Preface

Book of abstracts includes the scientific program and the abstracts of papers will be presented at the Final COST Action FP0904 Conference on “Recent Advances in the Field of TH and THM Wood Treatment” at the Luleå University of Technology, Division of Wood Science and Engineering, in Skellefteå, Sweden on 19–21 May 2014.

The main objective of COST Action FP0904 is to achieve a better understanding on mechanical and chemical transformations of wood during Thermo-Hydrous (TH)/Thermo-Hydro-Mechanical (THM) processing through collaborations between different researchers from the wood and material sciences. This Action provides cooperation and encourages research between research groups from academia and industry to help to overcome the challenges in scaling-up research findings, improving full industrial production, process improvement, in understanding the relations between the processing parameters, material properties and the development of new products. The COST Action FP0904 consists of three Working Groups (WGs):

WG1: Identification of chemical degradation of wood under Thermo-Hydrous treatment
WG2: Modelling of Thermo-Hydro-Mechanical behaviour of wood during processing
WG3: Innovation and new products by Thermo-Hydro-Mechanical processing

We wish the conference provides a forum and an opportunity for experts and young researchers from worldwide academia and industry to present their latest research, exchanging and developing new ideas within the field of TH and THM wood treatment. The objectives of this conference are to present and discuss the state-of-the-art of TH/THM wood treatment in open and closed systems and the challenges in wood characterization and scaling-up from laboratory to full industrial production, through a discussion of the latest research results and new ideas. The key objective of this Final Action FP0904 Conference is to present the main results of the Action, to summarise the scientific progress achieved and to formulate open questions and further challenges. This conference will include an evaluation session with representatives of COST and Action Management Committee members.

Luleå University of Technology (LTU), established in 1971, is the northernmost University of Technology in Scandinavia and is known for its education and research within the field Wood Science and Engineering. The research area of Wood Technology, Wood Physics and Wood Products Engineering is since 1982 established in the city of Skellefteå. Northern Sweden is one of the most important areas in Europe when it comes to forestry and the wood industry. The Wood Science and Engineering group at LTU are engaged in a wide range of fields within the entire chain from forest to finished product.

On behalf of the COST Action FP0904 Management Committee I would like to thank everybody that kindly contributed to this meeting: all the authors and specially the keynote speakers; Callum Hill, Eiichi Obataya, Otto Th. Eggert and Kevin Candelier.
I gratefully acknowledge the help of the Scientific Advisory Committee in reviewing the abstracts and preparing the scientific program.
I express my sincere gratitude to Dick Sandberg and Mojgan Vasiri for their works in preparing the “book of abstracts” and also as the local organizer.

Parviz Navi
Chair of COST Action FP0904
Scientific Advisory Committee

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Registrarion
Mrs Inger Lindbäck                                       NEX meeting & event
## General Informations

**DATE**  
18-21 MAY

**VENUE**  
Arenan at Campus Skellefteå

**OFFICIAL LANGUAGE**  
The official conference language will be English.

**BADGE**  
Delegates must report to the registration desk to collect their name badges and conference materials. Every participant including his/her accompanying person is requested to wear a name badge during the conference period.

Venue: Lobby Arenan  
The desk will also be operating during the following schedule

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**SPEAKER’S RECEPTION DESK**  
Regarding Oral Presentations, please note that:  
It is expected that all presentations will be presented in English using Microsoft PowerPoint with a common computer provided by the conference organizers. We encourage you to check your PowerPoint file compatibility in advance. An overhead projector will be available by special request.

**IMPORTANT!** All speakers are required to check in at the Speaker’s Reception Desk by 18 & 19 May in order to hand over the CD or USB with the PowerPoint file, to be downloaded on the conference computer. All speakers during Tuesday must hand in their presentations during Monday May 19. The opening times for the Speaker’s Reception Desk are the same as for the Information Desk, Sunday 14:00-16:30 and Monday 7:20-11:30.

During Tuesday and Wednesday only by request in advance, (please contact the General Information Desk for further assistance)
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Water vapour sorption properties and surface chemical analysis of thermally modified wood particles, p. 92 |
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## WEDNESDAY  
**MAY 21**

<table>
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<td>08:00-08:30</td>
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| 08:30-10:30| Management committee meeting with the COST representative  
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Abstracts
Thermally Modified Wood – the role of hemicelluloses

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Keywords: water vapour, sorption, hydroxyl groups, hemicelluloses, mechanical properties

Thermal modification reduces the hygroscopicity of wood. The reason for this is generally attributed to a reduction of hydroxyl groups, as a result of degradation of the thermally labile macromolecular components of the cell wall (primarily the hemicelluloses). But how important is this mechanism? Degradation of the hemicelluloses is also responsible for changing the modulus of the cell wall; thermally modified wood is stiffer (and more brittle) than unmodified wood. The three macromolecular cell wall components of wood have specific roles to play in determining the properties of the wood cell wall and in this paper, attention will be paid to the hemicelluloses and the roles that they play in determining the properties of wood. The cellulose microfibril comprises the tensile reinforcement of the wood cell wall, exhibiting exceedingly a high modulus of elasticity (of the order of 145 GPa) under tension. However, although strong in tension, cellulose microfibrils buckle easily when subjected to a compressive load. Lignin has the role of providing a rigid enveloping matrix for the microfibril in order to provide resistance to compressive loads. But the surface of the microfibril is highly polar, with a high density of OH groups on the surface; whereas the lignin matrix has a much lower OH to carbon ratio. This results in a low adhesive interaction and hence poor interfacial stress transfer between the microfibril and the surface. One role of the hemicelluloses is to act as an interfacial coupling agent between the surface of the microfibril and the lignin matrix. Some microfibrils have a molecular geometry in regions of the structure that allows for close contact with the surface of a microfibril (or at least a cellulose chain) where there are chains of β-(1,4)-linked pyranose monomers. Examples of such structures include the glucose backbone in xylan which are the most abundant hemicelluloses in dicotyledon cell walls. Xyloglucan molecules have been isolated with lengths of up to 700 nm, long enough to easily span the intermicrofibrillar spaces in the cell wall many times. The β-(1,4)-linked glucose backbone has frequent substitutions at the glucose C-6 position by α-D-xylosyl residues, along with side chains of galactose, fucose and arabinose and acetylated side chains in addition. It is thought that a minimum chain length of 12-16 of glucose residues is required to allow for an interaction between xyloglucan and cellulose.

The most abundant hemicellulose of softwood species is galactoglucomannan, which has a backbone of β-(1,4) linked glucopyranose and mannopyranose residues. There are α-D-galactose residues linked to the 6-C(OH) of the backbone and there are two forms of the hemicellulose with a high or low galactose content, meaning that one hemicellulose motif is highly branched and the other is not. The C-2 and C-3 (usually of the mannose) is often acetylated. In the mannose residues, one of the OH groups is axially oriented (unlike in glucose, where all of the OH groups are equatorially oriented) which means that there is a mismatch in the H-bonding interactions between a mannose unit and a glucose unit. There is evidence to suggest that the glucomannan is aligned with the microfibril. This alignment is facilitated by the matching structure of the cellulose and the glucomannan backbone. Where regions of acetylation occur, hydrogen bonding between the backbone and cellulose is prevented.

Glucuronoxylans (xylans) are the most abundant hemicellulose in hardwoods and are also present in softwoods. Xylans have a backbone comprised solely of β-D-xylanopyranosyl residues, which are randomly substituted with 4-O-methylglucuronic acid. Softwood xylans are also substituted with α-L-arabinofuranosyl groups. In D-xylpyranose there are two equatorially oriented OH groups, but the C-6 carbon (and associated OH group) is absent. Although the backbone has the right structure to interact with the cellulose molecule, substitution of the backbone will prevent close association and the absence of one OH group will result in a weaker interaction (compared to cellulose-cellulose) in those regions where substitution is absent. Removal of substituents has been found to increase the affinity of xylans for cellulose. In the cell wall of maize, xylans with a low degree of arabinosyl substitution are found closely associated with microfibrils, whereas xylans with a high degree of arabinosyl substitution are found in spaces between the microfibrils. This work also indicated that an unsubstituted region of less than 15 contiguous xylosyl residues was sufficient for sorption onto a cellulose surface. As the degree of substitution of xylans by arabinosyl groups increases, the backbone tends to adopt a random coil geometry. The bonding between the surface of the microfibril and the hemicellulose is dipole-dipole, or due to dispersion forces, but the linkage between the hemicelluloses and lignin is primarily covalent in nature. It is well-established that there are chemical bonds (ester, ether) between hemicelluloses and lignin in the cell wall forming the lignin carbohydrate complex (LCC).

Water molecules act as plasticiser for wood because they are able to increase the chain mobility of the hemicelluloses and to a lesser extent the lignin. This is because the water molecules create void volume around segments of these polymers, allowing for greater freedom allowing the adoption of different configurations. Hemicelluloses are mainly responsible for imparting plasticity to the wood because they are able to adopt more configurations because of their open structure compared to lignin (which has a high cross-link density) and cellulose (which has extensive hydrogen bonding within the microfibril). The lower interaction energy at the interface between the microfibril and the matrix (a property of the interaction length) also allows for crack diversion when a propagating crack approaches the microfibril.
Many investigations of the thermal modification of wood determine the relative mass loss of the different species and these studies show that the hemicelluloses exhibit the most rapid rate of thermal degradation. However, using standard wet chemical techniques combined with gravimetric determinations can lead to misinterpretations. Degradation of amorphous cellulosic and potentially some lignin fragmentation could produce alkali-soluble components; furthermore there is a strong possibility of the hornification of hemicellulosic components, especially under hygrothermal conditions, which will reduce alkaline solubility. The combination of gravimetric methods with other analytical methods, such as sugar analysis, is therefore strongly advised. The results obtained from these studies are also highly dependent upon the experimental conditions. Experiments can be performed in air, under an inert atmosphere or vacuum (all in dry conditions), or under a steam blanket (hygrothermal) at atmospheric pressure or using higher pressures. The experiment may be open (where all degradation products and volatiles are lost from the chamber) or closed. Rate of heating, time at constant temperature and temperature at which the modification takes place, all affect the results. Wood species, moisture content, density, juvenile/mature, heartwood/sapwood, normal/reaction wood, earlywood/latewood and extractives are all variables affecting the experimental outcome.

Thermal modification is well known to lead to changes in mechanical properties of wood. With relatively short exposure periods, an increase in modulus is observed, although this is observed to decrease with longer treatment times. More dramatic is a large decline in toughness of the wood. This can be rationalised by consideration of the function of hemicelluloses at the microfibril-matrix interface.
Characterisation of thermally modified wood by a novel means of moisture sorption isotherm analysis

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Keywords: thermally modified wood, moisture sorption isotherm, hysteresis, structure relaxation

It is possible to determine $\theta(h)$, the occupancy of accessible water sorption sites in wood, at a given relative humidity $h=\text{RH}/100$ and temperature, by measurement of the change in moisture content (MC) in response to a quantitative change in oxygen content by mild thermal wood modification [1]. The found $\theta(h)$ is approximately given by $\theta(h)=h^n$, with $n\approx0.73$ at room temperature. Using this expression, the accessible water sorption site density can be simply calculated as a function of $h$ from experimental sorption isotherms. Applying this analysis to adsorption and desorption isotherms (Figure 1), one finds that the accessible water sorption site density in the adsorption line is relatively constant at low $h<0.6$ and increases progressively for $h>0.6$. The latter is attributed to inter-polymer H-bond breaking in the swelling cell wall, making hidden sorption sites accessible by the softened cell wall. The desorption line does not immediately return to the adsorption line, because the restoration of the broken H-bonds is a slow relaxation process, making a non-equilibrium excess of sorption sites available at lower humidity. Sorption hysteresis results from the occupancy $\theta$ of these excess sorption sites. Without any degree of relaxation, the upper desorption line can thus be directly calculated [1] from the accessible sorption site density at the start of the desorption at $h=0.95$ on the adsorption line (see broken line Figure 1, left) and the occupancy function $\theta$. This allows one to determine the amount of relaxation from the experimental adsorption-desorption loop, as the vertical difference between the calculated broken line and the measured desorption line.

Hysteresis and relaxation are clearly strongly $h$-dependent (Figure 1, left), hence for comparison, the corresponding values of the non-equilibrium excess accessible sorption site densities are taken by division of the hysteresis moisture and relaxed hysteresis moisture by $\theta(h)=h^{0.73}$, evaluated in the constant region below $h=0.6$ (Figure 1 right).

![Figure 1](image.png)

**Figure 1.** Dynamical Vapor Sorption isotherm adsorption-desorption loop (Accacia data [2]) in the humidity range $h=0.05-0.95$ with relaxation (dark-shaded) and hysteresis (light-shaded) contributions (left); and calculated density of accessible water sorption sites in the cell wall (right)

Applying this analysis to a series of heat treated Accacia specimen (Figure 2) one can observe that the maximum (relaxation-free) hysteresis follows the same decreasing trend as the equilibrium moisture content, but that the observed hysteresis moisture content seems almost independent of the (mild) treatment intensity: the decreasing hysteresis maximum is balanced by an equally decreasing relaxation. For more severe treatment intensity, there is very little relaxation and the observed hysteresis will approach the maximum value.

$\text{MC, MC/h}^{0.73}$
Figure 2. Changes of relaxed (dark-shaded) and hysteresis (light-shaded) non-equilibrium sorption site densities at h=0.50 with increasing heat treatment intensity (left to right). Accacia sample coding (labels on the horizontal axis) indicates temperature (°C) and treatment time (hrs) in oil, data from [2].

REFERENCES

ACKNOWLEDGMENT
The author wants to thank Prof. C.A.S. Hill for providing the raw DVS-isotherm data on oleo-thermally treated Accacia from the study of reference [2].
Water diffusivity of thermally modified beech wood

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Keywords: Fick’s law, convective boundary condition, sorption experiments, inverse identification

Thermal modification improves wood durability due to the reduction of its hygroscopic properties. The majority of studies were focused on determining sorption isotherms of the thermally modified wood. Much less attention was paid to determining water transfer in the modified wood. In the present work bound water diffusion in European beech (Fagus sylvatica L.) wood was in focus. The diffusivity was determined for both untreated and thermally modified wood. In the present study, a moderate treatment intensity was chosen (220°C for one hour) in order to observe distinct influence of the property alteration. The diffusion process was investigated in the radial direction. An example of input data for determining the bound water diffusivity is presented in Figure 1. Both the analytical methods, i.e. the initial sorption method [2] and Liu method [3] as well as the inverse identification [5] were applied to determine the diffusivity. The inverse identification considered two options for diffusivity (constant value and dependence on bound water content) and two options for the boundary condition (i.e. the convective boundary condition and the modified boundary condition with a relaxation time). It resulted in analyzing four identification options which are presented in Table 1.

The results obtained with the use of the analytical methods and the inverse identification was analyzed. The diffusivity dependence on the bound water content was discussed. The significant influence of the modification on the diffusivity values was found. The importance of the modification of the convective boundary condition was emphasized. The increased delay for obtaining the hygroscopic equilibrium of the modified wood was clearly shown. The determined diffusivity values were validated and the accuracy of the diffusion modeling was quantified. A significant reduction in mass diffusivity of wood after the thermal modification was clearly depicted and discussed in relation with the alteration of sorption equilibrium [1, 4] as well as wood ultrastructure [6].

Table 1. Identification options considered for the diffusivity identification, where D₀, α, c, d and τ are coefficients to be identified

<table>
<thead>
<tr>
<th>Identification option</th>
<th>Coefficients of the bound water diffusivity</th>
<th>Coefficients of the convective boundary condition</th>
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</thead>
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<tr>
<td>PAR3</td>
<td>$D = D_0$</td>
<td>$\sigma$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$M_{\infty} = c$</td>
</tr>
<tr>
<td>PAR4</td>
<td>$D = D_0 \cdot \exp(-\alpha \cdot M)$</td>
<td>$\sigma$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$M_{\infty} = c$</td>
</tr>
<tr>
<td>PAR5</td>
<td>$D = D_0$</td>
<td>$\sigma$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$M = c + d \cdot [1 - \exp(-t/\sigma)]$</td>
</tr>
<tr>
<td>PAR6</td>
<td>$D = D_0 \cdot \exp(-\alpha \cdot M)$</td>
<td>$\sigma$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>$M = c + d \cdot [1 - \exp(-t/\sigma)]$</td>
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</tbody>
</table>
Figure 1. The input data for the diffusivity determination (bound water content vs. square root of time). Thermally modified beech in the radial direction, sample thickness 6 mm, air relative humidity change 34-76%.

REFERENCES

ACKNOWLEDGMENTS
The work was financially supported by the Ministry of Science and Higher Education as the N N309 2876 3 research grant as well as the National Science Centre as the 2011/01/M/NZ9/00296 research grant.
The impact of elevated steam pressure during the thermal modification of Scots pine and Norway spruce

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Keywords: steam pressure, process conditions, open/closed system

Thermal modification to increase the dimensional stability and biological durability of solid wood has been extensively studied over the course of the past decades and comprehensively reviewed by Hill [1], Militz [2] or Esteves and Pereira [3]. All thermal modification processes follow the same principle – the exposure of the wood to elevated temperatures (usually between 160 and 230°C) while minimizing the residual oxygen in the treatment chamber. Nevertheless, differences in the applied conditions (open vs. closed system; steam, nitrogen or oil as a heating medium; number of process steps, etc.) exist between the various thermal modification processes. These process conditions have a strong impact on the chemical changes of the wood during the process and the resulting properties of thermally modified wood. Thermal modification using superheated steam in an open system at atmospheric pressure does not enable the control of the relative humidity. Thus, the wood moisture content is usually decreased to zero percent in a high-temperature drying step prior to the actual thermal modification step and reconditioned at the end of the process by water spray. In contrast, the use of steam in a closed reactor allows for the control of the relative humidity by increasing the steam pressure, as suggested by Willems [4]. By avoiding the absolute dry state of the wood, shrinkage stresses are reduced on the one hand [4], while certain chemical reactions (e.g. the formation of acetic and formic acid) are accelerated [5-7]. Although a vast number of publications on thermally modified wood exist, the impact of elevated steam pressure and the presence of water during the process are still not fully understood and have been the subject of recent investigations [6, 8-12].

This study includes the first results for the thermal modification at laboratory scale in a high-temperature autoclave at university of Göttingen that is equipped with an external steam generator and thus enables the control of the steam pressure and the relative humidity during the process. Small slats with dimensions of 32x60x800 mm³ were cut from logs of Norway spruce (Picea abies (L.) Karst.) and Scots pine (Pinus sylvestris L.), while separating between sap- and heartwood. The slats were conventionally kiln-dried to approx. 15% moisture content and thermally modified either at atmospheric pressure in an open system with a constant steam flow and a high-temperature drying step or at elevated steam pressure in a closed system without drying step but with a constant relative humidity during the process. The effect of the process conditions, peak temperature (160-230°C), peak duration (1-12 h) and maximum pressure (1-8 bar abs.) were investigated. Mass loss during the process and final moisture content were recorded for each process run. Furthermore, physical and biological properties (i.e. equilibrium moisture content and resistance against Rhodonia placenta) as well as chemical changes (i.e. by means of ESR-spectroscopy, acidity and phenol content) were investigated. The results give further insights into the impact of elevated steam pressure during the thermal modification on the final product.

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Effects of water re-saturation conditions and associated extractives leaching on thermal softening of wet wood

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Keywords: DMA, destabilization, glass transition, hardwood, specimens’ histories

Thermal softening, or thermo-activated viscoelasticity, is involved in several industrial and/or traditional craftsmanship techniques processing of wood. More fundamentally, it represents a very useful probe of chemical differences and/or of physical histories of wood samples [1,2,3,4,5]. Thermal softening of wet (= water-saturated) wood occurs at much lower temperature than that of completely (anhydrous) dried wood [6,7]. Softening of oven-dry wood happens at temperatures above 150°C, a temperature level at which wood amorphous polymers start to degrade [3,8]. Therefore, the characterisation of wet wood is the unique method that allows thermal softening to be distinguished from thermal degradation. Thermal softening of wet wood is also easier, or at least less energy consuming, in several applied processes. In many cases, it is not always practically feasible to start from green (e.g. never-dried) wood, either for scientific experiments or in real-scale applications, so that air-dry wood may need to be re-saturated. However, it is known that water re-saturated wood behaves differently from never-dried wood [4,5,9]. In addition, soaking wood in water is susceptible to remove secondary metabolites (extractives), some of which are known to modify viscoelastic properties of various wood species [10,11]. The objective of the present work is to provide insight on how thermal softening (studied by Dynamic Mechanical Analysis – DMA) of several hardwoods is affected by conditions (duration and temperature) of water re-saturation, and by additional effects of extractives leaching during these re-conditioning processes.

The study is based on five wood types from four hardwood species, chosen for contrasted chemical composition: black locust, oak, poplar, and beech (including normal wood and tension wood). Radial specimens for DMA (40×4×0.6mm3, R×T×L) were cut from naturally air-dried wood and prepared following a careful matching procedure. Three replicate specimens were tested for each modality of pre-treatment. Nearly 100 specimens were tested by DMA in total. Each DMA specimen was matched with a specimen designed for monitoring oven-dry weight losses due to a given pre-treatment. Pre-treatments included (see Figure 1): cold water (just after re-saturation, after 2 months and 10 months), 40°C water (4h and 96h), 70°C water (6h), 98°C water (2h), all cooled down slowly to ambient temperature again, and an overnight extraction in cold acetone followed by exchange with water. Tests were conducted in tension, using a TA Instrument Dynamic Mechanical Analyser, at frequencies 0.1, 1, 10 and 25Hz. Evolution with temperature, from 20°C to 100°C, was studied using 5 minutes isothermal steps every 5°C.

![Figure 1](image)

Figure 1. Illustration of the different pre-treatment modalities tested in this work, through an example of the evolution with temperature of loss tangent (tanδ, at 0.1Hz) for black locust wood. Error bars = 2×standard deviation between 3 specimens.
Results show that the thermal softening behaviour just after re-saturation appears to include a kind of “secondary transition” in the range of 50-60°C (Figure 2c for tanδ), which is consistent with previous findings [4,5]. However, the amplitude of this phenomenon is not similar for all tested wood types (see Figure 2). This disappears after re-conditioning above the glass transition temperature Tg (pre-treatments at 70°C or 98°C), which also leads to a reduction of the maximum of loss modulus and loss tangent and an apparent increase in Tg. Longer pre-treatments at lower temperature partially lead to similar results but even 10 months in cold water did not remove the “secondary transition”, whereas 40°C pre-treatment did but could not reduce as much the maximum of viscosity. Pre-treatments in water removed between 2 and 8% of extractives, depending on species and conditions. Results without re-conditioning but after cold acetone extraction show species-dependant effects notably on loss tangent tanδ, which strongly suggests an additive effect of extractives, and of physical re-conditioning, on the thermal softening behaviour of hardwoods.

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Presented experiments were conducted by the first author (I. Brémaud) while at LERFoB Nancy, and subsequent analyses conducted while at LMGC Montpellier in collaboration with LGPM Ecole Centrale Paris. Our thoughts and thanks go to the late Patrice Marchal for his generous and high quality technical help at LERFoB.
Flammability tests on thermally modified and untreated timbers

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Keywords: flammability, combustibility, standardised test, fire resistance

INTRODUCTION
This study investigates the flammability of thermally modified timber (TMT) compared to that of untreated timber. When natural timber is treated with temperatures of 160-250°C for a specific time period, its chemical, physical and biological properties change. While the improved resistance of TMT against fungi and insect attack has already been thoroughly investigated, there have been very few studies concerning its flammability. In this investigation a flammability test according to DIN EN 13501 [1] was made to determine whether TMT could be classified into the same category E (normally flammable) as most untreated timbers.

EXPERIMENTAL
A total of 20 samples of oak (*Quercus robur* L.), ash (*Fraxinus excelsior* L.) and pine wood (*Pinus sylvestris* L.) were tested, which were either untreated or thermally modified at 180°C or 200°C, as shown in Table 1. Measurements of samples were 190x90x25 mm. The testing of flammability in accordance to DIN EN 13501 requires a 15-second flame impingement at the lower leading edge of the samples. As long as a gauge mark 15 cm above the contact point is not reached by the flame within 20 seconds of time, the grade E test is considered passed. Furthermore the points of time of flame extinction and end of glowing combustion, as well as flaming droplets are also recorded.

Table 1. Number of tested samples; species and level of thermal treatment

<table>
<thead>
<tr>
<th>Species / Treatment</th>
<th>Untreated</th>
<th>180°C</th>
<th>200°C</th>
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<tr>
<td>Oak</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Ash</td>
<td>3</td>
<td>3</td>
<td>-</td>
</tr>
<tr>
<td>Pine</td>
<td>2</td>
<td>-</td>
<td>3</td>
</tr>
</tbody>
</table>

RESULTS
While all samples have clearly passed the grade E test, the durations of burning and afterglow greatly varied (see Figure 1). The samples of untreated oak were the only ones not inflamed. Oak samples modified at 180°C were burning with tiny flames for more than six minutes; still the gauge mark was not reached at any time. The untreated pine wood samples would have been completely combusted during the examination, which is why the test was prematurely aborted.

The reaction of TMT to fire differs by trend from that of untreated timber, greatly depending on the type of wood, and the level of thermal treatment. While untreated pine seems to even accelerate burning assumingly due to its high portion of resin (see Figure 2), these substances are no longer contained in that amount in the thermally modified samples, which is why their results are more similar to those of other timber of equal level of modification (see Figure 3).
CONCLUSIONS
The process of thermal modification of timber changes many of its substances of content as well as its physical properties. Although the fire resistance of TMT can be expected to be altered too, all samples in this examination have passed the requirements of the grade E test for flammability according to DIN EN 13501. While the 200°C treated pine wood samples showed a much higher resistance to fire than their untreated counterparts, these results cannot be transferred to hardwood species, maybe not even to other types of softwood.

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ACKNOWLEDGEMENT
The authors would like to thank the Federal Ministry for Education and Research for funding.
Chemical modification during heat treatment of Tunisian soft wood species

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Keywords: chemical analyses, heat treatment, tunisian wood, volatile compounds

Wood heat treatment is a good alternative method to improve natural durability wood species. The aim of this study is to determine chemical compounds of different heat treated samples of wood. For this purpose, several Tunisian softwood species (Aleppo pine, Radiata pine and Maritime pine) have been treated under vacuum atmosphere and performed from 200°C to 230°C to obtain mass losses resulting from wood thermal degradation of 8, 10 and 12%.

For each wood species and treatment intensity, chemical analyses were performed by measuring O/C ratio. The intensity of thermal degradation was evaluated by TD-GC-MS. Treated and untreated samples were subject to thermal degradation directly in the thermodesorption tube under nitrogen at 230°C during 15 minutes. Volatile compounds resulting from wood degradation were analysed.

The correlation between O/C ratio and intensity level of treatment (ML %) show an important increase of heat treated wood carbon content, while oxygen content significantly decreased. Elemental composition was strongly correlated with the heat treatment intensity depending from treatment duration, which directly conditioned the mass losses due to thermal degradation (Figure 1). Previous studies were shown similar results [4]. For each wood species, O/C ratio decreased linearly with the increase of the mass loss indicating that O/C ratio is a good indicator to estimate the mass loss of wood after thermal degradation. Therefore elemental composition seems to be a valuable parameter to evaluate the mass losses due to thermal degradation reactions which were directly connected to treatment intensity, for each wood species. This evolution of O/C ratio can be attributed to polysaccharides thermal degradation. In the literature, hemicelluloses are easily de-acetylated to form acetic acid catalyzing dehydration and depolymerisation reactions leading to anhydromonosaccharides like furfural [1-3].

TD-GC-MS analyses show that Tunisian softwood species contain less residual volatile products from hemicellulosic and lignin degradation like acetic acid or vanillin, according to heat treatment intensity increasing, characterized by mass losses. Chromatograms of thermodesorbed volatile products present in the different treated and untreated wood samples (Aleppo pine) are presented in Figure 2. The results concerning Aleppo pine were similar to those of the two other softwood species, Radiata pine and Maritime pine (data not shown). For the untreated sample, chromatogram showed (Figure 2) several natural extractives, mainly terpenoids, like hexanal (retention time = 3.43 minutes), beta-pinene (rt = 4.68), camphene (rt = 4.96), nonanal (rt = 6.99), isobornyl acetate (rt = 8.56), caryophyllene (rt = 11.40), humulene (rt = 11.84) and caryophyllene oxide (rt = 13.46) as identified major compounds. Most of these extractives disappeared after thermal treatment. For a 8% WL treatment, some remaining monoterpenes were still detected like pinene (rt = 4.74) or terpinyl acetate (rt = 5.97) with the natural caproic acid (rt = 5.71) and a thermal treatment product, furfural (rt = 3.85). For the most severe treatments, only acetic acid (rt = 2.42) due to hemicelluloses decomposition, vanillin (rt = 11.40) and guaiacylacetone (rt = 13.09), the first lignin degradation products, were observed. Quantitatively, since all these experiments were performed with the same weight of sawdust, it was obvious than the overall amount of extractives was strongly decreased by thermal treatment.
In conclusion, for the Tunisian wood species studied, we can observe a good correlation between the O/C ratio and the mass losses generated by the thermo-degradation. This O/C ratio evolution is clearly correlated with the chemistry of the thermo-degradation. As the European species, it’s possible to predict the treatment intensity, from the elemental analyses (O/C ratio) and predict the durability conferred to these species.

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ACKNOWLEDGEMENTS
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Effect of steam saturated atmosphere on some physical and mechanical properties of poplar wood

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Keywords: thermal treatment, saturated steam

Poplar wood is a species that is and it was widely used in Italy as solid timber or, more recently, as a part of wood-engineered products. Many important Italian heritage objects (e.g. panel paintings, wooden sculptures, old timber structures) were made in poplar. According to recent studies [1], nowadays the yearly need of poplar wood in Italy is around 3.9·10^6 m^3 from whom about one half are home grown.

Because poplar wood is very suitable for applications where the lightness is essential such as for the caravan sector, where the wood durability is essential as well, a research was planned in order to understand 1) the effects of the hydro-thermal (HT) treatment on modification of some poplar wood properties, and 2) for studying the suitability of HT as a means for producing artificial accelerated ageing of wood to be used in the restoration of wooden Cultural Heritage.

In this work, the effect of HT treatments in saturated moisture conditions is studied and the treatments were performed according to the experimental protocol presented in Table 1.

Table 1. Experimental matrix and main results in terms of colour variation (ΔE), mass variation (ΔMASS), MOE variation (ΔMOE) and moisture content variation (ΔMC) at standard environmental conditions (20°C and 65% RH) after hydro thermal treatment in saturated vapour conditions for the given temperatures and treatment periods

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>1h</th>
<th>2h</th>
<th>3h</th>
<th>6h</th>
<th>12h</th>
</tr>
</thead>
<tbody>
<tr>
<td>100°C</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>4.90</td>
<td>-0.9%</td>
<td>2.2%</td>
<td>-0.4%</td>
<td>-0.47%</td>
</tr>
<tr>
<td></td>
<td>-0.9%</td>
<td>-1.3%</td>
<td>-0.4%</td>
<td>-0.49%</td>
<td></td>
</tr>
<tr>
<td>120°C</td>
<td>8.30</td>
<td>-1.6%</td>
<td>2.1%</td>
<td>-1.01%</td>
<td>2.1%</td>
</tr>
<tr>
<td></td>
<td>-1.6%</td>
<td>-2.2%</td>
<td>1.3%</td>
<td>-0.96%</td>
<td>3.4%</td>
</tr>
<tr>
<td>140°C</td>
<td>15.27</td>
<td>-2.9%</td>
<td>4.7%</td>
<td>-1.30%</td>
<td>4.7%</td>
</tr>
<tr>
<td></td>
<td>-2.9%</td>
<td>-3.6%</td>
<td>9.4%</td>
<td>-1.40%</td>
<td>3.4%</td>
</tr>
<tr>
<td>150°C</td>
<td>17.00</td>
<td>-3.4%</td>
<td>7.4%</td>
<td>-1.59%</td>
<td>7.4%</td>
</tr>
<tr>
<td></td>
<td>-3.4%</td>
<td>-7.4%</td>
<td>-5.7%</td>
<td>-3.43%</td>
<td></td>
</tr>
</tbody>
</table>

Different samples of poplar wood (Populus x euroamericana Guinier) were prepared from the same board after a first cut by cleavage. The properties variations were determined as the difference between the treated and untreated state (20°C and 65% RH) and referred to the untreated state. The properties variations were studied on: colour, mass, MOE and moisture content. All these features, except the moisture content which is compared to a reference sample, were measured as a variation on the same specimens. The measurement of the colour was performed according to the CIE L*a*b* 1976 definitions and by a Konica-Minolta mod. CM700d colorimeter. The MOE was measured by a tensile test on small samples (8x3x150 mm) up to a 30% of ultimate load. Qualitative chemical analyses of the samples were performed by FTIR after extraction in Soxhlet as from the method defined by [3]. In Figure 1 is reported the mass variation for different temperatures and periods of treatment.
The mass losses are clearly depending on the treatment intensity and the reaction kinetics in saturated steam is very fast if compared to treatments performed for similar temperatures in conventional oven [2]. A mass loss of 8% (at 20°C and 65% RH equilibrium state) in the former case is attained after 3 hours of treatment at 150°C while in the latter after about 20 hours at 180°C. The equilibrium moisture content variation at 20°C and 65% RH result in the same trend than mass loss. The volume have shown an opposite behaviour if compare to oven treatments and result increased. The colour variations present trends in line with the mass loss. MOE show a tendency to increase up to 10% until a mass loss of 4% is attained. For larger mass losses MOE tends to reduce again getting to the same value of untreated samples at 5%. For higher mass losses MOE gets again to lower values if compared to untreated samples. From a chemical point of view results shown that a significant loss of glucoxylans starts after 6 hours of treatments at 100°C, and they completely disappear at 120°C for 12 hours, 140°C for 6 hours or 150°C for 1 hour. The other wood components do not seem to be very affected by the treatment.

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Soluble degradation products in thermally modified wood

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Keywords: thermal modification, birch, spruce, extract, phenol, methanol

It is well known that by thermal modification, wood products with higher resistance to swelling and higher durability can be produced. Detailed knowledge of such properties and how they are related to process conditions are still lacking. Characterization of chemical changes going on in wood during thermal modification processing as well as formation as soluble material is therefore of great interest. In this paper, we will present results from studies on extracts isolated from birch and spruce treated at superheated (Thermowood) and at pressurized/saturated steam (WTT) conditions.

Silver birch (B) and Norway spruce (S) was thermally modified at superheated conditions at 170, 185 and 212°C (SU170, SU185, SU212) as well as with pressurized and saturated steam (160-170°C) in a mainly closed process (SA160, SA170) [1]. Wood meal from treated wood was soaked in water (1:30) over night, filtered off and percentage weight of dried extract per dry wood was calculated [2]. Water extracted wood was further extracted in methanol in a similar way and yield of extracted material based on starting amount of dry wood was calculated. Analysis of UV-absorbance of water and methanol extract diluted in water (10-50 times) at 280 nm was performed. Difference UV-spectrum was obtained by comparison of UV-absorption spectra of diluted extract with corresponding diluted extract at pH 12.

In Table 1 content of dried material in extracts from thermally modified wood is presented. In our experiments less soluble material was found in the wood modified under superheated steam conditions than in the other type of process (Table 1). This indicated that degradation of wood was lower in wood treated under superheated than under saturated steam conditions.

Table 1. Content of dried material in extracts as well as content of hydroxymethylfurfural (HMF) and furfural in water extracts from thermally modified wood (%)

<table>
<thead>
<tr>
<th>Extract</th>
<th>SA160B</th>
<th>SA170B</th>
<th>SA170S</th>
<th>SU185B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.8</td>
<td>14.2</td>
<td>7.2</td>
<td>6.0</td>
<td>3.3</td>
</tr>
<tr>
<td>Methanol</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>4.9</td>
<td>10.4</td>
<td>2.6</td>
<td>2.1</td>
</tr>
</tbody>
</table>

| HMF     |        |        |        |        |
| 0.059   | 0.087  | 0.266  | 0.792  | 0.013  |
| Furfural| 0.017  | 0.031  | 0.071  | 0.048  | 0.110  |

With the exception of birch treated under saturated steam conditions at 170°C, smaller amounts of methanol extractable material was left after extraction with water than the amount of material removed by water extraction (Table 1). Soluble carbohydrates mostly of oligomeric type have been found in the water extracts [2]. Water extract isolated from birch modified under saturated steam conditions in our experiment contained more soluble carbohydrates at 160°C (SA160B) than at 170°C (SA170B) [2].

Degradation products, hydroxymethylfurfural (HMF) and smaller amounts of furfural were found in the water soluble material (Table 1) [2]. Content of furfurals were highest in spruce modified under saturated steam conditions (SA170S). This is probably due to the higher content of hexosans in spruce than in birch which preferably give hydroxymethylfurfural on heating and that the conditions here were more severe under saturated than superheated steam conditions.

Furfurals have strong UV-absorption at 280 nm and it could be seen that UV-absorptions at 280 nm in water extracts was strongest in case of spruce modified in saturated steam (Table 2). By obtaining difference UV-spectrum absorptions from phenolic compounds can be studied at 250 nm. Phenolic compounds contributed also to the observed absorption in UV-spectra (280 nm), especially birch treated under saturated steam conditions (Table 2).

Methanol soluble material from modified wood after water extraction was more colored than corresponding water extracts. It can be seen in Table 2 that UV-absorption at 280 nm, except for Thermo-D of spruce (SA170S), was stronger in the methanol extract than in the corresponding water extract even though neither HMF nor furfural was found in methanol by analysis with HPLC. By using difference UV-method, results pointed to that content of soluble phenolic compounds was larger in methanol than in water extracts (Table 2).
**Table 2.**  UV-absorption at 280 nm and at 250 nm in difference UV-spectra in water and methanol extracts per gram dry wood

<table>
<thead>
<tr>
<th>UV-method</th>
<th>Extract</th>
<th>SA160B</th>
<th>SA170B</th>
<th>SA170S</th>
<th>SU185B</th>
<th>SU212S</th>
</tr>
</thead>
<tbody>
<tr>
<td>280 nm</td>
<td>Water</td>
<td>0.47</td>
<td>0.92</td>
<td>1.30</td>
<td>0.28</td>
<td>0.28</td>
</tr>
<tr>
<td>280 nm</td>
<td>Methanol</td>
<td>0.83</td>
<td>2.27</td>
<td>0.55</td>
<td>0.33</td>
<td>0.46</td>
</tr>
<tr>
<td>250 nm (diff) Water</td>
<td>0.35</td>
<td>0.38</td>
<td>0.09</td>
<td>0.12</td>
<td>0.09</td>
<td></td>
</tr>
<tr>
<td>250 nm (diff) Methanol</td>
<td>0.55</td>
<td>0.89</td>
<td>0.14</td>
<td>0.15</td>
<td>0.21</td>
<td></td>
</tr>
</tbody>
</table>

It could be concluded that thermal treatment under saturated steam conditions in this investigation gave more of soluble degraded material than under superheated conditions especially for birch. Careful control of process parameters is necessary to reduce degradation reactions during pressurised/saturated steam conditions such as in the WTT-process. Moisture content was relatively high (MC ca. 15%) during the modification of wood with saturated steam and should be avoided.

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An NIR and XPS study of the lime wood samples modified for different periods at lower temperature and relative humidity

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Keywords: near infrared spectroscopy, X-ray photoelectron spectroscopy, hydro-thermal treatment

The degradation of lime (Tilia cordata Mill.) wood components during wood heat treatment under low temperature at about 140°C and relative humidity of 10% percentage were evaluated. The NIR coupled with second derivative, principal component analysis and two dimensional correlation spectroscopy were used in order to understand how these techniques are able to evaluate structural differences resulted under hydro-thermal treatment of the wood over a period of 504 h. The NIR spectra of treated samples were compared with the untreated one. Due to the broad bands in the NIR spectra (Figure 1), the assignment and modifications occurring during treatment is difficult, therefore the second derivative (Figure 1) and the principal component analysis were also used [1].

Figure 1. NIR spectra and their second derivative spectra of hydro-thermal treated lime wood (Tilia cordata Mill.)

The used analyses indicate transformations in the chemical composition and structural changes induced by the degradation of wood components.

The wood components degradation (hydrolysis, oxidation, and decarboxylation reactions) was observed by differences in the bands intensities or position from NIR and second derivative spectra.

By 2D COS was possible to predict the sequential order of the variation of the structural groups using the applied treatment time as external perturbation. Therefore, was observed as first moment changes the OH and C–O groups from carbohydrates and lignin, followed by C=H, C–H and CH2 groups from lignin, cellulose and hemicelluloses.

For the treated samples up to 168h, a decrease of the OH stretching and deformation bands of water was observed, wood becoming more hydrophobic, but for a longer treatment period the structure of wood become again hydrophilic.
On the other hand, X-ray photoelectron spectroscopy is particularly well suited for the study of surface chemistry of complex organic materials as wood and the most studies up to now focus attention on the surface chemical composition of different lignocellulosic materials. The modification in hydro-thermal treated wood was evidenced both from the carbon (C 1s) and oxygen (O 1s) high resolution spectra.

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Effect of thermal treatment on XRD, ATR-FTIR AND SEM analysis of several wood species

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Keywords: Thermal treatment, ATR-FTIR, SEM and XRD, Chemical properties

The objective of this study is to determine the effects of chemical properties of three wood species after thermal treatment (Pinus brutia, Quercus infectoria, Platanus orientalis L.), where obtained from Dohuk forestry in Iraq. Those species are subjected to thermal treatment for duration of two hours and a temperature of 200ºC. Swelling, anti-swelling efficiency and shrinkage as physical properties are evaluated. For chemical properties used (ATR-FTIR, SEM and XRD) analysis. Chemical properties were compared to the untreated samples. The scanning electron microscopy (SEM) was used to identify the morphology of the wood samples. The ATR-FTIR spectrometer was used to supply superior quantitative and qualitative information for routine analysis of solids and liquids of wood.

The results show that the crystallinity ratio in XRD analysis were identified as 74.69% and 86.42% in Platanus orientalis, 52.18% and 71.37% in Quercus infectoria, 63.29% and 41.41% in Pinus brutia for respectively before and after thermal treatment. The Crystallinity ratio of the Platanus orientalis and Quercus infectoria increased after heat treatment while the Crystallinity ratio of the Pinus brutia decreased after heat treatment. The SEM results indicated that there were significant differences in the three wood species after thermal treatment it was observed that the fibre had become cleaner and the roughness was reduced which almost all impurities have been removed from the wood surface. The water absorption and swelling of treated samples also decreased with increasing temperature and duration time of heat treatment process. This is due to the fact that thermal treatment affects the wood cell polymers which consist of hemicellulose, cellulose and lignin according to ATR-FTIR spectrometer

Thermal treatment affected the chemical properties of the wood which changes the chemical composition of wood leading to weight loss and increased the dimensional stability due to cross-linking in lignin, due to destruction of several hydroxyl groups and due to decrease affinity with water in the case of treated wood. As a result of the thermal treatment it was created a dark colour and prevents the emission of odours compared to untreated wood. All of the three wood species can be utilized using proper thermal treatment, where stability is important. Also thermal treatment wood species can be used for many applications like furniture, doors, interior and exterior decoration.
Recoverable effects of heat treatment

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Keywords: hygroscopicity, equilibrium moisture content, shape fixation, recovery

INTRODUCTION
Heat treatment is a useful method to improve the practical performance of wood with simple procedure and without any harmful chemicals. The reduced hygroscopicity achieved by the heat treatment gives excellent dimensional stability [1]. Stress relaxation induced by the heat treatment enables effective shape fixation of compressed wood [2]. The heat-treated wood is also useful for the restoration of antique wooden items because the dark colour of aged wood can be precisely reproduced by heating [3]. In general, the effects of heating are explained by irreversible chemical changes such as crystallization of cellulose and decomposition of hemicelluloses. However, careful discussion is needed to characterize the heat-treated wood because the effects of heating is sometimes reversible i.e. recoverable by mild after-treatments such as moistening and soaking in organic solvents. This paper briefly introduces some reversible phenomena around the heat treatment of wood, for clear understanding and reliable evaluation of heat-treated wood.

RECOVERY OF HYGROSCOPICITY DUE TO MOISTENING [4-6]
The hygroscopicity directly determines the dimensional stability of wood, and also, it indirectly affects most of wood properties strongly depending on its moisture content. Figure 1 shows the equilibrium moisture content (ECM) of heat-treated spruce wood plotted against the loss in weight (WL) due to heating. By heating in the absence of moisture, the ECM of wood decreases with increasing WL. However, the ECM is significantly recovered after the heat-treated wood experiences humid condition (97% RH). Such a recovery is almost completed by the 1st moistening process and thereafter the ECM remains unchanged during the following humidity cycling. This fact suggests that the reduction in ECM due to dry-heating include 1) irreversible effect resulting from chemical changes in wood constituents and 2) temporary annealing-like effect recoverable by moistening. Similar recovery of hygroscopicity has been reported by Mitchell et al. [7].

The degree of recovery depends on the moistening humidity as well as WL. The reversible change is enlarged by delignification, but reduced by the removal of hemicelluloses. Similar reversible change is recognized in xylan and regenerated cellulose, but less significant in paper and milled wood lignin. From these results, it is speculated that some rearrangement of amorphous polysaccharides is responsible for the reversible reduction in ECM due to dry-heating. Although such rearrangement cannot observed directly, it is worth trying to compare the heat-treated wood before and after moistening, with respect to their viscoelastic properties or the degree of orientation of amorphous polymers detectable by IR measurement [8].

Reversible or irreversible, that is a question when we evaluate the practical performance of heat-treated wood. Many data will need to be re-examined in respect of its reversibility. For example, particular reduction in hygroscopicity due to heating at moderate humidity has been reported [9], but that result have possibly included reversible effect.
Figure 2. Effects of heating humidity on the ECM of heat-treated wood at 25 °C and 60% RH. Crosses, unmodified; Open plots, heat-treated at 120 °C for 24 h followed by vacuum drying at room temperature; closed plots, heat-treated and moistened at 25 °C and 100% RH

SORPTION CHARACTERISTICS AND WET VOLUME OF HEAT-TREATED WOOD [10]

By using Hailwood-Horrobin adsorption equation, the adsorbed water can be separated into hydrated (Mh) and dissolved (Ms) water. Figure 3 shows the changes in Mh and Ms due to dry-heating and steaming as a function of WL. Note that the sorption isotherm used for this analysis does not include “temporary effect” because the samples had been previously boiled. Different effects of dry-heating and steaming are characterized by the amount of Ms: the reduction in Ms due to dry-heating is much greater than that due to steaming, while the Mh decreases monotonously with increasing WL regardless of heating methods. These results suggest that the dry-heating is more effective to restrict the swelling of wood at high RHs. The restriction of swelling is also recognized in the wet volume of wood. As shown in Figure 4, the wet volume of dry-heated wood is always smaller than that of steamed wood at the same WL. Interestingly the wet volume reduced by dry-heating is recovered after soaking in dimethyl-sulfoxide (DMSO), whereas such recovery is not recognized in the steamed wood. This fact implies that some hydrophobic structure is temporarily formed during dry-heating and it restricts the swelling of wood.

During dry heating, irreversible chemical changes such as crystallization and condensation as well as temporary formation of hydrophobic structure proceed in a shrunk state wherein the intermolecular spacing is minimized. On the contrary, such a tight structure cannot be formed during steaming because the chemical changes occur in a swollen state wherein the intermolecular spacing is maximized with adsorbed water. In respect of the changes in Mh and Ms, dry-heating is similar to formaldehyde treatment whereas the steaming is similar to acetylation. Different sorption characteristics of hydrothermally-treated should be considered to evaluate their dimensional stability.

Figure 3. Relative amount of hydrated (Mh) and dissolved (Ms) water adsorbed onto the heat-treated spruce wood plotted against WL. Circles, dry-heated at 120~180 °C for 1~24 h; Squares, steamed at 120~180 °C for 10~60 minutes
Figure 4. Relative volume of heat-treated spruce wood in water as a function of WL. Circles, dry-heated at 120–180°C for 1–24 h; Squares, steamed at 120–180°C for 10–60 min; open plots, after the heat treatment; filled plots, heat-treated, soaked in DMSO and leached in water. The volume is normalized by that in unmodified dry state.

RECOVERY OF SHAPE DUE TO SOAKING IN ORGANIC SOLVENT

When a moistened wood is compressed at high temperatures, its compressed shape is effectively fixed and remains unrecovered even after boiling in water. Such a “permanent” shape fixation is accompanied by stress relaxation, and it is usually explained by irreversible chemical changes such as depolymerisation of wood constituents [2] and recrystallization of cellulose [11]. On the other hand, Higashihara et al. has found that the “permanently-fixed” shape of hydrothermally-treated wood was much recovered when it was soaked in pyridine or DMSO [12]. As those organic solvents do not induce serious degradation in wood polymers, the significant shape recovery in those solvents suggests that some hydrophobic structure is formed during hydrothermal treatment and it is partly responsible for the shape fixation of compressed wood. It might be a kind of hornification, but detailed mechanism is still unclear.

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An incremental model for wood behaviour including hydro-lock effect

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Keywords: mechanosorptive behaviour, hydro-lock effect, viscoelasticity, incremental model

BASIS OF THE STUDY
The mechanical behavior of wood is highly dependent on environmental conditions and rheological effects. It is governed by mechanosorptive effects which result from a complex interaction between mechanical loading and moisture content variations. Tests carried out under variable humidity and loading have shown that this behaviour can be analysed on the base of strain partition [1]. Hence, the evolution of the total strain is given by the following differential equation

\[ \ddot{\varepsilon} = \dot{\varepsilon}_w + \dot{\varepsilon}_e + \dot{\varepsilon}_{HL} + \dot{\varepsilon}_v \]  

(1)

\( \varepsilon_w = \alpha \varepsilon \) is a free strain, which depends on the shrinkage-swelling coefficient and moisture content. \( \varepsilon_e = \sigma / E(w) \) is an elastic strain, where \( E(w) \) is the elasticity modulus which depends on humidity; \( \dot{\varepsilon}_e = \dot{\varepsilon}_e^\sigma + \dot{\varepsilon}_e^w \), \( \dot{\varepsilon}_e^\sigma \) is due to a stress variation and \( \dot{\varepsilon}_e^w \) is due to a moisture content change. \( \varepsilon_v \) is a viscoelastic strain which depends on the level and duration of loading; its value is given by Boltzmann’s integral, where the creep function is represented by a Dirichlet’s series [2], whose parameters depend on the moisture content. The hydro-lock strain \( \varepsilon_{HL} \) is defined below; it results from a strain-locking effect during a drying phase [3,4].

HYDRO-LOCK STRAIN
Mechanosorptive tests were carried out on silver fir specimens (\( \textit{Abies alba} \) Mill.), in order to study the process of creation and recovery of an hydro-lock strain [1]. Humidity varied stepwise, so as to describe a sine-like results (Figure 1) show that the hydro-lock of stress variations at constant humidity, phase under stress, it appears so as to term \( \dot{\varepsilon}_e^w \). During a moistening phase, it until it disappears when the moisture content \( \overline{w} \) at the beginning of the drying phase under these evidences and on thermodynamic proposed to represent its variation as follows

\[ \dot{\varepsilon}_{HL} = -\frac{E'(w)}{E^2} \overline{w} \tilde{\sigma} \text{ where } \tilde{\sigma} = \frac{E(w) \text{ } E(\overline{w})}{E(w) - E(\overline{w})} \varepsilon_{HL} \]  

(2)

INCREMENTAL NUMERICAL MODEL
Equation (1) is represented by an analogue model which the viscoelastic behaviour is represented by a in parallel with a Maxwell’s branch. This model the effects of creep as well as relaxation. Based on the differential equation is integrated over any finite interval\([t, t + \Delta t]\).

\[ \forall \ t, \Delta t \quad \Delta \varepsilon = \dot{\varepsilon}_{HL} + \varepsilon_{hist} + \ddot{\varepsilon} \]  

(3)

Equation (3) is the incremental constitutive law of wood under variable stress and moisture conditions. \( \dot{\varepsilon}_{HL} \) is a fictitious compliance which depends on the viscoelastic properties, time interval and hydro-lock effect. \( \varepsilon_{hist} \) is a history term which accounts for viscoelastic memory. \( \dot{\varepsilon} \) is a term which depends on the evolution of the free strain and the hydro-lock strain during the time step \( \Delta t \).

Figure 1. Evidenced hydro-lock strain from test (solid), from Eq.2 (dashed)

Figure 2. Analogue model for coupled hydro-mechanical effects in wood behaviour
As an example, results given by the model in the case of cyclic stress and moisture variations are shown in Figure 3 in the form of stress-strain diagrams. The two diagrams differ in the phase shift between the cyclic stress and the cyclic moisture. The dashed lines show the evolutions of the elastic (2), viscoelastic (3) and hydro-lock (4) strains during one cycle. Their sum (solid line 1) is the total strain minus the free strain (see Equation (1)). A significant difference is observed between lines (1) in the two diagrams. This difference mainly results from the difference between the hydro-lock strain evolutions (lines 4), which is a consequence of the phase shift between moisture and stress cycles. This exemplary simulation shows the necessity to account for coupled hydro-lock effects in wood in order to properly describe its behaviour under variable loading and moisture conditions.

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Elastic cell wall modulus and hardness of S2 layer and middle lamella in viscoelastic thermal compressed wood

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Keywords: mechanical behaviour, nanoindentation, hybrid poplar

Low-density hybrid poplar (Populus deltoides × Populus trichocarpa) specimens were densified with Viscoelastic Thermal Compression (VTC) process [1]. The width and length of the specimens were 56 mm and 170 mm, respectively. The initial thickness of the specimen varied, depending on the intended level of densification to be achieved by the VTC process. The specimens were compressed to a target thickness of 2.5 mm. Since three different initial thicknesses were used (4, 5 and 6 mm), VTC specimens with three degrees of densification (63, 98 and 132%) were created, Table 1.

<table>
<thead>
<tr>
<th>Initial thickness (mm)</th>
<th>Initial density (D1) at MC=0% (g cm⁻³)</th>
<th>Density after compression (D2) at MC=0% (g cm⁻³)</th>
<th>Density ratio (D2-D1)/D1</th>
<th>Degree of densification (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>0.339</td>
<td>0.552</td>
<td>0.63</td>
<td>63</td>
</tr>
<tr>
<td>5</td>
<td>0.341</td>
<td>0.676</td>
<td>0.98</td>
<td>98</td>
</tr>
<tr>
<td>6</td>
<td>0.340</td>
<td>0.792</td>
<td>1.32</td>
<td>132</td>
</tr>
<tr>
<td>6 (control)</td>
<td>0.331</td>
<td>0.331</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

A Hysitron Triboindenter equipped with a three-sided pyramid cube corner diamond indenter tip was used to determine the mechanical properties of the fiber cell walls of control and VTC specimens. From each group of specimens a 3 x 3 x 3 mm specimen was cut and embedded with Spurr epoxy resin. Axial indentations were performed in fiber across the transverse surface of the secondary cell wall. A multi-load cycle for indentations with a maximum force of 100 μN, total loading time 104 seconds with 10 segments of loading and unloading (1 second each) was used. The increasing loading steps were: 0.13, 2.04, 6.25, 12.76, 21.56, 32.65, 46.05, 61.73, 79.72, and 100 μN. From the indentation experiment, the peak load, the penetration depth at peak load and the initial slope of the unloading curve were obtained. The load-displacement data from the nanoindentation test were then used to calculate hardness and elastic modulus of the cell wall at the indentation location, S2 layer and middle lamella [2].

The hardness of S2 layer and middle lamella increased in the VTC specimens, while degree of densification had limited effect, statistically insignificant effect (Figure 1). The results showed that the cell wall hardness in S2 layer increased significantly from 0.34 GPa for the control to a maximum of 0.39 GPa in VTC specimens. Furthermore, cell wall hardness in middle lamella increased from 0.35 GPa in control specimens to 0.45 GPa in VTC densified specimens. Differences in elastic modulus among control and VTC specimens were found as well (Figure 1), however the changes in elastic modulus in the S2 and middle lamella of VTC specimens were not statistically significant.

It appears that the degree of densification is a secondary effect, or even has no effect on cell wall hardness and elastic modulus. Any change is due to the influence of steam and temperature. The nano-indentation results are indicating that during the VTC treatment there is some cross-linking. Namely, the VTC treatment causes weight loss, which alone should reduce elastic modulus and hardness. However, if some of the degradation products are chemically reactive and form chemical bonds with the cell wall polymers, this should make the cell wall stiffer. If the degree of densification caused some compression of the polymers within the cell wall, such that they were brought close enough for new chemical bonds to form, or more entanglements of the molecules, then the cell wall should become stiffer. This latter case is not supported by the data. However, the results showed that the increase in mechanical properties of VTC wood could be also due to an increase in the mechanical properties of the cell wall and not only due to an increase in wood density [3].
Figure 1. Modulus of elasticity and hardness of middle lamella (left) and S2 layer (right) of control and VTC wood

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ACKNOWLEDGMENTS

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Wood heat treatment modifications: effects of initial moisture and air exchange rate on kinetic and final product characteristics

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Keywords: heat treatment kinetic, air exchange rate, time-temperature equivalency, rheology, wood properties

This contribution aims to present the kinetic of heat treatment as well as the effect on some physical and mechanical properties of poplar wood (*Populus alba* L.). Some tests were performed at different treatment temperatures from 180ºC to 260ºC. Moreover two different air ventilation settings qualified as low and high air exchange rate (*AER*) with the exterior were applied during the treatments.

The treatment kinetic is studied, starting from the oven dry state condition, by following the mass loss during time and highlighted a possible time-temperature equivalency, see Figure 1 (increasing the temperature has the same effect than treating for a longer time).

![Figure 1](image-url)

**Figure 1.** Mass loss during heat treatment at different temperatures and different air exchange rates (low and high *AER*). The markers are the experimental points and the plains lines are the model outputs for each condition. The horizontal time-axis is in logarithmic scale

The complete analysis of the kinetic, using for instance the complex plot (Cole-Cole) to avoid the time parameter and evaluate the model parameters [1], supports the Time-Temperature Superposition Principle [2]. The mass loss *versus* time is formalized through a power law arising from rheological considerations and a master curve of the heat treatment build. The Arrhenius law was checked and used to achieve the complete formulation including the temperature and the time effects [3]. This work clearly evidences how the heat treatments at low or at high *AER* presents different kinetics even if almost similar activation energy values are found.

In addition, some physical and mechanical properties of wood after treatments performed up to a mass loss of 7 and 10% and starting from oven dry state or standard environmental conditions were measured. All the treated samples have shown statistically significant differences compare to the untreated one. The statistical analysis have shown how the treatments performed starting at dry or wet condition up to a dry mass loss of 7 and 10% present very similar effects on almost all the analysed parameters except *MOE*. For *MOE* in fact starting the heat treatment from the wet condition results in a lower reduction if compared to heat treatment started from the oven dry state condition.
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ACKNOWLEDGMENTS
The authors acknowledge the financial support of Toscana Regional Administration with the POR CReO projects funding line as well as the ERDF funding line. Authors would like to acknowledge Mr. Giacomo Del Bianco for the help in the measurement of physical and mechanical properties.
A comprehensive dual-scale computational model able to simulate the heat-treatment of a thick-bed of particles or boards

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Keywords: biomass, mild pyrolysis, simulation, multiscale, industrial process

Thermal modification, a process able to alter several wood properties whose effects depend on the final use. For example, in the case of solid wood, it improves both durability and dimensional stability thanks to the reduction of the hygroscopicity and in the case of BtL chain (biomass to liquid) it is a pre-treatment able to drastically reduce mechanical resilience, hence the grinding energy [1-3].

At the local scale, this process involves chemical reactions, thermally activated and with significant exothermic effects, especially above 250°C. At the particle scale, these reactions occur with a strong coupling with the coupled heat and mass transfer inside the particle. The coupling is two-ways:

- the thermal activation depends on the temperature field inside the particle which is the result of internal and external heat and mass transfer. In this coupling, the mass transfer is always important as it involves the latent heat of vaporisation,
- the chemical reaction provides sink or source terms in the energy balance, which affect the temperature field.

A comprehensive model able to account for most of the phenomena involved at the level of a macro-particle was proposed some years ago [6]. For example, the model perfectly predicts the plateau at the boiling point of water when dealing with humid particles. In the heat treatment stage, the model successfully predicts the double pressure peak: one due to water evaporation followed by a second one due to volatiles production. The temperature overshoot due to exothermic reactions is also well predicted. This phenomenon is very important in the domain of mild pyrolysis, where the temperature level is low (typically in the range 200 to 300°C), but the residence time is very long (up to several hours). In such conditions, the effect of some tens of degrees might have a dramatic impact on the heterogeneity of the heat treatment.

However, at the industrial scale, the configuration is even more complex, as the treatment often occurs in a configuration of fixed bed (stack of boards or bed of particles, Figure 1). Such a configuration renders the process control very difficult, as all couplings (heat/evaporation, heat/exothermic reactions) are strengthened in a thick bed configuration. Based on our extensive knowledge in the field drying and dual-scale modelling [7], we proposed a comprehensive dual-scale model of heat and mass transfer able to deal with any MC field inside the product, including liquid water. This model allows the gradual modification of gas conditions through the bed to be accounted for (Figure 2a). During the drying phase, the water evaporation cools down the gas flow along the bed and increases its humidity. Consequently, the particles situated at the bed outlet dry slower. This effect is particularly obvious if the initial moisture content is high (20% in Figure 2b, against 3% in Figure 2a). During the heat treatment phase, due to exothermic reactions, the gas temperature increases along the bed. Consequently, the exothermic reaction are more and more activated from bed inlet to bed outlet.

A comprehensive model is an excellent tool in R&D, either to design new configurations / new process control or to help in addressing problems encountered in running plants.
Figure 2.  

a) Evolution of temperature (surface and core) and dimensionless pressure (core) for boards 1, 6 and 11 (from inlet to outlet). Case study A (MC=3%, Thickness=35 mm).  
b) Evolution of temperature (surface and core) and dimensionless pressure (core) for boards 1, 6 and 11 (from inlet to outlet). Case study B (MC = 20%, Thickness = 35 mm) [5] 

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Numerical analysis of timber fracture due to mechanical and thermal loads: an approach based on invariant integral $A$

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Keywords: fracture, mixed mode, thermal fields, path independent integral, finite element method

Due to the advantages provided by its mechanical behaviour particularly under extreme loading conditions such as fire and seismic events, in addition to its aesthetic and environmental effects, timber is commonly employed in building and civil engineering structures. As well known, timber elements exhibit micro-cracks, which can propagate due to fatigue, overload or creep loading and cause failure of the structure. In addition, timber is a hygroscopic material whose mechanical behaviour is very sensitive to climatic changes such as temperature and moisture variations. For example, drying process accelerate the crack growth, while wetting process induce the delay of the crack propagation. To predict the crack growth process many numerical methods were developed to characterize the mechanical fields in the neighbourhood of the crack. These methods are inefficient when dealing with mixed mode crack growth problems because it is necessary to separate the displacement field into a symmetric and antisymmetric parts. In this work a new formulation of the $A$-integral is propose based on the analytical work developed by [1]. This formulation takes into account the effects of thermal field induced by temperature variation and mechanical load applied on the crack lips which can be caused by fluid action or contact between the crack lips during the crack growth process.

Experience has shown that integration through curvilinear path induce some inaccuracy on numerical results. To overcome this problem, the curvilinear path is transformed into surface domain by introducing a vector field $\vec{\gamma}$. This mapping function is continuously differentiable and takes these values: $\vec{\gamma} = (1,0)$ inside the ring $\mathcal{V}$, and $\vec{\gamma} = (0,0)$ outside it. Hence, the use of the Gauss-Ostrogradsky’s theorem enables us to obtain the $A$-integral given by:

$$A = \int_{\Omega} \frac{1}{2} [\sigma_{ij} \gamma_{ij} - \sigma_{ij} \gamma_{ij}] \phi_{ij} dS + \int_{\Omega} \frac{1}{2} [\sigma_{ij} \gamma_{ij} - \sigma_{ij} \gamma_{ij}] \phi_{ij} dS + \int_{\Omega} \frac{1}{2} \gamma_{ij} \phi_{ij} dx_1$$

where $\phi_{ij}$ and $\phi_{ij}$ are stress tensor components deduced from the real displacement field $u$ and the virtual displacement field $\psi$, respectively. The first integral represents the effect of mechanical loads applied far from the crack where $\sigma_{ij}$ and $\sigma_{ij}$ are stress tensor components and $\gamma_{ij}$ and $\gamma_{ij}$ are the corresponding virtual components. The second integral represents the effect of thermal load induced by temperature variation $\Delta T$, with $\psi$ is a virtual displacement field as defined by Bui et al [2] and $\gamma$ is a real coefficient function of material parameters. The third integral represents the effect of pressure on crack lips.

The $A$-integral is implemented in the finite elements software Cast3m. In order to illustrate the accuracy of the numerical results it has been decided to deal at first with isotropic material having a Young’s Modulus $E = 20000 \, \text{daN/mm}^2$, Poisson’s coefficient $\nu = 0.3$ and linear expansion coefficient’s $\alpha = 5 \times 10^{-5} \, \text{K}^{-1}$. The first problem deal with rectangular plate ($2b = 400 \, \text{mm}, 2h = 1200 \, \text{mm}$) with a central crack ($2a = 200 \, \text{mm}$) subjected to a uniform tension $\sigma = 1 \, \text{daN/mm}$ applied on the lips. The second problem deal with rectangular plate ($2L = 800 \, \text{mm}, 2W = 200 \, \text{mm}$) with edge crack ($a = 100 \, \text{mm}$) subjected to linear temperature field $\Delta T = 2T_0 \gamma$, where $x \in \left[\frac{W}{2}, \frac{W}{2}\right]$ and $T_0 = 100 \, \text{C}$. We note that the finite elements analysis is performed under the assumption of plane strain conditions. Table 1 compares the numerical results and the reference solution taken from the literature [3, 4] concerning the stress intensity factor $K_p$ representing the opening mode. As can be seen, the numerical results obtained by Finite Elements Method (FEM) are in good agreement with the reference solution since the error does not exceed 3.4%. Hence, the accuracy of the numerical implementation of the $A$-integral is approved.

Table 1. Comparison of the results obtained by Finite Elements Method to reference solution

<table>
<thead>
<tr>
<th>Problem</th>
<th>Reference</th>
<th>FEM</th>
<th>Error (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Problem 1</td>
<td>17.725</td>
<td>17.544</td>
<td>1</td>
</tr>
<tr>
<td>Problem 2</td>
<td>130.15</td>
<td>125.72</td>
<td>3.4</td>
</tr>
</tbody>
</table>

Now we are interested to orthotropic material. Thus, we employ a timber CTS (Compact Tension Specimen) of side of 50 mm. The crack length is 25 mm. The external load is an unitary loading applied to a steel contour as depicted in Figure 1c. The points $A_i$ and $B_i$, $i \in \{1, \ldots, 7\}$ represent the location of holes where unspecified forces can be applied with angle $\beta$ directed according to the crack in trigonometrical direction.
Unfortunately, it is difficult to find an analytic solution for this problem. Thus, the accuracy of the finite elements implementation is evaluated through the study of the convergence of the numerical results with respect to the number of crowns around the crack tip taken in the calculation of the crack driving forces. The number of crowns represents the surface of the integration ring used in the definition of the $A$-integral. We note that, the numerical analysis is performed under plan stress conditions and based on the finite elements mesh depicted in Figure 1c. The timber CTS specimen is made on pine spruce having the following elastic mechanical properties: longitudinal modulus $E_1 = 1500 \text{ MPa}$, transverse modulus $E_2 = 500 \text{ MPa}$, shear modulus $G_{12} = 700 \text{ MPa}$, and Poisson’s coefficient $\nu_{12} = 0.5$.

Figures 1a and 1b illustrate the evolution of stress intensity factors $K_I$ and $K_{II}$ with respect to the number of crowns and for different loading orientations $\beta$, respectively. As can be seen the convergence of the quantities $K_I$ and $K_{II}$ is well achieved with increasing the crown sizes. Good accuracy is observed for all loading orientations when the integration path is greater than the third crown size. Hence, the independence of the $A$-integral to the integration path is verified. We have also shown that the singularity of the mechanical fields induces inaccuracy on the numerical results. This disturbance is clearly observed when the integration process is based on the first crown, the nearest one to the crack tip. In this paper, all the analysis are achieved for elastic materials. In the coming work, viscoelastic effects will be taken into account in $A$-integral and coupled to moisture variation in order to investigate the mechano-sorptive law of wood under fracture process.

![Figure 1. Path independence verification of the stress intensity factors (a) $K_I$, (b) $K_{II}$, CTS specimen (c).](image)

REFERENCES


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Stochastic analysis of mixed mode fracture in timber material using polynomial chaos expansion

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Keywords: fracture, mixed mode, material uncertainty, stochastic analysis, polynomial chaos

Modelling the mechanical behaviour of the timber material has been among the most interesting topics of research in the last decades. Indeed, timber is widely used as building material, especially for its good mechanical properties. Due to its natural origin, timber elements exhibit micro-cracks, which can propagate by fatigue, accidental or creep loads during the structure’s lifetime. Hence, understanding the real physics related to the crack growth process is an important issue when analysing the timber structural integrity. Unfortunately, this is not a trivial task when dealing with orthotropic material such as timber. In addition, mixed mode crack growth is usually observed in timber elements where initial cracks have random shape and orientation. The crack growth initiation and the crack propagation is driven by the energy release rate by taking into account a separation between open and shear modes. In this context, the M-integral initially proposed [1] for isotropic materials and adapted later for orthotropic material [2] is a good alternative. In this paper, the mathematical formulation of the M-integral have been improved in order to take into account the effect mechanical load applied on the crack tips which can be result from fluid action or contact between the crack lips during the crack growth process. Let be a cracked body and is continuously derivable vector field defined around the crack tip, the following expression is thus obtained:

\[ M = \int_0^1 \left[ \frac{1}{2} \sigma_{ij}^{ij} u_i - \sigma_{ij}^p v_{ij} \theta_{ij} dS + \int_1^\infty \frac{1}{2} F_i v_{ij} \theta_{ij} dx \right] \]  

(1)

The first integral is the classical term of the M-integral which represent the effect of mechanical loads applied far from the crack, where and are stress tensor components deduced from the real displacement field and the virtual displacement field, respectively. The second integral represent the effect mechanical load applied perpendicularly to the crack lips . Note that, equation (1) represent the energy release rate. The mixed mode separation can be obtained by performing two distinct computations of the real stress intensity factors and for particular values of virtual stress intensity factors and such as:

\[ K_i^O = \frac{M(K_i^O = 1, K_j^S = 0)}{C_i}, \quad K_i^S = \frac{M(K_i^S = 0, K_j^O = 1)}{C_i} \]  

(2)

where and denote the elastic ampliances in opening and shear modes, respectively.

Due to its natural origin timber is a heterogeneous material and its mechanical properties exhibit a large dispersion. Hence, it seems to be essential to evaluate its effect on the variability of the fracture parameters. In this context, stochastic approaches have been proposed in the literature. The main objective of these methods is to propagate uncertainties through a mechanical model. Experience has shown, that the variation of a random parameter can be efficiently characterized by evaluating the statistical moments and the probability density function. Unfortunately, this is not a trivial task when dealing with orthotropic material such as timber. In addition, mixed mode crack growth is usually observed in timber elements where initial cracks have random shape and orientation. The crack growth initiation and the crack propagation is driven by the energy release rate by taking into account a separation between open and shear modes. In this context, the M-integral initially proposed [1] for isotropic materials and adapted later for orthotropic material [2] is a good alternative. In this paper, the mathematical formulation of the M-integral have been improved in order to take into account the effect mechanical load applied on the crack tips which can be result from fluid action or contact between the crack lips during the crack growth process. Let be a cracked body and be a continuously derivable vector field defined around the crack tip, the following expression is thus obtained:

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(1)

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\[ \mathbf{y} = f(\mathbf{X}) = f \circ \mathcal{T}(\mathbf{U}) \approx \sum_{k=0}^{p-1} \mathcal{a}_k \mathbf{\Psi}_k(\mathbf{U}) \]  

(3)

where \( \mathcal{T} \) denotes the probabilistic transformation, \{\( \mathbf{\Psi}_k(\mathbf{U}) \)\}_{k=0}^{p-1} \) are multivariate Hermite polynomials which form an orthonormal basis with respect to Gaussian probability density, and \( \{ a_k \}_{k=0}^{p-1} \) are unknown real coefficients of the polynomial chaos expansion defined as N-dimensional integrals. Once the polynomial chaos expansion is constructed, the statistical
moments of the random variable \( Y \) can be analytically obtained. The first two statistical moments, namely the mean \( \mu_{Y,Z} \) and the standard deviation \( \sigma_{Y,Z} \) respectively, are computed by:

\[
\mu_{Y,Z} = E \left[ \sum_{|\alpha| = 0}^{p} a_{\alpha} \psi_{\alpha}(U) \right] = \bar{a}_{0} \quad \sigma_{Y,Z}^2 = V \left[ \sum_{|\alpha| = 0}^{p} a_{\alpha} \psi_{\alpha}(U) \right] = \sum_{|\alpha| = 1}^{p} \bar{a}_{\alpha}^2 \quad (4)
\]

where \( E[] \) and \( V[] \) Denotes respectively the expectation and the variance.

Consider the timber CTS specimen of side 50 mm and containing a crack of length 25 mm as depicted in figure 1. The CTS specimen is subject to an external load \( F = 50 N \) applied through the steel arms with angle \( \beta = 45^\circ \) directed according to the crack in trigonometrically direction. In this study the uncertain parameters are longitudinal modulus \( E_L \), transverse modulus \( E_T \), shear modulus \( G_{12} \) and Poisson’s coefficient \( \nu_{12} \). These parameters are modeled by independent lognormal random variables, with the statistical characteristics (the mean value \( \mu \) and the coefficient of variation \( COV \)) given in Table 1.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Distribution</th>
<th>( \mu )</th>
<th>COV</th>
</tr>
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<tr>
<td>( E_L )</td>
<td>lognormal</td>
<td>15 GPa</td>
<td>20%</td>
</tr>
<tr>
<td>( E_T )</td>
<td>lognormal</td>
<td>600 MPa</td>
<td>15%</td>
</tr>
<tr>
<td>( G_{12} )</td>
<td>lognormal</td>
<td>700 MPa</td>
<td>15%</td>
</tr>
<tr>
<td>( \nu_{12} )</td>
<td>lognormal</td>
<td>0.5</td>
<td>8%</td>
</tr>
</tbody>
</table>

The statistical moments of the crack driving forces in mixed mode \( G_I \) and \( G_{II} \) are estimated by the stochastic approach presented above. We use a second order polynomial chaos expansion (\( p = 2 \)), and the unknown coefficients are computed by Gauss-Hermite cubature rule based on 81 finite elements model calls. The numerical results are given in table 2. As can be seen, the effect of the uncertainty of the material properties on the variability of energy release rates \( G_I \) and \( G_{II} \) is significant since the coefficients exceeds 12%. The mean and standard deviation, give an idea about the magnitude of the range of the variability of fracture parameters. This new information is very useful for the designer to avoid the structural failure.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>( \mu_{Y,I} )</th>
<th>( \sigma_{Y,I} )</th>
<th>COV</th>
</tr>
</thead>
<tbody>
<tr>
<td>( G_I )</td>
<td>0.1598</td>
<td>0.0203</td>
<td>12.7%</td>
</tr>
<tr>
<td>( G_{II} )</td>
<td>0.0133</td>
<td>0.0016</td>
<td>12.03%</td>
</tr>
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ACKNOWLEDGMENTS
The authors wish to strongly acknowledge the National Agency of Research (ANR) for its financial support of this work through the project CLIMBOIS N° ANR-13-JS09-0003-01 labeled by ViaMeca.
Experimental evidence of water diffusion gradient in wood using the grid method

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Keywords: early wood, grid method, late wood, strain, water diffusion

At a time when the scientific community seeks lasting solutions to regulate environmental problems, wood remains an alternative. Indeed, trees can fix certain greenhouse gas emission such as CO₂ and carbon wood material balance shows that this latter slightly impacts environment. However, this solution can be discussed due to wooden structures and their maintenance in the short and long term. In fact, the strain is increased by the effect of moisture content variation, and is reinforced by the viscoelastic behavior and the anisotropic characters of this material [1]. This work aims to assess the heterogeneity of timber during humidification by the use of the grid method [2].

The goal is to study the phenomenon of water diffusion and its effect on the deformation of wood sample. For that purpose, a suitable experimental device is designed. It offers the possibility to impose a direct contact of one side (tangential direction) of the material with water. The wood sample used belongs to the essence of pine of Landes. Its dimensions are 49 * 46 * 8 mm³. The experimental device consists of a plastic cube on which a rectangular opening is cut. The sample is bonded on this hole through a silicone mastic ensuring sealing around the perimeter. The opposite front of the surface that is in contact with the water surface is equipped with a grid. Grid images are grabbed at different times by a CCD camera to monitor the diffusion phenomenon of the material under adsorption. Strain maps during the process of water diffusion are deduced from the grid images using an in-house dedicated software. Figures 1(a), 1(b) and 1(c) show the front side of the sample on which the grid is bonded as well as the strain map 12 and 177 hours after the beginning of the experiment respectively.

![Grid Image](image)

Figure 1. Pine of Landes sample: (a) - scan of the front in contact with the grid. (b) - strain map after 12 hours exposure. (c) - strain map after 177 hours exposure

As shown in Figure 1(b), after 12 hours of exposure, the face in contact with the grid still is in compression, but tension progressively settles as water diffuses into the material (c). Indeed, initially, when the side in contact with the water reaches a more pronounced hydrous state, it tends to expand there by opposing the compression. The latter will be put in tension when the diffusion front advance much more pronounced in the material. Deformation gradients clearly appear between the annual growth rings, highlighting the different diffusion properties at the scale of a growth ring thus between the early wood and the late wood.

The results are also used to highlight the changes in moisture diffusivity material during the test. Indeed, when considering (for example the step during which the contact face is affected by the tension) the kinetic deformation maps obtained from images taken during two successive time intervals, we note that this one decreases gradually as the exposure lasts. These results indirectly realize that under adsorption, with imposed water diffusion conditions, the diffusion coefficient decreases along the duration of exposure. This result is quite in perfect agreement with what happens when subjecting the wood material to the adsorption phenomenon. This is because the intrinsic water status of the material is close to saturation, and it is quite obvious that in the vicinity of the latter the degree of freedom, which had water to spread in the wood, diminishes gradually.

To illustrate this, Figure 2 presents the deformation maps for images taken respectively after t₁ = 177 hours (map N° 21), t₂ = 200 hours (map N° 22) and t₃ = 600 hours (Map N° 24) of exposure. A particular point (X = 600 pixels, Y = 400 pixels)
corresponding to spring wood is studied. Between times $t_1$ and $t_2$, the strain at this point evolves from 1.431 % to 1.488 %; therefore increases by 0.057 % during 23 hours. Let us consider now the deformation at the same point between $t_2$ and $t_3$: the deformation varies from 1.488 % to 1.672 %; thus increases by 0.184 % for a period of 400 hours (Figure 2). It is important to note that if we had the same kinetics of diffusion between $t_1$ and $t_2$ and between $t_2$ and $t_3$, it would have taken just about 75 hours to reach the same level of deformation instead of 400 hours. This phenomenon confirms that the diffusivity has decreased significantly.

![Figure 2. Strain $\varepsilon_{RR}$ for images at time: $t_1=177$ hours (map N°21); $t_2=200$ hours (map N°22); $t_3=600$ hours (map N°24)]](image)

REFERENCES
Evaluation of roughness and hardness of heat treated wood species

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Keywords: thermo-hydro-mechanical processing, oak, pine, poplar

One of the most important positive effects of heat treatment of wood is to enhance resistance to biodegradation [1] and improve overall dimensional stability [2] in addition to discoloration having attractive dark colour [3]. High temperatures and long span time of heat exposure decrease most of the mechanical properties of wood [4], but provide enhanced surface quality [5]. The objective of this study was to evaluate the effect of heat treatment on surface roughness and hardness of three wood species, namely red oak (\textit{Quercus falcate} Michx.), Southern pine (\textit{Pinus taeda} L.) and yellow poplar (\textit{Liriodendron tulipifera}).

It is expected that heat treatment would enhance properties of these species so that they can be used more effectively and efficiently to manufacture value-added products.

Twenty five defect free samples with dimensions of 55 mm by 38 mm by 19 mm were prepared from each wood species. Five groups each of five specimens were tested for the experiments. One group of non treated samples was kept as control samples while the others were exposed to heat treatment of two temperatures, 120°C and 190°C for 3 and 6 h in a laboratory oven. Surface roughness and hardness were measured before and after the heat treatment. Average roughness (\textit{Ra}) was measured by using a portable profilometer device of Hommel T-500 type equipped with a TK-300 skid diamond stylus with a 5 microns radius and 90° of tip angle. Six measurements were taken across the grain at a constant speed of 1 mm/s over a 15 mm tracing length. A Comten Universal Testing Machine was used to measure the hardness by embedding a steel sphere of 11.2 mm diameter into the sample perpendicular to the grain orientation. Four measurements recorded in kg were taken from each sample to evaluate the Janka hardness.

As expected and also found in literature the colour of all specimens became darker as the temperature and exposure time increased. Changes and degradation of hemicelluloses, lignin and some extractive compounds were found responsible of wood discolorations under heat treatment [3].

The most significant reduction considering both dependent variables under study has been determined for red oak samples. Figure 1 shows roughness and Janka hardness results before and after the heat treatment for red oak specimens. Hardness values for red oak samples exposed to a temperature of 190°C for 6 h were 41.7% lower than that recorded before the heat treatment, while the average roughness was reduced with 5.8% under the same heat treatment. Less significant differences were found between same type of Southern pine and yellow poplar specimens before and after the heat treatment in terms of their roughness and hardness values. Based on the statistical analysis hardness of the samples was mostly influenced by the heat temperature and the cumulative effect of treatment temperature and wood species on surface quality was also found significant (Sig≤0.05). As mentioned in literature heat treatment enhanced overall smoothness of the specimens. The decreasing trend in hardness was found similar to other studies in the literature [5].
This study investigated the effect of heat treatment on surface roughness and hardness of three different most commonly used wood species. It was found that surface quality of the samples was enhanced while hardness values resulted in decreasing trend with increase of temperature and time of heat treatment. The colour of all species became darker as the temperature and span time of heat treatment increased. The results of this work revealed that heat treatment could have potential value on these wood species to be used more effectively in furniture manufacturing.

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Influence of air parameters on drying time and energy consumption during thermo-hydro processing of wood

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Keywords: drying kiln, air parameters, energy consumption, drying time, response surface modeling methodology

The work aims to use response surface modeling methodology in order to determine the influence of factors interactions (temperature, relative humidity and velocity of air) on time and energy consumption during thermo-hydro processing of wood [1]. Our analysis was focused on wood drying process, which was simulated in an experimental kiln. The simulation was performed using TORKISM software [2].

We designed 64 drying experiments by combining three variable factors (parameters) of the drying schedule: velocity, dry-bulb temperature and relative humidity (three-factor experiments). The responses were approximated by a quadratic polynomial model. The regression analysis was applied to estimate the regression coefficients in a model that relates the factors \(x_1, x_2, x_3\), their interaction \(x_1x_2, x_1x_3\) and \(x_2x_3\) and quadratic terms \(x_1^2, x_2^2, x_3^2\) to a response \(y\). The models for the two responses (time and energy consumption) were calculated individually by using the software MODDE 9.1.1. [3], which was also employed for construction and analysis of experimental designs and response surface plotting. Quadratic and interaction terms that were not significantly different from zero, with 95% confidence, were excluded from the models.

The multivariate analysis showed that temperature and relative humidity were the two dominating factors. Drying time decreased with increasing temperature and decreasing relative humidity (Fig. 1). The energy consumption decreased with increasing temperature and relative humidity (Fig. 2). Again, the temperature and relative humidity had important influence. Of importance were also the quadratic terms and the factors interaction. Both models were characterized by a high accuracy, namely, \(R^2 = 0.99\) and \(Q^2 = 0.98\) in case of drying time and \(R^2 = 0.94\) and \(Q^2 = 0.90\) for energy consumption.

![Figure 1](https://example.com/fig1.png)

**Figure 1.** Response surface plot for drying time (boxed figures, hours) at different temperature and relative humidity values at 0.65m/s velocity (left) and regression coefficients with 95% confidence interval (right)

![Figure 2](https://example.com/fig2.png)

**Figure 2.** Response surface plot for energy consumption (boxed figures, kW/m³) at different temperature and relative humidity values at 0.65m/s velocity (left) and regression coefficients with 95% confidence interval (right)
The influence of the velocity was significant only for the drying time, even if its contribution was unimportant. A reason for the result could be the low velocity values, which were adopted according to the similarity conditions applied to the laboratory kiln [4], [5]. The test section of the laboratory kiln is larger than the air flow channel from the industrial kiln that was simulated in the wind tunnel.

In future research, the results obtained on the experimental drying kiln should be compared with those obtained on the actual kiln. The study can be extended furthermore with a multivariate analysis of the stress development inside the wood board during drying. In this way, all aspects of the thermo-hydro process efficiency would be covered.

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We acknowledge the National University Research Council for its support through the research project PNII-PCE, ID_851: The application of the irreversible processes thermodynamics method to the optimization of the capillary-porous materials drying process.
Determination of elastic behaviour of precious samples from large wooden structures of cultural heritage including screening potential in process treatment

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Keywords: multiscale, wood, mechanics, resonant ultrasound spectroscopy

In order to predict the mechanical behavior of large wooden structure of cultural heritage, it is necessary to test small samples all the way to the point of failure. Larger members can generally not be dismantled for testing. Mechanical tests usually induce some kind of irreversible deformation, i.e. damage, or even ultimate failure. Only small samples from non-critical parts of the structure can therefore generally be spared. Due to the preciousness of the material, necessitating small samples, some particular challenges arise: 1. The samples may not be representative. 2. Imprecision in machining induces non-linear load-displacement relations, e.g. non-parallel surfaces in compression of cubes. 3. Effects of barreling in compression. 4. Unreliable identification of shear moduli.

To obtain uniaxial stiffness parameters like Young’s and shear modulus, Poisson’s ratio static experiments have been performed. All of those experiments require strain measurement. It is common to use full field strain measurement techniques such as digital speckle photography (DSP). Measuring stiffness is a common procedure; however in our case we faced problems, such as inhomogeneous distribution of applied compression force. Poisson’s ratio can be measured along with the stiffness during the compression experiments and then another problem arises like barreling effect or the unequally distribution of expansion during the application of force. Shear experiments that are considered to be complex were performed using the method that has been proposed by Hassel et al [1]. Figure 1 is showing shear test device.

Ultrasonic resonance techniques for obtaining elastic stiffness can offer a good complement to static testing. The possibility of measuring mechanical properties without any direct contact and at relatively high speed is making this technique attractive. Longo et al [2] has proposed to measure all components of correspondent elastic tensor of wood material using a single specimen. The basic idea behind this method is to measure its own resonant frequencies of wooden specimen.

Micromechanical homogenization has been used to deal with the variability in density, anisotropy and material stabilization. Finite elements simulations allow reducing the unwanted effects of barreling and non-ideal cubic geometry of the specimens.

The proposed methodology is not only intended for archaeological wood but also for general applications, such as new treated wood materials and structures. E.g., screening of the effects of process parameters of hydro-thermal treated wood on stiffness properties, as a tool in process optimization efforts.

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Effect of hygrothermal treatments on the physical properties of wood

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Keywords: hygroscopicity, closed system, accelerated aging

For many applications, such as the conservation of wooden cultural heritage, wooden construction, understanding and modelling the behaviour of ancient wood is important. During the 1950s, Kohara has shown that aged wood had not the same physical, mechanical and chemical properties as recent wood [2]; the models currently developed are based on non-aged wood and do not fit for our studies. Thus, the aim of our study, to both implement our model and work on a mimic of ancient wood, is to reproduce the properties of this "aged" material by accelerating aging process of recent wood.

Thermal treatments are often used to improve wood durability thanks to a reduction of hygroscopicity. They are usually performed at high temperature (>180°C) and are accompanied by a degradation of post-linear properties: the material becomes more brittle, its transverse strength lowers. Matsuo and al. [3] have validated a procedure to reproduce the wood colour property of aged wood by thermal treatments. However Obataya [2] has shown that thermal treatments in oven-dry state fail to reproduce some important mechanical properties or behaviour such as anisotropy or viscoelasticity. Some recent results [1] suggest that mild temperature levels (<150°C) are preferable to reproduce the chemical changes observed in ancient wood.

The treatments were applied on poplar wood using a closed system. Almost 130 specimens were observed: 1/3 from heartwood and radially oriented (LxRxT = 3x40x5 mm), the rest tangentially oriented (LxRxT = 3x5x40 mm), 1/3 heartwood and 1/3 sapwood (Figure 1). Before treatments, mass and dimensions of specimens were measured at oven-dry state. Then, two types of pre-conditioning protocols were compared: a short ‘dry’ storage at low relative humidity (RH) in a closed box with silica gel, and few days ‘wet’ storage at a RH approaching the RH applied later. The samples were heated 24 hours at 120°C. The same amount of water was introduced inside the reactor to reach the desired RH: 0%, 50%, 75% and 100% for both pre-conditionings. Because of the moisture initially present in the specimen, the applied conditions were respectively: 8%, 62%, 79%, 89% RH for the dry and 19%, 68%, 82%, 91% RH for the wet pre-conditioning. These amounts were evaluated during the test using the pressure measured in the device. Mass and dimensions of samples were measured at different steps of sorption isotherms to evaluate the effect of both pre-conditioning and treatment on hygroscopicity and swelling/shrinkage rate. The results show some physical modifications due to these mild hygro-thermal treatments: on sorption isotherm, points at high RH present a reduced hygroscopicity and a reduced swelling/shrinkage rate, whatever the pre-conditioning protocol (Figure 2); nevertheless the introduction of moisture in the sample has an effect on this phenomenon (Figure 3). Figure 4 shows the equilibrium moisture content (EMC) and weight loss (WL) calculated based on the weight at 60% RH, 25°C:

\[
WL = \frac{M_{\text{before treatment at 60\% RH}} - M_{\text{after treatment at 60\% RH}}}{M_{\text{before treatment at 60\% RH}}}
\]

\[
EMC = \frac{M_{\text{after treatment at 60\% RH}} - M_{\text{oven-dry after treatment}}}{M_{\text{oven-dry after treatment}}}
\]

Based on these results, the range of hydro-thermal treatments able to mimic aged wood can be discussed.

Figure 1. Sample position and treatments
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Three-dimensional finite element modelling of heat transfer for linear friction welding of Scots pine

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Keywords: FEM, thermo-hydro-mechanical wood processing

The frictional welding technique has been shown to be viable for the fabrication of high quality wood joints without any adhesive. Mechanically induced vibrations can also be used to increase surface hardness and improve performance in the presence of polymerizing unsaturated oils [1]. New friction-based processing techniques are regularly introduced. In all these methods, heat and heat transfer are key parameters for the success of the process [2]. The even distribution and conduction of the generated heat over the processed area also play a major role in achieving a satisfactory result. However, more detailed consideration reveals that the thermal behaviour of the wood during mechanically induced wood fusion processes such as wood welding, surface modification etc. is still unknown. In order to understand the thermo-mechanical behaviour of welded joints, for example, efficient numerical models are necessary. Such models can also be used as a predictive tool to study the thermal behaviour of wood during other mechanically induced vibration processes. The only effort hitherto has been a 2D heat model [3], while Oudjene et al. (2010) [4] have proposed a 3D finite element model for wood-dowel rotation welding.

The objective of this work is to develop thermo-mechanical numerical models for use as predictive tools for the thermo-mechanical behaviour of wood. A finite element (FE) model and ABAQUS software were used to determine the 3D-heat flux generated from the friction on the wood pieces during linear friction welding (Figure 1).

![Figure 1. Three-dimensional model showing wood welding set up consisting of two samples with 25 mm thickness, 500 mm length and 48 mm width corresponding half of the specimen](image-url)

This model is based on the welding of small wood components of Scots pine (*Pinus sylvestris* L.) and has adequate accuracy for use in wood manufacture. The energy produced by the friction welding of the wood samples was determined by a thermocouple and used as input data in the model. The model is based on anisotropic elasticity and the thermal properties are modelled as isotropic. This numerical simulation gives information on the distribution of the temperature in the sample during the entire welding process. A good agreement between the simulation and experimental results shows the appropriateness of the model for the simulation of wooden structures (Figure 2).

The model can be adapted to simulate the temperature spread in the wood for different pressures and welding times. Once the FE models have been verified against experiments, parametric studies can be undertaken to improve and optimize heat generation during wood fusion welding. This model needs further development in order to include different wood species with different physical properties and dimensions.
Figure 2. Temperature progression at the welding interface for a welding time of 60 s. The average temperature calculated by model (Simulated) and average experimental temperature (Experiment) and its 95% confidence interval

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Solid wood bending – a stunning production system

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Keywords: thermo-hydro-mechanical wood processing

INTRODUCTION
Since Michael Thonet invented solid wood bending around 1840, it is considered to be the most important industrial production method for chairs.

At the beginning very successful but limited to chair production, it was the chair type No. 14, intended as a low cost consumer product, and its siblings in an expanding family of chairs and furniture. Later, solid wood bending conquered the life of people when keeping pace with the development of many products of daily life, serving perfectly well e.g. as frame for suitcases as well as for airplane fuselages or many other products that required light and solid frames or curved parts.

With polymers on the rise, solid wood bending lost most of these market segments to other production methods during the 1960s and 70s. The two products that remained in the markets are chairs and sleighs. Both these products are produced in large scale with a very lean set up of machines (one line for 5,000 chairs/week in one shift, 8 h). Today, steel frame chairs compete with bentwood, too, however this can be regarded as a fashion trend that come and go.

THE THONET PRODUCTION SYSTEM
Since the late 1980s, the Japanese car maker Toyota Motors is considered to play the leading role in terms of productivity and optimizing its production processes like no other. Still today, the Toyota Production System is regarded THE role model in the whole industrial/ mechanical production world.

The key to the success of Toyota is the seamless development of product and production process. It is surprising however, that what Michael Thonet did when developing his chair No. 14 and its production, is a consequent production system, although no books outside of the bentwood chair business mention his name. Both production systems from Toyota and Thonet are shown, the ideas behind and the analogies are explained.

THE THONET PROCESS
In this section, the solid wood bending process is explained, focussing on tensions and compressions in the wood-strap system and the transition between visco-elastic and visco-plastic behaviour during the stabilisation phase.

Last but not least, Thonet’s first plant that still produces chairs since 1861 in Bystřice pod Hostýnem (today TON, Czech Republic) is briefly introduced. There, experienced workers with a long term established production system make solid wood bent parts with a reject rate below 1 per thousand.

Apart from the fact that TON is using only beech this extremely low reject rate proves that cracks and damages during the solid wood bending process are first of all caused by deficits in knowledge about the process rather than the so called Thonet Method itself or quality variations in the used beech wood (Fagus sylvatica).

Looking at results from tests it can clearly be suggested that there are five (independent) reasons that lead to rejects in solid bent-wood production: insufficient or over- steaming (moisture content and temperature/ softening process); over- extension of the stress zone (e.g. clearance when using a strap/ bending process); releasing work- piece from compression (e.g. strap) while tensions are not yet settled (stabilizing process); over- heating from inside (stabilizing in a radio- frequency field); bending properties do not match to bending ratio (wood species).

Keeping these five criteria always in mind, the bending process produces high quality parts at any time [1].

REFERENCES
Investigations on the recovery behaviour of beech (*Fagus sylvatica*) wood densified transverse to the grain

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Keywords: tension test, digital image analysis, finite strains, strain distribution, stress-strain relation

INTRODUCTION

Thermo-hydro-mechanical processes can be applied to develop and manufacture novel wood products [0]. One of the first technical applications with huge economic success was the wood bending technique longitudinal to the grain developed by THONET. It is well-known that not all wood species are equally well-suited for this process. Beech (*Fagus sylvatica* L.) wood is, e.g., known to be suitable for bending longitudinal to the grain.

Regarding bending transverse to the grain as applied in the wood moulding process [0] developed at Technische Universität Dresden, Germany, the suitability of beech was not investigated, yet. In this process, heated wood is first densified transverse to the grain going along with folding of the cell walls. Beech has one of the highest densities of European woods, which limits the available densification strain $\varepsilon_{\text{imp}} = \Delta L / L$ ($\Delta L =$ deformation after densification; $L =$ initial length of sample in densification direction).

The recovery of the preliminary densification is then used for bend-forming. Therefore, the densified wood is cut and glued to boards where the densification direction is arranged in the board plane. After another heating and wetting cycle, the board can be moulded to curved profiles, such as hollow cylinders. Within this process, the imposed deformations are recovered - most at the convex surface of the board.

The maximum recovery strain is given in terms of the imposed deformation $\varepsilon_{\text{rec}} = (1 - \varepsilon_{\text{imp}})^{-1} - 1$. Larger tensile strains will lead inevitably to cracks in the wood and, thus, a deficient product. However, due to defects in the wood introduced during the densification or already existing before, the recovery potential might be considerably lower compared to the maximum value. In the following, experimental investigations regarding the recovery potential are presented.

EXPERIMENTAL INVESTIGATIONS

The primary interest of the investigations was whether the densification of the samples can be sufficiently recovered without preliminary failure due to damages in the cell structure of the wood introduced during the densification. Therefore, two batches of wood planks of a thickness of 8 cm and a width of 20 cm were densified by 30% and 50%, respectively, in a heating press at a temperature of about 130°C. Thus, the maximum recovery strain is 43% and 100%, respectively. Three different cases with densification in strictly radial and tangential grain direction as well as a diagonal orientation were tested. By cooling down to ambient temperature (< 60°C) the compressed shape is fixed.

Samples of about 5x2x2 cm³ (length x width x height) were cut subsequently from the compressed planks. At the ends of the quadratic cross sections, uncompressed beech blocks with the same size like the samples in the centre were clued but oriented.

![Figure 1](image_url)

**Figure 1.** Tension tests with finite strains; a) Sketch of the test setup, b) Specimen at the start of the test, c) Specimen at the end of the tests, d) Initiation of crack (90° view), e) Propagation of crack (90° view), f) Crack through entire width (90° view)
with the longitudinal grain direction parallel to the loading direction. In each of the blocks, one screw was drilled into the end grain side to transfer the load from the testing machine. The test setup is shown in Figure 1a. After storing the samples for about 10 minutes in a boiling water bath, the samples were placed in the testing machine. During the water bath, the samples were fixed in a steel clamp to avoid preliminary recovery. After fixing the specimens in the testing machine, the clamp was detached, which resulted in compressive stresses resulting from swelling and set recovery.

Each tension test was performed within about 90 seconds. During the test, the applied force was measured with a load cell and deformations of the specimen with two digital cameras. The cameras were placed with an inclination angle of 90° such that their image planes were parallel to two adjacent surfaces of the specimen. The measured surfaces were marked with twelve black points each for subsequent evaluation of the deformations and strains. In a post-processing, the images were evaluated with digital image analysis techniques regarding the moving of the gauge marks. This facilitated the determination of the mean strain between each two points and a correlation to the stress values calculated from the force \( F \) measured by the load cell.

Exemplary stress-strain relations are shown in Figure 2 for the specimen shown in Figures 1b-f. At the start of the test, the specimen is still in the state of compression due to the fixation during the preliminary storage in the hot water bath and the suppression of swelling. With increasing stretching of the specimen the stresses merge to the tensile region. The deformations are in general not uniform over the length of the specimen. This result in smaller failure strains of the entire specimen compared to the nominal maximum value, which is calculated from the imposed strain of the preliminary densification.

Failure occurs, in general, after a quite stable crack growth perpendicular to the loading direction, cf. Figures 1d-f. This is advantageous for practical application processes as this allows for stress redistribution without sudden failure of the work piece. However, the results reveal that the nominal maximum recovery strain is not reached in the entire sample, cf. Figure 2. Besides damages, whether existing before or introduced during the densification, also the cool-down of the specimens during the tests will have been responsible for preliminary failure due to the increasing stiffening of the material with decreasing temperature.

**CONCLUSIONS**

The determination of large deformations is not a common task within the field of wood research. In general, the failure strain under tensile loading is in the range of about 1-2%. The recovery deformation of wood densified previously transverse to the grain might be in the range of 100% where common deformation measurement devices like strain gauges or LVDT can, in general, not be applied. The presented results show the suitability of the developed optical non-contacting deformation measurement to investigate the stretching behaviour of wood densified transverse to the grain. The observed deformations have maximum values of about 40%. Despite the principle suitability, the test setup can be further improved, e.g. regarding reducing the cooling of the specimens during the test.

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Modification of surface veneer to reduce damage in laminated veneer products during manufacturing

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Keywords: densification, open system, THM processing, wood

Moulded laminated veneer products consist of veneers bonded together with adhesive into a predetermined shape. These products have many advantages, e.g. the lamination of the veneers makes it possible to create a great variety of strong and flexible shapes for furniture, interiors and construction purposes. 3D-forms, i.e. moulding in more than one direction, demand much greater skill in manufacturing because of the anisotropic structure of wood. A major problem in manufacture is the stretching and/or buckling of the veneer with the risk that the veneers may crack or damage if they are subjected to large deformations. The degree to which a veneer can be formed during moulding depends on e.g. the veneer thickness and preparation, species-related properties, product design and the mould itself.

It is possible to modify veneers to avoid undesired deformations or cracking of the veneer under moulding in four principally different ways. 1) Veneers can be formatted by removing unnecessary parts of the veneer in areas where there will be problems of stretching and/or buckling during moulding. 2) It is possible to bond a fabric, mesh, paper or other material on the back of the veneer to strengthen the veneer in the transverse direction. This method is often used for the visible outer veneers when they are thin or of a brittle type of species. 3) One can also use a so-called “3D-veneer” which is a veneer which is able to be formed extremely three-dimensionally. The most well-known 3D-veneer was developed by Reholz GmbH and has now been successfully introduced onto the market [1]. During the production of a 3D-veneer, narrow grooves spaced 0.1 to 1 mm apart and through the thickness of the veneer are cut in an ordinary veneer along the fibre direction. To keep the wooden strips together, lines of glue are spread on the rear of the veneer. 4) The veneers can be modified before moulding by thermo-hydro, thermo-hydro-mechanical or chemical action [2]. Traditionally, steam has been the most common way to soften veneers.

In thermo-hydro and thermo-hydro-mechanical processes, the glass transition temperatures (Tg) of wood and of the different amorphous components of wood are a key factor for a good result. The choice of the temperature of forming must be selected according to two criteria. Firstly, the minimum temperature under which the wood can be formed is usually considered to be at least 25°C higher than Tg of the lignin, i.e. approximately 110°C under moisture-saturated conditions and approximately 140°C at 80% ambient relative humidity, see e.g. [3]. Secondly, the maximum temperature, usually considered to be 200°C when air is saturated, limits thermal degradation of the wood components. The thermo-hydrous window for the process of forming wood is thus limited to temperatures and relative humidity varying respectively from 110 to 140°C and from 80 to 100%. Under these conditions, lignin, hemicelluloses and the semi-crystalline cellulose are relatively mobile.

The aim of this study was to compare the advantages and disadvantages of different modification methods for increasing the formability during the moulding of laminated veneer products and avoiding cracks in the surface veneers. Chemical treatments to plasticize veneers were not however considered in this study. Three different modifications (Table 1) have been performed to study how different parameters affect damage during moulding: 1) surface veneer bonded with paper with hot melt adhesive (HMA), 2) densification of the surface veneer, and 3) a reference group with untreated surface veneer. The densification of the veneer in group 2 was performed with a surface pressure of 5 MPa at 100°C after the veneers had been humidified to 20% moisture content. The selected methods were judged to be interesting from an industrial perspective.

The study was made on a seat shell. The orientation of the veneers according lengthwise (L) and transverse (T) directions was from front to back: L-T-L-L-L-L-L-L-T-L-L-L-L-L-L-T-L. The dimensions of the veneers were 1250x600 mm. The thickness was 1.5 mm for all veneers except for the L1-veneer that was about 0.5 mm thick, see Table 1. The species of all the veneers was beech, except for the L1-veneer that was oak. A urea formaldehyde (UF) adhesive system from Casco Adhesives Inc. was used. The adhesive spread was 164 g/m². The press time was 4 minutes, of which the first 2.5 minutes were with dielectric heating.

Table 1. Specifications for the respective modifications in the study

<table>
<thead>
<tr>
<th>Group No.</th>
<th>No. of samples</th>
<th>Group</th>
<th>Thickness of the L₁ veneer</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4</td>
<td>Veneer (sanded) and paper bonded with HMA</td>
<td>0.53</td>
</tr>
<tr>
<td>2</td>
<td>5</td>
<td>Densified surface veneer</td>
<td>0.41</td>
</tr>
<tr>
<td>3</td>
<td>4</td>
<td>Reference</td>
<td>0.53</td>
</tr>
</tbody>
</table>

The results showed that both densification and bonding with paper improved the formability and decreased the cracking. The next step in this study is to expose the products to variations in relative humidity and to study their behaviour.
REFERENCES
Effect of high temperature treatment on selected properties of beech, hornbeam and turkey oak wood

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Key words: thermal modification, shrinkage, equilibrium moisture content

Modification of wood using elevated temperatures is a widely used technique to enhance different properties of wood. The targeted upgrade is usually focusing on colour changes, higher biological durability and dimension stabilization induced by moisture content changes [1, 2].

In this study, three hardwood species were exposed to elevated temperatures in order to achieve dimensional stabilization. Changes in density, equilibrium moisture content were recorded and analysed.

Beech (Fagus sylvatica), Turkey oak (Quercus cerris) and Hornbeam (Carpinus betulus) boards with standing annual rings were dried in heat and vent semi-industrial dryer at max. 60°C. From the boards clear (free from any visible defects or irregularities) laths with dimensions of 300x140x25 mm (LxRxT) were cut and prepared for thermal modification. So called twin samples cut from the same board served as control.

The laths were treated at three different temperatures: 140, 160 and 180°C, and 2 different durations: 24 and 48 hours. It is known from literature that significant chemical changes (degradations) occur over 160 °C. One of the temperatures was set below this limit, while the other one exceeded it.

After the treatments the laths were conditioned at 20°C and 65% RH. Than small specimen for tests were prepared with size of 30x20x20 mm (LxRxT).

The dimensions and the weight of the specimen were measured at normal climate (20/65), in absolute dry condition and after full saturation of water. Data gained from this measurement allowed the calculation of the densities, the equilibrium moisture content and the dimension stabilisation.

In Figures 1-3 the maximal volumetric shrinkage values are depicted for all the three hardwoods. Compared to the control all the applied temperatures caused decrease in shrinking, thus a dimension stabilisation could be achieved. Longer duration (48h) caused (with some xceptions) more severe decrease in the shrinking compared to the shorter (24h) treatments. The hardwood species reacted differently to the elevated temperatures. Hornbeam, having the highest density showed significant dimension stabilisation already at the lowest temperature (140°C), while the other two (beech and turkey oak) needed higher temperatures to reach significant stabilisation values. Might the higher density contributed to faster heat conduction, which caused longer effective treatments times at the high temperature range, which caused more severe degradation in case of Hornbeam wood.

In Figure 4 the equilibrium moisture contents for control and treated Hornbeam wood are shown. EMC-changes are following the trends observed for shrinking values. The last was observed for the other two species too.

The conclusion can be drawn that density has a clear influence on the changes caused by thermal treatment. Higher densities resulted in higher changes. Important observation was made, that already at lower temperatures (at 140°C) some structural changes happen in wood, thus the EMCs are lower.

Figure 1. Max volumetric shrinking for Beech. (blue – 24h, purple 48h) density = 766 kg/m^3 (control)
Figure 2. Max volumetric shrinking for Turkey oak. (blue – 24h, purple 48h) density = 720 kg/m^3 (cont.)
REFERENCES

ACKNOWLEDGEMENT
This study was supported by the Environment conscious energy efficient building TAMOP-4.2.2.A–11/1/KONV-2012-0068 project sponsored by the EU and European Social Foundation.
Thermally modified wood for use in musical instruments – a review

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Keywords: thermally modified wood, dimensional stability, sound characteristics

The desire of musical instrument manufacturers to reduce negative characteristics of the raw material wood by special modification processes has existed for a century. An overview to different historical modification processes can be found in the paper by Martius [1]. Many of these processes were not further pursued. Today, however, the question arises whether some of these historical procedures, with consideration of new, further developed methods, can help to satisfy the increasing demand of high-quality instruments.

In some scientific papers, the effect of heat treatment upon the acoustic properties of wood are described. Gadd and D’Arcy (1986) state that there is no statistically significant effect upon characteristic values as a result of the heat treatment of spruce. Such treatments with temperatures between 110°C and 115°C result in slight changes in material properties only [2].

A study with several producers of guitars from Finland between 1998 and 2002 shows the use of thermally modified wood in musical instruments. This research results in the conclusion that when thermally treating tonewoods in a specific manner the changes in wood are the same as in naturally aged wood, whereas certain qualities of wood improve while the wood maintains its workability and strength. Further, the benefits of thermo-treatment seem more apparent among the higher quality wood species treated (results published in Thompson (2006) [3]).

The aim of work of Pfriem et al. (2005) and Wagenführ et al. (2006) was to compare thermally modified and unmodified twin samples of resonance spruce wood for sound boards. By a specific thermal treatment physical-technical characteristics of wood can be changed in such way that they correspond better to the requirements of wood used for sound boards than unmodified woods. More serious is the reduced moisture sorption, larger dimensional stability, better durability as well as acoustic characteristics. The effects of the relatively mild thermal treatment (180°C) can be compared with an “artificial aging”. Due to the thermal modification, objectively measurable parameters are improved which permit conclusions on improved sound characteristics [4, 5].

Pfriem (2006) and Pfriem et al. (2007) built and tested musical instruments made with thermally modified wood. As part of these work guitars, violins, and harmonicas (partly) made from thermally modified wood were prepared and tested. The analysis of possible application areas in musical instruments was not only in the foreground of this investigation. Rather, the physical description of the alteration of material behaviour due to the thermal modification with special reference to the general requirement of the instrument making was investigated [6, 7].

Mohebbey et al. (2007) investigated the influence of hydrothermal modification on musical properties of mulberry wood. This wood is used traditionally for instrument making in Iran. Due to the improved hydrophobicity and dimensional stability sound properties became better by hydrothermal modification [8].

For the use of thermally modified wood for statically and dynamically highly stressed components of musical instruments, appropriate investigations were carried out by Zauer and Pfriem (2010). The cross sections of thermally modified maple (Acer pseudoplatanus L.) were reinforced with carbon fibres reinforced epoxy resin in a single-stage process. The results clearly show that thermal treatment improves the sound quality of wood. Additionally, the reinforcement with carbon fibre increases the static and dynamical properties of thermally modified wood [9].

Apart from a change of the acoustically relevant characteristics, thermally modified wood exhibits advantages concerning the dimensional stability and sorption in regard to alternating climatic conditions. On the other hand, the modification of wood at high temperatures results in a reduction in strength and toughness. Certainly, a mild thermal treatment (modifying at 180°C in oxygen-poor atmosphere) leads to clear changes of the measurable acoustic characteristics, such as Young’s modulus, damping and sound velocity. In conclusion, mild thermally modified wood is a material with favourable characteristics for musical instrument making. A pre-selection of the raw materials is however absolutely necessary. Inappropriate materials can hardly be improved by thermal modification. Due to these investigations, traditional production technologies of musical instrument making can be transferred for use of thermally modified wood in musical instruments. For mild thermally modified wood three application areas are possible:

- Use in instruments, where high dimensional stability and low moisture sorption and reduced reaction to climatic variability are required.
- Use in musical instruments, where specific sound characteristics are required, which otherwise can only be attained by use of woods stored for a very long time. A reduction of storage time and in that way a significant saving on storage costs can be obtained. Thermally modified wood will not replace wood previously used, but can
through a process of modifying timber, resulting in a product that is similar in sound characteristics to naturally aged wood. This modified wood can be used for the restoration and reconstruction of old musical instruments. Additionally, the results obtained allow us to draw conclusions on the use of thermally modified timber for other applications. Because musical instruments represent the “upper class” in the wood processing industry, such statements regarding the workability and usability can be transferred to other areas of application.

REFERENCES
Compressive elasticity of compressed wood and its application to flexible wooden beam

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Keywords: cellular deformation, flexibility

INTRODUCTION
As wood is a cellular solid, it can be densified by large compression in its radial or tangential direction. The rigidity, strength and hardness of lightweight wood can be much improved by the densification. Therefore, many researchers have tried to fix the compressed shape of wood by hydro-thermal treatments, for the effective utilization of lightweight wood species.

On the other hand, wood becomes extraordinarily soft and elastic against compressive load in the radial or tangential direction, when its cell walls are previously buckled and folded. In this case, the folded cell wall acts as a “flat spring” rather than a rigid frame. In this paper, we exhibits the elastic nature of compressed wood under compression, and propose some new applications in which the compressed wood is used as a soft and elastic member.

MATERIALS AND METHODS
Cubic samples, 10-15 mm on each side, were made by balsa (Ochroma pyramidale, 171 kg/m³), falcata (Albizia chinensis, 253 kg/m³), paulownia (Paulownia tomentosa, 255 kg/m³), Japanese cedar (Cryptomeria japonica, 359 kg/m³), Japanese cypress (Chamaecyparis obtusa, 403 kg/m³), Japanese red pine (Pinus densiflora, 447 kg/m³), Sitka spruce (Picea sitchensis, 468 kg/m³), Japanese magnolia (Magnolia obovata, 482 kg/m³) and beech (Fagus crenata, 594 kg/m³). Those cubes were conditioned at 25°C and 60%RH and then compressed in their radial direction. The compression test was repeated for 4 times to observe the elastic recovery of compressive deformation.

RESULTS AND DISCUSSION
Figure 1 shows the stress-strain curves of uncompressed and compressed cedar wood samples. Uncompressed wood showed clear yield point at small strain level (0.01-0.03) due to the buckling of the cell walls, and the following stress plateau indicated the stepwise folding of the cell walls. Consequently 70% of initial deformation remained unrecovered after the removal of load. That is, wood is plastic under radial compression. On the other hand, when a wood was previously compressed by 60%, it deformed easily and smoothly without showing clear yield point, and its shape was recovered almost completely after the removal of load. Such an elastic behaviour is explained by the flexural deformation of flat spring i.e. folded wood cell wall.

Figure 2 shows the compressive Young’s modulus (E) and elastic strain (εe) of compressed cedar wood in the radial direction plotted against the initial compression ratio. By the previous compression above 10%, the E of wood dropped by a factor of 4-20, while its εe increased with increasing the initial compression ratio. In general, the elastic strain of uncompressed wood is only a few percent irrespective of loading direction. However, the elastic range of compressed wood is 28% at most. This fact suggests that the compressed wood is really soft and elastic against the radial compression.

The improved compressive elasticity of compressed wood allows wider application of wood. For example, an appropriate combination of compressed wood enables to realize an extremely tough wooden beam: when the compressed wood supports the compressive stress, the beam becomes softer (lower rigidity), more flexible (greater elastic deflection), and tougher (greater impact bending strength) than the solid wood in the L direction.
Figure 1. Compressive stress-strain curves of uncompressed (narrow line) and compressed (heavy line) cedar wood in the radial direction. Compressed wood was previously compressed by 60%, and compression test was repeated for 3 times.

Figure 2. Compressive Young’s modulus ($E$, filled plots) and elastic strain ($\varepsilon_e$, open plots) of compressed cedar wood as a function of initial compression ratio.
Accelerated artificial ageing of thermally treated ash wood

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Keywords: ash wood, heat treatment, flooring

Relevant properties of wood floorings are dimensional stability, hardness and strength, dependant on wood species, where also their workability, permeability and impregnability have the important role. Despite all the advantages, heat-treated timber has some undesirable properties comparing to the natural wood used for flooring, which depends also on the degree of thermal modification [3, 4, 6]. A lot of research on UV degradation of natural wood was done, using various surface coatings systems [1, 2, 5]. There is less information about the behaviour of thermally treated wood in outdoor exposure under different weather conditions using adequate environmental-friendly surface protection.

Nine flooring panels of dimensions 300×300 mm were tested, where construction consists of polymer network 4.5 mm thick, with spacers and mounting holes, all together 11 mm thick. Upper layer was made of industrially heat-treated ash wood (Fraxinus excelsior L.), having nominal thickness of 20 mm. A single panel was composed of 6 elements having nominal width of 46 mm and length 300 mm. The testing group consisted of nine panels in 3 subgroups: A – unprotected heat treated ash wood, B – heat-treated ash wood protected with a "Bona Deck Oil" and C – heat-treated ash wood, surface protected with "Saicos Wood Oil".

14-days laboratory conditioning (T = 20 ± 2°C, RH = 35 ± 5%) preceded the exposure the artificial accelerated aging (AA) by intense UV and IR radiation (P_{UV} = 50 W/m²), and the artificial rain – liquid water spraying in an automatic AA-device. The exposure has lasted a total of 5 weeks of one hundred 1hour AA-cycles. The single 1h cycle consisted of artificial rain (22 min), inaction phase (9 min), UV and IR radiation at 60°C (3 min), UV radiation (21 min), UV and IR radiation at 60°C (3 min), and inaction phase (2 min). Samples were inclined at the angle of 84° to the source of artificial rain, UV and IR radiation. Each 1-week interval included 2-days inactive conditioning phase after 100 AA-cycles (T = 20°C, RH = 50% to 70%), used for quality control. We have determined therein the mass of the panel (Δm = ± 0.01 g), the cross-section dimensions of individual elements (Δx = ± 0.01 mm), surface visual changes and colour of the individual elements according to standardized CIE/L*a*b* system.

The tested experimental values (mass change, dimension change and total colour change (ΔE*) followed the First-Order-System response, the greatest at the beginning of the AA-process, continued by exponentially time-dependent reduced rate to reach their final change. The fastest response was noticed at mass change, slightly slower were dimension change, whereas total colour change had the slowest response.

The final mass increase of 9.2% was noticed at the control samples, and was significantly reduced using oil surface treatment, by -34% at C-samples, and even by -54% at B-samples. This caused also the highest increase of final cross-section dimensions of control samples (A), namely +0.85%, and was significantly 49% lower at C-samples, and even 71% lower at B-samples. The effectiveness of surface protection to reduce the dimension changes was especially proved at ends of tested flooring elements. The successful sealing effect of oil-treatment was in this case noticed at B-samples, where change of elements end-dimension was reduced even by 93%, and only by 43% at C-samples.

The total colour change (ΔE*) at the end of the AA-process was similar at all tested groups, ranging from 8.8 (A-samples) to 10.7 (B- and C-samples). The highest response was always noticed at the L* colour axis, where all tested samples were finally significantly lighter. The b* colour parameter was also significantly changed during AA-process at oil treated samples, whereas a* colour parameter was changed alike at non-treated samples. Visual surface structural changes are observed at A-samples after 100 cycles of AA-process, after 200 cycles at the C-samples, and after 300 cycles at the B-samples. The end cracks have been visually noticed also at the elements, especially of group A and C, where the water is retained at these places for a long time.
REFERENCES
Comparative durability tests on TMT Beech – preliminary results

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Keywords: natural durability, thermally modified timber, outdoor exposure

INTRODUCTION
In this study the durability of thermally modified timber (TMT) was investigated. In the production of TMT natural timber is exposed for a specific period at a temperature of 160-250°C in a closed treatment chamber with inert atmospheric conditions. The heat treatment causes changes in the chemical, physical and biological properties of wood. Because of the improved material properties TMT is primarily used outdoors, e.g. as cladding or as garden furniture. In field test (DIN EN 252 1990) a variety of wood-dwelling and wood-destroying organisms occurs simultaneously, while in lab-test (EN DIN 113 1996) idealized test conditions and the use of monocultures may lead to a different classification of the durability [1].

EXPERIMENTAL
The samples were produced in nitrogen atmosphere in the pilot plant at University for Sustainable Development Eberswalde. Twelve samples were taken from each of the batches, as shown in Table 1.

Table 1. Experimental test set-up, treatment parameters, batch size

<table>
<thead>
<tr>
<th>Species</th>
<th>Format [mm]</th>
<th>Temperature [°C]</th>
<th>Duration [h]</th>
<th>Specimen/Batch</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beech</td>
<td>1450 x 75 x 25</td>
<td>180, 200, 220</td>
<td>2, 4, 6, 8, 10</td>
<td>40</td>
</tr>
</tbody>
</table>

Either the durability was determined directly by the median fungal mass loss after a 16-week incubation period or by the durability factor [1] (Table 2).

Table 2. Assessment scheme for determining the natural durability of wood against fungi

<table>
<thead>
<tr>
<th></th>
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<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>very durable</td>
<td>x ≤ 0.15</td>
<td>≤ 5</td>
<td>f &gt; 5</td>
</tr>
<tr>
<td>2</td>
<td>durable</td>
<td>x ≤ 0.15 ≥ 0.30</td>
<td>&gt; 5 to ≤ 10</td>
<td>3 ≤ f &lt; 5</td>
</tr>
<tr>
<td>3</td>
<td>moderate durable</td>
<td>x ≤ 0.30 ≥ 0.60</td>
<td>&gt; 10 to ≤ 15</td>
<td>2 ≤ f &lt; 3</td>
</tr>
<tr>
<td>4</td>
<td>slight durable</td>
<td>x ≤ 0.60 ≥ 0.90</td>
<td>&gt; 15 to ≤ 30</td>
<td>1.2 ≤ f &lt; 2</td>
</tr>
<tr>
<td>5</td>
<td>non-durable</td>
<td>x &gt; 0.90</td>
<td>&gt; 30</td>
<td>f ≤ 1.2</td>
</tr>
</tbody>
</table>


As test fungi Gloeophyllum trabeum (strain BAM 109), Oligoporus placenta (strain FPRL 280) and Trametes versicolor (strain CTB 863) were used. The Degradation rate, respectively the Durability factor (f) of the samples was calculated according to Eq.1 and 2.

Degradation rate [ ] = Degradation assessment value / Exposure time (Eq. 1)
Durability Factor (f) = Degradation rate Reference* / Degradation rate Test sample (Eq. 2)

RESULTS AND CONCLUSION
Despite barely visible damages on specimens Trametes versicolor had the highest mass loss compared to the other test fungi. The final assessment of the samples, which were exposed for 2 years was performed according to the prescribed period of 5 years. However, an analogue trend concerning all batches can be recognized in comparison of the two current test results. Within batches of lower treatment intensities the detectable infestation was higher and deviations were greater compared to increased intensities. The smaller the median weight loss (Figure 1) and the degradation rate (Figure 2) of the specimens, the lower and thus better the durability classification. Both tests indicated that the changes of beech wood samples need to be considered during the classification of durability classes.
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ACKNOWLEDGEMENTS
The authors would like to thank Dr. Christian Welzbacher (HPI, Hannover) and the Federal Ministry for Education and Research for funding (BMBF, FKZ: 1728X09).
Colour, MOE and MOR of silver birch and European aspen wood after compression and thermal modification in an industrial scale modification chamber

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Keywords: CIEL*a*b*, densification, heat treatment, modulus of elasticity, modulus of rupture

Combination of compression and thermal modification of wood is a potential innovation for improving the utilization rate of light weighted wood species in solid wood products [1]. The objective of the study was to determine the effect of compression and thermal modification on the colour, modulus of rupture modulus of elasticity of Silver birch (Betula pendula) and European aspen (Populus tremula) wood. The modifications were carried out in industrial sized pilot device capable of simultaneous drying, compression and heat treatment of sawn timber. Combinations of two different degrees of compression and two different temperatures of thermal modification were used. Likewise in ThermoWood® process the combined compression and thermal modification reduced the lightness of birch wood and increased its redness compared to a conventional kiln drying at low temperature. With both species discoloration was the strongest in the surface layer when the compression was started at green state, but at the depth of 3 mm or more, when the compression was started at 20% MC (Figure 1). Both modulus of rupture and modulus of elasticity were clearly affected by compression and thermal modification. The relative improvement in mechanical properties as a result of densification was larger for less dense aspen than for birch. To conclude, combined compression and thermal treatment shows high potential to improve the mechanical performance of Silver birch and European aspen wood.

REFERENCES

ACKNOWLEDGMENTS
Korwensuun Konetehdas Ltd. co-funded the study, and performed the modifications. The Vocational Training Association of Woodworking Men co-funded the study. Mr. Juha Metros and Mr. Hannu Koivunen collected the wood material and made the gravimetric, stereometric and visual analysis of boards. The authors wish to present sincere thanks to all these institutions and individuals.
Effect of heat treatment conditions on the pH value and buffer capacity of poplar wood

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Key words: wood modification, heat temperature, polysaccharide, hemicelluloses

Wood modification can be defined has a process that improves the properties of wood, producing a new material that when disposed at the end of the product life cycle doesn’t present an environmental hazard any greater than unmodified wood. The heat treatment of wood changes its chemical composition by degrading cell wall compounds and extractives. The chemical changes due to heating depend on the duration and temperature of treatment.

Poplar wood as a fast growth and important tree species has attracted a lot of attention recently in Iran and used mostly for furniture manufacture. The effect of heat-treatment conditions (final treatment temperature: 170, 185 and 200\degree C, initial moisture content of sample: 6, 9 and 12\%) on the pH value and buffer capacity of Poplar wood (Populous alba) was studied. The heat treatment was applied during 24 hours at 150\degree C and finally for 2 hours at final heat temperature. PH value increased up to 4.1 for samples treated at 185\degree C and 9\% initial moisture content (Figure 1).

![Figure 1. Effect of heat-temperature and initial moisture content on pH value](image)

Buffer capacity of samples (at initial moisture content of 6, 9 and 12\%) reaching upon values of 1, 1.2 and 1.6 and at treatment temperature of 170, 185 and 200\degree C these values were 1, 1.2 and 1 and showed significant differences with those of control samples (Figures 2). Overall results showed that the pH value as well as buffer capacity of heat treated samples increased.

The heat treatment of wood changes its chemical composition by degrading cell wall compounds and extractives. The hemicelluloses are the first structural compounds to be thermally affected. The degradation starts by deacetylation, and released acetic acid acts as a depolymerization catalyst that further increases polysaccharide decomposition. Acid catalysed degradation leads to the formation of formaldehyde, furfural and other aldehydes. According to the result the degree of change in pH value and buffer capacity of poplar wood during heat treatment depends on treatment conditions used. Therefore great care should be taken to select the treatment conditions.
Figure 2. Effect of heat temperature (upper) and initial moisture content (lower) on buffer capacity

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Effect of thermal modification of beech wood on its MOE and other mechanical properties

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Keywords: beech wood, ultrasound method, TMW

The study presents the results of testing thermally modified beech wood in the atmosphere of overheated steam. The wood was modified at the temperature of 190 °C, for 2, 6 and 10 hours in laboratory drier.

The following mechanical properties of the modified wood were tested: the compressive strength in radial and tangential direction, along fibres as well as hardness determined using Brinell’s method in tangential and radial directions and along fibres. Additionally, MOE was determined using four point bending test and the dynamic modulus of elasticity was determined using ultrasound non-destructive methods. It was found that most mechanical properties of thermally modified beech wood had been reduced, with one exception which was compressive strength along fibres. Strong correlation was observed between results of non-destructive and destructive test in bending.

Table 1. Properties of dry (MC=0%) beech wood thermally modified in laboratory drier

<table>
<thead>
<tr>
<th>Tested properties</th>
<th>Parameter of modification in the atmosphere of overheated steam</th>
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<tr>
<td></td>
<td>Control 190°C, 2 hours</td>
</tr>
<tr>
<td>Density [kg/m³] MC=0%</td>
<td>713</td>
</tr>
<tr>
<td>Compressive strength</td>
<td>26.0</td>
</tr>
<tr>
<td>radial [MPa]</td>
<td>19.4</td>
</tr>
<tr>
<td>tangential [MPa]</td>
<td>102.3</td>
</tr>
<tr>
<td>along fibres [MPa]</td>
<td></td>
</tr>
<tr>
<td>Hardness (Brinell)</td>
<td>57</td>
</tr>
<tr>
<td>radial [MPa]</td>
<td>47</td>
</tr>
<tr>
<td>tangential [MPa]</td>
<td>129</td>
</tr>
<tr>
<td>along fibres [MPa]</td>
<td></td>
</tr>
</tbody>
</table>

Figure 1. Static modulus of elasticity MOE EN (according PN EN 408) in relations with dynamic modulus of elasticity MOE dyn (determined with application of ultrasounds) for beech wood thermally modified at 190 °C, 2 hours in overheated steam
Figure 2. Static modulus of elasticity MOE EN (according PN EN 408) in relations with dynamic modulus of elasticity MOE dyn (determined with application of ultrasounds) for beech wood thermally modified at 190°C, 6 hours in overheated steam

Figure 3. Static modulus of elasticity MOE EN (according PN EN 408) in relations with dynamic modulus of elasticity MOE dyn (determined with application of ultrasounds) for beech wood thermally modified at 190°C, 10 hours in overheated steam

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Characterization of physical and chemical changes occurring during wood thermal degradation: Influence of treatment intensity, wood species and inert atmosphere

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Keywords: heat treatment conditions, kinetic reactions, mild pyrolysis, volatile products, wood properties

For several years, heat-treated wood is more and more used because of its non-biocide behaviour and its low environmental impact [1]. This sort of treatment is based on biopolymer chemical degradation by heat transfer [2, 3], without additional chemical products impregnation; re-utilization or recycling of these products are possible at the end of life of the material. This process improves the dimensional stability and the decay resistance of wood [4, 5]. It allows valorizing European wood species with low durability for an exterior utilization. These improvements come at the expense of wood mechanical properties of wood which weak [6]. Several types of heating processes exist currently differing mainly by the nature of the inert atmosphere [7] used during treatment: smoke, steam, vacuum, nitrogen, oil. The durability of this new wood material is strongly correlated to the degree of polymers thermal degradation depending on the conditions and the treatment intensity (time-temperature) [8].

A conducting heat treatment pilot using nitrogen or vacuum and allowing dynamic record of mass loss is used to understand better the atmosphere influence on the chemical degradation reactions. The results on industrial size wood samples show (Figure 1) that utilization of vacuum permit the elimination of volatile products formed during heat treatment and accumulated in oven, leading to lower extractives and Klason lignin contents due to the non recondensation of thermal degradation products. Elimination or limitation of the formation of recondensation products generates a lower mass loss for same treatment intensity and explains the lower polysaccharides degradation during a vacuum process than a nitrogen treatment [9].

These chemical modifications, according to the heat treatment conditions have been confirmed by the decrease of corresponding heat treated wood mechanical properties [10]. Figure 1 shows that vacuum heat treated beech is less mechanically affected than the nitrogen heat treated wood. Vacuum’s influence during wood heat treatment process has been confirmed on one other softwood species (silver fir). Finally, vacuum has also an important impact during the wood drying step, thereby considerably reducing the process duration.

Fine chemical analyses by TD-GC-MS system analysis and the study about thermal degradation reaction kinetics have allowed confirming the higher sensibility of hardwood than softwood to thermal degradation (Figure 2) [11].

Figure 1. Effect of inert atmosphere on chemical composition and mechanical properties of beech treated wood

In addition, these analyses have permitted the volatile thermal degradation products identification related to the treatment intensity (Figure 3). Subsequently, activation energies of the formation of these different volatile products, issued from wood thermal degradation, have shown a higher thermal sensibility of lignin than holocelluloses for temperatures below 230°C [12]. This trend is reversed at higher temperatures.
Thermal degradation of wood appears mainly be characterized by: hemicelluloses degradation + lignin depolymerisation and recondensation reactions (confirming by vacuum effect). Moreover, the degree of acetyl groups initially present in the wood could be an important parameter to determine the necessary treatment conditions for a fixed thermal degradation level (putative marker).

The result of this work is a significant increase in basic knowledge about the mechanisms of wood thermal degradation as well as their relations with the processing parameters.

Figure 2. Thermal Desorption of beech and silver fir at different temperature during 15 minutes

Figure 3. Identification of volatile compounds formed during beech wood thermal degradation at 230°C during 15 minutes
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The authors gratefully acknowledge the financial support of the CPER 2007-2013 “Structuration du Pôle de Compétitivité Fibres Grand’Est” and the French National Research Agency through the Laboratory of Excellence ARBRE.
Restorative environmental design: a design paradigm for thermally modified wood

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Keywords: naturalness, life-cycle assessment, environmental impact, sustainable building, biophilic design

The objective of this paper is to present the prospect of using thermally modified wood as an element restorative environmental design (RED) and to discuss the developments in thermal modification processes needed to achieve this goal. RED combines sustainable and biophilic design principles \cite{Der12, Kel08}. Sustainable design principles emphasize reducing the impact of building construction, location and utilization on the environment by minimizing the impacts of material choice, site choice, and energy use across all phases of the buildings lifetime \cite{Sin13}. Biophilic design is the incorporation of the principles of biophilia into building design \cite{Kel08, Has02}. These principles are built around the concept of an innate human attraction to life, and life-like processes \cite{Kel08, Has02}. RED, therefore, is a model to improve not only the impact buildings have on nature, but also the impact buildings and nature have on occupants.

In order to be successfully integrated into RED practices, thermally modified wood must have minimal environmental impacts (or positive environmental impacts) and be a recognizable element of nature. Therefore, Life Cycle Assessment (LCA) of thermal modification process and the resulting products must be completed to document the environmental impacts. Furthermore, modifications to wood should minimize any changes that reduce its apparent naturalness.

Natural materials can meet many of the specific criteria of both biophilic design and the restoration theories of environmental psychology. In both cases, patterns, colours and shapes are thought to provide occupants with a connection to nature, if those aspects of the environment reflect what is found in nature \cite{Kel08, Har042}. This connection is thought to have positive health benefits including reduced stress levels, faster recovery from stressful events and reduction of sick building syndrome symptoms \cite{Kel05, Wen06}. Therefore, the perceived naturalness of a material may make it more or less suitable for RED.

Wood satisfies both general tenets of the RED paradigm, sustainability and a connection to nature, and is therefore an ideal material for RED. Wood from healthy, well-managed forests is a renewable material, and provides carbon storage \cite{Has02}. Emphasizing the aspects of wood people recognize as natural, such as grain patterns and colour, in appearance applications provides building occupants with a connection to nature \cite{Mas04, Nyr10, Nyr101, Ric06, Nyr10}. By thermally treating wood to extend its service life, lower maintenance costs and improve durability the carbon storage period can be extended and the greenhouse gas impacts of maintenance reduced. Thermally modified wood, therefore, may be an exceptionally fitting material for RED. However, LCA should be used to ensure the sustainability goals of RED are achieved.

Finding a balance between enhanced sustainability and maintaining the naturalness of the product should be a key objective for thermal modifications of wood. Achieving this objective will allow thermally modified wood products to be used in this emerging design paradigm.
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ACKNOWLEDGMENTS

The authors would like to acknowledge the Slovenian Research Agency for financial support within the frame of the project Z4-5520 and the Italy-Slovenia Cross-border Cooperation Programme 2007-2013 for financial support within the project EnergyViLLab.
Rapid assessment by PTR-ToF-MS of the effect volatile compounds emission of different heat treatments on larch and spruce

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Keywords: thermo-vacuum; Superheated stream; VOCs; PTR-ToF-MS

Heat treatment of wood is used to improve its durability and dimensional stability [1], but it might affect the emission of volatile organic compounds (VOCs) [2], and consequently indoor air quality.

Indoor air quality has become a public health issue of increasing concern during the last decades [3], since frequently air pollutants belonging to VOCs are more concentrated indoors than outdoors [4].

Proton transfer reaction time-of-flight mass spectrometry (PTR-ToF-MS) is a rapid and high sensitivity method employed for the detection of VOCs [5]. The purpose of this study was to use the PTR-ToF-MS to compare the effect on VOCs emission from Norway spruce (Picea abies Karst.) and larch (Larix decidua Miller) treated with Thermovacuum technology at two different temperatures (220 and 165°C) in vacuum condition, with the same species treated with ThermoWood system at 220°C with superheated steam. Moreover, not treated samples were analysed as reference.

Thin slices of wood samples were collected after removing the external layer of the wood and about 0.3 g were placed into 20 ml glass vials. In order to standardize the analysis, the collected sample’s slices occupy the first 3 cm of the vials.

The headspace of the samples was analysed by direct injection on the PTR-ToF-MS 8000 (Ionicon Analytik GmbH, Innsbruck, Austria) in its standard configuration. The drift tube was conditioned at 600 V, with a pressure of 2.25 mB and a temperature of 110 °C. Samples were analysed automatically by a Gerstel autosampler connected to the PTR-ToF-MS inlet. The rapid PTR-ToF-MS analysis allowed the identification of more than 400 mass peaks. In the following, only the peaks with an average concentration higher than 0.5 ppbv (parts per billion by volume) were considered. ANOVA and post-hoc Tukey test were performed at 0.01 confidence level in order to evaluate the differences between treatments. Mass peaks tentatively identified as important VOCs, such as formaldehyde, methanol, acetaldehyde, acetone, acetic acid and monoterpenes had a higher concentration in the head-space of superheated steam treated samples compared to samples treated with thermovacuum technology and showing an increasing trend from not treated samples to those treated with Thermovacuum technology at 220°C. Almost all the significant mass peaks were higher in superheated steam treated samples.

In order to graphically visualize the ANOVA and Tukey results, principal components analysis (PCA) was performed using the significant mass peaks (Figure 1). As displayed in the figure, PC 1 and 2 explain together 75% of the total variance. PC 1 allowed for a good separation of the samples from superheated steam treated timber, and for the separation of the samples from wood treated under Thermovacuum technology against not treated samples. On the other hand, PC 2 allowed for the separation of Therмowood samples from both types of wood.

These results indicate a higher VOCs emission from superheated steam treated samples regarding thermowood and not treated samples. In addition, wood treated with the Thermovacuum technology seems to have a VOCs emission close to not treated samples, and therefore much lower than Thermowood samples.

PTR-ToF-MS allows the rapid and non-invasive measurement of VOCs emission also in this context providing valuable information on the effect of wood treatment. Being an online technique the possible application to the real time monitoring of the treatment process is envisaged.
Figure 1. Score plot of the PCA performed with significant mass peaks from ANOVA-Tukey analysis. NS: Norway spruce. L: Larch. NT: Not treated. TV-165: Thermovacuum 165 ºC. TV-220: Thermovacuum 220 ºC. SHS: Superheated steam

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Soft and white rot degradation resistance of thermo-hydro-mechanical processed hardwood evaluated by infrared spectroscopy

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Keywords: biodegradation, THM processing, infrared spectroscopy, wood

Previously was established that the Thermo-Hydro-Mechanical (THM) processing can improve the intrinsic properties of wood, by producing new materials with new characteristics and properties. A closed THM system has been used to densify lime (Tilia cordata Mill.) wood samples. During THM processing, lime wood undergoes chemical transformations depending on the processing parameters. The treatments were carried out under steam saturated atmosphere and under three different temperatures, and for each sample a post treatment has been carried out at different times and temperatures. At the end of the post-treatment, the samples were cooled down.

After application of different treatments (including densification or/and post-treatment at different temperatures), the samples densified and post-treated at 140°C for 210 min series were chosen for the biodegradation tests. The action of two different fungi (Chaetomium globosum a soft rot fungi and Phanerochaete crisosporium a white rot fungi) has been studied for different durations and the comparison with non-treated wood has been done.

The degree of decay was determined by weight loss and the changes in the chemical structure of the wood were investigated using FT-IR and 2D IR correlation spectroscopy.

In both cases the weight losses of the THM treated lime wood samples are lower when compared with the non-treated wood, proving the efficiency of the treatment.

The modifications induced in the wood structure due to the biodegradation process for both, non-treated and treated samples are presented and the comparison between them was made. The relative changes in intensities of spectral bands associated with carbohydrates and/or lignin were determined (Figure 2).
In the non-treated wood samples spectra the intensity bands assigned to different vibrations from cellulose and hemicelluloses show a decrease, especially the C=O stretching vibration of carboxyl and acetyl groups in hemicelluloses and C-O stretching vibrations [1]. At the same time, for the same bands from the THM treated samples was evidenced a decrease in intensity with increasing the exposure time, but to a lesser extent.

A more detailed interpretation of the analysed samples was made by using 2D correlation spectroscopy.

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Wood thermal-modification at Luleå University of Technology

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Keywords: wood Physics group, wood modification, thermal modification, oil impregnation

The Wood Physics group at Luleå University of Technology (LTU) has the vision of transforming Swedish solid wood into the material of choice for the renewable economy of the future. To realize that vision, the group believes, stability and durability of local softwood species must be enhanced at a reasonable cost without jeopardizing the natural beauty of this environmentally friendly material. One of the methods for enhancing stability and durability of solid wood is thermal modification, and LTU’s Wood Physics group has vast experience in developing and evaluating thermal modification processes. In simple words, thermal modification involves exposing the wood to relatively high temperatures, between 160°C and 240°C depending on the products and technologies used, and in the absence of oxygen to avoid degradation of the wood by combustion. It has been proved that these relatively high temperatures modify the chemical structure of the wood polymers (cellulose, hemicellulose and lignin), and wood becomes less prone to absorb moisture from the environment and more resistance to biological degradation.

There are a number of thermal modification methods that have been implemented in Europe at the commercial level, such as ThermoWood® and WTT thermo-treatment. ThermoWood® process is performed under normal atmospheric pressure with superheated steam containing as little oxygen as possible. The wood is first dried to almost 0% moisture content with steam temperatures up to 130°C, and then exposed to steam temperatures between 185°C to 212°C for a few hours. Afterward the vapor temperature is reduced to below 90°C to saturate the steam and allow the wood to regain moisture. The WTT thermo-treatment is performed with saturated steam under pressure up to 20 bars and temperatures between 160°C and 210°C, so the wood is not dried during the process. In the last years, LTU's Wood Physics group has performed several studies in collaboration with local wood producers interested in the evaluation and optimization of thermal modification processes.

To study thermal modification in laboratory, LTU's Wood Physics has built pilot scale kiln/thermal-modification unit that fits through the field of view of a CT-scanner unit specially adapted for wood material studies. This combined equipment allowed measuring wood density profiles through entire thermal modification process, thus providing valuable information about the effect of the process conditions in the material. More recently, LTU's Wood Physics group became interested in the process of thermal modification by boiling in linseed oil for 2 to 4 hours. This technology is available in the market, but the novelty at LTU was the implementation of an additional oil impregnation cooling phase in which the wood is submerged in cool oil after thermal modification. This creates a sudden contraction of the gases inside the wood, which in turn draws considerable amounts of oil into the wood. The authors believe that this combined thermal-modification/oil-impregnation treatment offers a simple but effective methodology for simultaneously: 1) enhance the stability and durability of solid wood, 2) impregnate the wood surfaces with oil for increasing the repellency to moisture.

This presentation includes an example of the combined thermal-modification/oil-impregnation treatment applied to common Swedish softwood and hardwood species. Both species were treated by using the WTT heat treatment technology and impregnated with different types of preservative oils. After impregnation, the samples were tested for water repellency, dimensional stability, and resistance to mould. Water repellency and dimensional stability were assessed for both liquid water and air relative humidity, and the resistance to oil leaching was determined by exposing the treated wood to cycles in which the samples absorbed water by immersion and then release the water under vacuum. As expected, the treatments showed a significant improvement in the water repellency and dimensional stability of the wood. Overall, untreated wood was more stable after thermal modification, and thermally modified wood was more stable after oil impregnation.

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The resistance to mould was evaluated by using an accelerated technique also developed by the Wood Physics group at Luleå University of Technology. The technique consists in placing the wood samples in the upper zone of a conditioning chamber in which there are other pieces of wood already infected by mould in the lower zone. Typically, the source of mould is pine sapwood infected with mould of aspergillus, rhizopus, penicillium genus along with other various species, and the test samples are exposed approximately 20 days to the infected environment. After incubation, the incidence of mould over the surfaces is graded in scale from 0 to 6 based on the visual assessment of two independent observers. The results of the study showed that some of the oil impregnation treatments did not significantly improved mould resistance, and it was still questionable whether the oil would not leach from the wood when the products are in service. Future research in wood modification would be certainly needed to find the right thermal-modification/oil-impregnation combination for the right application, as well as to realize the vision of transforming solid wood in the material of choice for the renewable economy of the future.
Self-bonding of veneers with heat and pressure
– a full scale test

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Keywords: beech, moulding, hot-pressing, THM processing

Making boards from veneers does not have to involve glue; high temperature and high pressure together can suffice to produce a self-laminated board with excellent mechanical properties. In earlier studies, small boards of beech veneers were shown to self-bond in a small laboratory press 1. In this study, the self-bonding capacity of full-size beech veneers of dimensions 1200x1200x2 mm were tested.

Two electrically heated plates were mounted in a conventional 100 ton hydraulic, see Figure 1. Each panel consisted of five peeled veneers overlaid in a parallel grain direction. The veneers were taken from the regular production and conditioned to an equilibrium moisture content of 7% before being subjected to a pressure of 5 MPa and a plate temperature of 220°C for 5 minutes. The evolution of temperature in two of the bond-lines was recorded by thermocouples, see Figure 1. A total of 10 panels were produced. After pressing, the boards were allowed to cool in a vertical position. Dry and wet shear strength tests were run to check the bonding quality.

The pressing procedure was a success and proved that bonding solely with heat and pressure is a method that can be applied to veneers with large surfaces areas. The temperature recordings as well as the shear strength tests showed that the heat was not evenly distributed within the plates. The samples therefore exhibited different bonding properties although they were taken from the same board. The heating devices should be improved to achieve a uniform heat distribution in the laminate during pressing. A well-defined cooling procedure should also be introduced to avoid boards warping after pressing.

Figure 1. Schematic representation of the press, including five veneers and thermocouples in two of the bond-lines: (a) press plate, (b) electrically heated plates, (c) heating elements, (d) veneers, (e) thermocouples

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Thanks are due to “Innovationsbron Västerbotten” and “Frans och Carl Kempes Minnestiftelse 1984” which supported the project.
Estimation of heat-treated beech wood properties by FT-NIR spectroscopy: effect of radial and cross sectional surface

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Keywords: thermal modification, near infrared spectroscopy, PLS regression

Near infrared spectroscopy can be used as an inexpensive and non-destructive method to determine the properties of heat-treated wood [1-4]. However, limited information is available concerning the effect of wood surface on the model performance to estimate properties of heat-treated wood. Hence, the aim of this study was to investigate the influence of the radial and cross sectional wood surface on the performance of the partial least squares regression to predict bending properties and colour change of heat-treated beech wood by FT-NIR spectroscopy.

After kiln drying (max temp. 50ºC), the beech boards with red heartwood were planed to a final thickness of about 2 cm. The samples (1 untreated + 3 for heat treatment) from the central part of each kiln dried board were cut into specimens that were used to determine the mechanical properties (modulus of rupture-MOR and modulus of elasticity-MOE) and colour change (ΔE). Specimens were divided into two groups: sapwood and red heartwood. Heat treatment was carried out in a laboratory chamber, where groups of specimens were exposed to temperatures of 170ºC, 190ºC or 210ºC for 4 hours. The untreated (84 from sapwood and 84 from red heart wood) and heat-treated (108 from sapwood and 108 from red heart wood) specimens were conditioned at 23ºC and relative humidity of 50% during eight weeks. The wood properties were determined after conditioning. MOR and MOE were determined by a three-point bending test. Colour coordinates were measured by colorimeter on cross sectional and radial surfaces of untreated and heat-treated samples. Measurements were done at four places and average values were used for further calculations. The coordinates L*, a* and b* measured before and after the treatment, were used to determine total color difference – ΔE. FT-NIR measurements were made on the cross sectional and radial surface. Spectra were collected before and after heat treatment with a Nicolet Nexus 670 FT-IR spectrometer (11000–3300 cm⁻¹). For each scanning point, 100 scans (4 cm⁻¹) were collected and averaged into a single spectrum. Prior to multivariate analysis, four spectra collected from both radial and cross sectional surface were averaged producing one spectrum per surface. PLS analysis was done by The Unscrambler® 9.7. (CAMO AS, Norway). PLS calibration was done with two subsets: calibration set (60 – untreated, 78 heat-treated) and validation set (24 – untreated, 30 – heat-treated) with a maximum 10 latent variables. Model efficiency was then tested by the validation set. This study tested two models: the HT-HT model (that was obtained based on the regression of FT-NIR spectra collected from the surface of heat-treated wood and the properties of heat-treated wood) and the NT-HT model (that was determined based on the regression of FT-NIR spectra collected from the surface of untreated wood and the properties of heat-treated wood). For comparison the models, RPD (ratio performance to deviation) was used. According to obtained results, the PLS-R model parameters of heat-treated cross sectional surface differ from the radial surface of beech samples (Table 1). For sapwood, the assessment of modulus of elasticity (MOE) yielded the best results with the spectra from the cross sectional surface of untreated wood (NT-HT model, RPD=1.82). The RPD values obtained for red heartwood were higher than for sapwood, and the best assessment of MOE can be obtained with the spectra recorded from the radial surface (HT-HT model). Generally for sapwood, a good assessment of modulus of rupture (MOR) was obtained by the FT-NIR spectra from the cross sectional surface (HT-HT model – Figure 1). This conclusion is also valid for red heartwood, with RPD values being greater in sapwood. The poorer results of the radial surface for MOR can be related to the influence of wide rays on the acquisition of the NIR spectra. The results of assessment of colour change (ΔE) with FT-NIR were better than those with MOE and MOR. Radial surface showed better results as compared to cross sectional surface in both sapwood and red heartwood. The assessment of colour change after heat treatment with spectra from untreated wood was inadequate, as is shown by the very low values of RPD.

Table 1. Values of RPD (and the coefficient of determination – Rp²) for the models of estimation of the properties of heat-treated beech wood by FT-NIR spectra

<table>
<thead>
<tr>
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<th>NT-HT</th>
<th>HT-HT</th>
<th>NT-HT</th>
</tr>
</thead>
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<tr>
<td></td>
<td>Sapwood – cross sectional</td>
<td>Sapwood – radial</td>
<td></td>
<td></td>
</tr>
<tr>
<td>MOE</td>
<td>1.34 (0.40)</td>
<td>1.82 (0.68)</td>
<td>1.62 (0.64)</td>
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<td>MOR</td>
<td>2.66 (0.85)</td>
<td>1.86 (0.70)</td>
<td>1.96 (0.76)</td>
<td>1.80 (0.68)</td>
</tr>
<tr>
<td>ΔE</td>
<td>3.50 (0.93)</td>
<td>1.17 (0.26)</td>
<td>3.78 (0.95)</td>
<td>1.12 (0.24)</td>
</tr>
<tr>
<td>Red heartwood – cross sectional</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Red heartwood – radial</td>
<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td>MOE</td>
<td>1.50</td>
<td>2.15</td>
<td>2.39</td>
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</tr>
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<td>(0.79)</td>
<td>(0.74)</td>
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<tr>
<td>MOR</td>
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<td>1.80</td>
<td>1.91</td>
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<tr>
<td></td>
<td>(0.72)</td>
<td>(0.66)</td>
<td>(0.65)</td>
<td>(0.54)</td>
</tr>
<tr>
<td>ΔE</td>
<td>3.56</td>
<td>1.18</td>
<td>4.13</td>
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</tr>
<tr>
<td></td>
<td>(0.91)</td>
<td>(0.29)</td>
<td>(0.96)</td>
<td>(0.29)</td>
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</table>

Figure 1. Predicted vs. measured values of MOR in sapwood by FT-NIR spectra collected from cross sectional surface of heat-treated beech wood

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Densified and thermally modified wood as outer layers in light-weight panels for furniture and joinery

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Keywords: thermo-hydro-mechanical processing, THM, shape stability, recovery

A modified softwood product would enable the use of softwood in areas where it today fails. Wood densification is a physical process based on compressing solid wood in the transverse direction to increase its density and thereby related properties. Densified wood that has undergone no further chemical or THM-based treatment has the capacity to recover its initial form under the influence of heat and/or moisture. This recovery is one of the major problems when densified wood used in products. Another problem is the great weight of the material.

The purpose of this study is to present a new light-weight panel with surfaces of densified and in some cases also thermally treated Scots pine, and to discuss the challenges and potential of this product.

In total, eight three-layered light-weight panels with dimensions of 400x200x36 mm, (L1xW1xT3) were prepared, Figure 1. The panels had four combinations of outer layers as shown in Table 1. As raw material for the outer layers, clear finger-jointed pieces of Scots pine measuring 45x95x600 mm (T, R and L directions respectively) and with a moisture content (MC) of 6 % were used. For the densification, the wood pieces were compressed inside a steel mould that restrained the wood in the tangential and compressed the pieces in the radial direction, see 1 for details. The compression was performed at 20°C to 50 % of the original size in the radial direction.

Table 1. Combinations of outer layers (T1 and T2 according to Figure 1) of the light-weight panels, TT - thermal treatment

<table>
<thead>
<tr>
<th>Test group No.</th>
<th>No. of panels</th>
<th>Outer layer, T1</th>
<th>Outer layer, T2</th>
<th>Notation</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2</td>
<td>Untreated</td>
<td>Untreated</td>
<td>Reference</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>Densified</td>
<td>Untreated</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>2</td>
<td>Densified +TT</td>
<td>Untreated</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>2</td>
<td>Densified +TT</td>
<td>Densified +TT</td>
<td></td>
</tr>
</tbody>
</table>

Figure 1. Principal sketch of the light-weight panel. T1 = 6 mm, T2 = 6 mm, T3 = 36 mm, T4 = 24 mm, L1 = 400 mm, W1 = 200 mm, L2 = 96 mm, W2 = 25 mm, W3 = 42 mm
The untreated and the compressed pieces were split in the radial direction to lamellae. Thermally treated pieces were produced from compressed lamellae in an open system under the conditions specified in Table 2. The lamellae were edge-glued to 6 mm thick panels for the outer layer of the light-weight panels. The light-weight panels described in Table 1 were thereafter produced with webs of untreated Scots pine forming the middle layer.

The light-weight panels were tested for shape stability in two different climates: 20°C/40% relative humidity (RH) and 20°C/85% RH, respectively.

Table 2. Process and material parameters for the thermal treatment (TT), MC – moisture content

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Time (min.)</th>
<th>Pressure (MPa)</th>
<th>MC before TT (%)</th>
<th>MC after TT (%)</th>
<th>Thickness before TT (mm)</th>
<th>Thickness after TT (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>160</td>
<td>180</td>
<td>1</td>
<td>8</td>
<td>1.5</td>
<td>18</td>
<td>17</td>
</tr>
</tbody>
</table>

The results show the well-known fact that symmetry in the design of the light-weight panel is important for shape stability, so that good shape stability was obtained for panels in groups 1 and 4. The shape stability is greater in the panels with the densified and thermally treated outer layers than in the panels with an outer layer of only densified wood.

Thermal treatment of the densified wood is shown to reduce the recovery. Whether or not it is necessary to completely eliminate the recovery of the densified wood when used as outer layer in these panels remains to be evaluated further. Such tests will be performed together with studies into how potential consumers experience the product.

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Densified wood as a resource for novel nail-like connectors

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Keywords: wooden nails, densification, mechanical properties

The hardwood connectors such as a nails, wedges, dowels and pegs have been used in traditional timber structures as reinforcement of carpentry joints or to fix wooden ceiling matts or floor boards. In the study reported, the possibility to rely on enhanced mechanical and technological properties of densified wood, for the production of wooden nails is discussed. The wood used for connectors were domestic wood species; European ash, European beech, Black locust and poplar. Wooden blocks were exposed to densification procedure with the purpose of increasing the material’s density, dimensional stability and possibly, improve durability and selected mechanical properties. The densification ratio varied between 50 and 67%. A dedicated research has been performed in order to determine the effect of densification on the compression behaviour of wood in the form of nails. Chemical/physical changes to wood occurring due to densification were also investigated.

The process of preparation of wooden nails included shaping the nail core (3x3mm²) and sharpening the tip (30°) in order to allow straightforward pushing of the nail in to joint, without pre-drilling. The progress of pushing force during insertion of the wooden nails into wood samples was also monitored and served for insertion process control.

A preliminary set of samples (as shown on Figure 1) was prepared and series of push-out tests have been carried out on timber-to-timber joints assembled with the densified nails.

![Figure 1. Configuration of densified wood nail-like joint as used for push-out test (dimensions in millimetres) Note: the joined wood made of Norway spruce (Picea abies)](image)

The specimens used in the monotone tests were pushed up until failure. The load–slip curves acquired in tests provide the ultimate load of the connection and are presented in Figure 2. In addition they give supplementary information regarding stiffness and ductility. The maximum load noticed in test varied between 300 and 832N what corresponded to slip range of 5 to 10mm.
CONCLUSIONS

1. A research report described here proposes utilization of densified wood for production of wooden connectors. The main goal was to improve original mechanical/physical properties of the material, to obtain connectors for specific uses in timber assemblies.

2. A relevant increase of mechanical properties, in terms of resistance to compression, was observed for all the tested species, especially for ash and beech samples.

3. The resulting enhanced mechanical properties allow insertion of densified wooden nails into timber members, without damaging the nail itself, with the occasional exception of head crushing.

4. In terms of load-carrying capacity of timber-to-timber joints, average values of about 550 MPa were reached using nails of beech densified at 60%. These values can be appropriate for assemblies of complementary systems in wooden structures, such as floor boards, ceiling mats, etc.

5. Even if preliminary results are encouraging, further research is required, in order to develop a standardized procedure for nail production/insertion and joint design.

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Characterization of thermally modified wood by oxygen bomb calorimetry

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Keywords: higher heating value, elemental composition, heat-treated wood, characterization

The objective of this study was to verify oxygen bomb calorimetry, for accurate higher heating value (HHV) measurements, as a potential characterization method for thermally modified wood. The choice for this technique was based on the known strong correlation of relative fungal degradation rates of thermally modified wood with its elemental composition [2, 3, 4] and the independent literature correlations between the elemental chemical composition of torrefied biomass with its higher heating value (HHV) [5]. A series of heat-treated Tunisian grown wood species, radiata Pine (Pinus radiata), Aleppo Pine (Pinus halepensis) and Zen Oak (Quercus canariensis) at 4 different vacuum heat treatment intensities (0, 8, 10 and 12% ML, thermal mass loss), described in [1], as well as a number of samples of Poplar (0, 5, 10 and 15% ML), Beech and Ash (6% ML) from heat-treatment in atmospheric nitrogen [2], were taken as the samples for the test.

The sample preparation is fast and simple, requiring 100 to 500 mg of sawn wood slices per calorimetric determination, of quite arbitrary shape and size. The HHV (specific heat) is calculated using the determined oven-dry weight of the sample prior to each calorimetric measurement. At least 3 replicate calorimetric measurements were taken from each sample type.

Figure 1, left, shows that the O/C ratio correlates well with the HHV, but dependent on the used heating method in atmospheric N₂ or vacuum. Untreated samples did not generally fit into either of these two correlations. These results may relate to a different extractive content in untreated, N₂ heated and vacuum heated specimens. The vacuum heated samples showed however an anomaly in the elemental composition when plotted in a van Krevelen diagram [4], with some indications of (unintentional) oxidative charring.

Surprisingly, all samples fitted well in one multivariate linear regression with elemental composition (Figure 1, right), which significantly differs from the known correlations for torrefaction, found in literature. This might be related to the relatively mild heat treatment of our samples in comparison to torrefied biomass.

In conclusion, oxygen bomb calorimetry could sensitively resolve the increasing treatment intensity in a series of samples obtained with the same heating technique, which is a very promising result. Further research is needed to systematically investigate the observed differences between samples from different heating methods.

Figure 1. Higher heating value (HHV) in MJ/kg as a function of the elemental O/C-ratio of the specimen (left). All samples fit well in a common multivariate linear regression between elemental composition and HHV (right). Increasing heat treatment severity monotonically decreases the O/C ratio and increases the HHV. Untreated specimens are indicated by solid symbols.

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Influence of temperature of thermal modification on compressive densification of spruce

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Keywords: thermally treated spruce, set recovery, bending, hardness

The thermal treatment of wood improves its durability, but decreases its density and mechanical properties [1]. In this work, mechanical compression was used to increase the surface density of previously thermally modified wood to enhance its mechanical properties for applications like flooring, where hardness is important. Spruce wood lamellae, thermally treated at 170, 190, 210 and 230°C were surface densified by compression at a temperature of 150°C and to three degrees of compression. The immediate springback after press opening set recovery after soaking/drying, hardness and bending strength were measured to determine the effect of thermal modification on compression.

The disadvantage of densification is the recovery of compressive deformation, which occurs immediately after opening the press is opened (springback) and after exposing wood to soaking/drying cycles (set recovery) [2]. The least immediate springback after press opening occurred in the control wood (20% for most compressed samples). Springback generally increased with the degree of thermal modification and reached 30% for wood thermally treated at 230°C (Figure 1).

The set recovery of compressive deformation after soaking in water for 24 h and drying at 103°C exhibited the opposite trend - it was found to decrease as the degree of thermal modification increased. Five cycles of soaking and drying was carried out, but most of the set recovery occurred in first soaking/drying cycle and did not change much with additional cycles.

The 3-point bending and Brinell hardness tests were used to determine the effect of the temperature of thermal treatment on the mechanical properties of compressed wood (Figure 2).

Figure 1. Immediate springback after opening the press (left) and set recovery of compressive deformation after 1st cycle of soaking in water for 24 h and drying at 103°C (right)

Figure 2. Brinell hardness (left) and bending strength (MOR) of samples (right) depending on the degree of thermal modification and compression degree
The surface densification increased hardness and bending strength in all cases. The highest increase in both mechanical properties was seen in the non-treated wood; tending to decrease with increasing thermal modification temperature. The MOR of compressed wood was larger than un-compressed wood and tended to decrease as the degree of thermal modification increased.

The results indicated that thermal treatment of wood before surface densification resulted in physical and chemical changes that influenced the compressive deformation and stress relaxation during the surface densification process. The equilibrium moisture content of thermally treated wood is known to decrease with increased temperature and/or time of thermal treatment. Furthermore, specimens that were thermally treated prior to surface densification had lower moisture content, which resulted in higher elastic deformation induced by the mechanical compression and bigger springback after press opening. Smaller set recovery after the water soaking/drying cycles indicated that the thermal treatment of wood before surface densification could reduce the set recovery of compressive deformation, however parameters of surface densification process would have to be adapted to thermally treated wood and/or specimens would need to be conditioned in environments leading to higher moisture content of wood before surface densification. In order to confirm this assumption more research examining process parameters in relation to properties of thermally treated wood has to be performed.

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Emissions from TMT products

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Keywords: gas chromatography, thermally modified timber, volatile organic compounds

INTRODUCTION
Wood emits volatile organic compounds (VOCs). The applicability of wood in the museum environment, e.g. as material for display cabinets, is almost entirely restricted to use due to the required controlled climate and air purity, which is very different from normal indoor air, e.g. in dwellings, offices, schools etc. A variety of test methods for determining formaldehyde (FA) emissions from wood and wood products have evolved over the time. As reference method the California Air Resources Board [3] and the chamber method according to DIN EN 717-1 [4] recommend large chamber tests. A large chamber test is expensive, time consuming and requires a large amount of samples. Therefore it is impractical for quality assurance in commercial production [2] and for lab-scale-investigations, too. In this study the measurements of the emissions from wood were performed with a Field and Laboratory Emission Cell (FLEC) according to ISO 16000-10 [6], which has a proven correlation to the chamber method [4]. This set up provides a simulation of realistic indoor air conditions with respect to temperature, relative humidity and air exchange rates.

EXPERIMENTAL
The samples were recovered from two 60 year-old alder stems (Alnus glutinosa (L.) Gaertn.) from North-East of Germany. The preparation of the fresh-sawn specimens (210 x 210 x 20 mm³) was performed at the Eberswalde University for Sustainable Development. The set up consisted of four variations with three specimens per batch. The samples were dried in a fresh-air-kiln respectively modified at low temperature (45°C). Further samples were impregnated, with a buffer-solution and then kiln dried respectively modified. In order to quantify any interference of organic compounds from the clean air system, the atmosphere (background) inside the empty FLEC was determined. The wood samples were prepared according to ISO 16000-11, formatted (Ø14.8 cm) and stored in a conditioning room (23 ± 2 °C and 50 ± 5 %). Prior to the tests fresh surfaces were planed and the edges were sealed with an alloy tape. The stainless steel cell and subunit allowed a controlled climate at 23 ± 1 °C and 50 ± 3 % RH. The exposed wood surface of 0.177 m² was large relative to the cell volume of 35 ml ensuring thus a high loading factor of 506 m²/ m³ for sufficient analytical sensitivity.

RESULTS AND CONCLUSIONS
The emissions obtained from the micro-chamber measurements were calculated as concentrations for the first day (Figure 1 left) and the third day (Figure 1 right). Lower-molecular-weight aldehydes, i.e. formaldehyde (FA) and acetaldehyde (AA) were detected in small amounts. FA values were below the detection limit of 2 [µg/m³] according to the BEMMA scheme [1]. In addition, the formation of formaldehyde was not catalysed by the acetic condition of the untreated specimen. The content of terpenes and aldehydes, and thus the sum of VOCs (sum of aldehydes > C2 and terpenes) increased for the impregnated and modified samples. However, these compounds possess very little corrosivity towards materials like textiles, paper and metals. Contrary to expectation, VOCs essentially lower-molecular-weight aldehydes were still found after the impregnation and/ or modification process. In addition to these prior results, additional samples of further wood species (e.g. oak) and buffer-systems with varying pH values will be investigated subsequently. It is expected to receive more information about the formation of FA during the thermal process in response to pH values by use of wood species with higher initial acid content in natural stage.

![Graph 1](image1.png)

**Types of samples**

![Graph 2](image2.png)

**Types of samples**
Figure 1. Concentration [µg/m$^3$] of emissions from specimens (Alder, n = 3 /batch); left: values after 1st day; right: values after 3rd day

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Set-recovery and micromorphology of surface densified wood

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Keywords: Scots pine, SEM, UV laser ablation

It is well known, that the mechanical properties of wood are positively correlated with its density. An increase in density can be achieved by compressing the porous structure of wood between heated plates. If the densification can be targeted to the surface, where the property improvements are most needed, rather than the entire bulk of the material, less process energy may be required whilst the desired property improvements, such as increased hardness, can be obtained. In this study Scots pine (Pinus Sylvestris L.) sapwood was surface densified by compressing in the radial direction in an open press with one heated plate using different temperatures (100, 150 and 200°C) and closing speeds (30, 10 and 5 mm/min). The original thickness was 20 mm and the target thickness was 15 mm, giving a densification ration of 25 %. Further details of the densification process and the equipment used can be found in [1] and [2]. For the set-recovery measurement, the samples were pre-dried in an oven at 40°C for 24 h, after which the samples were soaked (20°C, 24 h) and oven-dried again (40°C, 24 h). The set-recovery (SR) was calculated according to Equation 1.

\[
SR = \frac{t_s - t_o}{t_o} \times 100
\]

where \(t_s\) is the oven-dry densified thickness after soaking, \(t_d\) is the oven-dry densified thickness before soaking, and \(t_o\) the original thickness before densification.

Set-recovery was found to be significantly affected by the compression temperature, with the recovery decreasing with higher temperature. The average set-recovery values for temperatures of 100, 150 and 200°C were 80.8, 73.5 and 67.1 %, respectively. This result was expected, since higher temperature would increase stress relaxation during compression, and therefore, more permanent deformations may occur. Closing speed did not affect the set-recovery significantly; only a minor decrease in set-recovery was recorded with a slower closing speed. This might result from enhanced relaxation during slower closing due to longer exposure to elevated temperature.

Sample preparation for micromorphology analysis was carried out using UV-laser ablation technology to avoid mechanically induced artefacts on the surface or moisture recovery of the densified wood during preparation [2]. The cross-cut surfaces were analysed with a scanning electron microscope after densification and also after the soaking (20°C, 24 h) and drying (40°C, 24 h) of the samples. Cross sections of an untreated sample, a densified sample and densified sample after soaking and drying are presented in Figure 1.

![Cross-sections of (a) an untreated sample, (b) surface densified sample (100°C, 5 mm/min) before soaking and (c) surface densified samples after soaking](image)

Even though a high level of set-recovery was observed after the soaking and drying of the densified samples, deformation at the microscopic level was not completely recovered and the cells were left distorted (this is visible in Figure 1c). There were no significant fractures found in the microscopic analysis, therefore, this permanent deformation most likely does not result from fracture of the cell-wall, but rather from a viscous flow and plastic deformation of the cell-wall components. Temporary fixation may be achieved during compression as the hydrogen bonds (between cellulose and hemicelluloses) are broken and re-formed in a new position [3-5]. After compression, as water molecules enter the cell-wall, the re-formed hydrogen bonds are broken and the deformation is recovered [6]. However, in the light of this study, it may be expected that part of the hydrogen bonds re-bond permanently in the new position possibly as a result of the so-called Velcro-mechanism [7, 8] or even hornification [9, 10]. It is suggested that this permanent deformation occurs within the cell-wall between the microfibrils [7, 8], as well as between cells, that is, in the middle lamella [7, 11].
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Water vapour sorption properties and surface chemical analysis of thermally modified wood particles

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Keywords: dynamic vapour sorption, humidity cycling, wood modification, X-ray photoelectron spectroscopy (XPS)

There is an increased interest in the use of modified and thermally modified wood within different fields of application, such as building materials. As a result of this, new material combinations and systems can be produced through recycling and reusing of waste products, which would have advantages in terms of environmental aspects. Such a material could for instance be produced by methods including gluing, coating or any other composite application. In this process and in the application area of the material, knowledge about water sorption properties as well as surface properties is of interest to be able to predict the behaviour of the material and possible also to tailor new optimal material combinations. The aim of this work was to study the water sorption properties of thermally modified wood particles exposed and not exposed to dry-humid cycling. Furthermore, to perform chemical surface analysis of thermally modified wood samples exposed and not exposed to increased humidity conditions.

Water vapour sorption properties of various materials sensitive to moisture changes can be studied using a dynamic vapour sorption (DVS) instrument. With this technique the mass of the sample is measured with the change in vapour concentration which gives a quantification of the absorbed moisture. The sorption kinetic behaviour can be explained by the use of the parallel exponential kinetics (PEK):

\[ MC = MC_0 + MC_1[1-\exp(-t/t_1)] + MC_2[1-\exp(-t/t_2)] \]

MC is the moisture content of the sample at time t during exposure to a constant relative humidity (RH) condition. The equation includes a fast process which is explained by \( MC_1[1-\exp(-t/t_1)] \) and a slow process \( MC_2[1-\exp(-t/t_2)] \). MC₀ is referring to the moisture content of the sample at time zero. The terms MC₁ and MC₂ describes the moisture content of the sample at infinite time where the first one is related to the fast process and the latter one to the slow process.

Sorption isotherm measurements were performed on thermally modified wood and unmodified wood particles. The wood particles had previously been exposed to dry-humid cycles using inverse gas chromatography for surface characterization measurements. The thermally modified wood particles were exposed to three cycles of dry-humid cycling at 30°C, 0 and 75% RH, and then three cycles at 30°C, 0 and 25% RH. The unmodified wood particles were exposed to three cycles of dry-humid cycling at 30°C, 0 and 75% RH. Furthermore the samples were conditioned for about 4 weeks in a climate chamber (30°C and 75% RH). These tests were performed to study the properties of the surface energetics during dry-humid cycling. The water vapour sorption measurements were performed using samples exposed to dry-humid cycling and samples not exposed both for thermally and unmodified wood particles.

The samples exposed to increased humidity conditions went through a relaxation process during the dry-humid cycling. The intention of this study was to get information about the water sorption properties of the thermally modified wood particles and to compare the results of samples exposed to an alternative dry-humid cycling and samples that were not exposed. Previous studies have shown that thermally modified wood, exposed to sorption cycles (using DVS), shows a reduction in hysteresis after the first sorption isotherm cycle [2].

The surface chemistry provides important information about the possibilities of interaction with other materials or composites. A technique used for surface chemical analysis of the outermost surface is X-ray photoelectron spectroscopy (XPS), some examples of applications of surface chemical analysis of wood and modified wood can be found in references [1,3-4]. The results obtained from such measurements gives information about the carbon bindings and the amount of carbon present at the surface of the material. Thermally modified wood samples and unmodified wood samples, both exposed to humidity conditions at 30°C and 75% RH and not exposed were analysed using XPS.
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