing, the eggs and the larvae deposited in depth are not destroyed). So, the exit holes will appear also onto the icon front side, increasing the deterioration of the pictorial layer. At the best, the final result would be a reinforced solid panel, usually very heavy, on which the pictorial layer – the image intended to be preserved – will would deteriorate even more rapidly than before consolidation.

Taking into account the above facts, the restorer Dinu Savescu suggested a completely different restoration method, based on the following considerations:

- the pictorial layer has an insignificant weight comparing to the wood panel itself, thus a very solid panel is unnecessary;
- the xylophages biological attack, especially on soft wood, is directed to the areas between the wood fibers, the panel preserving its mechanical resistance to a greater extent than expected;
- the consolidation of some areas is feasible using soft material, like balsa wood. Thus, the density of the support material is not essentially modified;

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- some local consolidations may be done (but not by impregnation), depending on support resistance and the intended exhibition manner, or the exhibition on horizontal plane is recommended for museum icons;
- another possibility is the construction of an aluminum straps structure, onto which the back of the icons is attached with mobile cup holders, sliding on the straps. This system allows a prevention of strains resulting from the dimensional variations of the support (method initially proposed by Istituto Centrale del Restauro, in use today in most European museums);
- avoiding the impregnation, the access of biocide substances to xylophages insects is facilitated and can be repeated in various phases of the insects life cycle;
- even in the case of adult individuals growth the number of exit holes on the icon front side will be much smaller.

The Iconostases of the churches of Humor and Arbore Monasteries have more common points concerning the restoration problems: both exhibit the same deterioration, which is caused by a widespread biological attack, resulting in an important degradation of the structural resistance. All these degradation forms were identified as produced by *Hylotrupes Bajulus*.

At Humor Monastery the most virulent biological attack is confined to the central area of the Iconostasis, the whole Apostles register being affected. The right pillar and all the bottom area of the wood mounting frame behind the Iconostasis had been affected by a biological attack, creating an unstable connection between the elements in the Iconostasis.

The conservation state of icons from the central area of the Apostles register – Deisis scene – is very poor, showing extreme degradation caused by the biological attack. In fact, the inside of the panel had turned into a fine wood dust and the wooden support under the paint layer had been converted into a very thin skin. The whole panel painting is in danger of inner collapse.

The backside is painted in a decorative manner and some traces of axe working of the panel can still be noticed. These are significant elements for art historical studies, and have to be preserved.

The icons display an extremely thin support, under which, as in a closed case, the wood material had turned into dust (turning the icon upside-down the inner content is running down as in an hourglass).

The icons display an extremely thin support, under which, as in a closed case, the wood material had turned into dust (turning the icon upside-down the inner content is running down as in an hourglass).

The Arbore icons generally exhibit the same problems. Nearly 80% of the back side of the wooden structure on which the ensemble had been constructed has been destroyed by the biological attack. The traverse linking the north and south walls of the church is the only one unaffected and could be used as a carrying element on which a supporting structure would be constructed.

The Arbore Iconostasis support had been constructed in situ and it is composed of 5 registers joined together with wood plugs. The whole surface was then primed with a ground layer and on the ground the paint layer and metallic foils were laid. This is an important remark, as the detachment of some constituent elements is not possible without destroying the intimate links created between elements by the preparation layer. Thus the whole Iconostasis back side, together with the weakened and deteriorated supporting structure, had to be submitted to a restoration process without detaching the constituent elements.

The degradation of wooden structure due to biological attack requires a technical evaluation, bearing in mind the resistance details.

If the supporting structure of the iconostasis is discontinuous (braked off), resulting in degradation of the pictorial layer, an intervention is necessary. The intervention must however be limited to restoring the connection of the carrying structures.

The preservation of the pictorial layer and its wooden support should take into consideration the relationship between the various elements and the whole. The treatment of a wooden structure deteriorated by a general biological attack has in view to strengthen the total structure. Local putty filling, wood replacement or locally added new wood or natural/synthetic consolidants will often not be enough.

For the above presented Humor Monastery icons, bearing on the verso a pictorial layer meant to be preserved, considering the requirement of their exhibition in vertical position on the Iconostasis, an intervention on icons side was carried out. The aim was to restore the connection between the sound structures still existing inside and especially the structure of the lower part was consolidated. These structures are very important to the state of conservation of the icons. The sides of the icons were mounted, acting as a fastening structure between the back and the front sides. The necessity of mounting entire supporting structures on the sides of the icons complicated the problem, as the effect of new created strains had to be balanced by adverse forces. The increase of the weight because of the penetration of the consolidant requires the hardening of the scaffolding or the cogging of the back side to decrease the weight.

For the Arbore Church Iconostasis the restoration interventions took into account the specific deterioration of the pictorial layer caused by the bad state of the wooden support. The pictorial layer exhibited a good adherence to the nearby under layers (cca. 5mm depth), while deeper links were destroyed by biological attack. The most adequate approach was thought to be the joining of layers one to another, from the pictorial layer inwards.

The intervention was carried out by inserting some acacia wooden elements of smaller size than the exit holes, which would take over the bending strength tasks of the support.

### **Conclusion**

The consolidation by injection with various natural/synthetic consolidants does not solve the cohesion problems between the supporting layers deteriorated by a biological attack. The support consolidation by injection with consolidation solutions should be used only after a positive test effectuated over neutral materials, identically with the original, which would be sacrificed for the determination of the depth of penetration and the efficiency of hardening.

The transposing operation on a new wood or other kinds of support could result in major degradations of pictorial layer, accelerating the degradation process.

The treatment of wooden supports affected by a massive biological attack should be approached by focusing on connection between the various components and on strains between these components, depending on the exhibition position after restoration and depending on the micro- and macroclimate in which the ensemble will be preserved.

The above presented problems are illustrated by an ample photographic documentation, some photos being attached to this text.

## (E) STRUCTURES



# WHAT ONE NEEDS TO KNOW FOR THE ASSESSMENT OF TIMBER STRUCTURES

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## **Abstract**

The assessment of old timber structures is normally a great source of problems for building intervenors and a frequent justification for integral replacement of structures that would otherwise be kept in service.

It requires understanding the original structural system behaviour, the estimation of strength and stiffness properties of the timber in use, the evaluation of individual timber members' quality and effective cross section dimensions. Due to the natural variability of timber, unknown load and environment history, and common biological damage, assumptions have to be made, frequently with a high degree of uncertainty.

This paper discusses the common approach in the assessment of timber structures in service and tries to identify advances, problems, knowledge gaps and research needs related to this activity.

## **1. Introduction**

Repair, strengthening and upgrading of old buildings are and will represent in the future a large share of building contractors activity, as the urge for new construction is diminishing due to population stabilization in most developed countries, and the economical benefits, historical or environmental concerns justify the maintenance, as opposed to replacement, of existing structures.

Nevertheless, civil engineering and architectural teaching has been mainly directed towards new construction, thus ignoring the old materials and construction techniques. This is now changing, especially at the level of post-graduate studies or optional courses, but even so the time dedicated to timber structures is in most cases negligible.

At the same time, professional carpenters, in the traditional sense of long-gained practical experience and father-to-son transmitted knowledge, no longer exist. Often, so-called carpenters do not really understand the material, and frequently base their activity on erroneous principles applied with a frightening sense of self-confidence.

This scenario compromises the conservation of old timber structures where specific expertise and knowledge are essential for obtaining data on timber members and joints mechanical behaviour, which is directly linked with wood species used and structural system under evaluation.

Assessment of timber structures may be carried out under various circumstances. The easiest case includes the retrofitting of well preserved structure to its original condition, which can be handled with minor replacements or strengthening. In this case it is assumed that the number of years in service is a prove of its safety. However, in most cases, either due to conservation problems or change of use, judgement about material strength and damage quantification is required.

In the last situation, the lack of expertise on wood as a structural material often leads to replacement of suitable timber structures, by steel or concrete structures more adjusted to current engineer knowledge. One should say however that, despite the difficulties, there have also been numerous interventions where timber structures receive careful inspection and suitable consideration.

Several steps should be tackled before a final answer can be given about the safety of an existing timber structure. Inspection is a basic and crucial step upon which other steps and decisions rely. The purpose of this paper is to identify common methods, problems, knowledge gaps and research needs related to inspection of timber structures.

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## 2. Overall structural system evaluation

The original structural system behaviour, although sometimes complex and difficult to understand, can in most cases be handled by structural engineers.

One should have present that ancient timber structures are not always structurally sound, even disregarding possible degradation. Some exhibit an enormous degree of improvisation, some have basic conceptual/structural errors, some were poorly made and others have been altered disrespecting safety considerations.

Common problems in roof structures are erroneous geometry, eccentric loading at the truss supports and due to rafters placed away from the truss nodes, lack of bracing between trusses and missing elements due to previous interventions. In the case of floor systems, insufficient support length at the beam ends, lack or sloppy bracing between beams, removal of support walls and introduction of intermediate loading partitions are quite common.

Joints frequently have some kind of damage (metal corrosion, sloppiness, timber splitting or crushing) and original defects like missing plates or fasteners, minute edge and end distances of fasteners, too small washers, gaps between elements that should be in contact. The engineer should carefully inspect the joints, be able to understand them, identify failure or other dangerous situations like the consequences in the internal forces distribution of a failure in a joint, and propose remedial measures.

Structural defects must receive due consideration and the interventors must be aware of the need to guarantee suitable safety levels (sometimes by restraining the use of the structure to less demanding activities) despite the historical or architectural interest of the building.

## 3. Estimation of timber basic properties

Estimation of timber properties begins with the identification of wood species. This task although highly specialized, can be easily performed, especially when historical information is available (date of construction, region and wood species normally used in that period and region) thus the wood anatomist only has to confirm the species from a limited range of suspected species.

After wood species identification, the tricky task of allocating characteristic values (strength, stiffness) to the different timber structural members should be performed. In general three different types of approach are adopted by contractors. These are discussed below.

### 3.1. Sampling

Building contractors are often tempted with sampling and testing timber from the structure, copying the approach followed for other construction materials. Their sampling may involve one or two structural members that for some reason are to be replaced; or involve a small amount of wood taken from different locations within the structure: either to check clear wood properties, or state of conservation, as an attempt to evaluate its effects in strength.

Due to the natural variability of timber, such sampling can't be considered representative. To account for variability, the determination of characteristic values for the mechanical properties of timber is generally based in several samples of 40 timber pieces each, of structural dimensions from a certain timber grade (exhibiting representative defects and features of that grade). This is obviously not feasible within the assessment of existing structures, but explains why strength values obtained by sampling from the structure can hardly be used with confidence. Besides, sampling of clear wood disregards the major influence of defects like knots, slope of grain or fissures, and the effect of local biological damage, which may vary considerably within the structure.

Sampling is however very useful to provide information on species, moisture content and density. It can also be used to check if strength values of clear wood fall within the expected range or to restrain the quality of wood in a certain visual strength grade; but in order to do that the collected information shall be used in conjunction with methods specially developed to derive strength values appropriate for design.

### 3.2. Load tests

Load tests may be performed on the whole structure or parts of it (individual floor beams, for instance). Loading is applied to the structure by using hydraulic jacks or, more commonly, dead loads like water tanks or cement bags. The measured deflection of the structure under load is compared to the predictions of a structural model (generally a finite element modelling), and the estimated mechanical properties of the elements and joints (especially stiffness) necessary for the model are adjusted/calibrated to match the measured deflections.

These provide an estimation of elastic modulus (MoE) of timber, which alongside with density serves to derive strength values. Bending strength (MoR) and compressive strength are derived from existing correlations of these with MoE+density for that specific species. The other properties are generally derived from more general correlations between different properties.

Load tests may also consist of vibration tests. In this case, an instantaneous load (hit) is applied to the structure and the resulting motion is measured by accelerometers. The vibration frequencies are a function of geometry, support conditions and material properties, namely the elastic modulus. This enables an estimation of the MoE, which is subsequently used to derive the other mechanical properties.

Both techniques are expensive and time consuming and the interpretation of results depends on how accurately the support conditions and the influence of load-sharing elements can be understood and modelled. Results are known to be affected by moisture content, therefore local variations of moisture content may be another source of errors.

Moreover, results depend on how good the correlations between different properties are. It may happen that for the specific sample of timber used in a particular structure, the actual correlation deviates from the average correlation established for that species. Furthermore, for some species such correlations were never determined. It should also be stressed that the whole approach assumes that the correlations between different strength properties obtained for new timber of a given species also apply for old timber, even if it has suffered unknown load and environment history. Studies conducted so far on mechanical performance of structural members removed from old constructions do not seem to support the idea that age on its own is a factor that should be considered.

### 3.3. The grading approach

This can be considered the most currently followed approach. It involves the identification of the wood species and a general evaluation of timber members' quality (density, defects).

In this process ideally the evaluation of timber quality should be reported to an existing stress grading standard (used to industrially grade timber for structural uses), for which strength values can be allocated. Stress grading procedures check if the defects (specially knots, slope of grain, fissures and density) of individual members are within the limits established for a certain grade.

This evaluation allows the allocation of an average grade to all members. After a first structural analysis, a more refined grading can be carried out on those members which importance in the structure and/or high stress level justify extra attention.

The grading approach naturally requires an extensive survey of the structure, both costly and time consuming. It also assumes that the strength values allocated to new timber of a certain species and grade also apply to old used timber.

The influence of moisture content cyclic variations in the strength and stiffness of timber has been extensively studied and most results indicate that these have a negative effect. Also load history previously applied to timber is known to reduce strength and stiffness, highly depending on the stress levels attained and the environmental conditions. Other studies suggest that time itself enhanced some of the timber properties, although we lack precise information about the properties of that same timber when it was new.

Survey of timber structures have to deal with the large range of wood species used for construction, natural variability of timber (combination of wood species, growth region, physical properties and defects) and unknown load and environment history which brings a high degree of uncertainty on the assumptions taken from inspection.

## 5. Biological damage and estimation of cross sections

Cross section dimensions are in principle easy to assess, although in the case of irregular round elements this may require some simplification.

If biological degradation of timber is identified its overall and local effects on strength and stiffness of members and joints must be judged. This is normally a great source of problems for building interventors and a frequent justification for integral replacement of structures that would otherwise be kept in service.

In the case of fungal attack, the actual strength loss varies with wood species, specific fungus, time and environmental conditions. The affected volume may be assessed by non-destructive methods. Models to predict strength and stiffness reduction have been tried, but the allocation of equivalent values for the affected section in a practical situation involves a high degree of uncertainty.

Consequently, it is common to assume in practice a negligible contribution of those cross sections seriously affected by decay and to completely or partially replace those members or strengthen them as if they were already lost. In those cases where decay is incipient and it seems to affect just the surface of a member, that member may be kept in place if it is clearly overdesigned by report to calculated stresses.

Timber affected by insects is a totally different matter, as the decrease of timber members' properties result from a reduction of material in the cross section (rather than from a chemical modification of the wood cells).

When attacked by beetles, it is common that only a surface layer of sapwood is destroyed. The depth of the attacked layer may be assessed with the help of a simple knife or screw-driver or by other non-destructive techniques.

A reduced cross section may then be estimated. Alike the procedure followed for timber quality, safety checking may be carried out by assuming the same cross section reduction for all members in the structure, followed by a more detailed assessment and refined verification for specific members of higher importance or higher calculated stress levels. This procedure may however be too conservative, since even the damaged layer is likely to contribute to the load carrying capacity of that cross section.

Although less frequent, insect attack may instead take the form of a diffuse damage throughout most of the cross section. This has been observed in softwood members with a small percentage of heartwood. Considering a reduced cross section is meaningless in this case. Alternatively, a reduced quality due to a reduced apparent density may be assumed for such cases.

However, both approaches regarding insect damage involve a high degree of uncertainty since the real influence of insect attack in the strength and stiffness of timber members has yet to be clarified.

## 6. Conclusions

The lack of proper methods to evaluate the strength of timber members on-site is a strong drawback shown by timber structures, as compared to concrete or steel structures.

The assessment of old timber structures requires specific expertise and knowledge, given the need to quantify individual timber members and joints mechanical properties and effective strength. Regarding the timber members assessment, possible approaches basically include load tests and grading approach. Load tests rely on existing correlations between the timber strength and stiffness in structural dimensions. Such correlations only exist for a few species. Even then, one has to assume that the same correlation found for new timber also applies for timber that grew in different conditions and that was subjected to unknown loading and environment through its service life.

In the above "direct evaluation" of individual timber elements strength and stiffness, one assumes that the correlation between *species + origin + (quality) grade* and *strength properties* established for new timber also applies to old aged timber. However, this is yet to be confirmed. Besides, grading operations may be difficult in practice when there is no visual access to the ends and some faces of the timber, or when it is dusty or stained. Grading randomly chosen members may be an alternative in these situations but it introduces even another source of uncertainty.



The estimation of the effective cross section dimensions represents a huge problem. Options like assuming a nil contribution of decayed cross sections, or a reduced cross section in the case of beetles surface layer attack have been widely used in practice but may be too conservative.

Although a number of approaches may nowadays be used to assess old timber structures, they involve steps that still require scientific validation. Wood Science should help to reduce the present degree of uncertainty associated to some necessary assumptions, thus improving safety while avoiding too conservative evaluations that may obstacle the maintenance of still safe structures.

# EXPERIMENTAL INVESTIGATIONS ON FLEXURAL STRENGTHENING OF OLD WOOD MEMBERS IN HISTORICAL BUILDINGS WITH GFRP

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## Abstract

The aim of this paper is to describe a new innovative technology for strengthening old wood members in historical buildings, which had lost part of their strength due to heavy loads, loading time factor, effects of fungal or insect attacks. Most of the ancient buildings throughout Iran have been constructed mainly using wooden members. Some of the structural members in such valuable buildings are partially defective and, therefore, they seriously need to be repaired or rehabilitated, in order to maintain appropriate strength. Present research is focused on strengthening the old wood beam members with glass fiber reinforced polymer (GFRP) composite materials, sampling from non-essential sections of historical buildings. A three-point loading test was used to study the behavior of the beam subjected to gradual static load under different configurations of reinforcement. The load-displacement response, ultimate strength, modulus of elasticity and the mode of failures were evaluated. The experimental results have shown an improvement in load capacity, strength and stiffness of the hybrid samples in comparison with un-reinforced ones. At the end, the experimental and analytical results were compared with each other.

Keywords: GFRP, rehabilitation, retrofit, stiffness, strength, wood.

## 1. Introduction

Before the era of steel and concrete technology wood was the most common structural material in residential, public and industrial buildings throughout Iran, particularly in the Northern provinces which own commercial forest. The majority of existing ancient buildings in this country are over one century old; some of them are registered as historical heritage and are still standing in a good functioning shape. At present, conservation and restoration of these valuable buildings are the main concern of Iran's office of historical heritage. Inappropriate rehabilitation of the defective structural elements in historical buildings has had a great influence on their beauty and dignity, See Fig. 1.



Fig. 1. Replacement of old stringers with new material .

Over the past 40 years both fiber reinforced polymer (FRP) and non-FRP materials have been used to reinforce wooden structural members.

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During the last two decades, a special prefabricated fiber reinforced polymer (FRP) composite was developed to repair and retrofit wooden elements in the field. The advantages of these reinforcing materials lie in their low weight, high tensile strength and resistance to corrosion. According to recent investigations, FRP materials have been successfully used to strengthen existing structural members. Svecova and Eden (2004) tested timber beams reinforced with GFRP dowel bars as shear reinforcement as well as flexural bars to avoid the tension failures observed in some of the specimens [1]. Amy and Svecova (2004) presented an economical rehabilitation scheme to strengthen creosote-treated timber beams in both flexural and shear with glass fiber reinforced polymer bars [2]. Borri *et al.* (2005) presented a method for flexural reinforcement of old wood beams with CFRP materials. Mechanical tests on the reinforced wood showed that external bonding of FRP materials may increase flexural stiffness and bending capacity [3]. Corradi *et al.* (2006) presented in-plane shear reinforcement of wood beam floors with FRP [4]. Buell *et al.* (2005) presented an investigation on reinforcement timber bridge beams with a single Carbone-FRP (CFRP). A 69% increase of bending strength was reported when compared with control beam and a compression failure mode [5]. Calderoni *et al.* (2006) presented an evaluation of flexural and shear behavior of ancient chestnut beams under both experimentally and theoretically. The results have shown that the simplified methods commonly used to evaluate the bearing capacity of wooden beams can be safely applied to ancient structural members [6]. Dagher (2005) introduced current state of the art of reinforced wood technology, new products, codes and specifications [7]. Corradi and Borri (2007) presented a study on Fir and Chestnut timber beams reinforced with GFRP pultruded elements and the tests showed that the reinforcement with GFRP beams produced significant increase in flexural stiffness and bending capacity [8].

The purpose of this study is to present a new innovative technology, based on glass fiber reinforced polymer composite (GFRP) sheets, for the on-site rehabilitation of the old wooden members in historical buildings, through testing of small size wooden members and comparing the experimental results with numerical calculation.

### **3. Materials and methods**

#### **3.1. Wood Materials**

This experimental test is based on more than 50 specimens of ancient *Zelkova serrata* (Celtic Occidentalis) which is a hardwood. These specimens have been obtained from newly replaced and crushed structural elements of a historical building, located in the town of Babol, near the Caspian Sea, built up almost 150 years ago. Because of the limitation of obtaining enough old wood for testing and also because of major defects present in the main wood, like longitudinal splitting, cracking, degraded zones and holes due to nailing and insects attack, the samples were obtained in size of 25 × 25 × 410 mm, and tested according to ASTM D143 for small clear specimens of reinforced timbers [9].

#### **3.2. GFRP Materials**

In recent years FRP has been used as a compatible reinforcement material for timbers and plywood. The physical/mechanical/chemical properties of the FRP are very versatile. The FRP may be engineered to match and complement the orthotropic properties of wood; consequently, incompatibility problems between the wood and the reinforcing FRP are minimized. A unidirectional pultruded glass fiber-reinforced-polymer (GFRP) sheet with a thickness of 0.16 mm in size of 25 × 410 mm was used, having the physical and mechanical characteristics reported in Table 1, as provided by the producer.

#### **3.3. Resins**

The adhesive used in this test was Mac epoxy glue, which consisted of two parts, resin and hardener, mixed in the ratio of 3:1 by volume volume. The mechanical properties of the epoxy resin are reported in Table 1.

Table 1. Mechanical characteristics of the reinforcement material (reported by producer)

Material	Modulus of elasticity (E) MPa	Failure stress ( $\sigma$ ) MPa	Ultimate strain ( $\epsilon$ ) mm/mm	Poisson's Ratio ( $\nu$ )	Density ( $\rho$ ) g/cm <sup>3</sup>
GFRP	76000	2000	2.60	0.22	2.60
Epoxy resins	2000-5000	35- 100	1-60	0.35-0.40	1.10-1.40

### 3.4. Test set-up

In this experimental investigation, three-point loading bending tests were performed as shown in Fig. 2 according to ASTM D-143 [9] for finding out the flexural strength. The simply supported test method was selected to simplify the experimental setup. The wood specimens were supported at the butt and the tip, and the load was applied at the centre through a bearing block on the tangential surface nearest the pith Fig. 2 (b).



Fig. 2 – Static bending test assembly. (a) Testing apparatus, (b) sample assembly and loading

The free span between the two supports was  $L_s = 360$  mm for a total length of the specimen of  $L = 410$  mm. These spans were established in order to maintain a minimum span-to-depth ratio of 14. Both supporting knife edges were provided with bearing plates and rollers of such thickness that the distance from the point of support to the central plan is not greater than the depth of the specimen Fig. 2 (b). The knife edges were adjustable laterally to permit adjustment for slight twist in the specimen.

During the process of loading, test data were transferred to a computer that was programmed to collect and analyse all the data received from a load cell, connected to the Instron testing machine as shown in Fig. 2. The bending tests have been carried out for a series of control (un-reinforced) beams and for GFRP reinforced beams with varying area fraction of fiber reinforcement, as shown in Table 2.

Table 2. Mean dimensions of mid-span cross section for timber beams, reinforced with GFRP.

Samples	No.of specimen	Width (mm)	Depth (mm)
Control	5	25.17	25.13
1 layer bottom	5	24.87	25.51
2 layers bottom	5	25.13	26.24
2 layers bottom +1 layer top + 2 strip	5	25.34	26.68
3 layers bottom	5	25.58	26.44
3 layers bottom + 1 layer top	5	24.79	27.70

The mid-span displacement, failure load, stress at rupture and modulus of elasticity have been measured automatically by a purposely written computer program . see Fig. 2 (a and b) and Table 3. The approximate moisture content of the samples at the time of reinforcement application was 12% and the relative humidity of the lab during the test was 50%.

## 4. Results and Discussion

### 4.1. Control specimens

According to the field of investigation, Zelkova serrata wood was the most useable material for structural elements in a high percentage of ancient buildings; therefore this wood has been selected for study. The results of the tests for control specimens are shown in table 3. In this test, the mid-span deflection, load capacity, modulus of elasticity in range of the 25% to 75% of the elastic region and the mode of failure were evaluated.

Table3. Experimental results of control (un-reinforced) samples.

Specimen Number	Displacement at rupture (mm)	Load at rupture (kN)	Stress at rupture (MPa)	Modulus of elasticity (Nmm <sup>-2</sup> )
1	16.46	3.92	131.80	9721
2	11.22	3.70	127	11740
3	11.59	3.22	110.10	9495
4	10.85	3.41	116.40	10440
5	12.79	3.32	111.20	8554
Mean:	12.58	3.51	119.30	9990
S.D.	2.29	0.29	9.70	1186

### 4.2. GFRP-Wood hybrids

It should be noted that at first, some samples were tested with short length reinforcement only on the high moment zone, but the results obtained were not satisfactory. For this reason, all the other tests have been conducted with full length bond with size of 410 mm long.

In this case, the reinforcement of wood, with glass fiber reinforced polymer caused a considerable increase in strength and stiffness under bending test. The load-displacement curves for different schemes of reinforcing are shown in Fig. 4 (a to e).

The results of this investigation have shown that the load-displacement behaviour of all the control specimens and low fraction of reinforcement samples, like one and two layers, are almost linear up to the failure point, as shown in Fig. 3a and 3b. However, for those with more than two layers of reinforcement the behavior changes toward nonlinearity, as shown in Fig. 3(c to e).

The samples under test experienced either a brittle tensile collapse or a ductile compressive failure. All control specimens and those reinforced with up to two layers of GFRP failed on the tension side. The samples reinforced with three or more layers of GFRP showed ductile compressive failure.

The test results have shown that the effect of GFRP composite reinforcement on the mechanical properties of GFRP-wood hybrid is positive. In all the experimental cases there was a significant increases in the strength and stiffness of the reinforced specimen, compared with the control. The most effective results in GFRP-wood hybrid were obtained when the specimens were reinforced with three layers of GFRP on the tension side, and one layer on compression side, as shown in Fig. 3e. In this case, load capacity, stress at rupture and the modulus of elasticity increased by 57.10, 31.26 and 9.61%, respectively when compared with control specimens, as shown in Table 4.

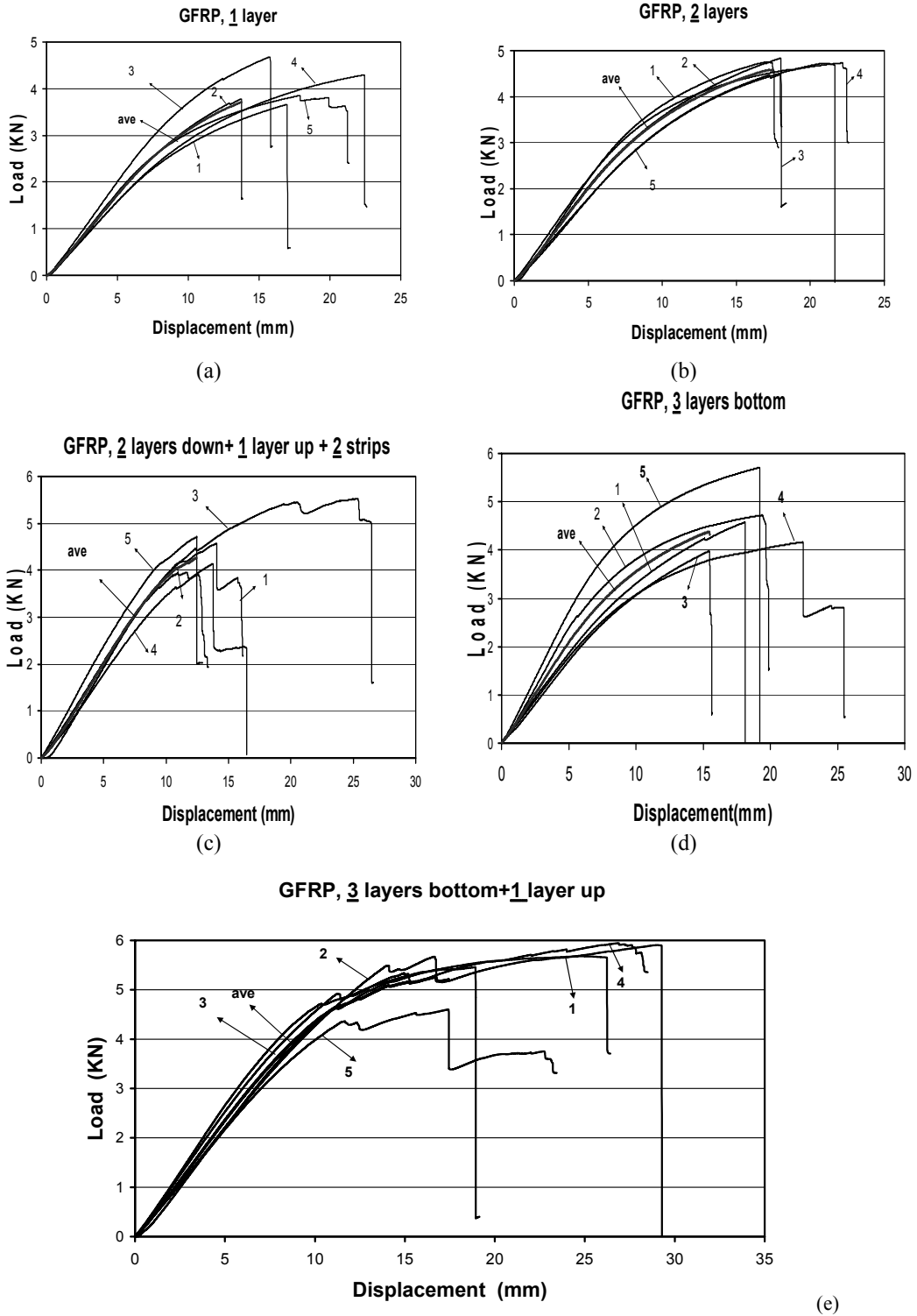


Fig. 3. Load-displacement curves for different configurations of GFRP – wood hybrids. (a) Specimens are reinforced with one layer of GFRP, (b) two layers reinforcement, (c) two layers down plus one layer up plus two strips rounded near the supports, (d) three layers reinforcement, (e) three layers reinforcement on bottom and one layer on up.

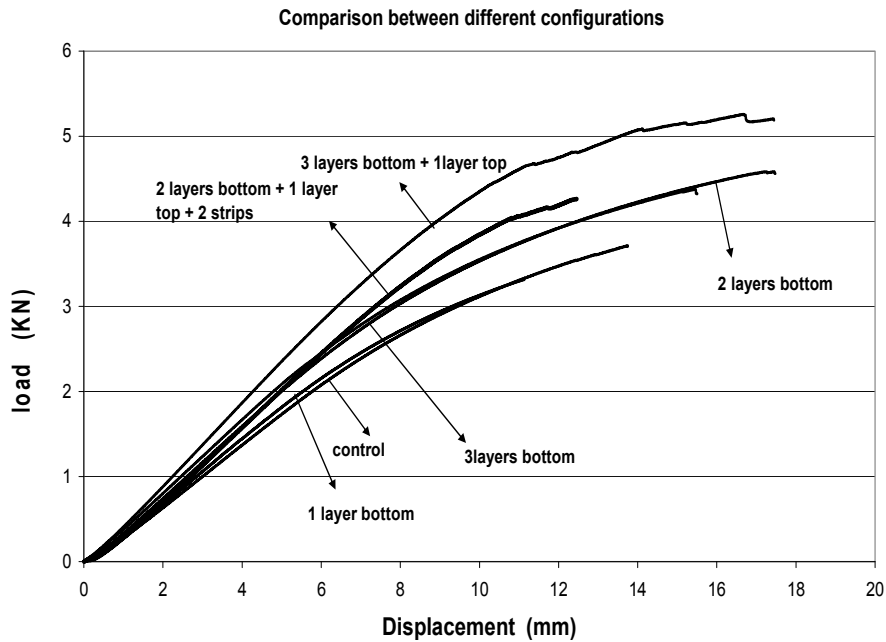


Fig. 4. Comparison between the results of average curves from each group of reinforcement.

Table 4. The mean value of the test results in different reinforced schemes of wood- GFRP hybrid

Samples		ultimate Load (KN)	Increase %	ultimate Stress (M Pa)	Increase %	Modulus of elasticity (E)	Increase %	Failure Mode
Control	Mean Value	3.51		119.30		9990		Brittle failure in tensile side
	SD.	0.29		9.70		1186		
1 Layer bottom	Mean Value	4.06	15.48	135.40	13.50	9551	- 4.40	Brittle failure in tensile side
	SD.	0.42		14.38		1435		
2 Layers bottom	Mean Value	4.71	34.02	147	23.20	9608	- 3.82	Ductile failure with debonding in tension side
	SD.	0.13		5.30		1160		
2 Layers bottom + 1 Layer top + 2 strip	Mean Value	4.58	30.46	137.40	15.17	10959	9.70	Ductile and tension failure
	SD.	0.61		19.60		1546		
3 Layers bottom	Mean Value	4.63	31.77	141.80	18.86	9558	- 4.32	Delaminated with tensile failure
	SD.	0.67		17.80		2457		
3 Layers bottom + 1 Layer top	Mean Value	5.52	57.10	156.60	31.26	10950	9.61	Ductile with compression failure
	SD.	0.55		12.80		865		

## 5. Conclusions;

In general, the results of the experimental tests have shown an interesting improvement in the strength, stiffness and failure behavior the reinforced specimens. In particular:

1. Generally, glass fiber polymer reinforcement increased the load capacity, strength and stiffness for all of the different configurations; also, it has a good advantage with respect to visual aspects of in restoration in historical members, since it becomes colorless after application.
2. Samples reinforced with three layers on the tension side and one layer on the compression side produced the best performing configuration, because, better strength and stiffness were exhibited and also all samples were fully plasticized at failure stage.
3. The samples reinforced with more than two layers of GFRP composite material provided a complete ductile failure on the compression side.
4. Full length bond between wood and GFRP provided a more ductile failure than other configuration of reinforcement with higher properties.

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# STRUCTURAL BEHAVIOUR OF TRADITIONAL MORTISE-AND-TENON TIMBER JOINTS

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## Abstract

Timber is one of the most used materials in the roofs and floors of monumental constructions in Portugal. However scarce information is available on the behaviour of traditional mortise-and-tenon joints and the effect that wood properties have on that behaviour. A study was then carried out on the assessment of ultimate strength capacity, global deformation and failure patterns of wood mortise-and-tenon joints. The study comprise three parts: 1) dealing with the mechanical testing of recent cut chestnut sawnwood and chestnut wood from old timber members; 2) with the experimental test of these joints; 3) with the development of a numerical model (taking wood properties from the results obtained in the first part) and comparison between model and experimental results. The validation of the non-linear model was performed by means of a comparison between the calculated numerical results and experimental results. The results seems to support that safety assessment of existing timber structures can be made using mechanical and physical properties data from current available chestnut wood. Also, the numerical results provide very good agreement with the experimental results.

## 1. Introduction

Timber is one of the most used materials in the roofs and floors of monumental constructions in Portugal. In particular, Chestnut (*Castanea sativa* Mill.) is usually present in noble constructions, given not only its mechanical and durability properties, but also its aesthetic characteristics. Careful conservation or rehabilitation of an existing construction implies extensive knowledge about the materials from which the structure is made, both from the mechanical point of view and from the physical point of view. This knowledge constitutes the support to evaluate the short-term structural behavior and to foresee the continuous adaptation and capacity of response of the material under adverse factors (long-term structural behavior).

In the past, timber structural design was dominated by the carpenter empirical knowledge about timber structural solutions that performed reasonable well (did not fall), either based upon his previous works or the works of others. Although empirical, the observation of old timber structures show evidence of perfect awareness that some members were subjected to tension and others to compression stresses. A key factor on a suitable structural behaviour of a timber structure deals with a proper design of timber joints. Joints hold on the structure, sustaining the stresses imposed on it and distributing the load between structural members and other parts of the structure. In traditional timber constructions load distribution through joints was made with empirical knowledge, transmitted and improved through generations. The early design rules or standards were built upon this empirical evidence.

A study on mortise-and-tenon joint type was selected because is one of the most commonly used and a typical example of an interlocking joint. Mortise and tenon joints were the basic components of joint craftery in Portugal for connecting two or more linear components, forming a “L” or “T” type configuration. The key problem found in these joints is the possible premature failure caused by large displacements given among other factors its limited shrinkage restraint capability. Unlike most timber joints, the load-displacement behaviour of these joints is generally very ductile.

The bearing capacity of mortise and tenon joints is a function of the angle of the connection, and length of the toe and mortise depth. According to the European building codes, joints are of crucial importance for the seismic design of timber structures. However, there are no recommendations on the design codes about general dimensions, such as length of the toe and the mortise depth in order to avoid structural failure of the connections, being design still very much based on empirical rules.

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Therefore, the present study on mortise-and-tenon joints looks into investigate the static behaviour of real scale replicates, considering joints made from new (NCW) or old (OCW) timber elements, characterize the ultimate strength and the global deformation of the joint as well as the respective failure patterns.

## 2. Experimental tests

The present work was carried out with two complementary phases: in a first phase, experimental work has been conducted on a total of 342 specimens of clear wood, with no visible chemical, biological or physical damage, which included standard compression tests, parallel and perpendicular to the grain, and standard tension tests, parallel to the grain (Fig. 1). For this purpose, it was decided to consider specimens from recently chestnut sawn timber (NCW), which is now available on the market for structural purposes, and specimens from old chestnut timber structural members (OCW) obtained from ancient buildings (in service for over 100 years). Tab. 1 shows the results obtained for the mechanical properties determined over the two different wood samples chosen.



Fig. 1. Test set-up for destructive tests: (a) compression parallel to the grain; and (b) tension parallel to the grain.

Table 1: Compression and tension parallel to grain results.

Mechanical properties			Wood type	
			NCW	OCW
Compression	Strength	Average (N/mm <sup>2</sup> )	42.9 (15)	47.6 (14)
		Characteristic value (N/mm <sup>2</sup> )	32.2	36.2
	Modulus of elasticity	Average (N/mm <sup>2</sup> )	7700 (16)	8800 (8)
		Characteristic value (N/mm <sup>2</sup> )	5700	7600
Tension	Strength	Average (N/mm <sup>2</sup> )	47.4 (29)	48.1 (23)
		Characteristic value (N/mm <sup>2</sup> )	40.1	41.4
	Modulus of elasticity	Average (N/mm <sup>2</sup> )	11500 (19)	13700 (19)
		Characteristic value (N/mm <sup>2</sup> )	8900	10600

(...) Coefficient of variation

Table 2: Comparison between compression and tension parallel to the grain characteristic values.

NCW		OCW	
$E_{t,0,05}/E_{c,0,05}$	$f_{t,0,05}/f_{c,0,05}$	$E_{t,0,05}/E_{c,0,05}$	$f_{t,0,05}/f_{c,0,05}$
1.32	1.41	1.18	1.23

In a second phase, experimental testing of mortise-and-tenon joints was carried out. A testing set-up was built aiming to test the specimens under compression being the procedure based on EN 26891 (1991) requirements, see Fig. 2a. Eight joints were tested (four for each group of wood) by joining two NCW (or two OCW) elements (brace and rafter). The joint had in mind the need to avoid “Shear Block” effect on the structural behaviour observed.

The results of the experimental joints tests are presented in Tab. 3. It can be seen that the results presents a huge scatter, ranging from an ultimate force of 98.5 kN up to a force of 161.5 kN. Specimen J\_7 can possibly be discarded because the value found is too low and is controlled by a local defect:

the large longitudinal crack in the rafter. In this case, the average ultimate force values of the groups NCW and OCW are almost the same, which is in agreement with the values of density found for the sample.

Table 3: Joints experimental results.

	Ultimate Force (kN)	Average (**)	Std. Dev.	Group
J_1	121.6	145.4	18.9	NCW
J_2	161.5			
J_3	159.7			
J_4	138.9			
J_5	126.4	133.8 (145.5*)	27.2 (16.7*)	OCW
J_6	157.1			
J_7	98.5			
J_8	153.0			

(\*) average discarding specimen J\_7

(\*\*)Average values of density (one specimen for each timber element)

Fig. 2b and Fig. 2c show typical individual load-displacements diagrams and envelopes of load-displacement diagrams. From the load-displacement diagrams obtained the following relevant remarks can be drawn. In a first phase, the diagrams always start with an upward curvature, exhibiting a nonlinear, non-recoverable, “bedding” response, which is due to the adjustment of the tenon and the mortise. In a second phase, within working stress levels, the response exhibits an approximately linear branch up to the conventional maximum load, which occurred at an average displacement of 8mm for the NCW group and 7mm for the OCW group.

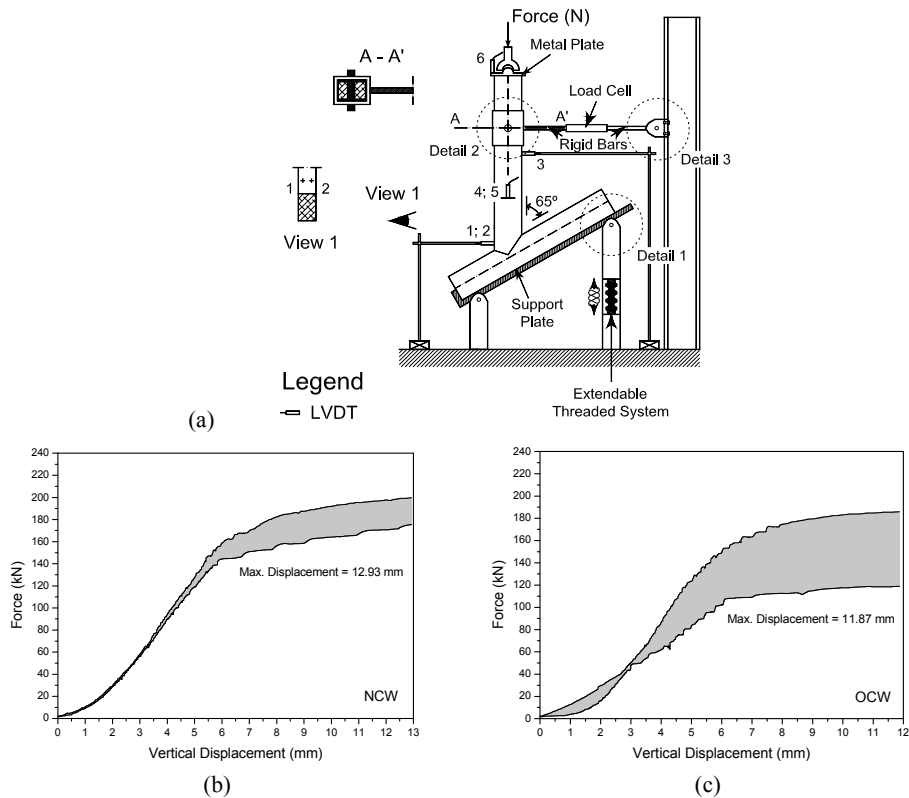


Fig. 2. (a) aspects of the destructive test set-up; (b) and (c) envelope of load-displacement diagrams for the NCW group and the OCW group, respectively.

The value of the displacement associated with the maximum load is lower for the NCW group joints in comparison with the OCW group joints, possibly indicating a slightly larger deterioration of the timber of the OCW specimens. Finally, after the conventional maximum load the displacement increases rapidly with a much lower stiffness, due essentially to the compressive failure of the wood in the rafter around the joint.

Having in mind the use of these results for numerical analysis, the true load-displacement diagrams were corrected with an offset that eliminates the upward curve related to the nonlinear behaviour of the joint previous to full contact (joint closure).

Finally, in a third phase, a finite element non-linear analysis of the joint was used. The multi-surface plasticity model adopted comprehends a Rankine type yield surface for tension and a Hill type yield surface for compression.

### 3. Numerical analysis

In structural mechanics, a problem is usually considered to be nonlinear if the stiffness matrix or the load vector depends on displacements. Nonlinear analysis is used to trace the equilibrium path up to and beyond the first critical point, at which the structure becomes unstable. There is one algorithm commonly used in the incremental iterative solution of nonlinear problems: the Newton-Raphson method. The full Newton-Raphson method, with stiffness matrix update in each iteration is used in the analyses carried out in this work. Two different finite elements were considered in the plane stress analyses carried out in this work: continuum elements (8-noded) to represent wood and line interface elements (6-noded) to represent the interface between rafter and brace. The integration schemes used are  $2 \times 2$  Gauss integration points for the continuum elements and 3 Lobatto integration points for the interface elements.

#### 3.1. The adopted anisotropic failure criteria

A plane stress continuum model, which can capture different strengths and softening characteristics in orthogonal directions, was formulated by Lourenço (1996). The proposed failure criterion consists of an extension of conventional formulations for isotropic quasi-brittle materials to describe orthotropic behaviour. It is based on multi-surface plasticity, and wood is an example of a material for which this criterion applies, having different strengths in the directions parallel and perpendicular to the grain. Formulations of isotropic quasi-brittle materials behaviour consider different inelastic criteria for tension and compression. In this formulation, and in order to model orthotropic material behaviour, a Hill yield criterion for compression and a Rankine yield criterion for tension were adopted.

#### 3.2. Adopted material parameters

A characteristic of the adopted model is that the tension strength, in a given direction, must be equal or lower to the compression strength in the same direction. This does not hold for wood. Here, the tensile part of the yield criterion was ignored due to the irrelevant contribution of the tensile strength in the global behaviour of the joint. This means that the yield surface reduces to the standard Hill criterion. The adopted elastic and inelastic material properties used in the analyses are detailed in Tab. 4.

Table 4: Adopted elastic and inelastic material properties.

$E_x$	$E_y$	$G_{xy}$	$\nu_{xy}$
800N/mm <sup>2</sup>	8500N/mm <sup>2</sup>	1500N/mm <sup>2</sup>	0.3
$f_{c,x}$	$f_{c,y}$	$\beta$	$\gamma$
7N/mm <sup>2</sup>	45N/mm <sup>2</sup>	-1.0	3.0

The shape of the adopted yield criterion in the compression-compression regime, features an extreme degree of anisotropy with a ratio  $f_{c,x}/f_{c,y} = 0.156$ .

### 4. Numerical vs. experimental results

A structured mesh is used for the rafter and the brace, whereas an irregular transition mesh is used in the vicinity of the connection between rafter and brace. Interface elements are also used between the

rafter and the brace. The thickness ranges from 62 mm to 93 mm. This aims at representing the thickness of the mortise.

A preliminary analysis with an infinite stiffness of the interface, assuming a fully rigid connection, indicated that such an assumption provided far too stiff results. Therefore, the stiffness of the interface elements was obtained by inverse fitting. A first conclusion is that the stiffness of the interface elements has considerable influence in the yield strength of timber joints. In Fig. 3a, three distinct situations are presented: a numerical simulation with infinite stiffness of the interface elements ( $k_{infinite} = k_n = k_s = 10^9 \text{ N/mm}^3$ ); a numerical simulation with an adjusted stiffness of the interface elements obtained by inverse fitting of the experimental results ( $k_{fit}$ ):  $k_n = 6000 \text{ N/mm}^3$  and  $k_s = 2308 \text{ N/mm}^3$ ; and a numerical simulation with a spring ( $k_{spring} = 10^6 \text{ N/m}$ ) located in the brace to simulate the reaction cell used in the experimental sets. The stiffness of the spring was again obtained by inverse fitting of the experimental results, keeping the adjusted stiffness of the interface elements.

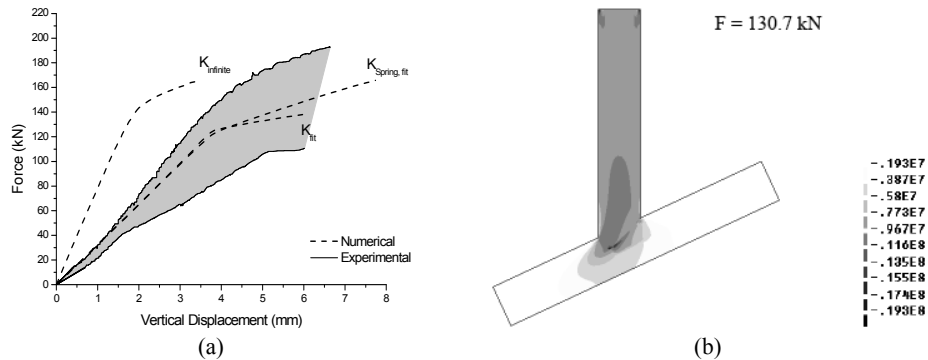


Fig. 3. (a) comparison between numerical and experimental load-displacement diagrams; and (b) minimum principal stresses (values in  $\text{N/mm}^2$ ).

The numerical results, in terms of force-displacement diagrams, with the adjusted stiffness for the interface elements, provide very good agreement with the experimental results both in the linear and nonlinear parts. The influence of the experimental horizontal restraint, simulated by a linear spring, is only marginal. The usage of infinite stiffness for the interface (rigid joint) results in an increase of the slope of the first part of the response, from  $30 \text{ kN/mm}$  to  $80 \text{ kN/mm}$  (+ 266.7%). The ultimate strength of the joint, given by an offset of the linear stretch by 2% in terms of strain values, also changes from  $130 \text{ kN}$  to  $152 \text{ kN}$  (+ 17%), once the joint becomes fully rigid. Fig. 3b shows the contour of minimum principal stresses at the end of the analysis. It is possible to observe a concentration of stresses in a narrower band with peak stresses at the joint (zone where the interface elements were placed).

With this concentration of stresses one may say that failure is clearly governed by wood crushing where, for a late stage of the analysis, the compressive strength of the wood in the joint is completely exhausted. This situation is also confirmed in the experiments.

## 5. Effects of the material parameters

A strong benefit of using numerical simulations is that parametric studies can be easily carried out and the sensitivity of the response to the material data can be assessed. There are a total of six key parameters in the present model and the effect of each parameter on the global response will be analyzed separately. It is noted that moderate variations ( $\pm 25\%$ ) are considered for the strengths and large variations (division/multiplication by two) are considered for the stiffness values. These assumptions are rooted in the fact that strength is usually better known than stiffness.

### 5.1. Normal and tangential stiffness of the interface

Fig. 4a shows a comparison between the results of the variation of the  $k_n$  parameter: with a reduction of 50% in  $k_n$ , the ultimate strength of the joint, given by an offset of the linear stretch by 2%, decreases from  $127.2 \text{ kN}$  to  $120 \text{ kN}$  (-6%).

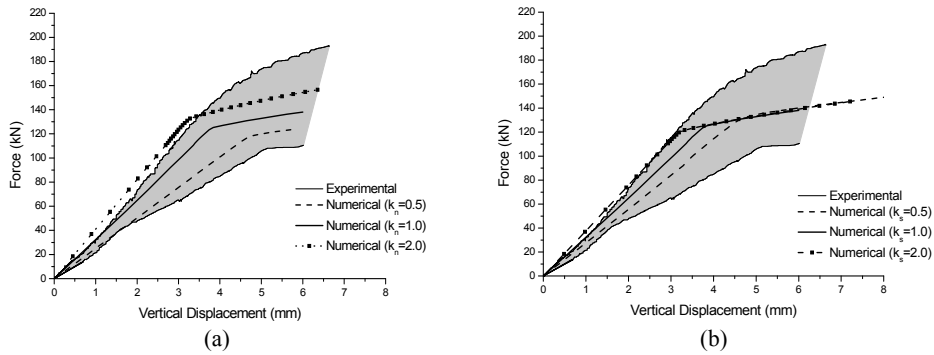


Fig. 4. Effect of the variation of parameter: (a)  $k_n$ , and (b)  $k_s$  on the model response.

Multiplying  $k_n$  by a factor of two the ultimate strength of the joint, given by an offset of the linear stretch by 2%, increases from 127.2 kN to 135.0 kN (+7%). The reduction/increase of the normal stiffness of the interface also affects the global stiffness of the joint: the global stiffness of the joint decreases as the normal stiffness of the interface decreases, being more sensitive to this variation when compared with the ultimate strength. The reduction of 50% of this parameter, results in a decrease of the slope of the first part of the response, from 32 to 26 kN/mm (-23%). On the other hand, the multiplication by a factor of 2 results in an increase of the slope of the first part of the response, from 32 to 41 kN/mm (+28%). Because this parameter sets the relation between the normal tension and the normal displacement, the obtained results were expected *a priori*.

Fig. 4b shows a comparison between the results of the variation of the  $k_s$  parameter. The ultimate strength is insensitive to a  $k_s$  variation, whereas the reduction/increase of this parameter affects the global stiffness of the joint: the global stiffness of the joint decreases as the  $k_s$  parameter decreases. The reduction of 50% of this parameter, results in a decrease of the slope of the first part of the response, from 32 to 28 kN/mm (-14%). On the other hand, the multiplication by a factor of 2 results in an increase of the slope of the first part of the response, from 32 to 37 kN/mm (+16%).

## 5.2. Elastic modulus and compressive strength

The effect of the variation of the elastic modulus of elasticity parallel and perpendicular to the grain was considered individually. Fig. 5a indicates that the ultimate strength is almost insensitive to the variation of the elastic modulus of elasticity for wood ( $\pm 4\%$ ).

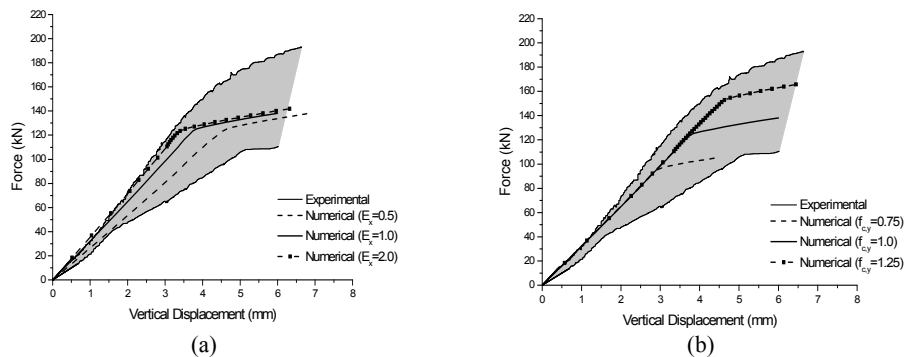


Fig. 5. Effect of the variation of the: (a) elastic modulus of elasticity ( $E_x$ ), and (b) compressive strength ( $f_{c,y}$ ) on the model response.

The inclusion of the effects of the elastic modulus of elasticity does change significantly the elastic stiffness of the joint. Therefore, decreasing the parameter  $E$  decreases the global stiffness of the joint. The reduction of 50% of the  $E_x$  parameter, results in a decrease of the slope of the first part of the response, from 32 to 28 kN/mm (-14%). On the other hand, the multiplication by a factor of 2 results in an increase of the slope of the first part of the response, from 32 to 36 kN/mm (+13%).

The ultimate strength and the global stiffness of the joint are insensitive to the variation of the compressive strength of wood in the parallel direction. Fig. 5b indicates higher sensitivity of the ultimate strength of the joint to the variation of the compressive strength of wood in direction perpendicular to the grain, as expected: with a reduction of 50%, the ultimate strength of the joint, given by an offset of the linear stretch by 2‰, decreases from 130 kN to 100 kN (-30%); multiplying by a factor of 2 the ultimate strength of the joint, given by an offset of the linear stretch by 2‰, increases from 130 kN to 160 kN (+23%). However, the global stiffness of the joint is insensitive to the variation of the compressive strength perpendicular to the grain.

## 6. Conclusions

From the results obtained in the first part of the study no significant difference was found between the mechanical characteristics of the old wood and new wood, although the former shown a slightly higher values (7-12%) is could be easily allocated to the natural variability of wood.

Comparing the characteristic tensile strength and modulus of elasticity values with the characteristic compressive results one can conclude that the characteristic strength values are slightly higher in tension parallel to the grain than in compression parallel to the grain ( $\approx 32\%$ ). The elastic modulus of elasticity results are also slightly higher in tension parallel to the grain than in compression parallel to the grain ( $\approx 25\%$ ).

The second part of the study shows that the difference in mortise-tenon joints results for the ultimate load between the two groups is very low, which is in agreement with the values of density found for the sample. Thus, the results seems to support, although more studies should be conducted on timber removed from members in-service, that safety assessment of existing timber structures can be made using mechanical and physical properties data from current available chestnut wood.

Regarding the third part, the different failure mechanisms observed in the experiments are well captured by the numerical model, which is the most important validation of any simulation. It is striking that such excellent agreement is obtained also in the load-displacement diagrams.

A preliminary analysis considering an infinite stiffness of the interface, assuming a fully rigid connection, indicates that such an assumption provides too stiff results. Another conclusion is that the normal stiffness of the interface elements has considerable influence in the yield strength of timber joints. The numerical results, in terms of force-displacement diagrams, with the adjusted stiffness for the interface elements, provide very good agreement with the experimental results both in the linear and nonlinear parts. The influence of the experimental horizontal restraint, simulated by a linear spring, is only marginal.

It has been shown that the parameters that affect most the ultimate load are the compressive strength of wood perpendicular to the joint and the normal stiffness of the interface elements representing the contact between rafter and brace. The tangential stiffness of interfaces and the Young's moduli of wood have only very limited influence in the response. The compressive strength of wood parallel to the grain has almost no influence in the response.

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# WOODEN LOAD BEARING STRUCTURAL ELEMENTS OF KULA TRADITIONAL HOUSES CASE STUDY OF ZABUNLAR AND GÖLDELİLER HOUSES

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## **Abstract**

In this paper, two important Kula houses are focused as a case study. These examples are chosen because one of the renovation works of these buildings is done and other is in process. These buildings are Zabunlar House, and Hikmet Bozkurt House. The wooden load bearing structural elements of these two main buildings are analyzed from the point of view of dimensions, construction methods, junction methods of elements, and renovation techniques of the buildings.

## **1. Introduction**

Throughout history, wherever wood has been available as a resource, it has found favor as a building material for its strength, economy, workability and beauty.

Wood remains one of our most important renewable natural polymers, providing shelter, supporting transportation, and even allowing us to effectively communicate via writing. However, wood is a biological material that can be degraded by a variety of organisms. In buildings, the agents of deterioration are primarily fungi and insects, and their occurrence is generally a function of conditions created through original construction, changes in designs, or the lack of rigorous maintenance [1].

The Turkish House can be defined as the types of houses which Turks have lived in throughout history. The basic-system of construction is the timber frame with infilling material or the lathe and plaster [2]. The timber frame construction is compatible with the forest cover of Anatolia and the Trace region and is also preferable to other type of construction methods because these regions are within the seismic zones. Furthermore, this method enabled quick construction and therefore suited the needs of an ever-expanding society, continuously on the move. For the same reason the details of wood construction are very simple; simple joints and nailed bindings have been preferred to complicate joint details. This construction method also facilitated the reconstruction, within a short time when whole quarters were destroyed instantaneously by fire [3]. The timber frame construction also facilitated opening more windows building projections and wide eaves. This provided control over climatic conditions, and enabled the building to breathe in humid climates, which, in turn, helped prevent condensation and moisture in the rooms.

## **2. Definition of wooden load bearing structural elements of kula traditional houses**

Kula is a traditional settlement, located in the middle of the Aegean Zone of Turkey, and has very important architectural heritage of wooden structures dating back to 2-3 hundreds of years. The construction system of Kula houses is a mixed type. First floors are made of load-bearing stones with mud mortar, which are supported by horizontal timber lintels embedded into bearing wall masonry. This system is called as Hatıl construction system [2]. Upper floors are made of timber frame construction. This timber frame construction seen in the territory constructed with two main techniques which are Bagdadi and Himis technique.

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Fig. 1: Horizontal Timber Lintels

Hımiş (Timber Framed structures) construction is simply described as a timber frame with masonry infill such as bricks adobes or stones. This type of construction is a variation on a shared construction tradition that has existed through history in many parts of the world. It is possible to classify traditional hımiş construction depending on the structural system such as; system contained bracing elements and no bracing elements [2]. But, since the Kula is in the seismic region of the country, the studs are supported especially by diagonal bracing elements to strengthen the building against horizontal forces. Bagdadi construction where the voids between timber framing members is filled with lighter materials or with trunk shells are transformed into a filling material by sand and lime mortar. The interior surfaces of walls are covered by lath and plaster work or wood, whereas the outer surfaces are either plastered or non-plastered or wooden plastered.(covered by wood planks) [2]. A timber frame with masonry infill which is defined as Hımiş Technique (timber framed structure), is the most commonly used technique in Kula Houses.



Fig. 2: Construction System of Kula Traditional Houses

### 3. Examination of the case study examples –zabunlar house and gödeliler house

Two important Kula houses are focused as the case study examples of renovation of historical timber structures. These examples are chosen because one of the renovation works of these buildings is done and other is in process. The first example is done by reconstruction method, and the second renovation work is replacing only the damaged timber elements with new ones and conserving the others in situ. Zabunlar house is a typical Ottoman house located in the territory and is renovated and turned into a hotel. Hikmet Bozkurt House, which is known as “Gödeliler House” once when it was built, and

owned by Armenians living in the city. This building is under renovation process and will be turned into a hotel as Zabunlar House. These two houses are located side by side, and after the renovation process of Hikmet Bozkurt House will be completed, the two courtyards of these two buildings will be used together.

### 3.1. Construction techniques of Zabunlar house - Gödeliler house

Both of these houses have the same plan scheme, and due to this factor same type of construction system is used in both cases. This system is the one that is commonly used in all the traditional houses in Kula settlement. These two houses are located in a courtyard, which is surrounded by high courtyard walls for obtaining privacy in the house. All the openings of the house the south façade (sofa façade of the building) is viewed from the courtyard. The main projected room to the street is called as “bas oda”. This room is the biggest room of the house which is owned by the elders of the family, and is reached from the differentiated part of sofa. All the rooms located in the first and the second floors are opened to a semi-open space called as “sofa” Sofa space has a unique part which has projection to the street, as well as the main room, this differentiated part is the specialized space which gives access to the main room. North facades of the houses have solid surfaces to prevent the interior of the houses from cold, and south facades have open surfaces to obtain sun and breeze in the interior of the houses.

The ground floors are used for service facilities such as barns, depots and because of the privacy item, the first floors and the courtyard of the house are covered with high and thick stone walls, which are made of kufeki (a type of stone) as the local stone element of the region. These walls are load-bearing elements and these walls continue till the second floors on the north facades to prevent the interior of the house from cold air, and wind. These walls are supported by wooden hatil elements timber tie plates regularly placed every 70–100 cm which strengthens the building against tensile stress (horizontal loads) especially during the earthquake loads.

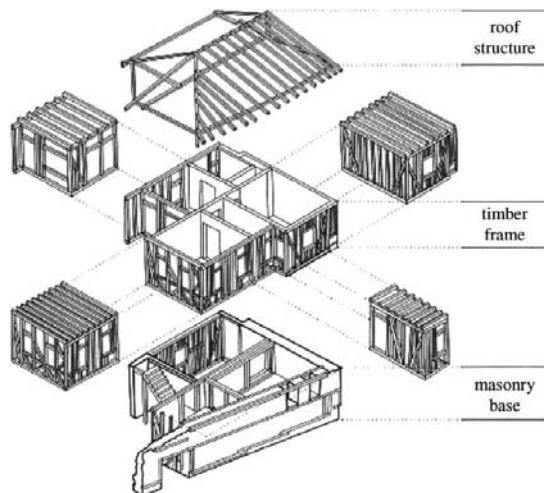


Fig. 3: Construction System of Timber Houses [4, pp. 849]

On the south-west-east façades the first and the second floors have timber framed structure with kufeki stone as infilling material. This system is used to obtain more openings on this façades, and also the timber frame system prevents the dead-loads affect the building. The first floor is made of kufeki stone masonry wall; these walls have 50-70cm thickness. The timber framed system is constructed on these masonry walls. Firstly a 12/12 cm timber beam for supporting secondary joists and as a base for the timber studs is placed on the masonry wall. Then approximately 10/12 cm studs made of pine tree are installed in approx. 1 meter distance with each other on the timber beam. These studs are connected with a wooden beam on the top level as well. These studs are supported with the secondary diagonal timber elements with 8/8 cm dimensions. In the cases of a need of openings such as windows or doors,

secondary studs are used for to obtain the necessary void for the openings. Two secondary timber beams, one in the bottom of the opening and one at the top of the opening (lintel) supports the void, and are connected to the main studs with nails. The walls are filled with kufeki stone to form the surface. The construction is plastered by “kıtıklı sıva(plaster)” which is made of mud and straw.



Fig. 4: Gödeliler House- Structural Elements of Timber Framed Structure (Ozgul Yılmaz Karaman 12.10.2008)

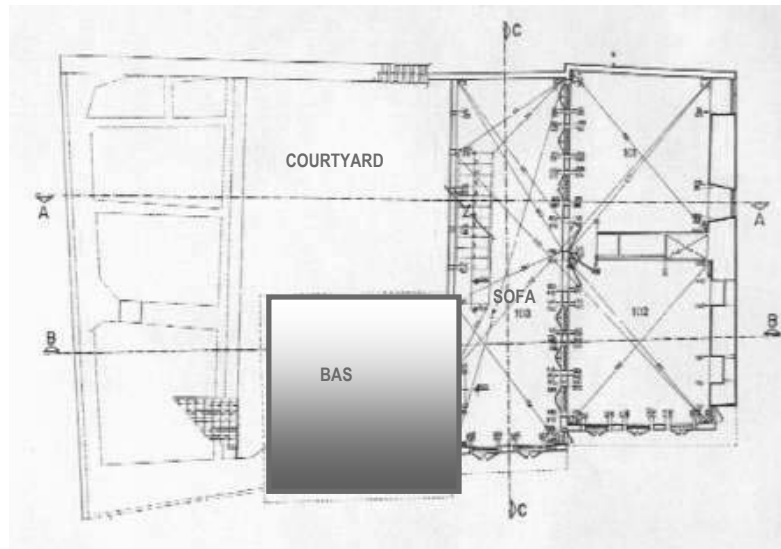


Fig. 5: Interior Plan Scheme of Turkish House

The masonry base (ground floor) fits the shape of the lot on which the building is located, whereas the upper floor is constructed in a regular geometrical shape with the projections built within the capabilities of the timber-framed system. Before the construction of the upper floor, the timber wall plates are placed on the inner and outer edges of the ground floor main walls. The free-standing posts placed in the semi-open circulation spaces known as tas-lik –sofa (Fig.-5) are connected horizontally to the main beams, forming a base for the upper floors. In the upper floors, the floor beams are placed on the wall plates below, forming the shape of the upper floor and the geometry of the room. The secondary floor beams (joists) which have 8/15 cm dimensions in sections, are spaced at approximately 50 cm intervals, parallel to the short side of the room. The spatial dimensions are usually determined according to the size of the available materials, whereas in some larger spaces such as the sofa — the main circulation hall in Ottoman houses — and tas-lik –sofa (Fig. 5) where a wide span is required, long timber beams with a relatively larger cross-section (like 15/20, 20/20 cm) are used. To built projection (cıkma in Turkish) on the upper floor, the load-bearing elements that carry the projection are constructed according to the type and extension of the projection and put in place at this stage. Main beam(s) is extended and the secondary joists placed on/between them. And the timber cantilever beams are supported by diagonal bracing elements (pit prop).

### 3.2. Deformation factors of Zabunlar and Gödeliler house

Deformation factors of the timber framed constructions are mostly the living organisms and rainwater containing the surfaces. Water serves a variety of functions in the decay process. It is a reactant, a swelling agent and a medium for diffusion of both degradative enzymes and wood degradation products. Most organisms that degrade wood require moisture content. The primary durability hazard with wood is biodeterioration. Wood in buildings is a potential food source for variety of fungi and insects. Timber frame structures that are commonly used in Kula region has less moisture content due to both the weather conditions of the settlement and the type of the wood that is found in the region.

The organisms that degrade Zabunlar and Gödeliler Houses are found only as “worms”.wood boring beetles Due to the moisture levels being low, an occurant type of fungal organisms had not been found attacking the pine wood structural elements used in the houses.

The rainwater is removed from the structure easily by the help of long eaves surrounding the buildings. These eaves are 80-100 cm long timber elements. Rain penetration control is sustained by these long eaves, minimizing the amount of water containing the building surfaces. .

When both buildings are observed structurally, it can be seen that Zabunlar House had corner cracking especially at openings, wall deformation, deformation of walls along the wooden beams and separation of walls from the beams, roof separation from wall, partial collapse in the structural system. Gödeliler House has beter structural conditions, less structural deformations than Zabunlar House.

## 4. Conclusion -renovation works of the cases

These two houses dated to same period and constructed about mid 19<sup>th</sup> century. The timber structures were about 200 years old. The deteriorations that happened within the wood structure were caused mainly of wood boring beetles Now that the territory had no humidity problems, the pine structural elements did not suffer from Fungi attacks. Now that the territory was not situated within a seismic zone, the wooden structure did not suffer from the tensional earthquake stress as well.

The habitants of Zabunlar House left the building earlier than Gödeliler House, so periodically renovations can not be applied to the building as it happened in Gödeliler House. As a matter of fact; firstly some structural deformations started to occur on the roof structure of Zabunlar House, by the help of the water coming from the top the building these deformations installed within the timber frame structure elements and masonry wall with mortar. The corners of the structure cracked especially from the openings, the masonry wall separated from the wooden structure, the building partially collapsed. In this case in order to stabilize the masonry building and the timber structure, the timber structure was all taken, and replaced with the new timber elements, and tied to the masonry which is conserved in situ. The restoration process of Zabunlar House is done with reconstruction technique. The north façade of the building-the load bearing masonry wall is protected because it needed no interventions, but the whole timber structure (pine joists, studs, secondary beam elements etc.) is displaced with new pine elements.

Gödeliler House is in better condition, the structure is well-preserved. The roof is partially damaged, so the roof elements are replaced with the news, so little amount of them preserved in situ. The first and the second floor slab elements are in good condition as well, there is no fungal or wood boring beetlestacks either but a high rate of deflection is obtained in the slabs, so in order to fix it, all the slabs were removed and replaced with new pine elements. The other structural elements studs, joists, beams etc. are mostly preserved in situ and the ones which have deformations, and biodeteriotions are replaced.



Fig. 6: Renovation Process of Zabunlar House (Kula Municipality Archieve)

These two examples can be examined in the meaning of preservation conception. Most of the the timber elements of Zabunlar House is renewed instead of conserving them just to make the building safe against earthquake loads. This is a common way of restoring a mostly damaged building in Turkey. Now that most of the elements of Zabunlar House are replaced, the building became a replica. In the second case the Gödeliler House now that it was partially damaged, only the damaged timber elements renewed. In this case instead of renewing the damaged timber elements, they could be conserved in situ or in a laboratory, if the tests were professionally done. But anyhow by this manner, the building has preserved its authenticity because most of the elements remained untouched. When comparing these two renovation projects, Gödeliler House can be defined as a more successful case than Zabunlar House.

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(F) OTHER CONTRIBUTIONS  
TO WOOD SCIENCE





# INVESTIGATION ON THE UTILIZATION OF POMEGRANATE WOOD FOR PARTICLEBOARD PRODUCTION

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## Abstract

In this investigation, particleboard manufacturing has been offered to consider of lignocellulosic residues usage from resection of pomegranate trees (*Punica granatum*) in particleboard production using pomegranate and poplar (*P.nigra*) wood with 4 different combinations (1. 100% of pomegranate wood 2. 75% of pomegranate wood and 25% of poplar wood 3. 50% of pomegranate wood and 50% of poplar wood 4. 100% of poplar wood as the control sample) and using 2 resin content levels UF (10 & 12 %) and 2 press time levels (4 & 5 min). According to these factors, 16 combination samples were collected and 3 boards from each treatment, totally 48 experimental sample boards were made. The result of physical and mechanical properties were analyzed using Randomized Complete Block Experiment Design (RCBD). The result of this investigation showed that Modulus of Rupture (MOR) in boards has effectively reduced while increasing the press time. On the other hand, resin content content and press time have a direct impression on Modulus Rupture (MOR) and Modulus of Elasticity (MOE) in boards so that MOR and MOE decreased during 10% resin content and increasing the press time. These results also indicated that MOR and MOE of the boards have been increased by adding poplar particles to the wood combination for board production. Indeed, Internal Bonding (IB) of the boards has been modified by 105 resin content and increasing the press time. Anyway, all the Internal Bonding average values of boards have been higher than the standard level. The results also showed that Thickness Swelling (TS) of boards has been increased by adding poplar particles to the wood combination to board production.

## 1. Introduction

In recent years, because of industrialization of most of developing countries, demand for raw materials and production inputs has grown up. Considering the fact that raw material especially in natural resources sector is limited, one of the most important challenges facing the human communities in recent decades has been supplying the wooden raw material needed by the expanding industries on one side and protection of the limited forest resources on the other side. Therefore, in countries such as Iran which in respect of wood and forestry resources are considered poor it is necessary that for providing wooden raw material a special attention be paid to agricultural residues. This is because particleboard industry is able to use and consume a vast spectrum of wooden and non-wooden lignocellulosic residues. However, identifying and introducing new lignocellulosic materials to particleboard manufacturing unit for production of particleboard and or other wooden combined products need studies and research. Thus, the aim of this research has been studying the possibility of using the wood resulted from pruning pomegranate trees and optimization of pomegranate orchards as raw material for production of particleboard. Presenting the best combination of pomegranate wood and one other wood species (which in this study has been poplar) for production of particleboard having desirable physical and mechanical properties in industrial scale has been one of other aims of this research.

According to the statistics published by the Ministry of Agricultural Jihad, in year 1384 (21 March 2005-20 March 2006) more than 60,000 hectares of the country's lands were covered by pomegranate orchards. These trees shall be pruned each year. The preliminary studies have showed that the lignocellulosic waste resulted from pruning these trees amounts to about one ton per hectare which is indicator of potentially 60,000 tons lignocellulosic materials per year. Using this lignocellulosic waste (which are burnt every year after completion of pruning operations) as raw material of particleboard enjoys high economic justification.

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## 2. Materials and Methods

In this study, several of the factors related to manufacturing conditions had been taken to be variable. These variables were as follow:

1. Resin content level in making the particleboard was at 10% and 12% (based on the weight of the particleboard).
2. In this research, the press time in making the particleboard was 4 and 5 minutes.
3. In this research, the third variable factor was the combination of pomegranate wood and poplar wood. Four combinations exhibited in Table 1 were used.

Table 1-Percentage of pomegranate wood and poplar wood in wood combination and their abbreviated names

Abbreviated Name of the Wood Combination	Percentage of Used Wooden Material	
	Pomegranate	Poplar <i>P.n</i>
A	100	Zero
B	75	25
C	50	50
D	Zero	100

Other manufacturing factors including specific gravity of the board (0.75 gr/cm<sup>3</sup>), pressure (30 kg/cm<sup>2</sup>), moisture content of particle wood mat (12%), press temperature (175°C), thickness of the board (15 mm), liquid urea formaldehyde (UF) (50%) were taken to be fixed in all treatments.

For making particleboard for this research, the wood of pomegranate trees of suburbs of Kashan City of Esfahan Province was used. After being transported to the laboratory, the pomegranate woods were chopped to smaller parts by one rolling laboratory chipper and then were converted by a ring mill into particles usable in making particleboards. The moisture content of the prepared particles was about 30%. Using a drier and temperature of 120°C, the particles were dried and the degree of moisture content lowered to 1%. They were packed in resistant bags which were moisture insulators. For including poplar wood in the combination of the wood material used in making particleboard, 3 poplar trees of *P. nigra* species were cut down and carried to the laboratory. In the laboratory, particles were prepared from the carried poplar wood. For applying resin to the particles, one laboratory gluing instrument was used. From a nozzle and by using compressed air, the resin solution and one catalyst were sprayed on the fibres which were rotating inside in the resin spraying compartment. The resin solution, catalyst, and fibres were mixed together thoroughly. For making the mat, a wooden mould with dimensions of 40 x 40 cm was used. The wood particles, treated with the resin and weighed in a laboratory scales, were sprayed into the mould uniformly.

When the mats were prepared, they were pressed with a laboratory press and experimental particleboard samples were made. In this research, by combing the three variables in different levels, 16 treatments were obtained. Each treatment was repeated three times. So, totally 48 boards were made. After termination of press stage, for conditioning and making the moisture of the boards uniform as well as for giving equilibrium to internal stress, the produced boards were kept in laboratory conditions (relative humidity 65±1% and temperature 20±3°C).

For determining physical and mechanical properties of the boards, experimental samples were prepared by using one round saw. First, the boards were trimmed and then in compliance with DIN-68763 standard, samples were cut for determining Modulus of Rupture (MOR), Modulus of Elasticity (MOE), Internal Bonding (IB), Thickness Swelling (TS) after 2 and 24 hours immersion in water (T.S.2 and T.S.24).

After performing mechanical and physical tests on prepared samples, the results were analyzed using Randomized Complete Block Experimental Design under 3-variable tests and through using Dunken Test (DMRT) and with the aid of variance analysis technique. By this statistical method, the independent and mutual effects of each variable factor on the studied properties were analyzed at the confidence level of 99 and 95%. Table 2 shows the dimensions and number of the specimens in each repetition and treatment.

Table 2- Dimensions and number of the experimental samples in each repetition and treatment

Type of the Test	Dimension (mm)			Number of Samples in Each Repetition	Number of Samples in Each Treatment
	Length	Width	Thick-ness		
Modulus of Rupture and Modulus of Elasticity	250	50	10	4	12
Internal Bonding	50	50	10	4	12
Thickness Swelling	50	50	10	4	12

### 3. Discussion and conclusion

In this study, particleboard was made from four different combinations of pomegranate wood and poplar (*P. nigra*) wood with two resin contents of 10% and 12% and two press times of 4 and 5 minutes. The four wood combinations were as follows: 100% pomegranate wood; 75% pomegranate wood and 25% poplar wood; 50% pomegranate wood and 50% poplar wood, and 100% poplar wood as control sample. The results of physical and mechanical tests performed on the manufactured particleboards may be discussed as follows and reach to following conclusions.

Considering the results, it was observed that when the press time increased from 4 to 5 minutes, the MOR of the boards decreased meaningfully (Fig. 1). Increase of press time from 4 to 5 minutes not only has deteriorated the adhesive quality of resin and wood but also caused thermal destruction of wood particles in surface layer. On the other hand, the mutual effects of resin content value and the press time on MOR and the MOE of the boards are meaningful and when the resin content is 4% when the press time is increased from 4 minutes to 5 minutes, the MOR and the MOE of the boards are decreased (Fig. 1, 2). This indicates that during the press time the resin particles have undergone more thermal destruction and when the resin content is 12%, the existence of more resin particles on the board surface will decrease the MOR significantly.

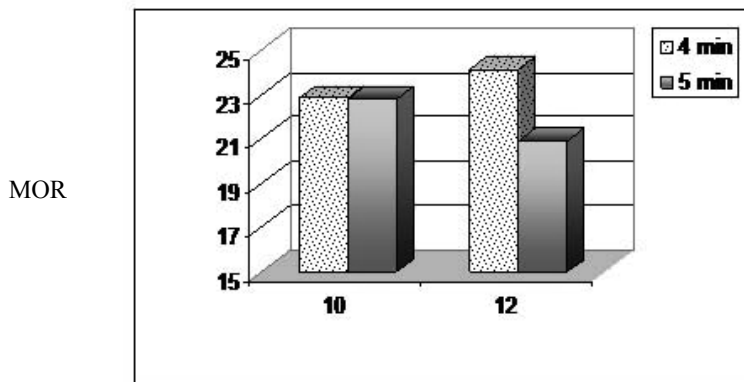


Fig. 1-Mutual effects of resin content and press time on modulus of rupture

The results of this study also showed that when poplar wood particles were added to the wood combination used for making the particleboards, the MOR and the MOE of the boards would increase and the highest MOR was gained when the wood combination was purely (100%) poplar particles. Since the pomegranate particles have higher specific weight than poplar particles, when poplar wood is added to the combination the compression coefficient of particles in the mat (especially on its surface) is increased, therefore addition of poplar wood particles improves the MOR and the MOE. In the meanwhile, even when pomegranate wood is used 100% in making the particleboards, the MOR and the MOE are higher than standard value.

The results of this study shows that increase in resin content will improve the IB of the boards because when more resin is used, more efficient connections are created between the particles and thus, higher

IB is created in the particleboards. However, all averages of IB of the manufactured particleboards were higher than standard (Fig. 3).

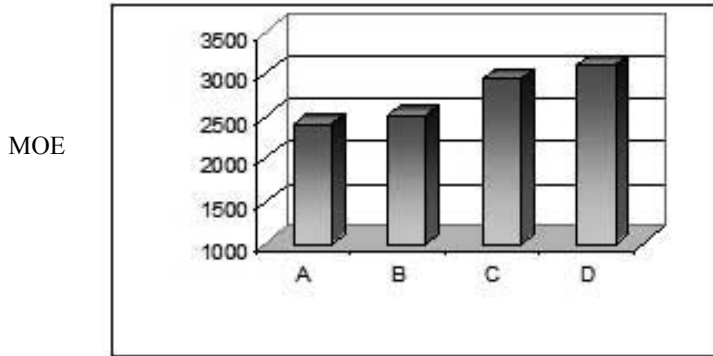


Fig. 2-Mutual effects of wood composition on MOE

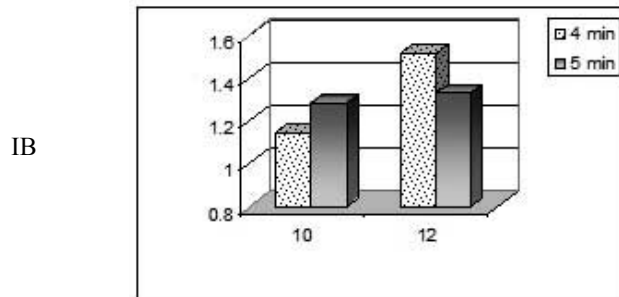


Fig. 3-Mutual effects of resin content and press time on internal bonding

The results related to the effect of variable factors on 2- and 24-hour TS too showed that increase in resin content on the surface statistically would decrease TS (Fig. 4). This decrease is mainly due to more IB of the boards because of more connection resistance between the wood particles which resulted from the higher amount of resin particles. Clearly, when the IB of the boards is increased, the TS of the boards will improve and will decrease. On the other hand, increase of the press time from 4 minutes to 5 minutes has caused more 2- and 24-hour TS of the boards. This can be result of the destructive effect of heat on resin connections during elongated press time.

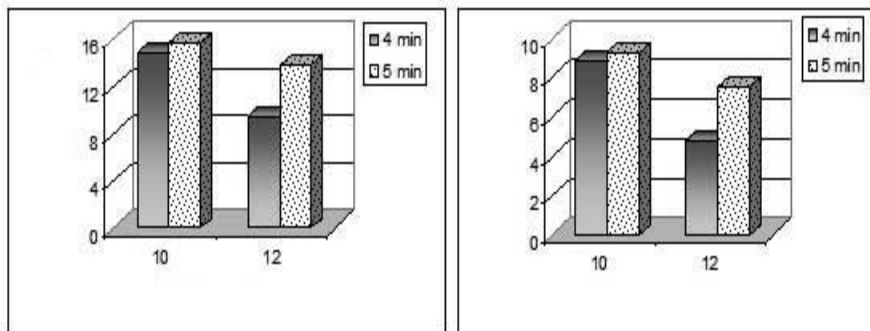


Fig. 4-Mutual effects of wood combination on thickness swelling: (left) TS24h; (right) TS2h

The results of this study also shows that when the percentage of poplar wood in the wood combination of the particleboard is increased, the 2 and 24-hour TS are increased and maximum TS is when the wood combination is purely poplar wood (100%). The reason is that the pomegranate wood has higher

specific weight than poplar wood and when poplar wood is increased less resin particles will exist on unit surface of the wood particle and consequently the connective resistance will decrease and the TS of the boards will increase.

In general with due regard to the results of measuring the physical and mechanical properties of the particleboards made in different conditions it can be said that pomegranate wood holds a lignocellulosic material which is suitable for making particleboard and may be used for making particleboards with standard physical and mechanical properties by itself or combined with poplar wood.

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# COLLAPSE RECONDITIONING OF *EUCALYPTUS CAMALDULENSIS* DEHN FROM ALGERIA (ARBORETUM BAINEM)

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## Abstract

This purpose of this study is based on the qualification in terms of physical properties (density, retractibility) of an Algerian wood (*Eucalyptus camaldulensis* Dehn) in order to use it as timber. The species *Eucalyptus* is particularly sensitive to the collapse phenomenon which occurs during drying. Collapse leads to checking and undesired structural deformations including appearance of cracks. A part of the collapse can be controlled by proper drying. It is possible to recover an important part of collapse during the drying process by a steam treatment. In this context, the objectives of this work will focus on the reconditioning of an Algerian wood, *Eucalyptus camaldulensis* Dehn from Bainem Arboretum located 15 km west of Algiers, Algeria. It is a hard and dense wood affected by collapse. The plant material consists in standard specimens extracted from a tree at different levels. The comparison of the results before and after reconditioning allowed a global evaluation of the resulting collapse phenomenon. The shrinkage anisotropy ratio is about 0.49. The axial shrinkage is small (0.11% before reconditioning and -0.17% afterwards) but constitutes a good indicator of tension wood, where it can reach 1%. The collapse recovery obtained is satisfying. It is more important in tangential direction (reconditioning ratio: 1.18) than in radial direction (0.96). The density reduction resulting from reconditioning is 4% and is also a good indicator of collapse phenomenon. The correlation found between the volumetric shrinkage and the basic density is negative and very significant ( $r = -0.49$ ). It indicates that the wood is less subject to the collapse phenomena with the increasing of density.

## 1. Introduction

The Australian *Eucalyptus* is particularly sensitive to collapse. However, at the present, no data are available concerning the technological characteristics of this wood in the purpose to endorse it. To overcome this lack, we started a preliminary study aiming at characterizing physical properties and applying the technology of reconditioning to Algerian wood samples from *Eucalyptus camaldulensis dehn*. It is well known that during drying there are two different phenomena: collapse and shrinkage. Both cause structural distortions and cracks during the drying process. The collapse is a phenomenon that occurs when drying from above the saturation point, where water in the fibers is evacuated from the empty cells. It results from the interaction between the distribution of free water in the microstructure on the one hand, the structure and strength of anatomical elements on the other [1, 2<sub>a,b,c</sub>]. The collapse can be limited by a well-controlled drying. A large collapse can be recovered by techniques such as applying steam during the drying process [3]. Studies by Chafe (1985 and 1986) on 69 species of *Eucalyptus*, Sesbou and Nepveu (1990) on *Eucalyptus camaldulensis* Dehn; have shown that there is a significant negative correlation between shrinkage due to collapse and wood basic density. The shrinkage without collapse is an intrinsic characteristic of the material and its measurement requires the use of small specimens [4-6<sub>a,b,c</sub>] [7].

## 2. Material and methods

### 2.1. Material

The raw material was kindly donated by an arboretum located in Bainem (Algeria), station of forested research of INRF (Table 1). A slice about 80cm in height from the base of the stem regeneration (length about 50cm) was used as a sample to carry out the measurement of physical properties of wood, as in this area the intensity of collapse is known to be the strongest. From this piece 2 orthogonal diametral planks (see Fig.1) were sawn, then cut into 36 cubes of 20 mm.

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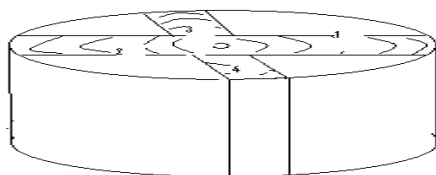


Fig. 1. Cutting of diametral planks

Table 1: Characteristics of the sampling station (Arboretum of INRF Bainem)

Geographic Location	Forest of Bainem
Description of settlement	
Plot surface	10.04 ha
From a reforestation	Artificial settlement
Composition	Eucalyptus
Average height	15m
Average diameter	24cm
Spacing between 2 trees	2.5 to 3m
Average age	35 Years (1957 source INRF)
Current treatment	Nil
Description of the station :	
Topography	Very low slope
Exposure	South West
Altitude	165m

## 2.2. Methods

The samples (36 cubics of 20x20mm) were analyzed; the following physical characteristics were measured: basic density, moisture content, shrinkage with collapse (before reconditioning); shrinkage without collapse (after reconditioning).

*Basic density* was obtained by dividing the oven-dry mass  $M_0$  (in kg) by the volume of the wood saturated in water  $V_s$  ( $m^3$ ):

$$Y = M_0/V_s \quad (1)$$

$V_s$  was estimated from the maximum weight  $M_{max}$  measured at saturation (*Keylwerth*)

$$Y = M_0/V_s = 1 / (M_{max} / M_0 - 0,347) \quad (2)$$

The *moisture content* (m.c.) was calculated as

$$H (\%) = (m_H - m_0) / m_0 \cdot 100 \quad (3)$$

where  $m_H$  and  $m_0$  are the mass (in gram) of the specimen before and after oven drying, respectively.

The *shrinkage* was measured for every cube, according to three orthotropic directions: axial (L), radial (R) and tangential (T), between successive states:

- Saturated state: immersion of samples in the water
- Air-dry condition, corresponding to 12% m.c.: carried out by drying the samples in a climatic chamber conditioned to 72% R.H. (dry temperature =50°C; wet temperature =44°C) during 48 hours.
- Anhydrous state: in the oven at 130°C±2°C, (oven dry base).

The analysis of this shrinkage is performed from the average dimensions of 5 measurement points by direction of shrinkage with a specific displacement indicator allowing a precision of 10µm (Fig. 2).

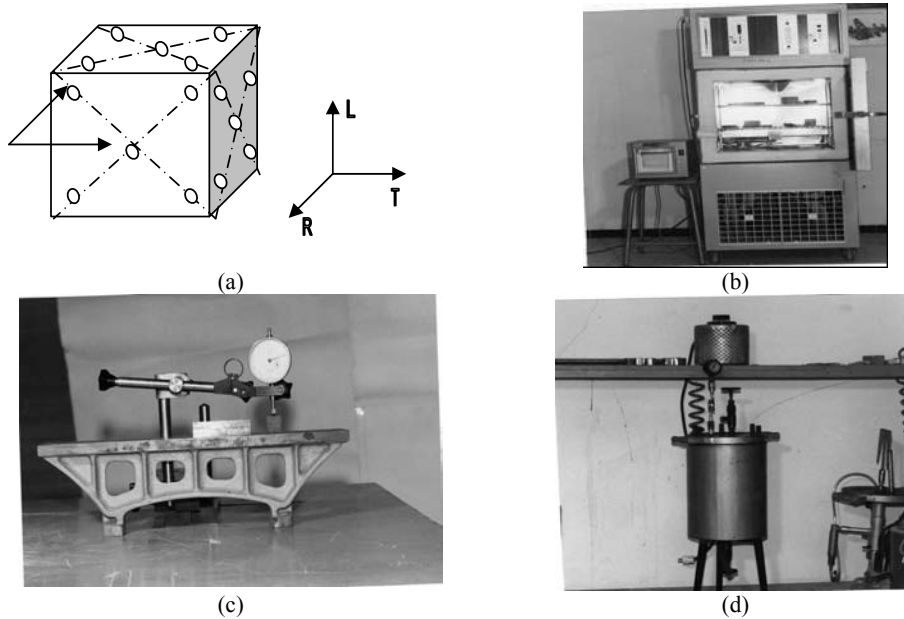


Fig. 2: Measurement of shrinkage  
 (a) Measurement points for shrinkage on 3 faces (TR, TL and RT), (b) Experimental set up,  
 (c) Experimental device for shrinkage measuring, (d) Autoclave for reconditioning of specimens

The *partial shrinkage* (expressed in %) is calculated before reconditioning by the following expression:

$$RXS = (LX_s - LX_{12}) / LX_s \quad (4)$$

where X stands for the direction (L, R or T),  $LX_s$  is the average specimen length in the saturated state,  $LX_{12}$  in the air-dry state. The *total shrinkage* in any direction X is defined as:

$$RXT = (LX_s - LX_0) / LX_s \quad (5)$$

*Volumetric shrinkage* is noted by replacing X by V and calculated by the following expressions:

$$RVy = 1 - (1 - RTy)(1 - RRy)(1 - RLy) \quad (6)$$

where y stands for S (partial) or T (total). *Shrinkage anisotropy* is calculated as:

$$RRy / RTy \quad (7)$$

*Coefficients of shrinkage* are also calculated. They are obtained for any direction X by dividing the partial shrinkage by the estimated loss of bound water:

$$CRXS = RXS / (H_s - H_{12}) \quad (8)$$

where  $H_{12}$  is the m.c. of the specimen measured in air-dry condition and  $H_s$  the moisture content at fibre saturation point, given arbitrarily the typical value of 30%.

Having been re-conditioned under vaporization at 110°C during 20 minutes in autoclave, the specimens were oven-dried, which allows to compare the shrinkage before and after reconditioning. Measurements in air-dry state in 3 directions and for the same length measured before reconditioning were done again; the same expressions were applied to calculate the tangential, longitudinal, radial and volumetric shrinkages as well as the transverse anisotropy. *Partial shrinkage after reconditioning*, corresponding to wood presumably without collapse, is noted  $RXC$  for any direction X (L, R, T and V) and obtained in the same way as the partial shrinkage before reconditioning, as well as shrinkage anisotropy  $RRC/RTC$ . The *reconditioning fraction* is defined as the ratio of shrinkage with collapse to that without collapse:

$$RXS / RXC \quad (9)$$

It is expected to be always superior to 1.



### 3. Results and discussions

To illustrate the drying operation, we have represented the m.c. kinetics of samples that have been regularly weighed to obtain the weight loss in the climatic chamber (Fig.3).

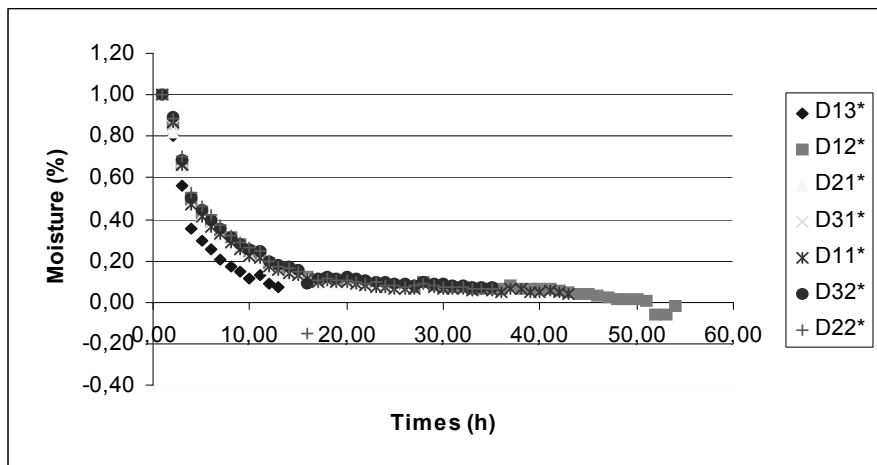


Fig. 3. Kinetics drying test witnesses

At the macroscopic level, the collapse is characterized by visible distortions of the specimen. Indeed, an initially prismatic specimen with right angles, once collapsed gets curved faces (with an inside convexity) due to the inward buckling of fibres (Fig. 4). The effect is much more pronounced in T direction than R direction. In L direction, the buckling of fibres is not observable thanks to the high rigidity of the cell walls in this direction. Besides, we can observe radial slits, which originate from the collapse of parenchyma rays. Hence, our results highlight the weakness of the anatomical features of this species.

Concerning the longitudinal drying between the saturated and air-dry states, it is typically a restrained shrinkage: the mechanical behavior during the drying is the predominant factor in this process. Between the air-dry and anhydrous states, we noticed a weak longitudinal shrinkage. From literature review this phenomenon is called "effect Coquille", (Kelsey, 1957): the wooden cube acts during drying as a rigid box, which opposes to the shrinkage and goes through a deformation comparable to Poisson's effect. The total shrinkage of distortion can be considered to be the combination of two processes: a **reversible elastic deformation** including the Poisson's effect noticed above, and non-reversible plastic deformation observed when applying to wood hydration-dehydration cycles, combined with collapse appearing during drying process.

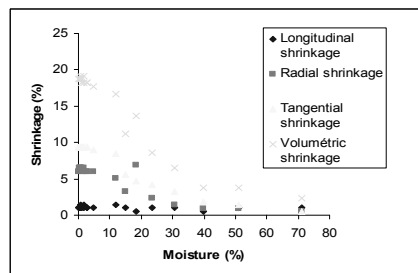


Fig. 4: Mean shrinkage of *E. camaldulensis* Dehn

#### 3.1. Shrinkage with and without collapse

The results of the shrinkage stocks are summarized in tables 2, 3, 4. In the case of the shrinkage with collapse: we noticed a relative shortening of 0.1, 3.9 %, 8.3 % and 12 % in L, R; T direction and in volume, respectively. After reconditioning, as was expected we clearly observe a drop in these values,

which fall to -0.17 % (slight elongation), 4.1 %, 7.0 % and 10.4 %, respectively. Kingston and Risdon (1961) found on Australian samples a T shrinkage of 8.9 % with collapse and 4.8 % after reconditioning; a R shrinkage of 4.4 % with collapse and 2.7 % without collapse. Wright [15] obtained similar values for *Eucalyptus* in Australia, with a shrinkage without collapse about 4.6 % in T direction, 3 % in R direction and a volumetric shrinkage of 7.7 %. Our results concerned coppice wood from 18 to 20 years old, while the Australian samples came from older grown-up trees. In Brazil, in the Institute of Sao-Paulo (1961), the following shrinkage values were obtained: 15.5 % for T, 6.8 % for R, 25.9 % for V. In Australia, among the commercialized eucalyptus trees, *E. Wandoo* 2 % gives the smallest shrinkage without collapse and *E. Diversicolor* the highest value with 12.5 %. For the L we observed 0.1%, negligible compared to other shrinkages, with a high variability (142%). This may be a good indicator for tension wood and juvenile wood (very important L shrinkage exceeding 1%). In our case, this indicator of tension wood is lower. The basic density was negatively associated with the axial shrinkage. The correlation was not found significant, but the trend was similar to that determined by Sesbou [16] on Moroccan and Italian *Eucalyptus* aged 9 years old.

Table 2: Basic density and partial shrinkage (from wet to m.c. = 12%) of *Eucalyptus camaldulensis* Dehn wood with collapse (before reconditioning)

Specimen number: 36	Shrinkage from wet do air-dry				anisotropy	Shrinkage coefficient				basic density (g/dm <sup>3</sup> )
	L (%)	R (%)	T (%)	V (%)		R/T	L (%/%)	R (%/%)	T (%/%)	
Direction	RLS	RRS	RTS	RVS	RRS/RTS	CRLS	CRRS	CRTS	CRVS	Y
Average	0.11	3.93	8.26	11.98	0.49	0.01	0.20	0.42	0.60	638.50
Standard deviation	0.16	1.93	3.71	5.08	0.17	0.01	0.10	0.23	0.31	43.51
Coefficient of variation (%)	141.7	49.1	45.0	42.5	34.2	141.3	51.9	54.7	50.6	6.81

Table 3: Total shrinkage of *E. camaldulensis* Dehn wood with collapse (Before reconditioning)

Samples: 36	RLT (%)	RRT (%)	RTT (%)	RVT (%)	RRT/RTT	CRLT (%)	CRRT (%)	CRTT (%)	CRVT (%)	Y (g/dm <sup>3</sup> )
Average	0.33	6.03	10.43	16.10	0.59	0.01	0.20	0.35	0.54	638.50
Standard deviation	0.12	1.34	1.93	2.67	0.12	0.00	0.04	0.06	0.09	43.51
Coefficient of variation (%)	37.59	22.19	18.49	16.56	20.39	37.59	22.19	18.49	16.56	6.81

Table 4: Basic density and partial shrinkage (from wet to m.c. = 12%) of *E. camaldulensis* dehn wood without collapse (after reconditioning)

Samples: 36	RLC (%)	RRC (%)	RTC (%)	RVC (%)	RRC/RTC	Y (g/dm <sup>3</sup> )
Average	-0.17	4.11	7.01	10.35	0.66	666
Standard deviation	0.27	1.77	3.40	4.29	0.38	34.72
Coefficient of variation (%)	157.1	43.1	48.5	41.4	57.7	5.2

### 3.2. Anisotropy

The average value found in our samples was 0.49, which is generally observed (0.50) between the two radial and tangential shrinkage. Wright [15] reported for *Eucalyptus* a transverse anisotropy of 0.5, the extremes being *E. maculata* with 0.7 and *E. viminalis* with 0.26. Clarke (1930) found for 200 samples of 60 species belonging to 21 genera, a ratio RR / RT ranging from 0.13 to 0.91. On the other hand,

Keylwerth (1951) noted for the European species an average anisotropy of 0.61 (Sesbou, 1981). This strong transverse anisotropy is one of the main causes of distortions resulting from wood drying.

### 3.3. Basic density

The average value found  $Y = 639 \text{ kg/m}^3$  is typical of this hardwood species. It presents a coefficient of variation accepted for wood (7%). The loss of density of 4% by reconditioning is also a good indicator of the phenomenon of collapse.

#### Correlations between specimens at the tree level

The correlation between basic density and partial L shrinkage is not significant at the 5% level. The L shrinkage remains low and has little influence on the V shrinkage. Concerning R and T directions, the correlation between basic density and radial shrinkage is not significant at the 5% level. However, the correlation ( $r = -0.582^{***}$ ) between basic density and shrinkage with T collapse is highly significant (Fig. 6). The regression equation is as follows:

$$\text{RTS} (\%) = -0.0497Y + 39.992 \text{ with } R^2 = 0.339 \quad (10)$$

The basic density is significantly negatively correlated to the V collapse (Fig.7) with  $r = -0.488^{**}$ . The regression equation is as follows

$$\text{RVS} (\%) = -0.0571Y + 48.403 \text{ with } R^2 = 0.238 \quad (11)$$

The obtained trend curves are consistent with Sesbou (1981) results: the higher the density, the lower the shrinkage, confirming that the phenomenon of collapse is less pronounced for the densest woods. The woods most affected by collapse are those with a medium density.

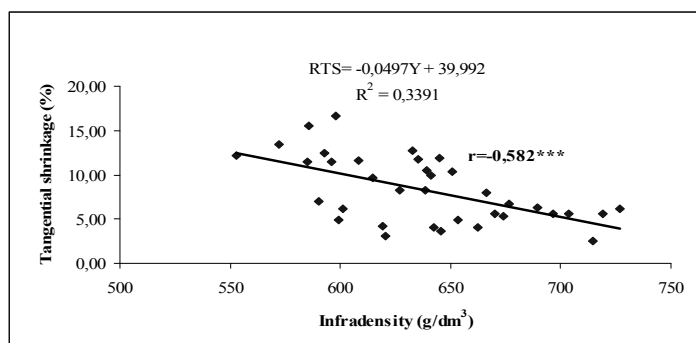


Fig. 5. Relationship between basic density and T partial shrinkage

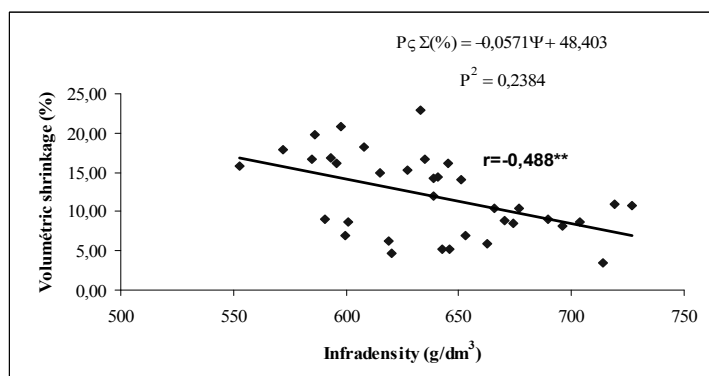


Fig. 6. Relationship between basic density and V partial shrinkage

The correlation between density and transverse anisotropy is positive with  $r = 0.475^{**}$ . The regression equation is as follows:

$$\text{RRS} / \text{RTS} = 0.0018Y - 0.6859. \text{ with } R^2 = 0.226 \quad (12)$$

The higher the basic density, the lower the anisotropy.

#### 4. Conclusion

Based on the results obtained of this preliminary study on Algerian *Eucalyptus camaldulensis* Dehn we can conclude that the wood is hard and dense, and affected by drying collapse. The negative and very significant correlation found ( $r = -0.488^{**}$ ) indicates that with increase of density, the wood is less subject to collapse phenomenon. Our results are consistent with the literature review. The intensity of collapse varies with the height in the tree; for all samples from the same tree it decreases from bottom to top. The reconditioning done on the specimens has reduced the collapse to a large extent. The first results, including a correlation between wood density and recovery are suitable. They demonstrate that the technique of reconditioning used on standard samples at the laboratory scale can be generalized and used at an industrial scale. The technique of reconditioning may be advantageously used for a better valuation of wood products from countries where eucalyptus have been introduced.

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# ANNEXES





## ANNEX 1: FACTS ABOUT COST ACTION IE0601

### COST Action IE0601: Wood Science for Conservation of Cultural Heritage (WoodCultHer)

#### Facts about the Action

(from COST website [http://w3.cost.esf.org/index.php?id=262&action\\_number=ie0601](http://w3.cost.esf.org/index.php?id=262&action_number=ie0601))

Chair of the Action: Professor Luca UZIELLI

Rapporteur: Professor Zsolt KAJCSOS

Science Officer: Ms. Caroline WHELAN

Administrative Officer: Milena STOYANOVA

Website: [www.woodculther.org](http://www.woodculther.org)

#### Action overview

##### Main objective

The main objective of the Action is to improve the conservation of our wooden cultural heritage by increasing the interaction and synergy between wood scientists and other professionals applying wood science and technology towards the study, conservation and restoration of wooden artefacts of artistic or historic interest (WCHOs, i.e. Wooden Cultural Heritage Objects).

##### Specific objectives may be identified as follows:

###### General

- To put into evidence how the modern scientific knowledge about wood may contribute to Diagnosis and Conservation of wooden Cultural Heritage.
- To favour meeting and interaction, at both scientific and practical level, of researchers in the field of wood, specialists in conservation of wooden artworks, manufacturers of equipment and products which might be successfully used for the diagnosis, restoration and conservation of wooden artworks.
- To acquire a deeper insight into several fields and processes concerning wood material (e.g. the ageing processes, their factors (physical, mechanical, biological, chemical, environmental, and their interactions), in order to improve the conservation of wooden artworks.
- To develop criteria for evaluating durability of interventions during very long time (centuries).
- To develop criteria for ensuring "re-treatability" (i.e. that present interventions will not impede future interventions, if and when needed).

###### Wood deterioration

- To develop new methods for the evaluation of new techniques and products for the conservation of wooden artworks.
- To acquire further understanding of the process of bacterial wood degradation in order to develop practical conservation methods to preserve historical wooden structures and remains in the soil.
- To further develop micro waves as a conservation method against insect degradation.

###### Diagnostic methods and equipments

- To develop and foster the implementation of the use of practical sensors to indicate risk to wooden objects in museums and at historic sites, or during the transportation of artworks.

###### Interactions between wooden artworks and environment

- To be able to better evaluate the interactions between individual wooden artworks and environment, also by direct monitoring physical changes and damage processes in objects.

###### Dendrochronology

- To stimulate the development of non-destructive high resolution scanners for in situ inspection of wooden objects to identify aging and degradation processes, that also allows tree-ring analyses (dendrochronology) for exact age determination.
- To disseminate results which obtained by applying "dendro-provenancing" techniques, in order to support further historical and technological studies.

Joseph Gril (edited by), *Wood Science for Conservation of Cultural Heritage – Braga 2008: Proceedings of the International Conference held by COST Action IE0601 (Braga - Portugal, 5-7 November 2008)*, ISBN 978-88-6453-157-1 (print) ISBN 978-88-6453-165-6 (online) © 2010 Firenze University Press

Non-destructive inspection of wooden objects

- To further develop non-destructive methods and equipments, for inspection and evaluation of both movable and non-movable WCHOs.

Numerically modelling of risk of damage

- To develop and validate mathematical models and computer simulations of short- or long-term phenomena, from the observation of past events and processes, aiming towards prediction of future behaviour.
- To develop methods for predicting by simulation the long-term result of present interventions (e.g. present tendency to provide panel paintings with flexible cross-ties or frames).

Long-term behaviour and "accelerated ageing"

- To further explore specific subjects such as the properties and behaviour of "old" wood, the influence of ageing on the properties of WCHOs.
- New principles, criteria, observation and evaluation methods need therefore to be developed in order to evaluate expected deterioration of WCHOs in the very long term.
- To acquire knowledge and establish methods for studying deteriorations that take place during very long time periods (decades and centuries), and for evaluating the long-term compatibility of interventions, treatments, products, aiming to improve the conservation of wooden artworks.
- To develop adequate models of the ageing and deterioration processes, deriving from the observation of past events and processes, aiming towards prediction of future behaviour.

Archaeological and archaeo-botanic wood

- To improve prevention of bacterial decay of wood in foundation piles and archaeological sites.
- To develop methods and standards for evaluating procedures and products for conservation of archaeological and archaeo-botanic wood.

Timber structures

- To develop specific safety factors for verification of WCHO timber structures.
- To develop appropriate load tests for WCHO timber structures.
- To produce guidelines about criteria for conservation (and reinforcement, if necessary) of WCHO timber structures.
- To produce guidelines and standard documents concerning (for various situations and types of structures) inspection, assessment of load-bearing capacity, use of visual versus instrumental methods, practices and responsibilities.
- To develop criteria for evaluating effectiveness and durability (during very long time, i.e. centuries) of interventions performed on WCHO timber structures.
- To foster development of national or local grading rules for existing "old" timber structural elements; to encourage, make available and compare results of test campaigns aimed to determine reliable strength and stiffness values for such timbers.

Wooden foundations

- To improve knowledge and techniques appropriate for conserving wooden foundations piles under historical buildings.
- To increase knowledge on the process of bacterial wood degradation under water (e.g. ship wrecks, foundations piles), and to define strategies to control the soil hydrology or water streaming in open water leading to a reduction or even to stop the wood degrading bacterial activity.

Standardization

- To put in active contact the European scientific communities dealing with conservation of wooden Cultural Heritage, in order to provide a very strong and wide scientific background, and an informed consensus throughout European countries, for standardization (particularly of CEN/TC 346) in the field of wooden artworks.
- To contribute to European Standardization in the field (inputs to CEN/TC 346 "Conservation of Cultural Property")
- It should be emphasized here that since in the field of Cultural Heritage each artwork (especially if made of wood) is different (materials, wood species, manufacture, history, environment(s), decay/deterioration,

interventions, etc.), each artwork needs/deserves a "personal" care, i.e. individual assessment, evaluation, solutions; therefore the technical standards should specify methods and criteria, not "standard solutions" to problems.

### Action's details

(updated 2009)

Domain : Materials, Physical and Nanosciences

Memorandum of Understanding: 317/06

CSO Approval date: 20/11/2006

Action started: 18/04/2007

End of Action: 17/04/2011

Countries having signed the MoU (Total 24): Austria, Belgium, Czech Republic, Denmark, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Italy, Latvia, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovenia, Spain, Sweden, Switzerland, Turkey, United Kingdom

Countries having expressed Intention to sign the MoU (Total 2): Estonia, Slovak Republic

Non-COST Institutions (Total 3): RISH (Research Institute for Sustainable Humanosphere), Japan; Scion (New Zealand Forest Research Institute), New Zealand; Agricultural University of Tirana, Faculty of Forestry Sciences, Albania.

### Management Committee of Action IE0601

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Vice Chair: Dr. Joseph GRIL

COST Participants

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#### The Action's Steering Committee

The Steering Committee (SC) is formed by the Action's Chairman, Vice-Chairman, Webmaster, and by the Leaders and Vice-Leaders of the three Working Groups; it mainly fulfils the following roles:

- to support the Chairman in making urgent decisions that will later be submitted for MC ratification (e.g. in approving Short term Scientific Missions, or in declaring if a proposed research project fits into the framework of our Action, when national Authorities, in order to allocate research funds to national Institutions, require an expression of formal support to such projects from Chairs of relevant COST Actions)
- to discuss and prepare policies and future activities to be later discussed by the MC, and
- to organize and steer future meetings and activities, according to the guidelines approved by the MC

## The Steering Committee of COST Action IE0601

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**The Action's Working Groups**WG1 “Wood Properties”

Mechanisms and characterisation of wood ageing, degradation and transformation processes, focussing on changes of properties and behaviour of wood and wooden artworks. Properties would include ultra-structural, physical and mechanical, chemical, rheological, biological, etc., including ageing processes of archaeological and water-logged wood.

WG2 “Assessment & Diagnosis”

Evaluation of destructive, non-destructive and non-invasive methods and equipments for in-situ and ex-situ assessments and diagnosis and study of Wooden Cultural Heritage Objects (WCHO's). Analysis of agents and causes of deterioration.

WG3 “Conservation & Restoration”

Evaluation of existing and new procedures, methods, techniques and products for interventions on wooden WCHO's (“acceptable, respectful and compatible”).

Note: WGs are useful for several organizational aspects, however the Action's activities – due to their interdisciplinary nature – are mainly carried out through Plenary or Focused (thematic) meetings.

## ANNEX 2: FINAL PROGRAM OF THE CONFERENCE

### International Conference on WOOD SCIENCE FOR PRESERVATION OF CULTURAL HERITAGE: MECHANICAL AND BIOLOGICAL FACTORS

Joint Meeting of COST Action IE0601 “Wood Science for Conservation of Cultural Heritage”, and the  
European Society for Wood Mechanics  
Museu D. Diogo de Sousa, Braga, Portugal, 5-7 November 2008

#### General program

##### **Wednesday, November 5, 2008**

- 16:00 – 16:15 Opening of the Conference  
16:15 – 17:00 *Keynote presentation 1 (plenary)* – Vasco Fassina  
17:00 – 17:45 *Keynote presentation 2 (plenary)* – Helena Cruz  
17:45 – 18:30 MC Meeting (part 1)  
19:00 *Verde de Honra (local wine “Vinho Verde” tasting)*  
19:30 Dinner (Museu D. Diogo de Sousa)

##### **Thursday, November 6, 2008**

- 09:00 – 09:40 *Keynote presentation 3 (plenary)* – Stefan Michalski  
09:40 – 10:15 *Keynote presentation 4 (plenary)* – Peter Niemz  
10:15 – 10:45 Coffee break  
10:45 – 12:45 *Parallel sessions (including Short oral poster presentations: 3 slides / 3 minutes)*  
- Parallel session 1 (WG1) Dimensional Response to environment and ageing  
(Coordinator G. Jeronimidis)  
- Parallel session 2 (WG2) Strength of Aged Load Bearing Structures (Coordinator H. Cruz)  
- Parallel session 3 (WG3) Biodeterioration (Coordinator T. Nilsson)  
13:00 – 14:30 Lunch  
14:30 – 16:00 *Parallel sessions*  
16:00 – 16:30 Coffee break  
16:30 – 18:00 *Parallel sessions*  
18:00 – 19:00 *Poster session (Free discussions in front of posters)*  
20:00 *Conference Dinner (Museu D. Diogo de Sousa)*  
21:30 *Popular Portuguese Music show from Banda Plástica*

##### **Friday, November 7, 2008**

- 09:00 – 09:45 *Keynote presentation 5 (plenary)* – Junji Sugiyama  
09:45 – 10:45 WG Meetings  
10:45 – 11:15 Coffee break  
11:15 – 12:00 *Keynote presentation 6 (plenary)* – Eero Ehanti  
12:00 – 13:00 Plenary session (including WG's and parallel sessions reports) - Closing of the Conference  
13:00 – 14:30 Lunch  
MC Meeting (Part 2) - (For non-MC Members: Museum visit, other visits around Braga  
14:30 – 16:00 historical center)  
16:00 – 16:30 Coffee  
16:30 – 19:00 Visit to Mosteiro S. Martinho de Tibães ([www.mosteirodetibaes.org](http://www.mosteirodetibaes.org))  
19:30 *Dinner (Museu D. Diogo de Sousa)*

#### Authors and papers to be presented at the Conference - List and scheduling

Notes:

- Orals had available 20 minutes for on-screen presentation + 10 minutes discussion
- Posters planned in Sessions 1-2-3 were presented on-screen (3 minutes / 3 slides + 2 minutes discussion) during the OSP period (Thursday November 6, from 12.15 to 12:45); further discussions could take place in front of the Poster itself, during the Poster session
- All Posters had time for free discussion in front of the Poster itself (Poster session - Thursday November 6, from 18:00 to 19:00)

**Keynote presentations**

	<b>Reference Author</b>	<b>Author(s)</b>	<b>Title</b>	<b>day</b>	<b>time</b>
IT	Fassina	Vasco FASSINA	Update on the activities of the CEN Technical Committee 346 "Conservation of Cultural Property" after a four year period	05-nov	16:15 – 17:00
PT	Cruz	Helena CRUZ, José SAPORITI MACHADO, Pedro PALMA	What one needs to know for the assessment of timber structures	05-nov	17:00 – 17:45
CA	Mihalski	Stefan MICHALSKI	The rationale of the ASHRAE guidelines for climate control in museums, especially as determined by collections of wooden objects	06-nov	09:00 – 09:40
CH	Niemz	Peter NIEMZ	Methods of non-destructive wood testing	06-nov	09:40 – 10:15
JP	Sugiyama	Junji SUGIYAMA, Suyako MIZUNO, Yoshiki HORIKAWA, Chiori ITO, Misao YOKOYAMA	Non destructive imaging for wood identification	07-nov	09:00 – 09:45
FI	Ehanti	Eero EHANTI	The Wreck of Vrouw Maria - Current Condition and Future Perspectives	07-nov	11:15 – 12:00

**PARALLEL SESSION 1 – Thursday November 6, 2008 – Oral presentations**

	<b>Reference Author</b>	<b>Author(s)</b>	<b>Title</b>	<b>time</b>
JP	Inagaki_1	Tetsuya INAGAKI, Katsuya MITSUI, Satoru TSUCHIKAWA	NIR archaeometry as a powerful tool for investigating the archaeological wood – Investigation of thermal degradation mechanism of softwood and hardwood	10:45 – 11:15
DE	Aderhold	Jochen ADERHOLD, Hiltrud BROCKE	Measurement and simulation of dimensional changes due to flows of heat and moisture in wood and wood-based materials: How objects of cultural heritage can be studied by methods developed for industrial applications	11:15 – 11:45
PL	Bratasz	Lukasz BRATASZ, Roman KOZLOWSKI, Bartosz RACHWA	Sorption of moisture and dimensional change of wood species used in historic objects	11:45 – 12:15
		Oral Short Presentation of Session 1 Posters (3 minutes / 3 slides - see separate list, below)		12:15 – 12:45
		<b>Lunch</b>		
FR/JP	Bremaud	Iris BREMAUD, Pierre CABROLIER, Kazuya MINATO, Jean GÉRARD, Bernard THIBAUT	Vibrational properties of tropical woods with historical uses in musical instruments	14:30 – 15:00
IT	Fioravanti	Marco FIORAVANTI, Paola MAZZANTI, Giacomo GOLI, Gabriele ROSSI ROGNONI	Physical and mechanical characterization of ancient wooden musical instruments for their conservation	15:00 – 15:30
FR	Dinh	Anh Tuan DINH, Gilles PILATE, Carole ASSOR, Patrick PERRE	Measurement of the elastic properties of minute samples of wood along the three material directions	15:30 – 16:00
		<b>Coffee break</b>		

IT	Uzielli	Luca UZIELLI, Elisa CARDINALI, Paolo DIONISI-VICI, Marco FIORAVANTI, Nicola SALVIOLI	Structure, mock-up model and environment-induced deformations of Italian laminated wood parade shields from the 16th century	16:30 – 17:00
GR	Moutsatso u	Anna P.MOUTSATSOU, E. KOULOUMPI, A. V. TERLIXI, M. GEORGES, C. THIZY, V. TORNARI, M. DOULGERIDIS	Effect of thermal accelerated ageing on structural defects of model panel paintings	17:00 – 17:30
FR	Marcon	Bertrand MARCON, David DURREISSEIX, Paolo DIONISI-VICI, Joseph GRIL, Luca UZIELLI	Experimental and numerical mechanical study of a framing technique for cupping control of painted panels combining crossbars and springs	17:30 – 18:00

### PARALLEL SESSION 2 – Thursday November 6, 2008 – Oral presentations

	Reference Author	Author(s)	Title	time
PT	Feio	Artur O. FEIO, Paulo B. LOURENÇO, José S. MACHADO	Structural behaviour of traditional mortise and tenon timber joints	10:45 – 11:15
PT	Brites	Ricardo BRITES	Strength assessment of in-service structural timber members through testing of meso-specimens	11:15 – 11:45
SI	Srpacic	Jelena SRPCIC	Biological deterioration of historical wooden roof and floor structures and their renovation	11:45 – 12:15
		Oral Short Presentation of Session 2 Posters (3 minutes / 3 slides - see separate list, below)		12:15 – 12:45
		<b>Lunch</b>		
NO	Flaete	Bernt-Håvard ØYEN, Hans NYEGGEN, Per Otto FLÆTE	Bending stiffness and strength of 300 year old salt(NaCl)-exposed wood from Bryggen in Bergen, Norway	14:30 – 15:00
CZ	Kucerova	Irena KUČEROVÁ, Martina OHLÍDALOVÁ, Jiří FRANKL, Michal KLOIBER, Alena MICHALCOVÁ	Defibring of historical roof beam caused by ammonium sulphate and ammonium phosphates based fire retardant	15:00 – 15:30
SI	Humar	Miha HUMAR, Bojan BUČAR, Franc POHLEVEN	Changes of mechanical and chemical properties of wood after brown-rot decay and blue staining	15:30 – 16:00
		<b>Coffee break</b>		
JP	Kawai	Shuichi KAWAI, Misao YOKOYAMA, Miyuki MATSUO, Junji SUGIYAMA	Research on aging of wood in RISH (Research Institute for Sustainable Human sphere, Kyoto University)	16:30 – 17:00
FR	Husson	Jean Marie HUSSON, Frédéric DUBOIS, Nicolas SAUVAT	Hygro-Lock Integration in a Kelvin Voigt model	17:00 – 17:30
DE	Kaliske	Michael KALISKE, Eckart RESCH, Jörg SCHMIDT	Numerical Simulation of the Strength of Wooden Structures: Application to Historic Pianoforte	17:30 – 18:00



**PARALLEL SESSION 3 – Thursday November 6, 2008 – Oral presentations**

	<b>Reference Author</b>	<b>Author(s)</b>	<b>Title</b>	<b>time</b>
RO	Bucsa	Livia BUCSA, Corneliu BUCSA	Romanian wooden churches wall painting biodeterioration	10:45 – 11:15
RO	Cutrubinis	Mihalis CUTRUBINIS, Khôi TRAN, Eugen BRATU, Loic CAILLAT, Daniel NEGUT, Gheorghe NICULESCU	Disinfection and consolidation by irradiation of wooden samples from three Romanian churches.	11:15 – 11:45
DE	Scheidig	Wolfram SCHEIDING, Katharina PLASCHKIES, Björn WEISS	Mould on organs and cultural heritage objects. Investigations of 8 churches in Saxonia	11:45 – 12:15
		Oral Short Presentation of Session 3 Posters (3 minutes / 3 slides - see separate list, below)		12:15 – 12:45
		<b>Lunch</b>		
LV	Irbe	Ilze IRBE, Ingeborga ANDERSONE	Wood decay fungi in Latvian buildings including cultural monuments	14:30 – 15:00
EE	Pilt	Kalle PILT	Biodeterioration of Wood in Estonia	15:00 – 15:30
PT	Nunes_1	Lina NUNES	Termite infestation risk in Portuguese historic buildings	15:30 – 16:00
		<b>Coffee break</b>		
AT	Grabner	Michael GRABNER, Maria KOTLINOVA	Ageing of wood – described by the analyses of old beams	16:30 – 17:00
NO	Mattsson	Johan MATTSON	Protection of historical wood by use of sodium chloride	17:00 – 17:30
SI	Pohleven	Franc POHLEVEN, Iztok VIDIC, Crtomir TAVZES	Degradation of melanin and biocides by ligninolytic fungi	17:30 – 18:00

**Posters in Session 1 (Oral Short Presentations) – Thursday November 6, 2008 (12:15 – 12:45)**

	<b>Reference Author</b>	<b>Author(s)</b>	<b>Title</b>	<b>Nº.</b>
FR	Leconte	Sandie LE CONTE, S. LE MOYNE	Limit default detection of the acoustic holography technique in the cultural heritage	1
MT	Formosa_1	Michael FORMOSA, Martin MUSUMECI	Prediction of linear dimensional change of unrestricted wood at different levels of Equilibrium Moisture Content	2
PL	Lukomski	Lukasz LASZYK, Michal LUKOMSKI, Lukasz BRATASZ	Simple Electronic Speckle Pattern Interferometer (ESPI) for the investigation of wooden art objects	3
IT	Macchioni	Benedetto PIZZO, Ana ALVES, Nicola MACCHIONI, Antonio ALVES, Gianna GIACHI, Manfred SCHWANNINGER, José RODRIGUES	Characterization of waterlogged wood by infrared spectroscopy	4
JP	Inagaki_2	Tetsuya INAGAKI, Satoru TSUCHIKAWA	NIR archaeometry as a powerful tool for investigating the archaeological wood – Spectroscopic observation of the degradation process in thermal treated wood using a deuterium exchange method	5

JP	Inagaki_3	Tetsuya INAGAKI, Hitoshi YONENOBU, Satoru TSUCHIKAWA	NIR spectroscopic monitoring of water adsorption/desorption process in modern and archaeological wood	6
SE	Almkvist	Gunnar ALMKVIST (to be confirmed)	(Report about his STSM in Finland)	1b

**Posters in Session 2 (Oral Short Presentations) – Thursday November 6, 2008 (12:15 – 12:45)**

	Reference Author	Author(s)	Title	N°.
IT	Mazzanti	Paola MAZZANTI, Luca UZIELLI	Strength and MOE of poplar wood ( <i>Populus alba</i> L.) across the grain: experimental data	7
FR	Colmars	Julien COLMARS, Takato NAKANO, Hiroyuki YANO, Joseph GRIL	Creep properties of heat treated wood in radial direction: a preliminary study	8
PT	Bastos	Jorge DE NOVAES BASTOS	The XVI-th Century Rural House Restoration Project, in Milreu, Algarve	9
TR	Tanac	Mine TANAC KIRAY, Ozgul YILMAZ KARAMAN	Wooden load bearing structural elements of Kula traditional houses	10
JP	Matsuo	Miyuki MATSUO, Misao YOKOYAMA, Kenji UMEMURA, Junji SUGIYAMA, Shuichi KAWAI, Shigeru KUBODERA, Takumi MITSUTANI, Hiromasa OZAKI, Minoru SAKAMOTO, Mineo IMAMURA	Evaluation of the aging wood from cultural properties as compared with the accelerated aging treatment – Analysis on color properties	11
IR	Batebi	Yadollah BATEBI MOTLAGH	<del>Experimental Investigations on flexural strengthening of old Wood members in historical buildings reinforced with GFRP Composite</del>	42
IT	Dionisi-Vici	Paolo DIONISI-VICI	Report about his STSM in Malta (provisional)	13

**Posters in Session 3 (Oral Short Presentations) – Thursday November 6, 2008 (12:15 – 12:45)**

	Reference Author	Author(s)	Title	N°.
DE	Noldt	Uwe NOLDT (to be confirmed)	Researches on insects in Cultural Heritage Objects in Latvia	14
AT	Tscherne	Florian TSCHERNE, Bernhard SCHACHENHOFER, Karen ROUX	Research study on the effects of the Thermo Lignum® Warmair treatment on art objects with paint and gilt finishes	15
HU	Tolvaj	Laszlo TOLVAJ, Sandor MOLNAR	Photodegradation and thermal degradation of outdoor wood	16
FR	Leclaire	Charlotte LECLAIRE, Elodie LECOQ, Geneviève ORIAL, Franck CLEMENT, Faisl BOUSTA	Fungal decontamination by cold plasma: an innovating process for wood treatment	16b
RO	Negut	Constantin Daniel NEGUT, Laurent CORTELLA, Mihalis CUTRUBINIS, Khôi TRAN, Corneliu Catalin PONTA	Colour measurements intercomparison of disinfected by irradiation polychromed wooden objects	15b

AT	Unger	Merle STRATLING, Wibke UNGER, Karin PETERSEN	Orientated Investigation to kill the mycelia of the Dry Rot Fungus, <i>Serpula lacymans</i> , with microwaves (DE – P – 3)	14b
BR	Monteiro	Maria Beatriz BACELLAR MONTEIRO, Takashi YOJO, Gonzalo Antonio CARBALLEIRA LOPEZ	Wood Structure Biodeterioration – A Case Study on a Century Church in Piracaia, Brazil	13b

**Posters in Session 4 (presentation in front of Posters during the Poster session)  
Thursday November 6, 2008 (18:00 – 19:00)**

	Reference Author	Author(s)	Title	Nº.
MT	Formosa_2	Michael FORMOSA	On 18th and 19th Century Sacristy Furniture in the Maltese Islands: Materials and Techniques	12b
RO	Constantine scu	Bogdan CONSTANTINESCU	Romanian Architectural Wooden Cultural Heritage – The Present Status – A Survey	11b
RO	Georgescu	Gheorghe NICULESCU, Migdonia GEORGESCU	The consequences of wooden structures consolidation through traditional techniques on the resistance of support panels and paint layers (Some case study - Stelea Monastery Targoviste, Humor Monastery and Arbore Monastery)	10b
PT	Esteves	Lília Maria ESTEVES, Raul Adalberto LEITE	Ressurreição de Cristo – Panel from the XVI century, Museu de Arte Sacra da Sé de Évora. A repainted painting – dating and treatment (1974-2008)	9b
PT	Murta	Elsa MURTA, Diogo SANCHES	Nossa Senhora do Rosário and Anjo Ceroferário, two different cases in conservation and restoration of sculptures in wood support in the Department of Conservation of IMC.	8b
PT	Nunes_2	Teresa Sande LEMOS, Lina NUNES	Diagnosis of painted wood ceiling planks from an eighteen century building	7b

**Posters in Session 5 (presentation in front of Posters during the Poster session)  
Thursday November 6, 2008 (18:00 – 19:00)**

	Reference Author	Author(s)	Title	Nº.
BE	De Ridder	Maaik DE RIDDER, J. VAN DEN BULCKE, Hans BEECKMAN, Joris VAN ACKER	In the heart of the Limba tree ( <i>Terminalia superba</i> Engl. & Diels): detection methods for heart rot and false heartwood	6b
BR	Mascia	Nilson Tadeu MASCIA, Steven CRAMER	On the Effect of the Number of Annual Growth Rings, Specific Gravity and Temperature on Redwood Elastic Modulus	5b
DZ	Tazrout	Mansour TAZROUT, M.Tahar ABADLIA, Atika OUDIA	Repackaging Study of <i>Eucalyptus camaldulensis</i> Dehnh. wood from Bainem	4b
IR	Soleymani	Homayoun SOLEYMANI ASHTIANI, Abolfazl KARGARFARD	Investigation on the utilization of pomegranate wood for particleboard production	3b

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