**COST Action FP0904**
**Thermo-Hydro-Mechanical Wood**
**Behaviour and Processing**

# Program and Book of Abstracts

3rd Annual Conference

## "Evaluation, processing and predicting of THM treated wood behaviour by experimental and numerical methods"

April 9-11, 2013
Iasi, Romania

Edited by: Carmen-Mihaela POPESCU and
Maria-Cristina POPESCU
"Petru Poni" Institute of Macromolecular Chemistry of
Romanian Academy
41A, Grigore Ghica Voda Alley
RO700487
Iasi, Romania
http://www.icmpp.ro/

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# Preface

Book of abstracts includes the scientific program and the extended abstracts of papers presented at the COST Action FP0904 third annual meeting on “**Evaluation, Processing and Predicting of Thermo-Hydro-Mechanical Treated Wood Behaviour by Experimental and Numerical Methods” at “Petru Poni” Institute of Macromolecular Chemistry in Iasi, Romania** on 9–11 April 2013.

The main objective of COST Action FP0904 is to achieve a better understanding of 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

The objectives of this conference are to present and discuss: the state of art in THM and TH processing; examine new analytical methods in order to better understand chemical reactions occurring simultaneously within the wood constituents and to predict the performance of a product on the basis of processing parameters,which is a real challenge; to identify the problems in transferring the laboratory research findings to industrial production; and to get a better understanding of the processing needs, the development of new ideas, new products and opportunities for new markets. The conference will bring together experts and young scientists from European academia and industry as well as from other countries.

The third Action annual meeting will be held in beautiful city of Iasi one of the oldest cities in Romania at the “Petru Poni” Institute of Macromolecular Chemistry. This is an Institute of excellence of the Romanian Academy. Established in 1949, the Institute has a long tradition in fundamental and applied research in the field of organic and inorganic chemistry, polymer chemistry and physics.

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 Carmen-Mihaela Popescu, Dominique Derome, Wim Willems and Andreja Kutnar.
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 the Carmen-Mihaela Popescu and Maria-Cristina Popescu, for their works in preparing the “book of abstracts” and to the local organizing committee Carmen-Mihaela Popescu, Maria-Cristina Popescu, Cornelia Vasile, Irina Elena Raschip, Elana Parparita and SinzianaVlad.

Parviz Navi
Chair of COST Action FP0904

# Conference Organization

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## Conference Venue

Petru Poni Institute of Macromolecular Chemistry of Romanian Academy
41A, Grigore Ghica Voda Alley
RO700487, Iasi, Romania

**Iasi** is the second city of Romania after Bucharest, the national capital, in terms of both population and cultural, historical and academic terms. It is the second largest university centre in Romania.

Iasi is situated in the north-eastern part of Romania, very close to the border with the Republic of Moldova. Ancient churches, old European style houses and communist apartment buildings compete for space in this crowded city.

The "Petru Poni" Institute of Macromolecular Chemistry is located in Copou Hills, close to the centre of the city, connected by public transport. Its research achievements are recognized in Romania and abroad. For many years, the Institute has been in the first line of Romanian research and covers practically the whole area of macromolecular compounds science and technology, organic chemistry, as well as of polymeric materials.

## How to get to conference venue:

*Buses to/from "Petru Poni" Institute of Macromolecular Chemistry:*
Route ***41***: Rond Copou - Podu Ros - Rond CUG II
Route ***43b***: Rond Copou - Piata Independentei - Iulius Mall - T. Vladimirescu - CUG I
*Trams to/from "Petru Poni" Institute of Macromolecular Chemistry:*
Route ***1***: Rond Copou – T. Vladimirescu - Tatarasi – Rond Copou
Route ***13***: Rond Copou – Tg. Cucu - Tatarasi - T.Vladimirescu - Baza 3 - Bucsinescu

To reach the *Institute* you need to leave the bus/tram at the "Rond Copou" stop. Then you have a 5 min walk.


A – Unirea Hotel
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C – Best Western Astoria Hotel
D – Select Hotel
E – "Rond Copou" Stop
F – PPICM
Copou
Gară

# Scientific
Program

## Tuesday, April 09, 2013

| | | |
|---|---|---|
| 09:00 – 09:30 | Registration at the Desk (conference place) | |
| 09:30 – 09:45 | Opening Session | |
| Chairpersons: | Mark Hughes, Mathieu Pétrissans | |
| 09:45 – 10:25 | Keynote (Session 1) | |
| 09:45 | Two dimensional correlation spectroscopy applied for evaluation of the structural changes in wood<br>Carmen-Mihaela Popescu, Maria-Cristina Popescu | p. 13 |
| 10:25 – 10:50 | Poster presentation (Session 1) | |
| | Determination of dimensional stabilityof thermally modified beech and spruce wood,<br>Lothar Clauder, Alexander Pfriem | p. 73 |
| | Thermal behaviour of some wood species treated with ionic liquid<br>Cornelia Vasile, Manuela-Tatiana Nistor, Silvia Florica Patachia | p. 75 |
| | Evaluation of surface quality of wood composites as function of weathering<br>Aniela Garcia Perez, Emilia-Adela Salca, Bobadilla Maldonado, Salim Hiziroglu | p. 77 |
| | The correlation between wood moisture content and air state properties during drying<br>Daniela Sova, Venetia Sandu, Ioan-Bogdan Bedelean | p. 79 |
| | Wood anatomical changes on thermally modified surfaces of Norway spruce and Scots pine<br>Michael Altgen, Jukka Ala-Viikairi, Antti Hukka, Timo Tetri, Holger Militz | p. 81 |
| 10:50 – 11:30 | Coffee break and Poster Session | |
| 11:30 – 12:50 | Oral Presentation (Session 1) | |
| 11:30 | Comparison of chemical composition of heat treated wood cured at a same temperature under different inert atmospheres: nitrogen, vacuum and steam pressure<br>Kévin Candelier, Stéphane Dumarçay, Anélie Pétrissans, Philippe Gérardin, Mathieu Pétrissans | p. 21 |
| 11:50 | On-line monitoring of hygroscopicity and dimensional changes of wood during thermal modification by means of neutron imaging methods<br>David Mannes, Walter Sonderegger, Eberhard Lehmann | p. 25 |
| 12:10 | Comparison of acoustic NDT for assessment of small stiffness changes during low temperature thermal treatment<br>Dan Ridley-Ellis, Barbara Keating, Carmen-Mihaela Popescu | p. 27 |

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| 12:30 | Modelling the isolated and combined effects of chemical modification and hygro-thermo-mechanical loading of wood. *Report of COST FP0904 Meeting, Paris, 19-20 December 2012*<br>Patrick Perré, Joseph Gril | p. 35 |
| 12:50 – 14:30 | Lunch | |
| Chairperson: Joseph Gril | | |
| 14:30 – 15:10 | Keynote (Session 2) | |
| 14:30 | The role of water in the HTM behavior of wood<br>Dominique Derome | p. 15 |
| 15:10 – 16:20 | Oral and short oral (Session 1) | |
| 15:10 | Physico-chemical characterization of THM modified wood using inverse gas chromatography (IGC)<br>Susanna Källbom, Magnus Wålinder, Kristoffer Segerholm, Dennis Jones | p. 33 |
| 15:20 | A pseudoelastic mechanosorptive model for wood material<br>Omar Saifouni, Rostand Moutou Pitti, Jean-François Destrebecq, Frédéric Dubois | p. 29 |
| 15:40 | Strength prediction of mild thermo-hydro treatments and extrapolation for natural aging<br>Julien Froidevaux, Joseph Gril, Parviz Navi | p. 31 |
| 16:00 – 16:15 | Poster presentation (Session 1&2) | |
| | Experimental and numerical detections of cracks appear in green wood during drying process<br>Rostand Moutou Pitti, Frédéric Dubois, Eric Fournely, Jean-François Destrebecq | p. 83 |
| | About some chemical changes to wood due to densification<br>Jakub Sandak, Anna Sandak, Dusan Pauliny, MariaPaola Riggio, Ilaria Santoni | p. 85 |
| | Reducing set recovery of densified wood with heat treatment<br>Miha Humar, Frederick A. Kamke, Andreja Kutnar | p. 87 |
| 16:15 – 17:00 | Coffee break and Poster Session | |
| 17:00 – 18:00 | Oral presentations (Sessions 2 & 3) | |
| 17:00 | Finite element modeling of deformation in fiber bundle during steam explosion of wood<br>Muhammad Muzamal, Anders Rasmuson | p. 37 |
| 17:20 | Effects of closed system hydrothermal treatment conditions on colour and hardness of European beech wood | p. 39 |

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| | Charalampos Lykidis, Petros Konstantakos, Stavros Tsalikis | |
| 17:40 | Adhesion of polyurethane coating on surface densified Scots pine wood<br>Kristiina Laine, Lauri Rautkari, Borut Kričej, Matjaž Pavlič, Marko Petrič, Mark Hughes, Andreja Kutnar | p. 41 |
| 18:00 | Day conclusions on Sessions 1 & 2 + Discussion on the THM modeling | |
| 19:30 | Wine tasting | |

## Wednesday, April 10, 2013

Chairpersons: Peer Haller, Christelle Ganne-Chedeville

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|---|---|---|
| 09:00 – 09:40 | Keynote (Session 3) | |
| 09:00 | From chemical process monitoring to direct control of thermally modified wood properties<br>Wim Willems, Holger Militz | p. 17 |
| 09:40 – 10:20 | Oral presentation (Session 3) | |
| 09:40 | The Behaviour of Sorption Hysteresis in the Water Vapour Sorption Isotherm of Thermally Modified Wood<br>Callum Hill, James Ramsay, Lauri Rautkari, Mark Hughes, Kristiina Laine | p. 43 |
| 10:00 | Effect of treatment medium on the colour change of heat treated wood during natural weathering<br>Róbert Németh, László Tolvaj, Miklós Bak, Diána Csordós | p. 45 |
| 10:20 – 10:50 | Poster presentations (Session 3 & 4) | |
| | Studies on post-welding heat-treated vibrational welded wood<br>Jussi Ruponen, Martin Rhême, Silvia Ferrari, Lauri Rautkari, Mark Hughes | p. 89 |
| | Continuous Wood Densification Process of Circular Profiles<br>Jörg Wehsener, Peer Haller, Jens Hartig, Tom-Egmont Werner | p. 91 |
| | Shape stability of THM processed laminated veneer products glued with bio-based adhesive systems<br>Lars Blomqvist, Jimmy Johansson, Dick Sandberg | p. 93 |
| | Effect of thermal-vacuum treatment on bond shear strength. A comparison among Norway spruce, White ash and Turkey oak wood<br>Luigi Todaro, Silvia Ferrari, Paola Cetera, Ottaviano Allegretti,<br>Nicola Moretti, Achille Pellerano | p. 95 |
| | Determination of Thermo-modified Oak Wood Emissivity Properties in the Infrared Spectral Wavelength Range 7.5-13 µm<br>Miljenko Klarić, Stjepan Pervan, Silvana Prekrat, Aleš Straže, Željko Gorišek | p. 99 |

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| | Characterisation of chemical and physical properties of thermo-modified wood by FT-NIR spectroscopy<br>Aleš Straže, Željko Gorišek, Stjepan Pervan, Anna Sandak, Jakub Sandak | p. 101 |
| 10:50 – 11:20 | Coffee break and Poster Session | |
| 11:20 – 12:00 | Oral presentation (Session 3 & 4) | |
| 11:20 | Experimental and numerical investigations of mechanical properties of welded joint using the Arcan setup<br>Martin Rhême, John Botsis, Joël Cugnoni, Parviz Navi | p. 49 |
| 11:40 | Challenges using dielectric heating for THM processing of solid wood<br>Jimmy Johansson, Lars Blomqvist, Dick Sandberg | p. 53 |
| 12:00 – 13:00 | STSM presentations | |
| 12:00 | Using Dynamic Mechanical Analysis (DMA) for fundamental understanding of thermo treatments of wood<br>Andreja Kutnar, Robert Widmann, Iris Brémaud | p. 61 |
| 12:10 | Durability and chemical modifications of four Tunisian wood species after heat treatment<br>Mohamed Elaieb, Kevin Candelier, Stéphane Dumarcay, Philippe Gerardin, Mathieu Petrissans | p. 63 |
| 12:20 | Experimental study of mechanosorptive hygro-lock effect in wood subjected to variable loading and relative humidity<br>Omar Saifouni, Rostand Moutou Pitti, Jean-François Destrebecq, Julien Froidevaux, Parviz Navi | p. 65 |
| 12:30 | Thermal and Analytical Characterization of Welded Beech<br>María Inés Placencia Peña, Carmen Mihaela Popescu, Frédéric Pichelin, Antonio Pizzi | p. 67 |
| 12:40 | Thermal behaviours of THM densified wood<br>Carmen-Mihaela Popescu, Gabriela Lisa, Julien Froidevaux, Parviz Navi, Maria-Cristina Popescu | p. 71 |
| 12:50 | 3rd Training School Anouncement<br>Kristiina Laine, Mark Hughes | |
| 13:00 – 14:30 | Lunch | |
| Chairperson: Dennis Jones | | |
| 14:30 – 15:10 | Keynote (Session 4) | |
| 14:30 | Environmental impact assessment of THM products<br>Andreja Kutnar | p. 19 |

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| 15:10 – 15:50 | Oral presentations (Session 4) | |
| 15:10 | Influence of thermo treatment on wood physical-mechanical properties and VOC and aldehyde emissions<br>Regula Moser, Thomas Lorenz, Philipp Hass, Frédéric Pichelin, Thomas Volkmer, Urs Von Arx, Peter Niemz | p. 55 |
| 15:30 | Structural, economic and environmental performance of fibre reinforced wood profiles vs. solutions made of steel and concrete<br>Cristoph Manthy, Edeltraut Guenther, Andreas Heiduschke, Peer Haller | p. 57 |
| 15:50 – 16:30 | Poster presentations (Session 3 & 4) + 1 announcement | |
| | Set-recovery of densified and thermally modified wood under repeated soaking-drying-cycles<br>Kristiina Laine, Lauri Rautkari, Mark Hughes | p. 97 |
| | Bonding performance of densified VTC beech bonded with liquefied wood<br>Andreja Kutnar, Aleš Ugovšek, Frederick A. Kamke, Milan Sernek | p. 103 |
| | Baltic Sea network for efficient and sustainable wood processing based on wood modification processes<br>Alexander Pfriem, Mario Zander, Lothar Clauder | p. 105 |
| | Fatigue behaviour of VTC and untreated beech wood<br>Georg Behr, Holger Militz, Frederick A. Kamke, Andreja Kutnar | p. 107 |
| | Gluing ability of Pinus Pinaster depending on its moisture content<br>Anne Lavalette, Gérard Elbez, Alain Cointe, Régis Pommier, Michel Danis | p. 109 |
| | Influence of hot-pressing regimes at MDF production<br>Viktor Savov, Julia Mihailova, Cvetelin Evstatiev | p. 111 |
| | Thermo-Hydric behaviour of dowelled and bolted steel-to-timber connections exposed to fire<br>Abdoulaye Samake, Patricio Palmili, Mustapha Taazount, Philippe Audebert, Maxime Audebert, Abdelhamid Bouchaïr | p. 113 |
| 16:30 – 17:00 | Coffee break and Poster Session | |
| 17:00 – 17:40 | Oral presentations (Session 3 & 4) | |
| 17:00 | Products meeting Needs: Applying Thermal Modification and Thermo-Mechanical Processing of Wood to its Fullest Potential<br>Dennis Jones, Edo Kegel | p. 59 |
| 17:20 | Preliminary tests of combined steaming and thermal-vacuum treatment on Turkey oak (*Quercus cerris* L.) wood<br>Silvia Ferrari, Luigi Todaro, Ignazia Cuccui, Ottaviano Allegretti, Mario Marra | p. 51 |
| 17:40 | Day conclusions on Sessions 3 & 4 + Discussion on the Products meeting Needs | |

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| 19:30 | Conference dinner |
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| **Thursday, April 11, 2013** | |
| 09:00 – 10:00 | WG groups meetings |
| 10:00 – 10:40 | General discussion with all the working groups |
| 10:40 – 11:10 | Coffee break |
| 11:10 – 12:20 | MC meeting |

# Abstracts

# Keynotes

**Session 1**

# Two dimensional correlation spectroscopy applied for evaluation of the structural changes in wood

**Carmen-Mihaela POPESCU, Maria-Cristina POPESCU**

"Petru Poni" Institute of Macromolecular Chemistry of Romanian Academy, Iasi, Romania
mihapop@icmpp.ro

**Keywords:** generalized 2D correlation spectroscopy, lime wood

Due to its unique and useful properties, wood has remained an important substance throughout history and is widely used (tools, paper, buildings, bridges, guardrails, railroad ties, posts, poles, mulches, furniture, packaging, and many other products [1]). The main components of the cell walls are degraded to different extents by various groups of organisms. Both the so-called soft rot and brown rot fungi, belonging to the *Ascomycetes* and *Basidiomycetes*, respectively, decompose mainly the polysaccharides. A third group, also *Basidiomycetes* and known as white rot fungi, attacks both lignin and polysaccharides either simultaneously or successively. Wood is also a good light absorber. Among the constituent polymers, lignin is an excellent light absorber leading to the formation of free radicals. These react with oxygen to produce chromophoric carbonyl and carboxyl groups, which are responsible for colour changes [2].

The characterization of wood involving different structural degradations/modifications is a complex procedure. Spectral techniques are widely used as simple methods for obtaining rapid information on the structure of wood constituents and chemical changes taking place in wood due these various modifications.

Generalized 2D correlation spectroscopy is a well-established method which can provide useful information in different spectroscopic studies of polymers. The original concept of multidimensional spectroscopy evolved in the field of NMR spectroscopy several decades ago. The successful applications of various forms of 2D NMR certainly influenced the early development of 2D correlation approaches in other field of spectroscopy such as: FT-IR and Raman spectroscopy [3], FT-IR ATR spectroscopy [4], fluorescence spectroscopy, UV-Vis spectroscopy, birefringence [5], circular dichroism, X-ray absorption, gel permeation chromatography (GPC), molecular dynamics[6] etc. In generalized 2D correlation spectroscopy, the change in the spectra alteration can be monitored as a function of any physical or chemical variable, e.g. time, temperature, pressure, tensile, chemical reaction, optical pumping or concentration. Following the pioneering work of Noda, many research groups have adopted 2Dcorrelation analysis to study e.g. self-associated molecules, polymers, liquid crystals, Langmuir–Brogett films and biological molecules, such as peptides, proteins, cellulose and wood, etc.

The fundamental concept governing 2D correlation spectroscopy is a quantitative comparison of the patterns of spectral intensity variation along the external variable t observed at two different spectral variables. The intensity of 2D correlation spectrum X(ν1, ν2) represents the quantitative measure of a comparative similarity or dissimilarity of spectral intensity variations y(ν, t) measured at two different spectral variables, ν1 and ν2, during a fixed interval.

The generalised 2D correlation function is described by:

$$\Phi(\nu_1, \nu_2) + i\Psi(\nu_1, \nu_2) = \frac{1}{\pi(T_{max} - T_{min})} \int_0^\infty \tilde{Y}_1(\omega) \cdot \tilde{Y}_2^*(\omega) d\omega$$

defines the synchronous and asynchronous correlation intensities [7].

The most important features of this technique are the simplification of the complex spectra consisting overlapped bands, enhancement of spectral resolution by spreading bands along the second dimension, unambiguous assignments through the correlation of bands selectively coupled by various interaction mechanisms, and determination of the sequence of the spectral modifications.

Generalized two-dimensional correlation spectroscopy can be also used to evaluate the chemical changes occurring when wood is subjected to different degradation factors or to different modification conditions. It is also possible to predict the mechanisms of the components degradation or modification during the applied treatments.

**References:**

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**Session 2**

# The role of water in the HTM behavior of wood

**Dominique DEROME**

Fluids and porous materials, Laboratory of Building Science and Technology, Empa, Switzerland
dominique.derome@empa.ch

**Keywords:** poromechanical approach, swelling, nonisothermal, vapor and liquid transport

Wood, due to its biological origin, has the capacity to interact with water. Sorption/desorption of moisture is accompanied with swelling/shrinkage and softening/hardening of its stiffness. The correct prediction of the behavior of wood components undergoing environmental loading or industrial process requires that the hygric, thermal and mechanical (HTM) behavior of wood are considered in a coupled manner. We propose a comprehensive framework using a fully coupled poromechanical approach, where its multiscale implementation provides the capacity to take into account, directly, the exact geometry of wood cellular structure, using computational homogenization. A hierarchical model is used to take into account the subcellular composite-like organization of the material [1]. Such advanced modelling requires high resolution experimental data for the appropriate determination of inputs and for its validation, as shown in Figure 1.

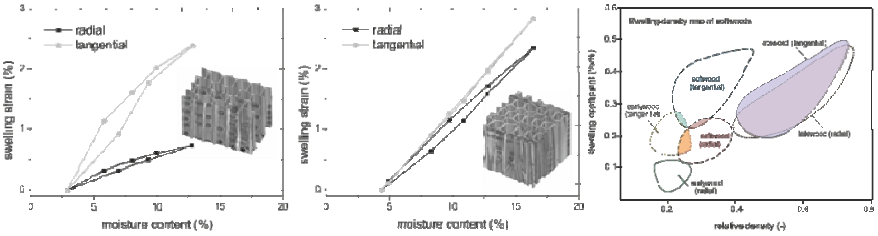

Figure 1. Swelling strains vs moisture content determined from synchrotron Xray tomography and DVS for earlywood (left) and latewood (center) [2,3]. Diagram of swelling coefficients [4].

As the application case, we consider friction welding of wood, a solid-state joining process due to the relative motion of the two wood pieces, under pressure normal to the welded plane, where the joint interface heats up, inducing plastic deformation and partial chemical degradation. Heat is generated by material deformation (plastic deformation) and friction. The bonding interface that results from friction welding presents a densification and some physical and chemical transformations, whereby entangled wood fibers are drowned into a matrix of softened and partly reticulated wood intercellular material, such as lignin and hemicelluloses. For this problem, the system of coupled nonlinear equations is solved by means of FEM method using our own Fortran code which follows an iterative total incremental approach [5] and is able to solve fully coupled thermo-hygro-mechanical problems. In the simulation, constant temperature at the bottom line simulates the boundary condition with the hot plate. Convective heat and mass transport occurs on the two side faces. In the experiment, a constant temperature of 250°C is maintained at the bottom of the specimen using a metallic foil connected to a power supply. The wooden sample is 40 by 40 mm2 and its initial moisture content is 68 kg/m3 (in equilibrium with 80%RH). Thermal neutron radiography allows the determination of the moisture content with a resolution of 100g/kg3. In the Figure 2, the modeling and experimental moisture content distributions are compared. The modeling results are in agreement with the experimental results. As a verification, the simulated change of total moisture content vs time agrees well with the experimental results. The modeling efforts are now aiming at including gas pressure at the weld line and plastic deformation for the prediction of the densification of wood at the weld line.

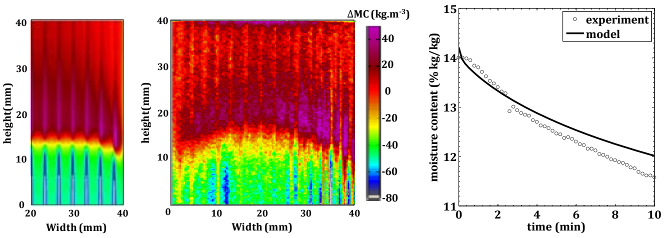

Figure 2. Simulation (left) and experimental (center) moisture content distribution and total (right)

In isothermal experiment at the cellular scale, we quantify the affine strains during the swelling and shrinkage using high resolution images obtained by phase contrast synchrotron X-ray tomography of wood samples of different porosities (Figure 1). The reversibility of the swelling/shrinkage is found for samples with controlled moisture sorption history. The deformation is more hysteretic for high than for low density samples. Swelling/shrinkage due to ad/desorption of water vapour displays also a non-affine component. The reversibility of the swelling/shrinkage indicates that the material has a structural capacity to show a persistent cellular geometry for a given moisture state and a structural composition that allows for moisture-induced transitional states. However, a collection of qualitative observations of small subsets of cells during swelling/shrinkage reveals a shape memory capacity, see Figure 3.

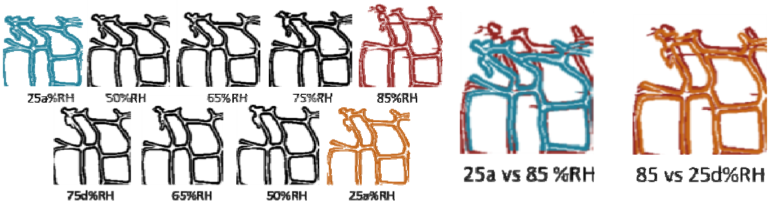

Figure 3. Cell configuration during successive moisture content states (left) with initial deformation that is mended during wetting, and comparison of states (right)[3].

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**Session 3**

# From chemical process monitoring to direct control of thermally modified wood properties

**Wim WILLEMS[1,2], Holger MILITZ[2]**

[1]Firmolin Technologies BV, Grote Bottel 7a, 5753 PE Deurne, The Netherlands
[2]Wood Biology and Wood Products, Georg-August University Göttingen, Germany
info@firmolin.com

**Keywords:** thermally modified wood, elemental composition, durability, moisture content, process monitor

The results from previous research [1,2] on the connection between elemental composition and properties of thermally modified wood are reviewed. Briefly, changing elemental wood compositions can be mapped into a van Krevelen diagram (Figure 1) and compared to pure carbohydrates, showing that the wood thermo-chemical reactions have an overall dehydration ($\Delta x<0$) as well as a chemically reducing effect ($\Delta Z<0$). It was shown that the x=O/C-ratio is a measure of the polarity of wood and that Z is equal to the average carbon oxidation state of wood. Parameter-free models were presented for the relation between x and the moisture content and between Z and a relative oxidation rate (a tentative measure of durability). Both simple models showed good agreement between predicted and experimental moisture sorption and durability data.

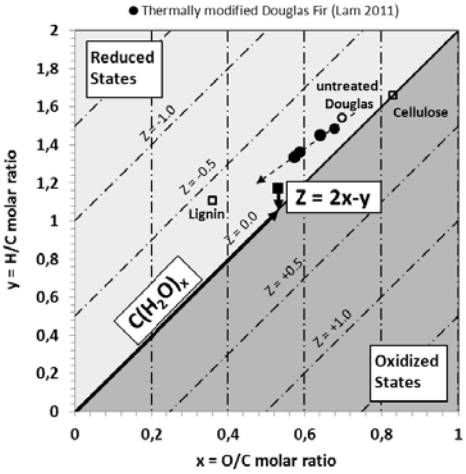

Figure 1. van Krevelen diagram for thermally modified wood, given by the empirical formula $CH_yO_x$. The coordinates are molar elemental ratios x=O/C and y=H/C. $|Z|=|2y-x|$ is the vertical distance of any point (x,y) to the carbohydrate reference line y=2x (Z=0). The broken line with arrow point indicates the composition trajectory for increased intensity of saturated steam heat-treatment on Douglas fir (data [3]: closed circles, untreated: open circle). The trajectory runs in the direction of $\Delta x<0$ and $\Delta Z<0$. The composition data from other sources appear to follow the same trajectory, universal to at least 9 widely different thermally treated wood species [4].

The overall composition change in thermally modified wood can be measured by CHN(O)-elemental composition analysis, but alternatively – as a consequence of conservation of mass and species – by analysis of exhaust gases during the modification process. Such an arrangement, which has recently been implemented in an industrial thermal wood modification plant in the Netherlands, is able to follow the development of ΔZ, a measure for the degree of modification, during the production run.

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**Session 4**

# Environmental impact assessment of THM products

**Andreja KUTNAR**

University of Primorska, Andrej Marusic Institute, SI-6000 Koper, Slovenia
andreja.kutnar@upr.si

**Keywords:** carbon footprint, EPD, heat treated wood, LCA

With sustainable design being a necessity, it is important to not only consider the performance of the thermo-hydro-mechanical (THM) treated wood, but also consider other performance metric to gain a holistic appreciation. These performance metrics are embodied energy over the product lifecycle, decay resistance, and structural performance. Sustainability forces us to take holistic approaches, which have not been addressed comprehensively. A THM treated wood that performs better than the untreated wood might have significantly higher embodied energy over its lifecycle. This will make it bad choice from an environmental standpoint.

With sustainability being the driving force in the creation of a building, environmental impact of selected materials should be included in planning, considering the life cycle and embodied energy of the materials used. Therefore, the Life Cycle Assessment (LCA) methodology should be used to reveal the environmental and energy performances of the used materials, as well as the developed products through the whole life cycle. Since the 1980s, when LCA analysis was developed, till today numerous methodologies to classify, characterize, and normalize environmental effects were developed. The most common, for example CML 2 (2000), IPCC Greenhouse gas emissions, Ecopoints 97 and Eco-indicator 99 (PRé Consultants, 2010), are focused on following indicators: acidification, eutrophication, thinning the ozone layer, various types of ecotoxicity, air contaminations, usage of resources and greenhouse gas emissions. At first, LCA analysis was mostly focused on environmental effects like acidification and eutrophication, while in the past years mostly on greenhouse gas emissions (GHG), which are also called carbon footprint. The carbon footprint is expressed in terms of the amount of emitted carbon dioxide or its equivalent of other greenhouse gases.

In Europe, carbon footprint is gaining immense importance and expected to be mandated to accompany products and services. Furthermore, environmental product declarations (EPD) of type III are increasingly being used for assessment of building products. An EPD is a standardized and LCA based tool to communicate the environmental performance of a product or system, and is applicable worldwide for all interested companies and organizations. Therefore, following the common LCA methodology (ISO 14044, PAS 2050) the goal of the study was to objectively prove environmental impact of thermally modified wood heat treated according to the process developed by Rep et al. (2012). The carbon footprint of the beech wood thermally treated at 210°C for 3 hours was calculated. Environmental impact of the thermally treated wood was analyzed with the “Cradle-to-gate” variant, an assessment of a partial product life cycle from manufacture ('cradle') to the factory gate (i.e., before it is transported to the consumer). The use phase and disposal phase of the product were omitted. Analysis included transportation of material to the factory, electricity and other energy sources in product production, and waste resulting from product production.

Carbon footprint calculates the amount of GHG emissions caused by a particular activity or entity, commonly also referred to as global warming potential (GWP). It is measured in tones (or kilograms) of carbon dioxide equivalent ($CO_2$eq.). Approximately 50 % of dry timber is elemental carbon; thus, 1 kg of wood contains approximately 0.5 kg of carbon, which equates to 1.83 kg of $CO_2$. When calculating a carbon footprint, whether to include this stored carbon in timber is a much debated issue. In this study, the carbon footprint and NET carbon footprint were calculated. In Table 1 the carbon footprint of emission sources and their contribution to the total carbon footprint of 1 $m^3$ of thermally modified beech wood are shown. The calculated carbon footprint of 1 $m^3$ of thermally treated beech wood was 161 kg $CO_2e$, while the carbon sequestrated in 1 m3 of thermally modified beech was 982 kg $CO_2e$. Therefore the NET carbon footprint was – 821 kg $CO_2e$.

Table 1. Carbon footprint of emission sources and their contribution to the total carbon footprint of 1 $m^3$ of thermally modified beech wood

| | Carbon footprint [kg $CO_2e$] | Contribution to the total carbon footprint [%] |
|---|---|---|
| Water | 0.79 | 0.5 |
| Electricity | 57.4 | 35.7 |
| Kiln-dried sawn wood | 79.2 | 49.2 |
| Transport | 23.3 | 14.5 |
| Carbon footprint | 161 | 100 |
| $CO_2$eq. sequestered | 982 | |
| NET carbon footprint | - 821 | |

The results showed that energy consumption of the thermal treatment considerably contributes to the carbon footprint of thermally modified beech wood. However, the improved properties during the use phase were not included in the study, which might diminish the environmental impact of the heat treatment. However, it can be concluded that in order to be environmentally friendlier, the heat consumption of the thermal modification process should be reduced by using lower temperatures and/or shorter treatments. Furthermore, the studies should focus on intelligent concepts for reuse and recycling of thermally treated wood at the end of single product life.

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# Oral presentations

**Session 1**

# Comparison of chemical composition of heat treated wood cured at a same temperature under different inert atmospheres: nitrogen, vacuum and steam pressure

**Kévin CANDELIER, Stéphane DUMARÇAY, Anélie PETRISSANS,
Philippe GERARDIN, Mathieu PETRISSANS**

Laboratoire d'Etudes et de Recherches sur le Matériau Bois, EA 4370, Université de Lorraine,
Faculté des Sciences et Technologies, BP 70239, F-54506 Vandoeuvre-lès-Nancy, France
kevin.candelier@lermab.uhp-nancy.fr

**Keywords:** Beech, chemical composition, durability, heat treatment, inert atmospheres

Wood heat treatment by mild pyrolysis is used to improve wood properties such as its decay durability and dimensional stability [1,2]. These new properties are the result of chemical modifications of wood cell wall polymers occurring during heat treatment which confer to wood new properties [3-5]. Nowadays, several types of thermal treatments of wood are used. These treatments differ mainly by the nature of their inert atmospheres: under nitrogen, under vacuum, under steam pressure, in oil ... Decay resistance of this new material is strongly correlated to thermal degradation of wood cell wall components and consequently of treatment intensity. Theses parameters undergo the chemical and physical modifications during the thermal treatment [6]. The comparison between these three processes should let knowing the vacuum influence on thermodegradation mechanisms. In fact, vacuum might modifies reaction equilibrium and change the reaction ways [7].

The aim of this study is to determine the effect of a new generation of treatment performed under vacuum on the chemical modifications occurring during treatment. For this purpose, one hardwood (beech) has been treated under different conditions (vacuum, nitrogen, steam). All treatments were performed at 230°C for mass losses resulting from wood thermodegradation of approximately 12%. Times of thermal treatment are about 4.5 hours and the dimensions of wood samples are semi-industrial: 110 x 65 x 2.5 cm (L x R x T). For each treatment condition, extractives, Klason lignin, hemicelluloses and a-celluloses content were determined as well as monosaccharide composition.

Decay resistance test were performed on untreated and treated wood in order to evaluate possible chemical changes. Intensity of thermodegradation was also evaluated by TD-GC-MS. Treated and untreated wood samples were subject to thermodegradation directly in the thermodesorption tube under nitrogen and at 230°C during 15 minutes. And volatile compounds resulting from wood degradation were analyzed.

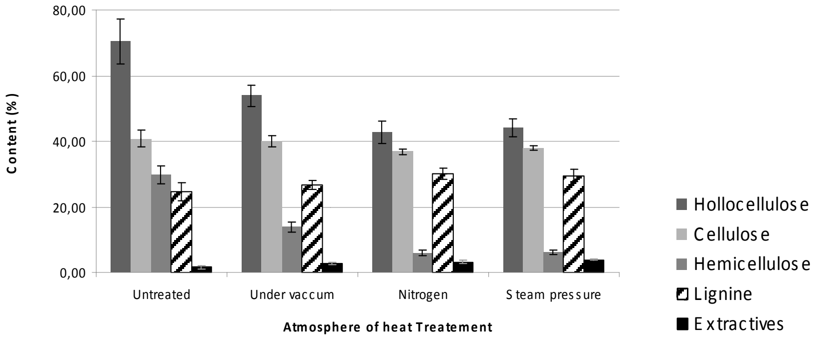

Figure 1. Chemical composition (%) of heat treated beech wood (ML= 12 %) according to the thermal treatment atmosphere.

Results show that extracts content were lower in the case of wood samples treated under vacuum, while lignin,hemicelluloses and a-celluloses contents were higher in the case of samples treated under steam and nitrogen, indicating lower wood degradation under vacuum treatment and neutral monosaccharides contents comparatively to wood heat treated under vacuum. These results may be explained by the effect of vacuum allowing removal of volatile degradation products limiting therefore acidic degradation of polysaccharides due to formation of acetic acid and recondensation of volatile degradation products within the wood structure. This hypothesis was confirmed by chromatography analyses according to the nature and quantity of thermodegradation volatile products formed during TD-GC-MS analyses.

These results may be explained by the fact that mass loss of 12% fixed in our experiments was probably underestimated in the case of samples treated under nitrogen and steam pressure due to the recondensation of degradation products which distorts the true mass loss value. Wood samples heat treated under nitrogen are consequently more degraded than those which where heat treated under vacuum. Decay resistance test were performed on untreated and treated wood in order to evaluate possible chemical changes. Results are presented on Figure 2.

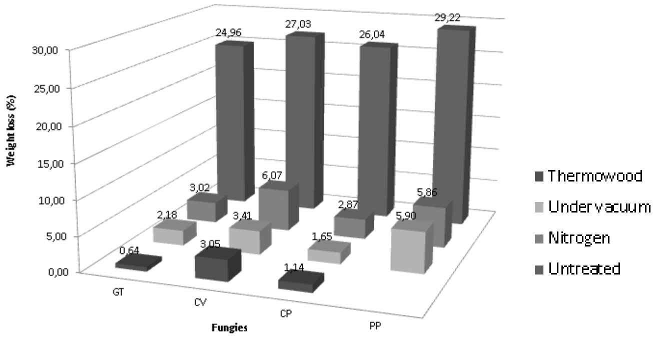

Figure 2. Correlation between weight loss (WL%) due to fungal attacks, different fungies and the atmosphere of treatment used (ML=12%).

Even if effect of vacuum versus nitrogen and steam pressure is not obvious, both treatments allow improving wood decay resistance, which is achieved for mass losses of approximateley 12%.

Further studies are currently under investigation to investigate the effect of inert atmosphere on wood mechanical properties.

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**Session 1**

# On-line monitoring of hygroscopicity and dimensional changes of wood during thermal modification by means of neutron imaging methods

**David MANNES, Walter SONDEREGGER, Eberhard LEHMANN**

Spallation Neutron Source Division, Paul Scherrer Institut, CH-5232 Villigen PSI, Switzerland
david.mannes@psi.ch

**Keywords:** Hygroscopicity change, neutron imaging, shrinkage, sorption isotherm, thermal modification

Hygroscopicity of wood and therefrom-induced dimensional changes are known to be reduced after high temperature heat treatment. However, the mechanism and level of this phenomenon is neither well-understood nor quantified, mainly due to experimental difficulties and equipment constraints.

The presented work is part of a research project supported by the Swiss Secretariat for Education and Research within the scope of COST FP0904 with the topic: “Investigation of thermo-sorptive aging of wood by means of neutron imaging methods“. Its main goal is to improve the knowledge on the hygroscopicity and dimensional changes occurring during wood modification (under high temperature / high pressure conditions) with special attention to thermosorption [1].

In contrast to other approaches this study deals with changes occurring during the actual modification process (in-situ study). Neutron imaging offers for this an unique opportunity as it is highly sensitive for hydrogen (and thus changes of the moisture content) while some metals (e.g. Al) are at the same time practically transparent to neutrons [2]. These facts allow to determine changes in hygroscopicity parallel to dimensional changes during the treatment in a high temperature / high-pressure environment inside a specially built testing chamber [3].

The neutron imaging investigations are carried out at the neutron imaging facility NEUTRA at the Paul Scherrer Institut (PSI), Villigen (CH) using relatively small samples of European beech (*Fagus sylvatica* L.) and Norway spruce (*Picea abies* [L.] Karst.), which are subjected to an in-situ treatment within the range of 70°C to 150°C and positive pressures up to 4 bar. The dimensions of the tested samples are relatively small (30 mm x 30 mm x 5 mm). On the one hand the samples have to be limited in size to allow rapid modification cycles. On the other hand the test sample thickness should not exceed few cm according to the attenuation behavior of the wood itself in order to maintain the best water sensitivity [4].

The sorption isotherms at 150°C of spruce and beech are shown in Figures 1 and 2 as first results. For this purpose, two specimens per species were exposed to 150°C at a positive pressure of 4 bar and 0, 20, 30, 40, 50, 60, 70, 80, 90, 70, 50, 30 and 0% relative humidity (RH) for at least half an hour at each RH-level. Neutron imaging measurements were performed (at least 15 images per RH-level) throughout the whole treatment. The neutron images were corrected with standard correction procedures (Mannes et al. 2009). To determine the wood moisture content (MC), the images were referenced to the initial state from the start of the experiment (0% RH = ovendry state). In this approach, the influence of swelling and shrinkage as well as the mass loss during the treatment are not considered resulting in a negative MC for the desorption curve at low RHs (Figures 1 and 2). In a further step, the dimensional changes and the mass loss during the treatment will be accounted for.

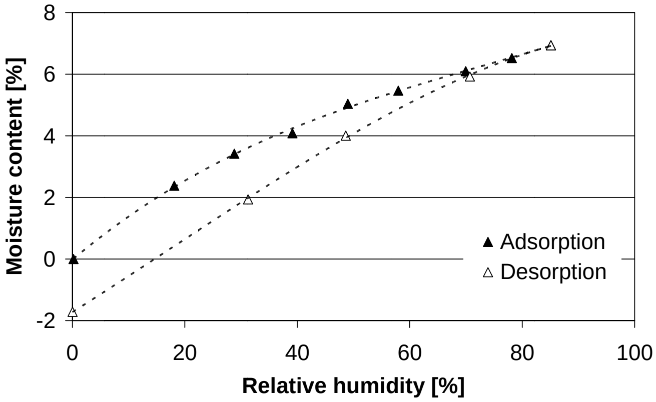
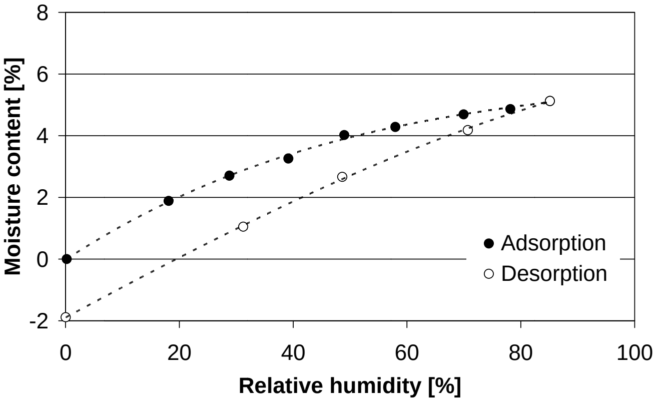


Figure 1. Sorption isotherm of spruce at 150°C (according to Sonderegger et al. 2012, modified)



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**Session 1**

# Comparison of acoustic NDT for assessment of small stiffness changes during low temperature thermal treatment

**Dan RIDLEY-ELLIS[1], Barbara KEATING[2], Carmen-Mihaela POPESCU[3]**

[1]Edinburgh Napier University, Edinburgh, UK
[2]University of Strathclyde, Glasgow, UK
[3]"Petru Poni" Institute of Macromolecular Chemistry of Romanian Academy, Iasi, Romania
d.ridleyellis@napier.ac.uk

**Keywords:** NDT, acoustic, resonance, ultrasonic, stiffness

This paper discusses the application of some simple, inexpensive, acoustic non-destructive testing techniques for measurement of changes in stiffness during thermal modification of wood. The ease of use, accuracy and reliability of the methods are compared using a case study of data obtained from an experiment in which clear samples of Scots pine (*Pinus sylvestris*) and Douglas-fir (*Pseudotsuga menziesii*) were heated in an oven at 110 ±2.5 °C for a total treatment time of 170 days. On a weekly basis the samples were removed from the oven and the small changes in longitudinal Modulus of Elasticity ($E_{dyn}$) and radial-tangential Shear Modulus ($G_{dyn}$) were tracked using:

- The impulse excitation method (flexural, longitudinal and torsional resonance)
- The ultrasonic time of flight method (24, 54, 150 and 250 kHz)

Impulse excitation and ultrasonic time-of-flight are both well-established methods for assessing the stiffness of a material non-destructively. Their use on wood has become common in both scientific and industrial applications although the results obtained need to be treated with some caution. The 'dynamic stiffness' as measured by an acoustic technique differs from the equivalent 'static stiffness' that would be obtained from a pseudo-static test because of the complex ways in which dynamic waves propagate in wood, its rate dependent material behaviour and the assumptions of linear elastic, homogeneous, isotropic material behaviour in the resonance equations. However, these errors are systematic and for a particular sample and testing method it is a straightforward matter to track changes in stiffness by acoustic methods.

Samples of planed clear wood (four of each species, dimensions in Figure 1) were heated in an oven, periodically removed, weighed hot and then allowed to cool in normal indoor conditions. They were then reweighed, measured with digital callipers and by the above acoustic methods. Finally the samples were weighed again and returned to the oven. The treatment time was recorded to be the time the samples spent in the oven.

For ultrasonic time-of-flight, it was found that all frequencies had similar transit times (within experimental error), but that the 54 kHz transducers produced the clearest results. Individual measurements typically ranged ±0.5 μs (equating to ±2% in the measurement of $E_{dyn}$), but by taking an average of 30 measurements this could be reduced to ±0.05 μs, the precision of the Pundit Lab plus instrument (±0.2% of $E_{dyn}$). In practice, signal noise and variability in instrument calibration reduced the measurement accuracy, as can be seen in Figure 1.

The impact excitation method employed was based on the standard test procedures for ceramic materials. The shape of the samples was a compromise so as to obtain flexural, longitudinal and torsional resonance modes on the same sample. The aspect ratios were outside the limits of the shape factor corrections in EN 843-2 and so the more complex corrections in ASTM E1876 were used. In order to provide better precision in the frequency domain, the sound of several hits was recorded and analysed together. Due to the shape of the samples there were many resonance peaks in addition to the basic longitudinal, flexural and torsional modes and their harmonics. In all cases the flexural mode was clear and unambiguous. The longitudinal mode was very difficult to discern in all cases as there were a multitude of overlapping peaks due to the complex modes of vibration for these sample shapes. By comparing spectra for a sample at different treatment points it was possible to use the cases where the longitudinal peak was clearest to discern the correct peak in other cases.

There was a high level of agreement between the methods, but the impact excitation technique outperformed the ultrasonic time-of-flight method in this case. All methods are capable of measuring the trend in stiffness and by tracking several measurement points a much better result was obtained than would have been from single before and after measurement points.

Even at this relatively low temperature, the wood underwent thermal modification, losing both mass (~0.02%/day for Scots pine and ~0.01%/day for Douglas-fir) and stiffness (~0.05%/day for Scots pine and ~0.04%/day for Douglas-fir). The change in $E_{dyn}$ was similar to the change in $G_{dyn}$.

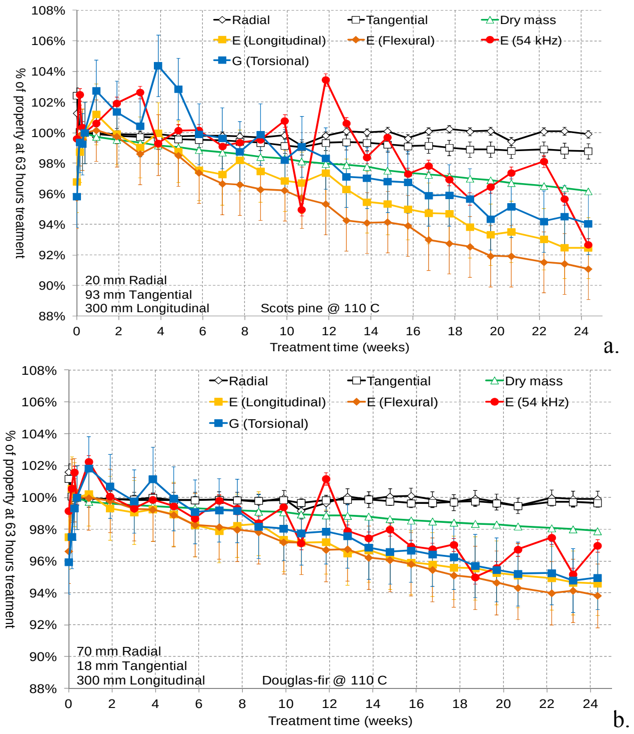

Figure 1. Average of four samples of Scots pine (a) and Douglas-fir (b)

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**Session 1**

# A pseudoelastic mechanosorptive model for wood material

**Omar SAIFOUNI[1,2], Rostand MOUTOU PITTI[1,2], Jean-François DESTREBECQ[1,2], Frédéric DUBOIS[3]**

[1]Clermont Université, Université Blaise Pascal, IP, UMR 6602, BP 10448, Clermont-Ferrand, France
[2]CNRS, UMR 6602, Institut Pascal, F-63171, Aubière, France
[3]GEMH, Département Génie Civil et Durabilité, Université de Limoges, 19300, Egletons, France
rostand.moutou_pitti@univ-bpclermont.fr

**Keywords:** wood, mechanosorption, thermodynamics, hygro-lock effect, rigidity approach

Understanding the combined effects between mechanical loading and moisture changes in wood material is important to accurately describe the mechanosorptive behaviour of timber structures. However, a difficulty in wood material is due to a strain locking during drying under load, known as hygro-lock effect [1]. In this paper, we propose a new stiffness approach of the pseudo-elastic mechanosorptive behaviour which takes into account the hygro-lock effect. The approach is based on a partition of the mechanical stress in an elastic part and a mechanosorptive part. The proposed model is developed for any variable loading and moisture content conditions, leading to a pseudo-elastic mechanosorptive law written in compliance and stiffness forms.

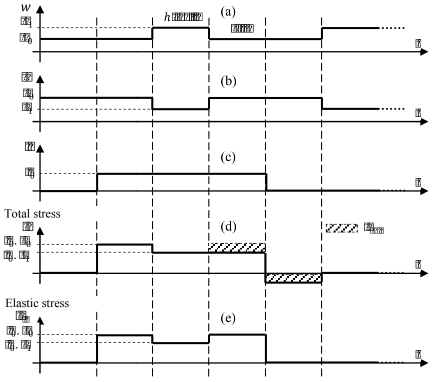

Figure 1. Mechanosorptive behaviour

When wood is subjected to a mechanical load under constant or increasing moisture content, its behaviour depends on both the mechanical and the hydric solicitations. In this case, it follows the Hooke's law (1.a). However, during a drying period while loaded, its behaviour depends only on the mechanical solicitation. Indeed, the relationship between stress and strain can be represented by the restricted Hooke's law (1.b), [2]:

$$(\text{a}):\ \dot{\sigma} = E(w)\,\dot{\varepsilon} + \dot{E}(w)\,\varepsilon \quad \text{with} \quad \dot{w} \geq 0; \quad (\text{b}):\ \dot{\sigma} = E(w)\,\dot{\varepsilon} \quad \text{with} \quad \dot{w} < 0 \qquad (1)$$

where $E(w)$ is the value of the elastic modulus corresponding to the actual moisture content. According to (1.b), a stress lock occurs when a decreasing moisture content variation is imposed to the strained wood, see Figure 1. Based on Figure 1, we propose to partition the actual stress as the sum of an elastic stress $\sigma_e$ and a mechanosorptive stress $\sigma_{ms}$ , as follows

$$\sigma = \sigma_e + \sigma_{ms} \tag{2}$$

We consider an isotherm transformation (for the sake of clarity, the hydric strain is omitted in the following). Based on usual assumptions of thermodynamics and continuum mechanics, a relationship is proposed between the mechanosorptive stress $\sigma_{ms}$ and the related internal variable $\varepsilon_{ms}$ , as follows

$$\sigma_{ms} = E(w)\,\varepsilon_{ms} \tag{3}$$

The approach is generalised for any imposed strain variation, by subdividing the total strain in a sum of finite increments $\Delta\varepsilon_i$ . After analytical developments, the mechanosorptive behaviour law obtained in stiffness form is given by equation (4a)

$$\mathrm{a}: \sigma = \sum_{i=0}^{N} H(t-t_i).\overline{E}_i.\Delta\varepsilon_i;\ \ \mathrm{b}: \varepsilon = \sum_{i=0}^{N} H(t-t_i).\frac{1}{\overline{E}_i}.\Delta\sigma_i \tag{4}$$

where $\overline{E}_i$ is the elastic modulus corresponding to the moisture content $\overline{w}_i$ at the beginning of the drying phase for the strain increment $\Delta\varepsilon_i$ , see Figure 2. Similarly, the dual complaisance approach, based on the strain partition, is given by equation (4b) [3]. The stiffness and the compliance analytic formulations are implemented in Matlab© software.

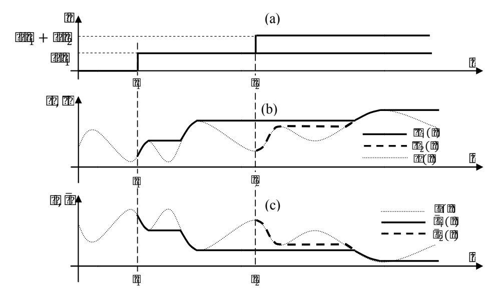

Figure 2: illustration of $\overline{E}_i$ and $\overline{w}_i$ in equation (4).

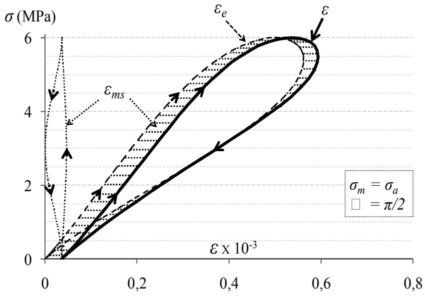

Figure 3: stress-strain curves under sinusoidal loading/humidity cycles.

Figure 3 shows the evolution of the total strain and his partitions (elastic and mechanosorptive) given by the model for a loading/humidity cycle ( $\sigma_m$ is the amplitude, $\omega$ is the pulsation, and $\phi$ is the phase angle)

$$w\% = 50 + 40.\cos\omega t \ \text{ and } \ \sigma = \sigma_m + \sigma_a.\cos(\omega t + \varphi) \ \text{ with } \ \sigma_m = \sigma_a = 3MPa \tag{5}$$

The stress-strain diagrams are shaped as stable hysteresis loops. $\varepsilon_{ms}$ appears at the beginning of the cycle (drying under stress); then it is released during the humidification phase. This result shows the ability of the model to depict the mechanosorptive behaviour of wood material under variable loading and humidity. Further work in progress is devoted to the expansion of the approach to the three-dimensional orthotropic case, and to the coupling with a viscoelastic model.

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**Session 1**

# Strength prediction of mild thermo-hydro treatments and extrapolation for natural aging

**Julien FROIDEVAUX[1], Joseph GRIL[2], Parviz NAVI[1]**

[1]BFH-AHB, Bern university of Applied Science, Switzerland
[2]UM2- LMGC, Université Montpellier 2, Laboratoire de mécanique et génie civil, France
julien.froidevaux@bfh.ch

**Keywords:** Aging, Thermo-hydro, Panel paintings

Thermo-hydro (TH) treated wood has been used to predict the behaviour of naturally aged wood. A first investigation on the comparison of the mechanical, physical and chemical properties of non-aged and natural aged spruce wood (*Picea Abies (L.) Karst*.) has been carried out [1] and [2]. A second investigation has been performed including the accelerated aging of wood [3]. Artificially aged samples have been produced by TH treatments. Two pressure vessels with controllable temperature, relative humidity (RH) and pressure have been designed and constructed. The treatment temperature range is between 100 and 150°C, the absolute treatment pressure between 0.1 and 8 bars and the RH between 1 and 95%.

Results indicate that in terms of mechanical behavior along the radial direction, only the strength showed a significant modification. For example, old wood samples of 500 years old show a decrease of about 25% of the strength compare to non-aged wood. No significant modification of the Young's modulus between the different samples has been seen. Loss of color lightness has also been seen. It was possible to reproduce similar loss of lightness and radial strength (without change in E-modulus) with TH-treatment.

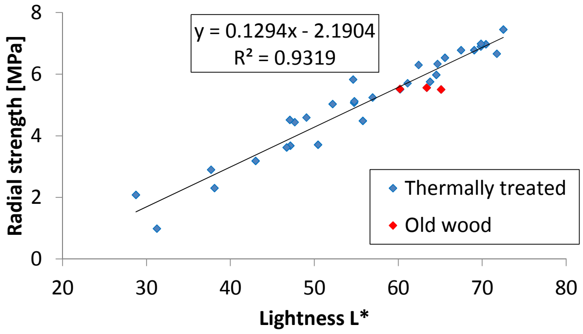

Figure 1. Linear relation between the radial strength and the lightness of thermally treated wood and old wood samples (200, 400 and 500 years old).

Figure 1 shows the experimental radial strength $\sigma_f$ against the lightness $L^*$. A linear relation can be seen between each other. Therefore, by knowing the color lightness $L^*$ of an old wood, the strength can be estimated simply with:

$$\sigma_f = 0.1294 \cdot L^* - 2.19 \tag{1}$$

A direct fitting has been made for the radial strength $\sigma_f$ and the color lightness $L^*$ on the kinetic law of chemistry, by minimizing the mean square difference by a gen*eralized reduction gradient algorithm* (GRG). The aging equations are:

$$\begin{cases} \sigma_f(t) = \sigma_0 e^{-k(T,m)\cdot t^n} \\ L^*(t) = L_0^* e^{-k(T,m)\cdot t^n} + L_\infty^* \end{cases} \tag{2}$$

where $\sigma_0$ is the radial strength of untreated wood, $L^*_0$ and $L^*_\infty$ are the lightness of untreated and treated at infinite time respectively, $n$ is the reaction order and $t$ the time. The rate constant $k(T,m)$ is dependent on the treatment temperature $T$ and the wood moisture content $m$ and it is given by:

$$k(T,m) = k_0 \cdot e^{Bm} \cdot e^{-\frac{E_a}{RT}} \tag{3}$$

where $E_a$ is the activation energy of the reaction and $R$ is the gas constant (8.314 J $K^{-1}mol^{-1}$) and $B$ and $k_0$ are fitted parameters.

The aging laws defined in equations (2) can be first used to predict the degradation of the radial strength and color lightness after thermal treatment. The range of validity of the laws is for temperatures between 110°C and 150°C, treatment RH between 0 and 25% and under atmospheric pressure. However, the law can be extrapolated to standard climate conditions, in order to predict the effect of natural aging.

Such analysis has been carried out on an old panel painting as shown in figure 2. Its color lightness has been measured and was $L^* = 53.74$. With equation (1) the radial strength can be estimated at about *4.75 MPa*.

A finite element model (FEM) calculating the stresses in panel paintings under RH variation [4] show that the radial stresses during a moving of the panel (quick change of RH from 65 to 45%) induce about *2.75 MPa* of radial stresses. By estimated the mean climatic condition in which the panel painting has been stored with the lightness (second equation in (2), using the age determined by dendrochronology), the radial strength will decrease at *2.75 MPa* in 1500 years!

Figure 2. Old wood panel paintings rented from the Swiss National Museum – Collection Centre. The age of the wood of this painting has been determined by dendrochronology.

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**Acknowledgments:** The financial support of the Swiss National Science Foundation (FNS) for the project (N° K21K-122336/1) is acknowledged with gratitude.

**Session 1**

# Physico-chemical characterization of THM modified wood using inverse gas chromatography (IGC)

**Susanna KÄLLBOM[1], Magnus WÅLINDER[1,2], Kristoffer SEGERHOLM[1,2], Dennis JONES[2]**

[1]KTH, Royal Institute of Technology, Building Materials, SE-100 44 Stockholm, Sweden
[2]SP Technical Research Institute of Sweden, Wood Technology,
EcoBuild Institute Excellence Centre, PO Box 5609, SE-114 86 Stockholm, Sweden
susanna.kallbom@byv.kth.se

**Keywords:** THM modification, Inverse Gas Chromatography (IGC), properties

A future vision of the Swedish market of building materials is to a larger extent utilize and increase the value of the by-products from the wood industry. The aim of this project is to widen the knowledge within the field of Thermo-Hydro-Mechanical (THM) modification of various wood components. One idea is to apply a production process based on non-ventilated process conditions carried out in the form of extrusion and pressing. The moisture content during this step will be higher than what is allowed in conventional processes. One specific focus will be on physico-chemical characterization of THM modified wood using inverse gas chromatography (IGC).

Inverse gas chromatography (IGC) is particularly useful to determine surface free energy and acid-base characteristics of materials in particle or fibrous form [1-8]. The method can provide an insight into changes in the surface chemistry of modified wood, in this case THM wood and its subsequent use, for example in the production of composite materials. These studies will assess the application of the IGC technique to study the surface energy characteristics of the treated fibres to provide valuable insight about the activities of the modified wood surface.

The principle of IGC is like regular gas chromatography where gases are injected into an inert gas stream passing through a packed column held in an environmentally controlled oven. As the gases reach the end of the column, they are detected using a flame ionization detector. In normal gas chromatography, the columns are chosen for their ability to separate gases adequately, whereas in IGC, known gas probes are chosen for their ability to interact with an unknown packing material. Figure 1 shows a sketch of the IGC principle.

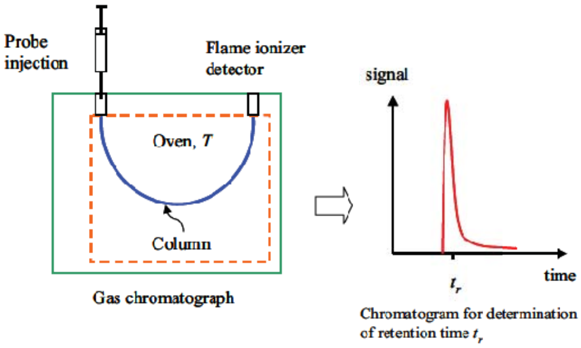

Figure 1. Schematic illustration of the principle for inverse gas chromatography (IGC) experiments.

Such information may be helpful to tailor and improve the THM modification of various wood species and its subsequent molecular bonding with binders in composites. This poster presentation covers some background information about the IGC technique as well as some initial trials to characterize surface energy parameters of ground thermally modified wood of different size distributions.

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**Session 2**

# Modelling the isolated and combined effects of chemical modification and hygro-thermo-mechanical loading of wood

## *Report of COST FP0904 Meeting, Paris, 19-20 December 2012*

**Patrick PERRÉ[1], Joseph GRIL[2]**

[1]Laboratoire de Génie des Procédés et Matériaux, École Centrale Paris, France
[2]Laboratoire de Mécanique et Génie Civil Engineering, Université Montpellier 2, France
joseph.gril@univ-montp2.fr

**Keywords:** wood, modification, modelling, constitutive equations

This meeting aimed at gathering a small group of experts in the field of modelling of wood modification and mechanical behaviour, in order to address the following issue: what kind of constitutive equations of hygro-thermo-mechanical behaviour should we use when the material itself changes over time due to chemical degradation? Although we have primarily in mind the degradation processes associated with thermal treatments, other modifications could be considered. The coupling in the reverse way, i.e. the influence of loading conditions of the degradation should also be considered. Obviously this is a complex question that involves as a preliminary to discuss the available knowledge on: (i) the processes of degradation resulting from modification treatments and the modelling of their kinetics; (ii) the physics and mathematical description of hygromechanical couplings, and their activation. Experimental validation of models should be also included in the discussions.

The meeting was attended by 20 scientists. It was organised in Ecole Centrale Paris, close to Paris, from December 19 noon to 20 noon, 2012, followed by an optional visit of LGPM (www.lgpm.ecp.fr) in the afternoon of the 20th. 16 Oral presentations by delegates during the afternoon of 19/12 allowed a first exchange of information. Most of the participants joined a dinner close to the venue where they had opportunities to continue the discussions. They were resumed during the next morning and stimulated by brainstorming discussions.

The contributions of the participants to the discussions, regarding open questions and research needs, can be summarized as follows:

- Characterisation of properties of native and modified/aged wood – e.g. mechanosorptive behaviour of heat treated wood, temperature dependence of sorption behaviour – is needed to assess the effect of treatments, or to find a treatment able to mimic aged wood.
- Finding a concept of constitutive equations appropriate during modification/degradation of wood is a fundamental challenge, associated with the need to develop specific techniques for characterisation of properties during the changes – e.g. heat treatments in a DMA allowing continuous monitoring of viscoelastic properties.
- A multiscale analysis is the key to understand the underlying phenomena and produce robust models; material characteristics should determined/predicted some from low level information –e.g. colour changes as result of chemical composition, creep modelled at molecular level, mechanosorption modelled at cell-wall level, effect of heat treatment explained by variations of the molecular structure and interaction, compression set explained either by cellular microbuckling or hygroscopic fatigue…

# List of oral contributions by participants

## 1. Experimental characterization

Structural and chemical changes during thermal and hygrothermal treatments – how to consider these?
*Lennart Salmén (SE)*

Influence of the THM processing parameters on the mechanical and chemical degradation of wood and size effect
*Navi Parviz (CH)*

Influence of thermal modification on mechano-sorptive creep, water diffusivity and on sorption characteristics of beechwood
*Ales Straže (SI)*

Why do we need to accelerate wood aging?
*Joseph Gril (FR)*

Near infrared spectroscopy as an experimental validation tool for characterization of chemical changes to wood during thermal treatment
*Jakub Sandak (IT)*

A water vapor sorption isotherm for thermally modified Douglas fir
*Wim Willems (NL)*

Use of dynamic mechanical analyse (DMA) data for the detection of thermal modification processes
*Robert Widman (CH), Andreja Kutnar (SI), Iris Brémaud (FR)*

Characterisation of a modifying material: mass, shrinkage and mechanical properties measured during heat treatment
*Patrick Perré, Giana Almeida (FR)*

## 2. Modelling

Numerical hygro-thermal modelling of wood during processes at high-temperatures
*Andrea Genoese, Alessandra Genoese (IT)*

Modelling service life of timber structures taking into consideration chemical alterations over time under permanent mechanical and weathering loads
*Wolfgang F. Gard*

Modelling of the hygromechanical couplings
*Frédéric Dubois*

The heat treatment of lignocellulosic material: modeling the coupling between heat&mass transfer and chemical reactions
*Romain Rémond (FR), Ian W. Turner (AU), Patrick Perré (FR)*

Modeling the compression set occurring during restrained swelling test
*Julien Colmars (FR), Paola Mazzanti*

Artificial aging versus natural aging of poplar wood
*Bertrand Marcon (IT, JP), Shuichi Kawai (JP), Miyuki Matsuo (JP, FR), Joseph Gril (FR), Luca Uzielli (IT)*

Natural ageing and heat treatment of lignocellulosic materials: evaluation and modeling
*Miyuki Matsuo (JP)*

Mild hygrothermal treatment to dissociate mechanical and hygroscopic ageing of wood
*Cécilia Gauvin, Delphine Jullien (FR)*

**Session 2**

# Finite element modeling of deformation of a fiber bundle during steam explosion of wood

**Muhammad MUZAMAL, Anders RASMUSON**

Department of Chemical and Biological Engineering, Chalmers University of Technology, Sweden
muzamal@chalmers.se

**Keywords:** Finite element model, fiber, steam explosion, elastic properties.

Steam explosion is an important wood pretreatment process in modern bio-refineries. Wood is difficult to digest by enzymes due to its compact structure. In steam explosion the wood is treated with high pressure steam for a certain time which makes the wood material soft and easily deformable. After processing, the wood is rapidly decompressed and vapors inside the wood fibers expand and create stresses on the fiber walls. These high stresses cause the wood structure to deform, break and thus open up. There is significant improvement in enzymatic digestion of the wood biomass due to steam explosion pretreatment [1]. Thus it is very important to study the mechanisms of wood deformation and breakage during steam explosion.

In this study a bundle of spruce fibers, composed of early wood and late wood fibers has been modeled using the finite element approach. The elastic properties of the fibers are calculated from the elastic properties of the basic wood components (cellulose, hemi-cellulose and lignin) at 12% moisture content and 20 $^{O}$C. The composition, thickness and micro-fibril angle (MFA) for spruce fiber layers have been taken from the literature [2]. The Halpin-Tsai equations and mixing rules are used to calculate the elastic properties of the fiber layers [3]. Since the steam explosion process is carried out at high temperature and with saturated steam, an estimation of elastic properties at 20% moisture content and 80 $^{O}$C has also been made using the trend which shows how elastic properties of spruce decreases with temperature and moisture content [4,5].

The load is applied as uniform pressure of 10 bar inside the fibers. Figure 1 shows the maximum principal strain in the deformed fiber bundle. It can be seen that the expansion of vapor inside the wood fibers creates uneven stresses. These stresses are not effective in the radial direction, but very effective in the tangential direction. Early wood fibers, with larger cross-section are more affected than latewood fibers. These results are also seen during the steam explosion experiments performed by Zhang and Cai (2006). Simulation results with 20% moisture contents and 80 $^{O}$C shows much deformation in fiber as compared to results obtained from simulation at 12% moisture content and 20 $^{O}$C.

Further studies are required to improve the model. These include determination of the elastic properties at high temperatures and extending the model to account for strength properties and breakage.

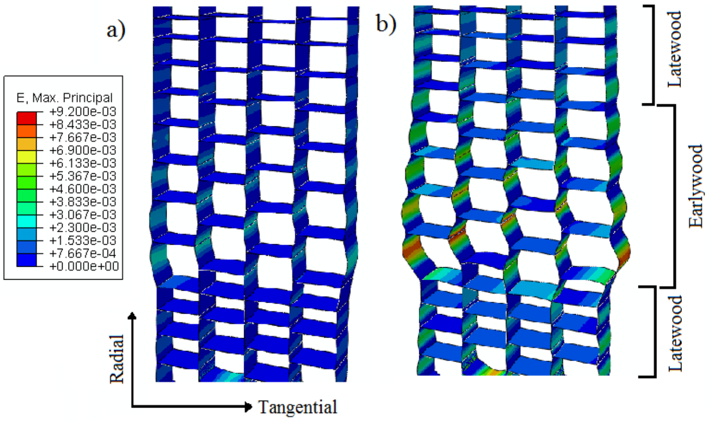

Figure 1. Max principal strain in fiber bundle at (a) 12 % moisture content and 20 $^{O}$C (b) 20% moisture content and 80 $^{O}$C

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**Session 2**

# Effects of closed system hydrothermal treatment conditions on colour and hardness of European beech wood

**Charalampos LYKIDIS[1], Petros KONSTANTAKOS[2], Stavros TSALIKIS[2]**

[1]Hellenic Agricultural Organisation "Demeter" Inst. of Mediterranean Forest Ecosystems and Forest
Products Technology, Lab. of Wood Anatomy and Technology,
Terma Alkmanos, 11528, Ilisia, Athens, Greece
[2]Tech. Educ. Inst. (TEI) of Larissa, Dept. of Wood & Furniture Design & Technology
11 V. Griva Str., 43100, Karditsa, Greece
lykidis@fria.gr

**Keywords:** wood, beech, hydrothermal treatment, colour, hardness

Hydrothermal treatment (steam-heat treatment) has been known for a long time as one of the most effective methods to balance colour differences and to improve the dimensional stability, the decay resistance and the durability of wood [1] but also as a useful tool in emerging methods of wood densification [2]. Along with the advantages, hydrothermal treatments are usually accompanied by intensive changes in the chemical composition and properties of wood, consequently increasing the interest in such information. Among other modifications, hydrothermal treatments result in colour changes [3, 4, 5] as well as degradation of all strength properties of wood [6]. Colour is determined non-destructively while mechanical properties usually require destructive methodologies in order to be evaluated. Hardness is related to other mechanical properties of wood and requires small specimens in order to be determined. For this reason, it is considered as one of the main indices of wood quality [7].

In an attempt to attain a relationship between treatment parameters (temperature and time) and colour as well as hardness of European beech (*Fagus sylvatica L.*) sapwood, laboratory-scale hydrothermal treatments were carried out in this study. Six hundred defect-free specimens with dimensions of 40mm x 40mm x 22mm were prepared from air-dried sawn boards which were climatised at normal climate (20°C, 65% RH) for at least 4 weeks. In order to facilitate hardness comparison between the treated and non treated ones, specimens were cut in pairs as explained in other works [8]. The specimens were subjected to hydrothermal treatments at 110, 140, 170 and 200°C for 10, 30, 60, 120 and 240min. The treatments were carried out in a 1,2l stainless steel reactor with steam temperature range of ±1°C. The treated and untreated test samples were then climatised at 20°C and 65% RH and were tested for the determination of Brinell hardness according to EN1534:2000 as proposed by Niemz and Stübi [9] using a Zwick 2020 Universal Testing Machine. The surface colour of all specimens was measured with a BYK Gardner tristimulus colourimeter with a 45/0 measuring geometry, circular measuring area of 20 mm in diameter, using a D65 illuminant and a 10° standard observer.

The results showed that hydrothermal treatments at temperature of 110-200°C resulted in a decrease up to 54,6 for $\Delta E^*$ and up to 60,6% for hardness. The higher treatment temperatures and the longer treatment times resulted to more intense changes. $\Delta E^*$changes at 110°C in the range of 10-240min followed logarithmic increase while at higher temperatures showed a more linear one. Moreover, multiple comparisons with one-way ANOVA (LSD, $\alpha$=0,05) showed that treatments at 110°C resulted to statistically significant (although marginally detected by the human eye) $\Delta E^*$ values for times up to 30min. The further increase of treatment time from 30 to 60, 120 and 240min did not cause significant further colour alteration. It was also found that treatment of 10min at 140°C resulted to the same ΔE* with treatments of 60, 120 and 240min at 110°C. Treatments at temperatures of 140, 170 and 200°C resulted to significant ΔE* values among all treatment intervals. It is obvious that the effect of hydrothermal treatment duration is milder than the effect of temperature. This effect seems stronger at higher treatment temperatures. The ΔE and L* values presented a severe reduction when the treatment temperature was 200°C and the duration was longer than 60min. The above specimens also showed high emissions of thermal decomposition products.

After similar statistical analysis, the hardness of the specimens showed significant reduction for hydrothermal treatments at 140°C and with durations longer than 60min but did not show significant change when duration increased from 120 to 240min. The above remarks can also be drawn from the related graph. Treatments at temperatures higher than 140°C induced more intense and duration dependent deterioration of wood hardness.

2-way ANOVA for independent samples showed that there is a significant influence of treatment temperature, treatment duration and interaction of both factors on the mean colour change ΔE* and hardness of the specimens. Lightness (L*) and ΔE* showed significant correlation (r=0,794 and r=-0,824 respectively, at the 0,01 level, 2-tailed) while the other colour parameters (a* and b*) were weakly to moderately correlated (r=-0,157 and r=0,489 respectively, at the 0,01 level, 2-tailed) to the hardness of the specimens.

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**Session 3**

# Adhesion of polyurethane coating on surface densified Scots pine wood

**Kristiina LAINE[1], Lauri RAUTKARI[2], Borut KRIČEJ[3], Matjaž PAVLIČ[3], Sergej MEDVED[3], Marko PETRIČ[3], Mark HUGHES[1], Andreja KUTNAR[4]**

[1]Aalto University, Department of Forest Products Technology, Finland
[2]Edinburgh Napier University, School of Engineering and the Built Environment, Joint Research Institute for Civil and Environmental Engineering, Forest Products Research Institute, Edinburgh, UK
[3]University of Ljubljana, Biotechnical Faculty, Department of Wood Science & Technology, Ljubljana, Slovenia
[4]University of Primorska, Andrej Marušič Institute, Koper, Slovenia
kristiina.laine@aalto.fi

**Keywords:** adhesion, Scots pine, polyurethane coating, surface densification

Since one of the intended uses for surface densified wood is for flooring, the characteristics of coatings on surface densified wood should be examined. Therefore, the aim of this study was to investigate the interactions between surface densified pine wood and commercially available solvent-borne polyurethane (SPU) coating for wooden flooring.

Specimens of kiln dried Scots pine (Pinus sylvestris L.) (150 mm in length (L), 55 mm in width (T) and 22 mm in thicknesses (R) that were conditioned at 20 °C and 65 % relative humidity (RH) to constant weight were surface densified using a specially designed heated press tool fitted to a universal testing machine (Zwick 1475) combined with an MTS Premium Elite controller. More details of the test set-up and densification process are given in [1]. The specimens were compressed from 22 mm to a target thickness of 15 mm in the radial direction, giving a compression ratio of 32 %. After conditioning, the surfaces of the densified and control specimens were manually abraded with 180 grit sandpaper and then coated with a commercial solvent-borne polyurethane wooden flooring coating (SPU; AMAL 4002 1K; AMAL d.o.o., Ljubljana, Slovenia). Coating was applied to the surface in two layers following the manufacturer's instructions; 0.6 to 0.7 g per surface was applied and the drying time prior to the second coating was 20 hours.

Adhesion of the coating to the modified surface was determined by the pull-off test described in [2]. The test dolly was glued using an epoxy adhesive to the coatings of seven specimens in each group. After 24 hours, the cured epoxy adhesive and the coating were cut to the substrate around the circumference of the dolly and the test conducted using a DeFelsco PosiTest AT Adhesion Tester. A tensile stress was applied at an increasing rate not greater than 1 MPa/s until the dolly was pulled-off. The breaking strength (in MPa) and the nature of the fracture were recorded.

The results of the pull-off test are presented in Table 1. It must be noted that the adhesion results given in the table should be considered very carefully because during the pull-off test there was an increase in the proportion of cohesive failure observed in the substrate of densified wood. To illustrate this, cohesive failure amounted to 79 % in pull-off tests on the pine control wood and this rose to 87 % in the case of the surface densified specimens. An increase in the proportion of cohesive failure in the densified substrate suggests that there is a decrease in the tensile strength of surface densified wood most probably due to cell fracture during densification. When a high portion of failure is cohesive, adhesion is not measured, but rather the cohesive strength of the substrate. In such cases, it can only be stated that the adhesion is higher than the value determined experimentally. The greatest adhesion, at 4.70 MPa, was observed in the pine wood control specimens coated with SPU. Although the observed adhesion of the coatings on the surface densified pine wood was significantly lower than on the control undensified specimens, the values determined are above the usual minimum requirements which a surface system should withstand under normal use. This minimum requirement, based on experience, was set to be 2 MPa. To conclude, the observed decrease of adhesion of coatings on surface densified wood, as measured by the test method employed, could be a combination of the ‘apparent’ adhesion, which reflects the cohesive failure of the substrate, and the ‘real’ adhesion which is a consequence of changes in the surface energy and topography following densification.

Table 1. Average adhesion and amount of cohesive fracture of the control undensified and surface densified pine specimens with the SPU coating

| Specimens | Adhesion [MPa] | Cohesive fracture [%] |
|---|---|---|
| Control | 4.70 | 79 |
| Surface densified | 2.57 | 87 |

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**Session 3**

# The Behaviour of Sorption Hysteresis in the Water Vapour Sorption Isotherm of Thermally Modified Wood

**Callum HILL[1], James RAMSAY[1], Lauri RAUTKARI[2], Mark HUGHES[2], Kristiina LAINE[2]**

[1]Forest Products Research Institute, Edinburgh Napier University, Merchiston Campus,
Edinburgh, EH10 5DT, UK
[2]Aalto University, School of Chemical Technology, Department of Forest Products Technology,
Espoo, Finland
c.hill@napier.ac.uk

**Keywords:** sorption, isotherm, hysteresis, glass transition temperature, thermal modification

The water vapour sorption behaviour of Scots pine (*Pinus sylvestris* L.) and Scots pine that was densified, thermally modified, or subjected to a combination of thermal modification and densification has been investigated. It was found that all modifications resulted in a decrease in the equilibrium moisture content (EMC) of the wood samples throughout the hygroscopic range. The water vapour sorption isotherms were reproducible for the unmodified wood samples, but changed between the first and subsequent sorption cycles for the densified, thermally modified and for wood subjected to a combination of the two treatments. This is the first time that changes in the sorption isotherm between the first and subsequent cycles have been reported for thermally modified wood.

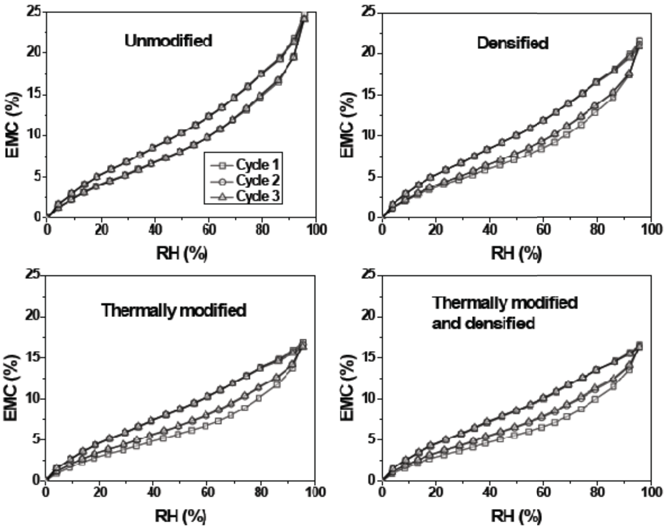

Figure 1. Changes in sorption isotherms between the first and subsequent cycles

Irrespective of the wood treatment the difference between the adsorption and desorption isotherm loops (sorption hysteresis) was the same and greater than that observed for the unmodified wood sample. After the first sorption cycle, the hysteresis decreased to the values observed for the unmodified wood, even though the isotherms were different (1).

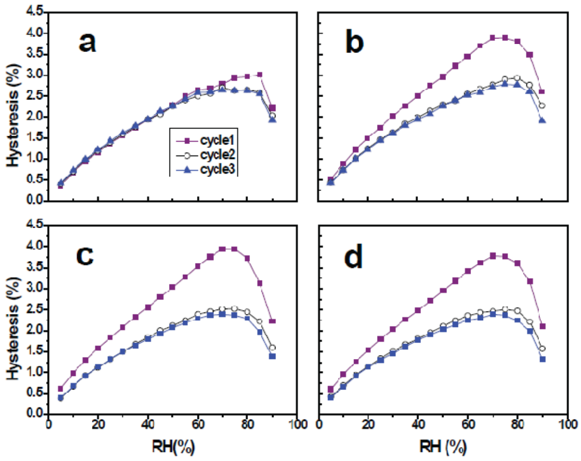

Figure 2. Changes in hysteresis between first and subsequent cycles for (a) unmodified, (b) densified, (c) thermally modified and (d) thermally modified and densified wood

The sorption kinetic behaviour was also investigated and found to be accurately described using the parallel exponential kinetics (PEK) model. The PEK model describes the dynamic sorption behaviour in terms of a fast and slow kinetic process and this has been interpreted in terms of two Kelvin-Voigt elements coupled in series (i.e. relaxation-limited kinetics). Data for the modulus calculated using this method is shown below.

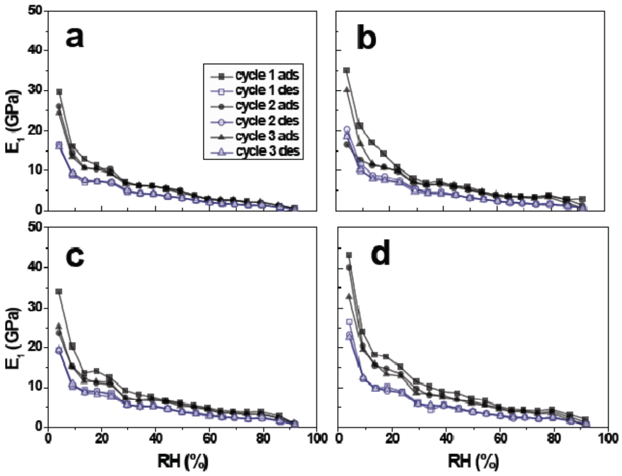

Figure 3. Changes in modulus calculated from the Kelvin-Voigt interpretation of the sorption kinetic data (letters are the same as in Figure 2)

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**Session 3**

# Effect of treatment medium on the colour change of heat treated wood during natural weathering

**Róbert NÉMETH[1], László TOLVAJ[2], Miklós BAK[1], Diána CSORDÓS[1]**

[1]University of West Hungary, Faculty of Wood Sciences, Institute of Wood Science,
Sopron, Hungary
[2]Institute of Physics, Sopron, Hungary
nemethr@fmk.nyme.hu

**Keywords:** paraffin, linseed oil, CIELab, weathering, heat treatment

The wood material is often preferred in outdoor using, nevertheless because of its aesthetic value. The appearance of wood is composed from the texture and the colour. But the latter one can be changed during the service life of the product due to exposure to sunlight, rain and temperature changes. The consequence of outdoor exposure is the well known greying effect. In order to prove the applicability of a material for using under outdoor conditions, weathering tests are recommended. The heat treatments in linseed oil (OHT) and in paraffin (PHT) modify the colour of the wood favourable, but the stability of the colour is a major factor of a product's value, thus it calls for special emphasises. The heat treatment procedure in paraffin bath and its effect on different material properties is given by Németh et al [1].

In the presented research work Pannonia Poplar (*Populus* × *euramericana cv*. Pannonia) wood was investigated. This wood species is wide spread in Hungary, with considerable shares in the forests. Poplar is one of the most important plantation grown species in Hungary, and considering the increasing area of new plantations their importance will even increase in the future [2]. The main target of this research work was to compare the colour stability of OHT, PHT and natural timber under outdoor conditions without coatings. The OHT treatment and its influencing effect on the material properties were described by Bak & Németh [3]. The colour change due to thermal treatment and due to exposure for different hardwood species was published [4-8].

Initial colour of OHT and PHT wood was similar, however yellow hue (b*) of PHT wood was significantly higher than by OHT wood. The lightness (L*) of the treated samples showed minor increase after the first month, and decreased continuously in the subsequent period. The initially different L* values (as the consequence of different treatment schedules) were equalized until the 12th month. Both of OHT and PHT samples darkened at the end of the experiments, but higher treatment temperature and longer durations resulted in less darkening. Significant differences could be found in terms of change in lightness between OHT and PHT wood. Namely, lightness of OHT wood decreased more than lightness of PHT wood during weathering. The lightness of the untreated samples decreased permanently, but it remained squarely higher as the lightness of the treated samples.

The red hue (a*) of the heat treated samples decreased continuously during the whole period. The untreated material underwent a slight decrease in this property; however it' starting value was very low before weathering. At the end of the experiment (after one year) a* values of the treated and the untreated samples got closer to each other, but the treated ones still had stronger reddish character. Significant differences could be found in terms of change in red hue between OHT and PHT wood. Namely, red hue of OHT wood decreased more than red hue of PHT wood.

After a slight increase during the first month the yellow hue (b*) of the treated samples declined remarkably and continuously. The measured differences in the initial b* values (different treatment schedules) were equalized at the end of the exposure time. At the end of the 12th month differences were diminished between treated and untreated samples in terms of yellow hue. The decreas in b* values of OHT and PHT wood at 160°C were similar, but heat treatment at 200°C resulted in different change of the yellowish character in case of oil and paraffin. In case of 200°C treatment yellow hue of PHT wood decreased more pronounced compared to OHT wood.

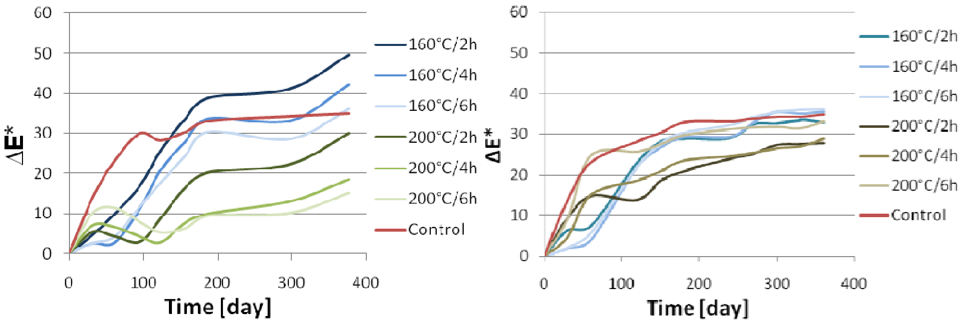
The ΔE* values of all samples increased continuously during the investigated period. By the end of the 3rd and 4th month of exposure time, in case of untreated and OHT wood at 160°C the differences exceeded the significant, with naked eye well visible value of 12. Whereas the OHT wood at 200°C only exceeded the value of 12 in terms of ΔE* after 7-10 months (Fig. 1/a). Similar tendencies could be obtained in terms of PHT wood; however some differences could be observed (Fig. 1/b). Because of the lower decrease in lightness and red hue, total colour change of PHT wood is smaller in by the 160°C treatments. But in case of the treatments at 200°C, total colour change of OHT wood is smaller, because of the minor decrease in yellow hue. In case of both OHT and PHT it is statable, that material treated at lower (160°C) temperature underwent more sever total colour changes compared to the higher temperature (200°C).

Different treatments (temperatures and durations) resulted in different colour values. These initial colour values changed during outdoor weathering remarkably. Regardless the applied treatment, the initially different colours became similar by the end of the exposure time. The colour of the untreated samples drew near to the treated ones, thus due to longer exposure time the differences likely will be diminished. This result calls for colour protection in case of applications where the colour is important.

An interesting result for Poplar was found, as the higher (200°C) treatment temperature resulted in lower total colour change values, compared to the lower temperature (160°C) due to weathering. This behaviour of Poplar can be explained by the low extractive content.

The colour changes of the treated samples did only exceed the changes of the untreated materials in case of 160°C treatment during the investigated period. It means that the colour stability of treated material is depending on the treatment parameters, mainly on the temperature. Higher treatment temperature is favourable for outdoor application however on long term colour protection is needed for aesthetical applications. By 160°C treatments PHT suffered less colour change during weathering, but by 200°C treatments OHT resulted in better colour protection. This result shows the importance of the treatment medium well.

Further investigations are running in order to evaluate the checks and cracks due to long term outdoor exposure. The results are promising, as no cracks could be observed on the surface of the treated samples so far.



a. b.

Figure 1. Total colour change (ΔE*) of OHT (a) and PHT (b) wood during 1 year of natural weathering

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**Session 3**

# Experimental and numerical investigations of mechanical properties of welded joint using the Arcan setup

**Martin RHÊME[1], John BOTSIS[2], Joël CUGNONI[2], Parviz NAVI[1]**

[1] Material and Wood Technology, Bern University of Applied Science, Solothurnstrasse 102,
CH-2500 Biel, Switzerland
[2]Laboratory of Applied Mechanics and Reliability, EPFL, LMAF-STI-EPFL, Station 9,
CH-1015 Lausanne, Switzerland
martin.rheme@bfh.ch

**Keywords:** Linear welding, Arcan test, Mechanical properties, Fibres orientation

Linear welding of wood is an assembly method based on friction welding technology. Simultaneous application of pressure and relative movement between two wood pieces, locally increases the temperature at the interface and enables molten material to flow between the pieces and therefore create a joint. It has been shown in some studies [1,2] that the mechanical performance of a welded joint is dependent on both welding parameters and wood fibres orientation.

A possible application for welded wood joint could be the fabrication of multi layers panels (for example cross laminated panels). In this type of assembly, the longitudinal directions of the different pieces are bonded in a perpendicular manner. Therefore it is of interest to characterize the mechanical strength and fracture properties of welded joints produced with wood pieces and fibres oriented at different angles.

The present work investigates the influence of, (1) the angle between the longitudinal direction of the welded pieces and, (2) the direction of the displacement in the plane of the weld. Furthermore, for a complete characterization, tensile, shear and mixed loads are investigated. For this purpose, the Arcan test is of particular interest because it enables to characterise several experimental conditions with the same setup. As mentioned by Valentin et al. [3], the Arcan test is also suitable to investigate fracture properties. An example of the Arcan setup used in this work with welded specimens is presented in Figure 1.

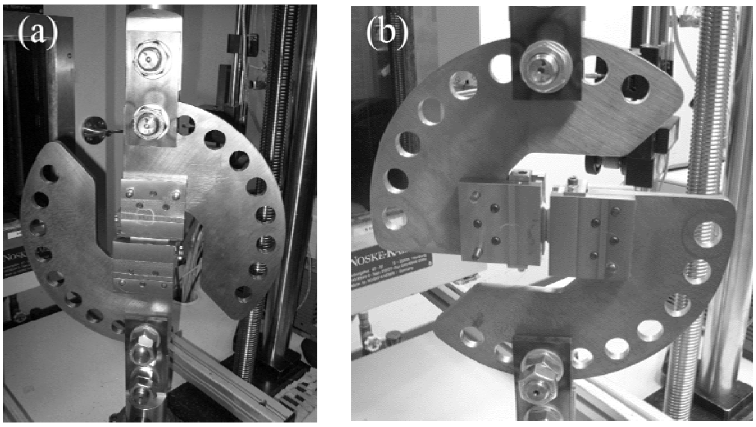

Figure 1. Experimental setup of the Arcan test. (a) in tension and (b) in shear.

In a first step, the test conditions are simulated with a finite element (FE) model for all configurations. The goal is to investigate the influence of the geometry and the possible out of plane effect due to the layers at different angles. The model takes into account the cylindrical orthotropic nature of wood and the orientation of the fibres. In a first approximation, the welded joint is simply modelled as a tied interaction between the surfaces of the two wood pieces. The total height of the specimen is set to 20 mm and its width to 10 mm.

The second step consists of producing the appropriate specimens and experimentally determines the mechanical strength of the welded joints in shear, tension and in an intermediate mixed mode. Fracture characteristics are also tested with the same experimental setup and specimens containing an initial crack.

Figure 2 gives an overview of how the geometry of the specimen influences the distribution of the shear stress on the interface. More precisely, the profiles along the middle of the surface are represented as a function of the specimen's shape. The stresses are relative to the maximum value in the middle of the profile. The results show that the introduction of grooves on the side of the specimen (parallel to the thickness direction) leads to more homogeneous stress distribution along the surface. Similar investigations show that in the case of tension testing, no groove is necessary to achieve a sufficient homogeneous profile of tensile stresses on the interface.

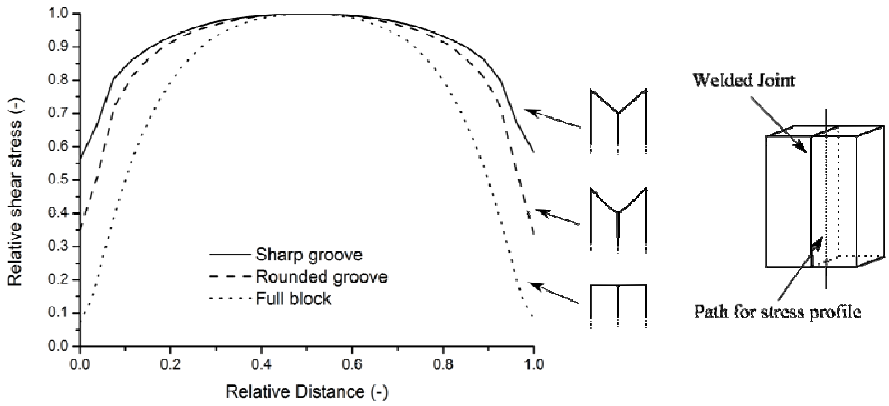

Figure 2. Simulated shear stress profile according to different groove shapes. The stresses and distance values are normalized.

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## Session 3

# Preliminary tests of combined steaming and thermal-vacuum treatment on Turkey oak (*Quercus cerris* L.) wood

**Silvia FERRARI[1], Luigi TODARO[2], Ignazia CUCCUI[1], Ottaviano ALLEGRETTI[1], Mario MARRA[1]**

[1]CNR-IVALSA, via Biasi 75, 38010 San Michele a/A. Trento, Italy
[2]University of Basilicata, V.le Ateneo Lucano 10, 85100 Potenza, Italy
ferrari@ivalsa.cnr.it

**Keywords:** Turkey oak, steaming, thermo-vacuum process

Turkey oak is a wooden species widely distributed in South-Eastern Europe and in Italy is mainly present in the Apennine Mountains. Compared to the other oaks, T. oak is less appreciated because of its easy tendency to crack, its lower technological quality and lower durability. The aim of the present work was to improve wood quality by treating T. oak through combined steaming and thermal treatment in vacuum conditions.

Two green logs were initially steamed in a vessel at 110° C for 24 hours and then cut into boards 32 mm thick. Steamed and also not steamed boards were then loaded into the dedicated machine in order to perform drying and thermal modification under low pressure conditions (200-250 mbar). The thermo-vacuum process, which has been already described in previous works [1], is characterized by a convection heat transfer and can be defined as an open system because all the volatile products of the process are continuously removed from the kiln by means of a pump which keeps the set pressure. Wood was dried until the 0% of moisture content and then it was thermally treated at 160°C for three hours.

Mass loss (ML), equilibrium moisture content (EMC) and color change (ΔE) were measured on treated and control material (not steamed and not treated), distinguishing between sapwood and heartwood. EMC was calculated at normal conditions, *i.e.* temperature (T) of 20°C and relative humidity (RH) of 65%.

The average ML value, as consequence of the thermo-vacuum treatment, was around 0.8 ± 0.3%. Concerning EMC, results are reported in table 1.

Table 1. EMC for T. oak sapwood and heartwood. ST= steamed; NST=not steamed; TT= thermally treated; NTT=not thermally treated

| Treatment combinations | EMC ± s.d (%) | ΔEMC (%) | EMC ± s.d (%) | ΔEMC (%) | $ID_{hyg}$ |
|---|---|---|---|---|---|
| | sapwood | | heartwood | | |
| Control | 11.39±0.05 | - | 10.40±0.08 | - | 6.95 |
| ST-NTT | 10.80±0.09 | -5.17 | 10.41±0.25 | -0 | 3.96 |
| NST-TT | 10.78±0.1 | -5.35 | 7.83±1.16 | -24.71 | 37.68 |
| ST-TT | 10.43±0.08 | -8.42 | 9.19±0.60 | -11.63 | 13.49 |

Steaming and thermal treatment significantly influence wood EMC by reducing wood affinity for water, particularly when both treatments occur. Sapwood and heartwoods behaved differently because the latter showed an EMC reduction more variable and higher than the first, *i.e.* -11.63 % and -8.42%, respectively in case of combined treatments. In table 1 is also reported the index $ID_{hyg}$ given by the ratio of the difference between the EMC of sapwood and heartwood and the EMC of heartwood. The index is an indicator of the material homogeneity in terms of hygroscopicity. The index decreased for steamed wood but increased significantly after thermal treatment determining a less homogeneous material compared to control.

Color of treated wood became darker, mainly because of the steaming process. In fact, the moist T. oak wood is affected by significant microstructural changes when it is treated with steaming processes already starting at moderate temperatures (100–120° C) [2].

Even the thermal treatment contributed to the darkening of the material, but with a minor effect compared to steaming, probably because of the low temperature adopted (160° C).

As consequence the *ΔE* value of not steamed material, calculated before and after the thermal treatments, is higher than the one calculated for steamed wood as shown in table 2.

Moreover the two treatments improved the color homogeneity of wood between sapwood and heartwood as already reported by Tolvaj *et al.* [3]. *ΔE,* calculated between sapwood and heartwood, shifted for not steamed material from 14.8 to 8.9 for not thermally treated and thermally treated material respectively, while *ΔE* remained constant at a value of 3 for steamed wood even if thermally treated.

Table 2. Color coordinates for sapwood (sap) and heartwood (heart)

| | Steamed material | | | | Not steamed material | | | |
|---|---|---|---|---|---|---|---|---|
| | Not therm.treated | | Thermally treated | | Not therm.treated | | Thermally treated | |
| | sap | heart | sap | heart | sap | heart | sap | heart |
| L*±sd | 60.1±1.3 | 57.9±2.1 | 53.1±0.6 | 50.9±0.9 | 76.8±1.0 | 62.3±1.4 | 63.6±2.0 | 54.3±0.9 |
| a*±sd | 9.9±0.3 | 8.5±0.5 | 9.9±0.2 | 7.5±0.3 | 5.6±0.5 | 7.0±0.2 | 7.7±0.6 | 7.5±0.3 |
| b*±sd | 19.6±0.7 | 18.0±1.0 | 18.5±0.3 | 16.6±0.8 | 21.5±1.0 | 19.3±0.8 | 22.7±0.9 | 19.9±0.3 |
| ΔE±sd | | | 6.6±0.6 | 7.0±0.6 | | | 14.0±2.4 | 9.3±0.9 |

The present work demonstrated that dimensional stability and color homogenisation of T. oak can be improved through steaming and thermo-vacuum treatment keeping at the same time the wood quality (no cracks or collapse were noticed). Future work will seek to thermally modify T. oak at temperature above 160°C with the aim of further improving technological characteristics and durability of this species.

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**Session 4**

# Challenges using dielectric heating for THM processing of solid wood

**Jimmy JOHANSSON, Lars BLOMQVIST, Dick SANDBERG**

Linnaeus University, SE-351 95 VÄXJÖ, Sweden
jimmy.johansson@lnu.se

**Keywords:** bending, beech, plasticizing, thermo-hydro-mechanical processing

A dielectric heating technique for solid wood bending, i.e. for heating, plasticizing and drying the wood to be bent in one step, is presented and the challenges to use this technique in practise is discussed. The main purpose to use dielectric heating is to decrease the time of the bending process. In practise this means reducing the time to bend and dry a straight piece of solid wood from a moisture content of about 25 % to 6-8 % from about 3 days to about 10 minutes. Plasticizing wood through dielectric heating for shaping wood has been studied for several years [cf. 1-7]. The theoretical approach is well documented by Torgovnikov [8]. Navi and Sandberg [9] have in their recent work described thoroughly a process for bending solid wood by using dielectric heating.

The short processing cycles gives high demands on controlling the moisture content, temperature and the strain fields that occur in the wood during processing. Figure 1 shows the temperature and moisture content in beech wood during a bending process (mean values for 21 wood pieces). The temperature was measured at the centre of each piece. Figure 1 also shows the rejection level of curved pieces, i.e. curved pieces that do not satisfy the requirements in bending radius or are not free from damage, cracks etc. The graph shows the rejected proportion as a function of time. During the heating stage, the rejection is by definition 100 % as the pieces are not yet curved. During the bending stage, the temperature is almost constant and the moisture content drops rapidly. At the end of this stage the moisture content is about 10 %. If the process were stopped at this time, the rejection proportion would be 100 % as a consequence of spring-back of the curved pieces.

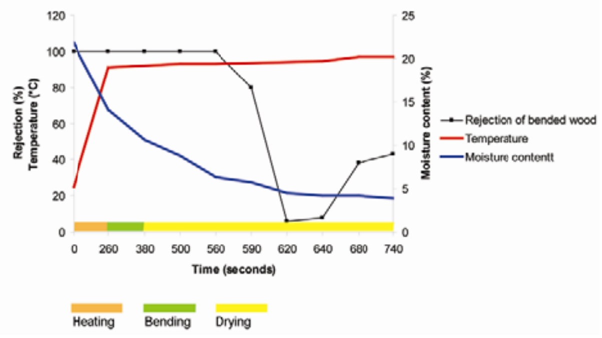

Figure 1. Temperature, moisture content and rejection as a function of time during the bending of solid beech wood using dielectric heating.

During the drying stage, the moisture content is lowered to a level where the curved shape is fixed at the required radius. In Figure 1, it is clear that the moisture content falls as long as the drying continues until no or a low amount of water remains in the wood. The temperature rises from 95°C to about 98°C during 360 seconds. The rejection falls from 100 % to about 5 % during a time period of about 60 seconds. The process should be stopped in this time period, to obtain a low rejection of wood pieces. If the drying continues, the rejection level of bended pieces increases again, and when the bending process is stopped, as in the example in Figure 1, the rejection rate is as high as 40 %. The curves in Figure 1 show that there is a relatively short time span during which the process should be stopped to ensure a low rejection rate of the bended pieces and not too low moisture content. The optimal processing time is of course dependent on the energy input into the wood.

Different types of damage that may occur in the wood during the bending process is related to rupture of cell walls if the steam generated within the cells of the wood finds difficulty in escaping, flash-over in the dielectric field, tensile or compressive rupture related to incomplete softening of the wood or to the structure of the wood (deviating fibre orientation, knots etc.).

**Conclusions:**

Dielectric heating technique can be used for plasticizing and drying wood in a single sequence high-speed solid wood bending process (open system). However, a good control on the moisture content of the raw material is needed and the equipment for bending must be designed to prevent flash-over in the electromagnetic field and to prevent steam in the wood from rupturing the cell walls.

To reach a low rejection level in the bending process, the process parameters, i.e. input energy and time for the different processing stages, have to be well controlled since the time-span for optimal bending is very small.

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**Session 4**

# Influence of thermo treatment on wood physical-mechanical properties and VOC and aldehyde emissions

**Regula MOSER[1], Thomas LORENZ[2], Philipp HASS[2], Frédéric PICHELIN[1],**
**Thomas VOLKMER[1], Urs von ARX[1], Peter NIEMZ[2]**

[1]Bern University of Applied Sciences, Architecture, Wood and Civil Engineering,
Solothurnstrasse 102, 2500 Biel, Switzerland
[2]Swiss Federal Institute of Technology Zurich ETH, Institute for Building Materials,
Schafmattstrasse 6, CH 8093 Zürich
regula.moser@bfh.ch

**Keywords:** thermo treated hardwood, ammoniac treated hardwood, volatile organic compounds, formaldehyde, sorption, elasticity modulus, bending strength

Thanks to its darker colour fumigated wood is highly demanded in the flooring industry. Thermal treated wood becomes more and more important as an alternative to tropical wood species. During the manufacture process for fumigated wood, wood is fumed with ammoniac steams during one week. Thus, this process consumes a lot of energy and exhaust ammoniac steams. The production in Europe is currently around 200’000 m$^3$per year [1]. Moreover, the production of thermal treated hardwood increases steadily. The main hardwood species used for thermal treatment are beech, ash and oak but also cotton wood and maple. The thermal treatment raises interest due of its resulting darker colour, which offers excellent opportunities for furniture and flooring.

In this work we investigated oak, which was thermal treated using two different, but common procedures: on the one hand, the oak wood was heat-steamed in an autoclave (< 2 bar and 120 -140 °C), on the other hand, the oak wood was fumigated with ammoniac at room temperature. The process at high pressure and temperature can be more environmental friendly as the fumigated wood as only water is needed.

The oak wood was investigated regarding the influence of the treatment method on the Brinell hardness, the porosity and the colour after treatment and the colour stability after artificial and outdoor weathering.

In terms of the porosity, the heat treatment showed slightly increased values, compared to the untreated samples and the ammoniac fumigated ones. This increase results from the slight thermal modification of the autoclave treated wood and is well known for heat treated wood in general. The results of the Brinell hardness showed no influence of the two types of treatment. The small differences in the mean values, which could be observed, originate mainly from the density differences between the three groups. With the autoclave treatment, the color of the oak wood could be darkened to a large extend and almost reached lightness and yellow values like the fumigated oak. Also the color stability of the autoclave treated wood and the fumigated oak behaved similar and differed from the untreated samples. This indicates that the treatment method in the autoclave could be an environmentally friendly alternative modification practice to the fumigation with ammoniac.

Another important topic which was investigated was the material emissions of treated oak wood. Government regulations require more and more that the total volatile organic compounds emissions (TVOC) are lower than 1000 µg/m$^3$ in the interior air.To evaluate the measured results, it is necessary to work with tools, made from toxicologists. The AgBB-Scheme in Germany is for example a common tool to assess the VOC emissions of a material. The AgBB-Scheme and also some labels claim also limits for single substances of the VOC emissions.

The VOC emissions were measured according to DIN ISO 16000, which includes the sample preparation, the emission chambers and the measurement of VOC by gas chromatography with mass spectrometry (GC-MS) and the aldehydes by liquid chromatography and UV-detection (HPLC-UV). Both, the heat steamed and the ammoniac steamed oak wood were put into the emission chamber, where two samples were taken on the third and on the 28th day after introducing the products into the emissions chambers.

Heat-steamed oak and ammoniac steamed oak do not differ significantly in the total VOC emissions. After 28 days the TVOC of heat-steamed oak is 323 µg/m$^3$ and of ammoniac steamed oak it is 334 µg/m$^3$ (Figure 1). However if the two procedures are compared by single components, a clear difference is visible: The main single component of heat steamed oak is furfural with a concentration of 186 µg/m$^3$ after 28 days in the emission chamber. Furfural is a well-known degradation product of hemicellulose in acid conditions [2]. The most important emission of a single compound in the ammoniac steamed wood is acetamine with a concentration of 159 µg/m$^3$ after 28 days. Acetamine as an emission of wood is known and discussed before [3].

The formaldehyde concentrations of both wood treating procedures are lower than 5 µg/m$^3$. During the treating process, no formaldehyde is produced.

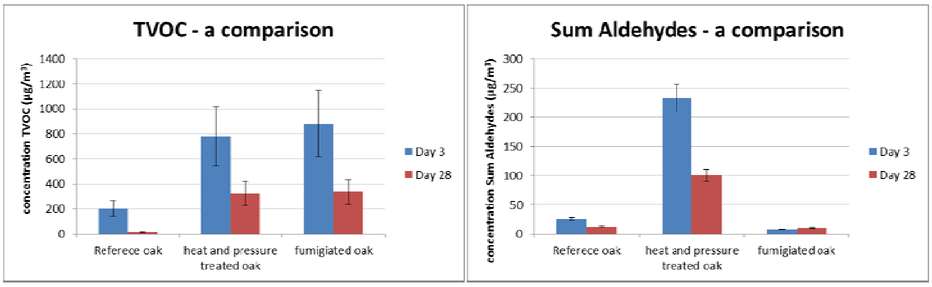

Figure 1: TVOC concentration of the three different samples according to DIN ISO 16000-6 (left) and the sum of the aldehyde concentration according to DIN ISO 16000-3 (right)

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**Session 4**

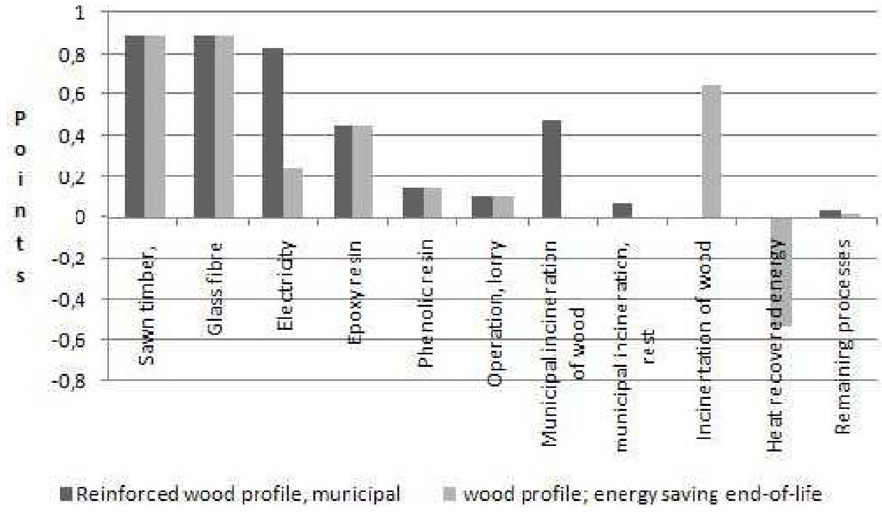
# Structural, economic and environmental performance of fibre reinforced wood profiles vs. solutions made of steel and concrete

**Cristoph MANTHY[1], Edeltraut GUENTHER[1], Andreas HEIDUSCHKE [2], Peer HALLER[2]**

[1]Technische Universität Dresden, Chair of Business Admin. – Env. Management and Accounting, Germany
[2]Technische Universität Dresden, Institute for Steel an Timber Structures, Germany
peer.haller@tu-dreden.de

**Keywords:**

The purpose of this paper is to evaluate the ecological performance of moulded fibre-reinforced wood profiles and to compare them with other building materials. The estimation is based on the concept of environmental Life Cycle Costing (eLCC). The moulded wood profile is a new product, the manufacturing of which is done in a therm-hydromechanical regime (Haller 2007). Fibre-reinforced wood profiles consist of such products reinforced by filament winding. The reinforcement copes with the drawbacks of wood such as low strength and durability and thus makes the product more applicable and reliable. Although wood has been proved to be a sustainable material in various researches, the use of resin and fibres has a negative effect on the environment. Therefore, the ecological performance of the entire compound is assessed. The load-bearing capacity, which is tested in compression, serves as the functional unit. Finally, the concept of life cycle assessment (LCA) is applied to evaluate the ecological performance. Here, the evaluation utlizes the Eco-Invent database to establish the flow diagram and the material inventory of the investigated object. Two methods, Eco-Indicator 99 (I) and CML, are adopted to create the LCA. Profiles from steel and concrete with the same load bearing capacity are designed according to the building code. Results of the LCA for moulded fibre-reinforced wood profiles are presented and compared to gluelam, steel and concrete. Data is taken from the Eco-Invent database. With the results from life cycle impact analysis environmentally sound products and processes can be engineered. Compared to Eco-indicator CML seems to be more applicable for products.With the existing knowledge and tools, it is possible to find the best environmental and economical solution for a specific building. In practice, however, these two criteria may be satisfied by different materials and solutions. It is important that design for life time becomes an accepted approach despite of the problems that are rather obvious. LCA will improve environmental awareness and will enhance innovation in the construction sector.



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**Session 4**

# Products meeting Needs: Applying Thermal Modification and Thermo-Hydro Mechanical Processing of Wood to its Fullest Potential

**Dennis JONES[1], Edo KEGEL[2]**

[1]SP Trä, SP Technical Research Institute of Sweden, Drottning Kristinas väg 67,
SE 114 28 Stockholm, Sweden
[2] Marketing Dept, Plato International BV, Arnhem, Netherlands
dennis.jones@sp.se

**Keywords:** Thermal modification, thermo-hydro-mechanical modification, marketing, business development, commercialisation of products

Whilst many paper on thermal modification (TM) and thermo-hydro-mechanical modification (THM) have dealt with technological advances, there has been limited consideration of market potential. Given the increasing trend towards commercialisation, this is a growing need for marketing to be considered. There is a need to critically assess why we are trying to develop new modification technologies, and what benefits can be gained from using those modifications already in the marketplace. These benefits can vary from country to country, even in some cases within regions in a country.

This paper will review many of the key parameters necessary for converting a good idea (in this case both TM and THM) to a commercial reality, including:

- Logistics – what is the need for the modification process;
- Benefits to be gained – how does the process increase the performance of the resulting material and/or product;
- Environmental considerations – such as an increased need for sustainable building, using where possible enhanced local resources, minimising risk of contamination as a result of leaching;
- Long term benefits gained – considering issues such as whole life costings, life cycle assessments;
- International marketing versus local marketing – developing niche markets in selected countries (giving examples). Corporate responsibility to address all these issues.

The overall aim of the paper will be an aid to future development and marketing of wood modification, and as an aid to the future will also consider some of the mistakes from the past, as well as come of the perceived hurdles for modified wood (such as cost). The paper will include several examples where some or all of these factors have been considered, corrected and applied. It does not aim to provide a ‘quick fix’ answer, and some of the conclusions may not apply to certain circumstances. It will also make suggestions on how to correct situations that have not been corrected so far.

# Short oral presentations

**STSM**

# Using Dynamic Mechanical Analysis (DMA) for fundamental understanding of thermo treatments of wood

**Andreja KUTNAR[1], Robert WIDMANN[2], Iris BRÉMAUD[2]**

[1]University of Primorska, Andrej Marusic Institute, SI-6000 Koper, Slovenia
[2]Empa, Swiss Federal Institute for Materials Science and Technology,
Structural Engineering Research Laboratory, CH-8600 Dübendorf, Switzerland
andreja.kutnar@upr.si

**Keywords:** DMA, mechanical behaviour, thermal modification, wood

Beech and spruce specimens were tested in Dynamic Mechanical Analysis (DMA) equipment in order to evaluate the possibility of integrating viscoelastic data into real time monitoring of a thermal modification process. Since wood is a composite polymeric system, application of techniques commonly used to characterize polymers may yield important fundamental information. The DMA as a function of temperature is a very useful tool as it provides simultaneously mechanical data, and indications of potentially occurring chemical modifications [1]. The aim of this study was to study with DMA the performance of thermally modified wood, which originates from complex changes at the molecular or microstructural level and degradation of hemicelluloses, lignin, and certain extractive compounds [2]. Therefore small wood specimens were thermally modified directly inside the DMA-equipment while, at the same time, real time mechanical and physicochemical information about the progress of the process was obtained. Specimens of beech (*Fagus sylvatica*) and spruce (*Picea* K. *abies*), 30 mm in longitudinal direction, 6 mm in radial, and 2 mm in tangential direction were used. Due to planed flatwise bending set-up the cutting of the specimens was made in such way, that the applied loading acted in radial direction and bending stresses resulted in longitudinal direction. DMA test were performed using the RSA III Rheometrics System Analyzer, which offers a rapid and sensitive means to simultaneously obtain an elastic modulus (stiffness) and mechanical damping (toughness) for materials. The dynamic temperature step method with 3-point bending test was used to test the specimens in bending. The distance between the support was 25 mm. DMA temperature scans were performed in a temperature range 50 to 250°C. A heating rate of 10°C/min with 1 Hz frequency and strain 0.02 were used for most DMA testing to obtain the storage modulus E', loss modulus E'', and loss tangent tanδ of the specimens. Under the same frequency and strain, DMA isothermal tests were performed at five temperature levels (200, 210, 220, 230, 240, and 250) for 2.5 hours.

The influence of temperature and time on the mechanical behavior of spruce and beech specimens showed similar relations. The results revealed that when the temperature is increased the free volume of the chain segment of the polymers in wood increases and consequently its ability to move in various directions. As the temperature continues to increase, the localized bond and side chain movements and further whole main chains can occur, resulting in a decrease in storage modulus. When the predefined target temperature level was reached, the storage modulus E' continued to increase with time, approaching a steady state value, for specimens tested at 200 and 210°C. In specimens tested at 220 and 230°C the increase was followed with short steady state and then decreased storage modulus E', which was more pronounced at 230°C. At higher temperatures, 240 and 250°C, the initial increase was followed with rapid decrease of a storage modulus E'. The decrease of the storage modulus at temperatures 220 to 250°C as the time is prolonged, might be due to the movement of some small groups attached to the main polymeric components, including hemicellulose, lignin, and cellulose, and breakdown of polymeric structures of the wood specimens, which was confirmed by the mass loss of the specimens during the DMA testing. In spruce specimens the mass loss was up to 29 % and in beech specimens 38 % at 250°C. Furthermore, with temperature the loss modulus E'' was increasing until the target level of the temperature was reached. With time the loss modulus decreased at all tested temperatures. Higher temperature of exposure resulted in bigger decrease in loss modulus E''. With temperature the rate of decrease was increasing. The results showed that higher temperature in isothermal tests affected the wood's ability to dissipate energy considerably.

To examine the interactive effect of thermal treatment on the viscous modulus and the elastic modulus, the tanδ, damping property of the wood, expressed, as the ration of the viscous modulus to the elastic modulus was determined. With increasing temperature the tanδ was increasing to approximately 90°C, which was followed with steady state value (most likely due to the removal of water) and after, at about 170°C with an increase for all examined specimens (Figure 1). In specimens that were heated to a higher target temperature the tanδ was higher. At isothermal conditions the tanδ was decreasing with time. Higher peak tanδ value indicates greater degree of molecular mobility and better damping characteristics. The influence of temperature and time on the mechanical behavior of beech specimens shows similar relations as for spruce specimens. However the storage modulus E' and loss modulus E'' of beech specimens were lower than for spruce specimens, while tanδ was higher for beech specimens.

Preliminary tests showed that it is possible to modify small wood specimens in the DMA-equipment while, at the same time, getting real time information about the progress of the process.

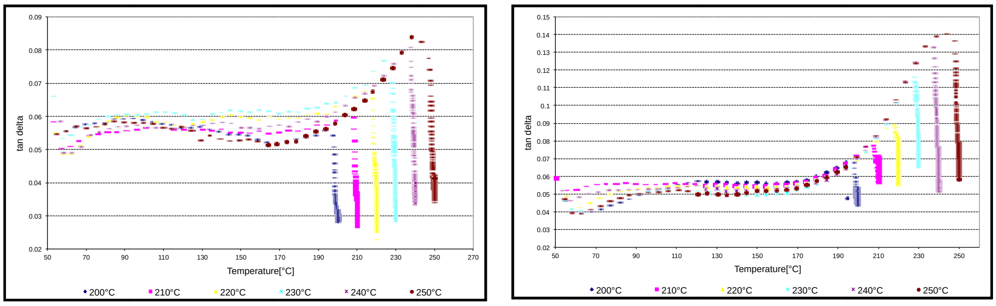

Figure 1. The tanδ of spruce (left) and beech (right) wood as a function of temperature for specimens tested with the isothermal test in the DMA equipment.

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**STSM**

# Durability and chemical modifications of four Tunisian wood species after heat treatment

**Mohamed ELAIEB[1], Kevin CANDELIER[2], Stéphane DUMARCAY[2],
Philippe GERARDIN[2], Mathieu PETRISSANS[2]**

[1]INRGREF, B.P. 10, 2080 Ariana, Tunisie
[2]Université de Lorraine, LERMAB, Bld des Aiguillettes, BP 70239 - F 54506
VANDOEUVRE les NANCY cédex France
ayeb2002@iresa.agrinet.tn/

**Keywords:** Chemical analyses, Decay resistance, Heat treatment, volatile compounds, Tunisian wood

Wood heat treatment is a good alternative method to improve decay resistance of natural durability wood species. Previous studies have showed that decay resistance was strongly correlated to thermal degradation of wood cell wall components and consequently of treatment conditions from which depend chemical modifications [1-5]. One of the main indicators of this treatment intensity was the mass loss generated by the thermodegradation.

The aim of this study is to determine chemical compounds produced during the treatment for different Tunisian wood species (Alep pine, Radiata pine, Maritime pine and Zeen oak). The heat treatment was realized under vacuum atmosphere and performed from 200°C to 230°C to obtain mass losses resulting from wood thermodegradation approximately of 8, 10 and 12%. The oven prototype allows having the dynamic Mass Loss (ML = fn(t)) during the treatment and following the kinetic of thermodgradation.

For each wood species and treatment intensity, wood chemical analyses were performed by measuring O/C ratio. Moreover test of durability was performed under different fungal attacks (*Poria Placenta*, *Coriolus Versicolor* …) at 22°C and 70% of relative humidity during 16 weeks. All of results are related to the Mass Loss.

Intensity of thermodegradation was also evaluated by TD-GC-MS. Treated and untreated wood samples were subjected to thermodegradation directly in the thermodesorption tube under nitrogen at 230°C during 15 minutes. The volatile compounds resulting from wood degradation was analyzed.

The correlation between O/C ratio and intensity level of treatment (ML %) showed an important increase of carbon content, while oxygen content significantly decreases. Elemental composition was strongly correlated with the heat treatment intensity depending from treatment duration, which directly conditioned the mass losses due to thermal degradations. Previous studies were shown similar results [5]. For each wood species, O/C ratio decrease linearly with the increase of the mass loss. Elemental composition appears therefore to be a valuable parameter to evaluate the mass losses due to thermo-degradation reactions which are directly connected to treatment intensity, for each wood species.

The TD-GC-MS analyses allow as finding the different compound hollocellulose and lignin degradation. We can see that cellulose and lignin are degraded After thermal treatment, The intensities of the pick of acetic acid (2,43: representing the hemicelluloses products) and the pick of vannilin (11,69: representing the lignin products) have decreased. Thermodegradation is faster for Zeen oack (hardwood) than pine (softwood). Correlations between weight losses recorded after fungal exposure and elemental composition indicated that carbon content and O/C ratio can be used to predict wood durability conferred by heat treatment

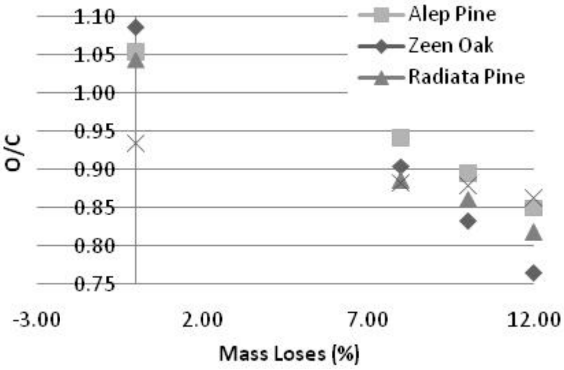
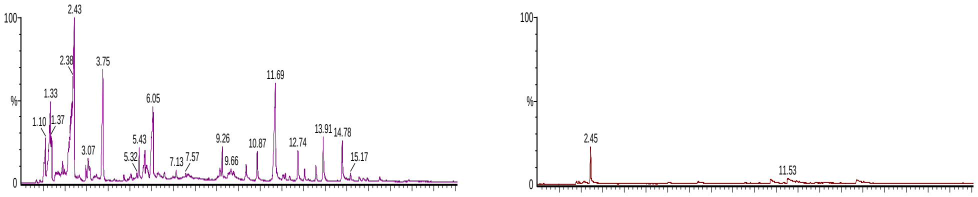


Figure 1. O/C evolution according to mass loses issued from heat treatment for different wood species



Not treated radiata pine

Treated (12%) radiata pine

Figure 2. TD-GC-MS chromatogrammes according to heat treated Radiata pine.

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**STSM**

# Experimental study of mechanosorptive hygro-lock effect in wood subjected to variable loading and relative humidity

**Omar SAIFOUNI[1,2], Rostand MOUTOU PITTI[1,2], Jean-François DESTREBECQ[1,2], Julien FROIDEVAUX[3], Parviz NAVI[3]**

[1]Clermont Université, Université Blaise Pascal, BP 10448, Clermont-Ferrand, France
[2]CNRS, UMR 6602, Institut Pascal, F-63171, Aubière, France
[3]Bern University of Applied Sciences, Biel, Switzerland
omar.saifouni@ifma.fr

**Keywords:** Wood, Mechanosorptive, Hygro-lock, Relaxation tests, Relative Humidity

Under stress and relative humidity variations, wood presents a complex behaviour called mechanosorptive behaviour. One of the main features of this behaviour relies in the locking of strain (or stress) during drying under variable or constant loading; this phenomenon is known as hygro-lock effect [1]. Several tests have been reported concerning the mechanosorptive behaviour [2] ; however, little is known about the hygro-lock effect which needs to be studied thoroughly, especially in the frame of a relaxation approach. In previous works, several models based on a partitioning of the strain into elastic, viscoelastic, mechanosorptive and swelling-shrinkage parts were developed in order to model this behaviour. Few of them take into account the hygro-lock effect.

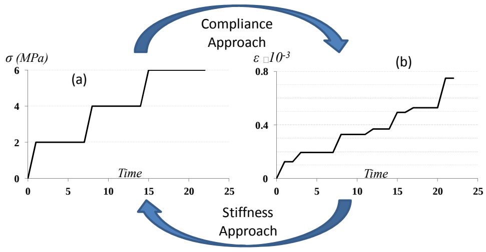

Figure 1. Solicitation-response cycle

In the present paper, an analytical model with hygro-lock effect is presented along with results of mechanosorptive relaxation tests which were performed in order to validate the model for various load and relative humidity variations. The approach is based on a stress partition into elastic, mechanosorptive and swelling-shrinkage parts. It is generalised for any imposed strain variation by subdividing the imposed strain into finite increments $\Delta\varepsilon_i$ . The behaviour relationships obtained from the present stiffness approach and from a dual compliance approach developed previously [3] are as follows:

$$\varepsilon = \sum_{i=0}^{N} H(t-t_i)\frac{1}{\overline{E}_i}\Delta\sigma_i \quad \& \quad \sigma = \sum_{i=0}^{N} H(t-t_i)\overline{E}_i\,\Delta\varepsilon_i \tag{1}$$

where $\overline{E}_i$ is the elastic modulus corresponding to $\overline{w}_i$ which represents the water content at the start of the drying phase for each imposed stress or strain increment.

The compliance and the stiffness analytical approaches are implemented in Matlab© software. As an application, a three steps uniaxial stress (Figure 1.(a)) is applied to wood material subjected to relative humidity variations. The strain response is computed using the compliance approach (Figure 1.(b)). Then, this strain response is used as the solicitation. The stress response calculated by the stiffness approach is the same as in Figure 1.(a). This result validates the equivalence between the both approaches.

In the scope of COST action FP0904, a series of relaxation tests were carried out at the University of Bern on the “Sapin Blanc” wood (Abies alba) according to figure 2. The mechanosorptive tests were performed in a specially designed apparatus [4]. These tests were focused on the study of mechanosorptive hygro-lock behaviour under variable loading and relative humidity. For such tests we used a specific loading and measurement device that allowed a good control of environmental conditions imposed to the samples. The samples were 0.7 mm thick in the radial direction, 50 mm long in the direction of the grain and 3 mm wide in the tangential direction; this helped reduce the effects of the hydric gradient and the duration of tests. Two sets of samples were prepared, the second tested once equilibrium was reached for confirmation that equilibrium was reached in the first set.

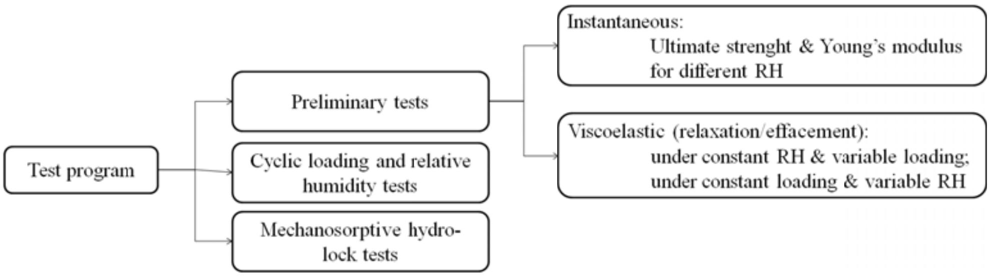

Figure 2. Tests program

Firstly, preliminary instantaneous tests were performed in order to determine the Young’s modulus and the ultimate strength $\sigma_r$ for different relative humidity levels. Then, the relaxation tests at a constant relative humidity at 50% and different imposed strain levels were performed; each test corresponded to a given relative stress $\sigma / \sigma_r$, in order to check the linearity of the behaviour. Secondly, the relaxation/effacement tests at a constant imposed strain level with a different relative humidity level for each test were carried out. Thirdly, the samples were tested under cyclic loading and relative humidity. The variations of relative humidity were maintained until the samples reached the equilibrium. Finaly, a mechanosorptive hygro-lock test in relaxation/effacement with a constant imposed strain level and a variable relative humidity in order to activate the hygro-lock effect was performed.

These tests provide the mechanosorptive viscoelastic parameters of the “Sapin Blanc” (Abies alba). The results allow us to better understand the hygro-lock behaviour and validate the analytical pseudo-elastic model; they give a basis for the analysis of the viscoelastic behaviour under any loading and relative humidity variation. In the coming work, the hygro-lock effect needs to be study in the case of compressive loading.

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**STSM**

# Thermal and Analytical Characterization of Welded Beech

**María Inés PLACENCIA PEÑA[1,2], Carmen Mihaela POPESCU[3], Frédéric PICHELIN[1], Antonio PIZZI[2]**

[1]Bern University of Applied Sciences, Bienne, Switzerland
[2]ENSTIB-LERMAB, University of Lorraine, Epinal, France
[3]"Petru Poni" Institute of Macromolecular Chemistry of Romanian Academy, Iasi, Romania
mariaines.placenciapena@bfh.ch

**Keywords:** linear welding of beech, FT-IR spectroscopy, thermogravimetry

Linear friction welding is a technology that enables assembling two wood specimens by means of the coupled effect of a perpendicular force and a continuous frictional movement that heats the interface, softens lignin, mills and mixes detached fibers, forming the welding interface. When friction is stopped pressure is still kept to allow the welding interface to cool down with the assembled join formed.

In the current study, the interface of welded beech at progressive welding times has been evaluated by FT-IR spectroscopy and thermogravimetry. The selected welding times (WTs) were 1, 1.5, 2, 2.5 and 3s. Frequency was 100Hz, amplitude 2.8mm, welding pressure 1.5MPa, holding pressure 2MPa and holding time 5s. During the welding process temperature increases as long as friction is applied between the two wood specimens. Temperature in the welding interface can reach between 300 and 350°C for a welding time of 2s. The "welded material" produced remains completely in the interface until a welding time of around 2s; for longer welding times small "extruded" fibers begin to appear at the edges of the welded zone, implying that "new" wood material is frictioned in the welding interface. The infrared spectra and thermo-gravimetric tests were performed strictly with the scratched material from the welded interface.

In Figure 1, the spectra of the welded material at different WTs are presented. Some of the main differences among the spectra are observed in the so called "finger-print" region from 1800 to 800 $cm^{-1}$. The band at $1740cm^{-1}$ in B_0s: reference sample spectrum, assigned to C=O stretching vibration of acetyl or COOH groups [1, 2, 3], undergo a decrease in absorbance and a shift to $1733cm^{-1}$ for the 3s of WT (B_3s) sample spectrum. These band modifications suggest a decrease in the concentration of hemicelluloses in welded beech, and/or the degradation or disappearance of acetyl groups of the glucoronic acid that is found as side group of the main chain of hardwood hemicelluloses. The bands at $1648cm^{-1}$, assigned to C=O in p-substituted aryl ketones (lignin), and at $1507cm^{-1}$, assigned to C=C stretching vibrations of aromatic skeletal (lignin), also show an intensity decrease. It can be interpreted as a lessening in the concentration of native lignin. Also, the band at 1507 is shifted to $1514cm^{-1}$ that shows possible condensation reactions of lignin with decomposition products of hemicelluloses and/or the cleavage of $OCH_3$ side chains of the aromatic ring. The other bands from the spectra are assigned distinctly to hemicelluloses, celluloses or lignin compounds.

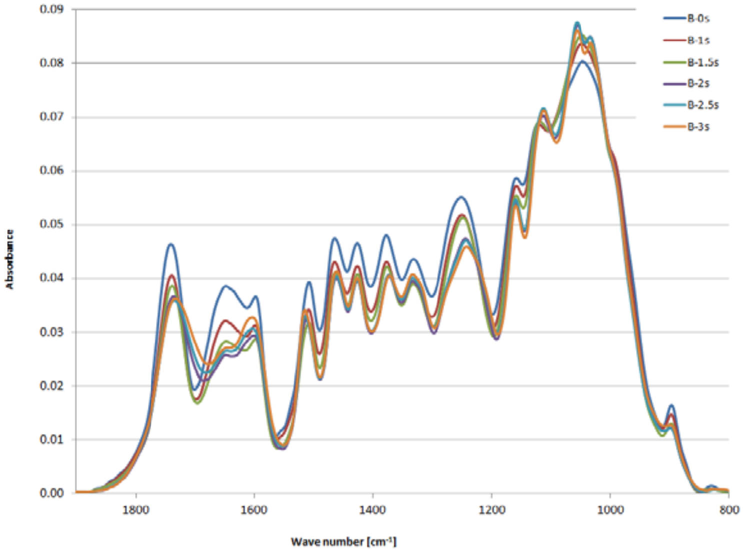

Figure 1. Finger-print region of the FT-IR spectra of reference and welded beech at different WTs

In figure 2, TG/DTG curves of beech and welded beech at 3s as well as their respective de-convolution curves are shown. The applied heating rate was 10°C/min. TG thermal curve gives the weight percentage of the original sample versus temperature [4], and DTG thermal curve gives the first derivative expressed as the rate of mass loss versus temperature. According to [5], assuming that the degradation of each component of a sample can be represented by one or the sum of few functions, the curve of the sample mass vs. time that is sensible to thermal degradation could be decomposed in several functions. A model of the TG and DTG curves with their respective de-convolution curves were obtained by using the equations mentioned in [5]. In thermo gravimetric studies of wood decomposition each de-convoluted curve is usually associated with one of the main components of wood [6]. In this case, curve "Peak 1" would represent water and low molecular weight components; "Peak 2" hemicellulose, "Peak 3" cellulose and "Peak 4" lignin. From these curves, the initial composition of the wood and welded wood samples can be obtained (% mass loss at around 31°C). A lower % of hemicellulose and celluloses and higher % of lignin respect to beech reference is observed at 31°C for the welded sample at 3s. It shows that due to the thermal effect of the welding process these materials (hemicelluloses, celluloses) have been partially degraded / eliminated / transformed, and probably formed compounds with lignin which initial composition at 3s of welding is higher. It can be also observed that the % of residue after the thermo-gravimetric tests of the sample welded at 3s is higher respect to the reference beech. From de-convoluted curves, important information can be also obtained, as the integral area of each curve that gives the composition at the temperature of maximal decomposition rate.

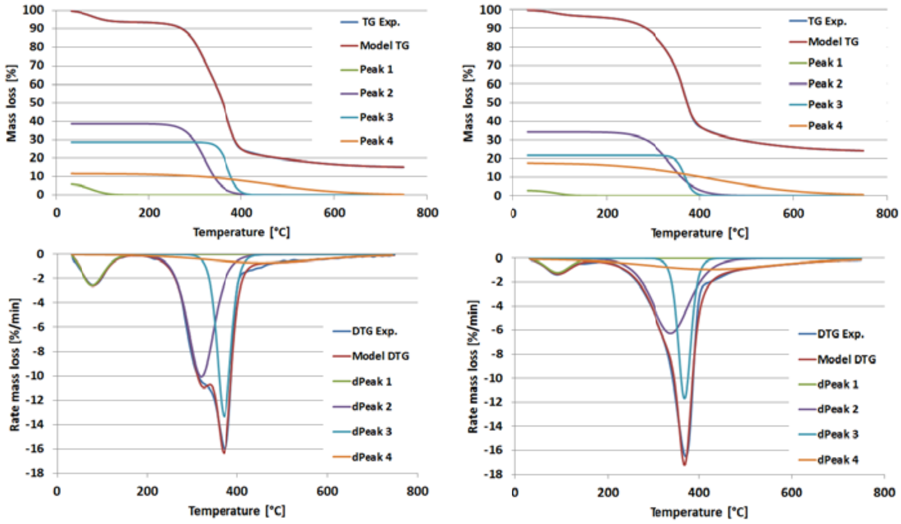

Figure 2. TG and DTG curves for reference (B_0s) (left), and welded (B_3s) beech

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**STSM**

# Thermal behaviours of THM densified wood

**Carmen-Mihaela POPESCU[1], Gabriela LISA[2], Julien FROIDEVAUX[3],
Parviz NAVI[3], Maria-Cristina POPESCU[1]**

[1]"Petru Poni" Institute of Macromolecular Chemistry of Romanian Academy, Iasi, Romania
[2]"Gh. Asachi" Technical University of Iasi, Department of Chemical Engineering, Iasi, Romania
[3]Bern University of Applied Sciences, Solothurnstrasse 102, 2500 Biel, Switzerland
mihapop@icmpp.ro

The effect of the thermo-hydro-mechanical (THM) densification in a close system on the set recovery and thermal stability of lime (*Tilia cordata*) wood was evaluated. The densified wood samples were subjected to three different post-treatment temperatures, and for each one, three different periods were used. The comparison between THM densified wood sample and post-treated samples at different temperatures and times were made [1].

During wood densification and particularly THM post treatments, the wood individual components undergo chemical degradations, depending to the processing parameters such as temperature, wood moisture content and processing time. Over the last two decades many research works have been reported on understanding the chemical degradation of TH wood aiming to correlate between this degradation of wood components and the macroscopic properties of TH wood. This has provided, on one hand, useful insights for the design of new wood based material and contributes to an improved predictability of the macroscopic behaviour of THM wood. On the other hand, it has strengthened the confidence of engineers and architects in THM wood and boosted the use of THM wood in conventional fields of application, such as the construction sector.

The set recovery tests showed that the post-treatment at 160 °C for 80 min or at 180 °C for 10, 15 and 20 minutes times, improve the dimensional stability of the samples suggesting a permanent fixation of transversal compression set. Also, it reduces their thermal stability, by having lower temperatures corresponding to the maximum decomposition rate and the kinetic parameters; but with lower values for the mass loss at the end of decomposition stage and higher values for the residual mass.

For the first step, the temperature corresponding to the maximum mass loss, and to the end of the stage decreased (from the S0 sample with no post-treatment) with increasing time of the post-treatment for the first two series of samples (Figure 1) and remain constant for the last series of samples.

The main decomposition step corresponding to wood components takes place from 180 to 410 °C, varying for each series. For the first series, the post-treated samples show almost the same temperature for the shoulder and for the maximum decomposition rate with the sample S0 and slightly higher temperatures for the end of the stage, increasing with the increasing time of post-treatment. The mass losses decreased for the samples S1-S4 comparing to S0, while the residual mass at 700 °C increased. The second series (S4-S6) show almost the same temperature values for the shoulder for the samples S0, S4 and S5 and smaller for the S6. The temperatures corresponding to the maximum decomposition rate and to the end of the stage are the same for S0 and S4-S6, with a small exception, the temperature of the end of stage for the sample S6 is lower than all others. The values of the mass losses are smaller for the samples with post-treatment at 160 °C for different time, comparing to S0 (no post-treatment). From Figure 1 it can be observed a decrease of the shoulder with increasing time of the post-treatment. Because this shoulder is assigned to hemicelluloses and amorphous cellulose, a decrease of it indicates an increase of hemicelluloses modification/degradation during wood treatment.

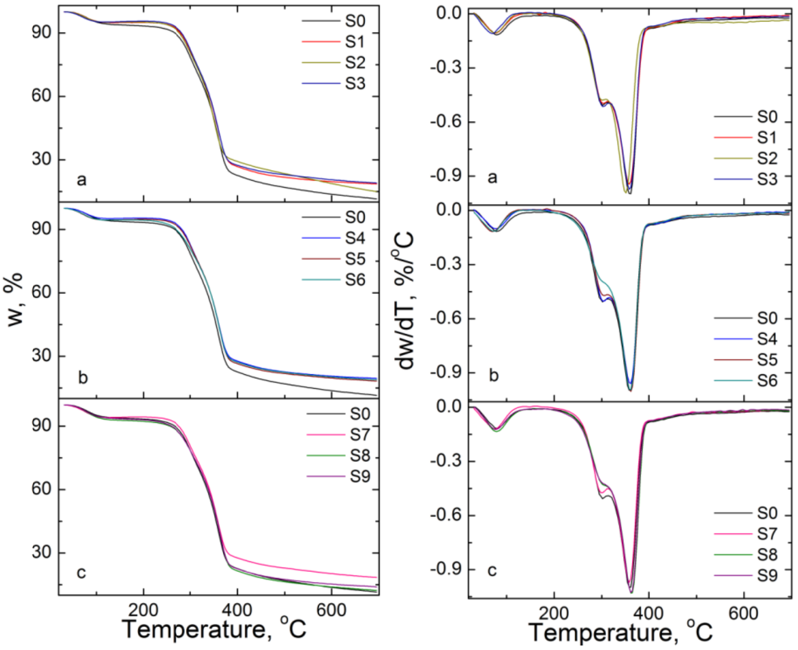

Figure 1. TG/DTG curves of the THM densified lime wood

For the samples S7-S9 (the third series) the temperatures for the shoulder and for the maximum decomposition rate increase in the series but are not higher than that of the sample S0. The values of the mass loss of this step increase in the series but also are not higher than that of the S0 sample. The values of the residual mass at 700 °C are comparatively higher for the post-treated samples, especially for the S7 sample, when comparing to the S0 sample. As reported before, the shoulder decreases with the increase of the post-treatment time, suggesting the occurrence of amorphous carbohydrates degradation/modification during this treatment.

The other samples showing high set recovery values, exhibited higher values for the temperatures corresponding to the maximum decomposition rate, lower values for the mass loss at the end of the stage and higher activation energies and reaction orders.

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# Poster presentations

**Session 1**

# Determination of dimensional stability of thermally modified beech and spruce wood

**Lothar CLAUDER, Alexander PFRIEM**

Eberswalde University for Sustainable Development - University of Applied Sciences,
Faculty of Wood Science and Technology, Friedrich-Ebert-Straße 28, 16225 Eberswalde, Germany
Lothar.Clauder@hnee.de

**Keywords:** Anti-Swelling-Efficiency, Beech, Dimensional Stability, Spruce, Thermal Modification

Moisture sorption behavior below the fiber saturation point is a critical aspect of wood and wood products during their service life. Loss and uptake of moisture can be caused by fluctuating relative humidity and temperature leads to dimension changes. In this study, the effect of heat treatment on the dimensional stability of beech (*Fagus sylvatica* L.) and spruce (*Picea abies* L.) wood is compared to untreated samples as reference. The heat treatment at 180°C, 200°C and 220°C was conducted in $N_2$ gas atmosphere in a prototype kiln of approximately 0.5 $m^3$ capacity. Matched 10 specimens (10x20x20 $mm^3$) of each treatment intensity and 10 untreated samples as reference were tested [1]. The applied cycle test is based on oven-dry-water-soak-cycle-system [2]. Results of a series of eight cycles are the best effective indicator of the effective achieved dimensional stability of a specimen. The schedule provides the following procedure for a single cycle: Oven drying at 103°C , once the mass is constant, water soaking at 50 mbar vacuum for 15 minutes. Then raising the pressure up to 8 bar and maintaining about 20 minutes. Afterwards keeping the specimens into the water for 24 hours till the mass is constant. The radial and tangential length change were measured by a dial indicator. The Anti-Swelling-Efficiency value (ASE) may be considered as a measure of the dimensional stability of wood. The determination of the ASE (Table 1) is based on the comparison of a treated specimen and an untreated specimen. The linear maximum swelling measure is determined in radial and tangential direction (Eq.1) for each specimen. Thus, the mean maximal swelling (eq.2) is calculated. The calculation of the ASE value is based on the equation 3.

Table 1. Max. swelling (eq.1), mean max. swelling-value (eq.2), ASE-value (eq.3)

| | | |
|---|---|---|
| eq. 1* | max. swelling | $\alpha = \frac{l_{\max} - l_{\min}}{l_{\min}} \times 100\%$ |
| eq. 2 | mean max. swelling | $\alpha_{mean} = \frac{\alpha_{radial} - \alpha_{\tan gential}}{2}$ |
| eq. 3 | Anti-Swelling-Efficiency | $ASE = \frac{\alpha_{mean\ reference} - \alpha_{mean\ \mathrm{mod}ified\ sample}}{\alpha_{mean\ reference}} \times 100$ |

* $l_{\max}$ = maximal length after water soaking; $l_{\min}$ = minimal length after oven drying

For specimens of both wood species the ASE values were calculated for each cycle as mean values in percent (Fig.1). The ASE values show the decline in swelling compensation during eight consecutive cycles, for both species. Therefor, in the advanced stage the effect of the dimensional stability of the thermally modified timber (TMT) is reduced by 12% for beech and 10% in the spruce compared to initial ASE after first circle.

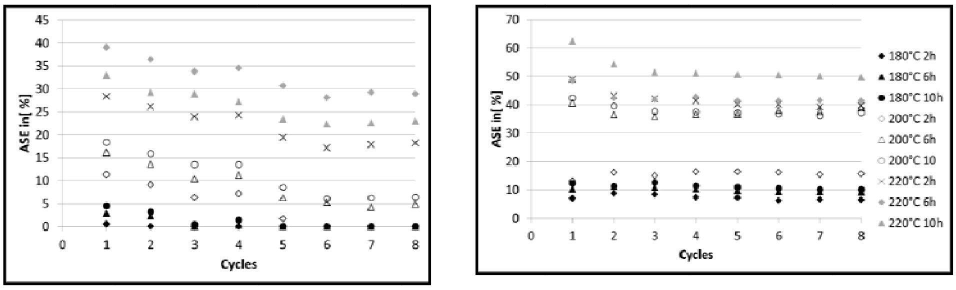

Figure 1. a) ASE values (spruce) b) ASE values (beech)

The increase in ASE due to the gradual increase of temperature and duration of treatment can be explained by the thermal degradation of cell wall components. In the temperature range of 180°C to 220°C, increasingly hemicelluloses are degraded, whose OH groups are responsible for the high hygroscopic behavior of the wood. Burmester [3] concluded that heat treatment of wood results in a high reduction in the hemicellulose content, and is thus an improvement of the dimensional stability of the wood. According to Fengel and Wegener [4] hardwoods are thermally less stable than softwoods. This may explain why the ASE values of beech reached higher levels than those of spruce. The significant increase in ASE values from a treatment intensity of 200 °C and 6 hours duration for the samples of both species is even after the eighth cycle still detectable. The step-change in ASE values suggests an increased influence of the treatment period. By correlating the ASE with the process-induced mass loss (dm) Welzbacher [5] specified a limit of dm for different treatment temperatures, beyond which no further increase in the ASE can be achieved by extending the treatment period. The ASE variation is detected in TMT, which can be used as a performance indication of the material in relation to its dimensional stability during the service.

Hygroscopicity of TMT was reduced compared to the untreated material. The results illustarte that die swelling compensation of TMT differs by a constant stress due to swelling and shrinkage of its initial value. This reduced swelling compensation is especially to be considered when evaluating the quality of TMT products for enhanced utilization as façade, flooring and other high-end joinery.

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**Session 1**

# Thermal behaviour of some wood species treated with ionic liquid

**Cornelia VASILE[1], Manuela-Tatiana NISTOR[1], Silvia Florica PATACHIA[2]**

[1]"Petru Poni" Institute of Macromolecular Chemistry, Department of Physical Chemistry of Polymers, 41A Gr. Ghica Voda Alley, 700487 Iasi, Romania
[2] Transilvania University, Chemistry Department, 29 Eroilor Str., 500036 Brasov, Romania
cvasile@icmpp.ro

**Keywords:** wood, ionic liquid, simultaneous thermal analyses, FT-IR, MS

In spite of obtaining a very large number of new synthetic materials, the wood remains one of the main classic sources of raw materials as such or to obtain various chemicals.

Ionic liquids (ILs) known as “green solvents” due to their very low vapor pressure, good thermal stability, non-explosivity and recyclability, have been proposed as suitable solvents for the components of wood, as well as suitable additives aiming to ameliorate the wood quality (wood plasticizers), wood surface hardening, to improve water resistance or thermal stability, as flame retardants and antifungal or biocide agents, UV stabilizers, etc. Some ILs can be used to take the place of currently widely-used phosphorous-containing fire retardant compounds. They are good alternatives to polluting compounds or technologies and to high energy consuming classical methods used for the mentioned aims. There are very large possibilities to combine organic cations and anions to obtain ionic liquids with tunable properties and capacity of interaction with wood based materials. Some imidazolium based ILs have been tested for dissolution of wood components and wood properties alteration.

Once is expected that the ILs treated wood products will gain the market, due to their improved properties and ecological technologies of producing and also the recovery possibilities of the ILs-treated wood wastes, aiming sustainable development it is important to know the thermal behaviour of IL-treated wood. It is important to know both thermal behaviour and compounds resulted by decomposition as a first step in application of the IL-treated wood at high temperature and also its recovery by pyrolysis/gasification processes.

Aiming to obtain composite materials from wood waste sawdust and considering the above mentioned reasons, the main goals of the present paper are: (1) to determine the influence of the 1-butyl-3-methylimidazolium tetrafluoroborate ionic liquid on the thermal behavior of two wood species (fir and beech); (2) to detect and identify the main species of volatiles evolved from the pyrolysis of the treated woods with ionic liquid and to understand their interaction

The thermal behaviour of two wood species treated with ionic liquid, 1-butyl-3-methylimidazolium tetrafluoroborate, was investigated using Thermogravimetry/Differential Scanning Calorimetry/Fourier Transform Infrared Spectroscopy/Mass Spectrometry coupled methods in order to elucidate the ionic liquid effect on the wood thermal behaviour. It was pointed out that the ionic liquid - treated wood samples show degradation in two processes. The effect of the ionic liquid on wood depends on the wood species. Untreated fir wood exhibited superior thermal stability compared to ionic liquid - treated fir, while the treatment of beech wood with ionic liquid has an improved thermal stability comparatively with untreated wood. New compounds have been identified in released gases during thermal decomposition because of ionic liquid - wood interaction.

The effect of the ionic liquid on the thermal degradation of the fir wood leads to the reduction of quaiacol, levulinic acid, levoglucosan fragments and gaseous compounds as ethylene, propanal, acetone and also furan derivatives. Similar effect had the ionic liquid on the beech thermal decomposition with delay of formation of the ionized fragments corresponding to the acetone, formic acid, vinyl acetylene (m/z 53), furan derivatives, 2-methylfuran and furfural.

It was also evidenced the increase of the emission of the fragments with m/z=18, 28, 42, 56 and the formation of new volatile compounds groups characterized by m/z=12, 34, 49÷59, 81, 85 when the thermal decomposition advanced. At high temperature they are no longer present. The compounds with high m/z ratio of 100, 104, 110, 163 are formed around 500 °C – Fig.1.

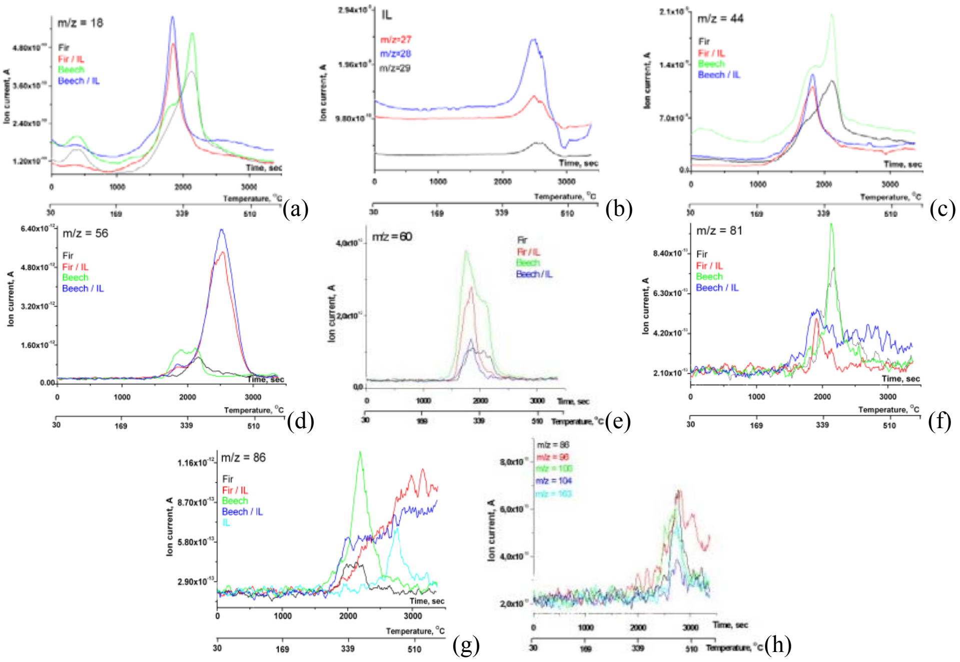

Figure 1. m/z versus time/temperature of the main representative volatile ionized fragments evolved from the decomposition wood samples untreated and IL-treated.

The ionic liquid increased the onset temperature decomposition for beech wood by changing decomposition reaction mechanism because of stronger interactions between components with new phase formation by mixing as it was evidenced by NIR analysis. At high temperature these interactions cause apparition of new compounds because of more possible secondary reactions of the decomposition products. The kinetic parameters indicated that the ionic liquid increases the overall activation energy of the thermal degradation.

New compounds were detected (m/z = 19, 27, 28) and time of evolution start of compounds from IL treated beech was delayed, while in the case of the fir sample an opposite situation was found (IL facilitates evolution of some light compounds).

**Acknowledgments:** We are grateful for the support received from COST FP0904 action and Marie Curie FP7 program grant BIOFUEL - PIRSES GA-2009-247550.

**Session 1**

# Evaluation of surface quality of wood composites as function of weathering

**Aniela GARCIA PEREZ[1], Emilia-Adela SALCA[2], Bobadilla MALDONADO[3], Salim HIZIROGLU[4]**

[1]Polytechnic University of Madrid - Department of Forest Engineering
[2]Transilvania University of Brasov – Faculty of Wood Engineering
[3]Polytechnic University of Madrid - Department of Forest Engineering
[4]Oklahoma State University – Department of Natural Resource, Ecology and Management
emilia.salca@unitbv.ro

**Keywords:** weathering, wood composites, roughness

Wood composites such as particleboard and MDF are widely used as substrate for thin overlays to manufacture cabinets and furniture pieces in many European countries including Spain and Portugal. On the behavior of these two composites as function of weathering there is little or no information [5, 6, 7, 8].

The aim of the study was to determine the effect of artificial weathering in terms of various cycles on surface quality of particleboard and MDF. Three weathering cycles of water soaking, freezing and heat exposures were applied to four types of panels with a view to establish their influence on the surface roughness.

Figure1. Control and weathered samples

The surface roughness of particleboard and MDF samples exposed to artificial weathering conditions was determined by using a stylus tracing method [2, 4]. Average roughness ($R_a$) and mean peak-to-valley height ($R_z$) were the two accepted roughness parameters used for the measurement of overall roughness changes of the samples [1, 3, 9].

The first cycle of weathering influenced the surfaces of both particleboard specimens, after that they were reconditioned and two more exposure cycles were applied. The surfaces of MDF samples suffered less influence compared to particleboard samples when refer to surface quality.

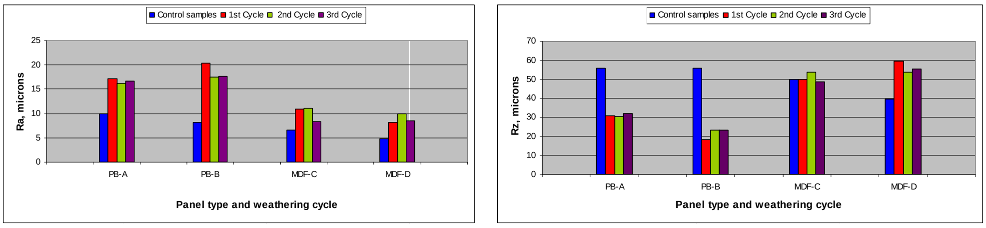

Figure 2. Average values of $R_a$ and $R_z$ parameters for panels as function of weathering cycle

The results on the behavior of these composite panels when exposed to outdoor conditions can be efficiently used to increase their service life.

Further study are to be conducted and would include not only surface quality but also dimensional stability as well as strength properties of such specimens, as a result of weathering cycles, to have a better understanding behavior of these two types of value-added composite panel products.

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**Session 1**

# The correlation between wood moisture content and air state properties during drying

**Daniela SOVA, Venetia SANDU, Ioan-Bogdan BEDELEAN**

Transilvania University of Brasov, Romania
sova.d@unitbv.ro

**Keywords:** drying kiln, softwood and beech timber, heat and mass transfer, drying schedule

The paper aims to develop a method for the determination of the drying agent state properties in respect with the wood moisture content and to apply it to a HD (Hildebrand) – type kiln designed for softwood and beech timber drying. The method is based on the steady-state heat and mass transfer problem, used in this case to a convective dryer, considering the first law of thermodynamics. By use of the equation of the first law of thermodynamics, the energy change in an open system during a thermodynamic process is calculated as a result of the thermal and mechanical energy transfer and the mass transfer [1]. The conventional wood drying kiln with forced air circulation corresponds to an open thermodynamic system that carries out through the control surface, a heat transfer by convection due to the air heater and a heat transfer by conduction through the walls, a shaft work due to the fans, a mass transfer due to the air that enters and leaves the kiln and due to the steam introduced to humidify the air [2].

The drying process is analyzed by use of the first law of thermodynamics as a rate equation in order to determine the rate by which the energy of the system changes as a result of the heat and mass transfer rate with the surroundings. For steady-state conditions and for a certain state of the air that enters the control volume (drying kiln), the state of the air that leaves the control volume is influenced by the drying velocity. Also, the enthalpy and moisture content of the air that leaves the control volume do not change during the constant-drying rate period, but diminish during the decreasing-drying rate period [3, 4, 5].

For the illustration of this analysis method, a simulation for an HD-type dryer has been carried out. The simulation has been performed for 38 mm thick softwood timber and 32 mm thick beech timber. The technical characteristics of the dryer are: overall dimensions of the stacks 6×1.6×2 m, number of stacks 8, volume flow rate of the axial fan 27000 $m^3_N/h$, number of fans 8, air circulation velocity through the stacks 2.5 m/s, critical moisture content (FSP) 30%, equilibrium moisture content 10% [6, 7]. The temperature, relative humidity and psychrometric difference of air - calculated and according to the drying schedule [8, 9] - at the exit of the drying kiln, as functions of the wood moisture content are shown in Fig. 1, 2 and 3.

With respect to the application of the thermodynamic analysis to the drying kiln, some observations should be made:

- the state of the humid air that leaves the drying kiln is influenced mostly by the fan's flow rate, the state of air at the kiln inlet (it is determined by the mixture ratio between the fresh and return air) and the state of air at the stack inlet, corresponding to the drying schedule of the wood material;
- the temperature and relative humidity values of air that leaves the drying kiln have been compared with those corresponding to the applied in practice drying schedules; the calculated temperatures have close values to the experimental ones, in turn, the calculated relative humidity values are larger than those encountered in practice. These differences can be explained by the fact that the moisture content of air at the control volume inlet has been adopted for each value of the wood moisture content, the mixture ratio between fresh and return air being unknown from practice. Also, there is no information

related to the change in the wood surface temperature with respect to the change of the heat-carrying agent properties. Therefore, the results were obtained by means of successive approximations. For exact calculations it is necessary to know these quantities which influence or are influenced, respectively by the drying schedule [10].

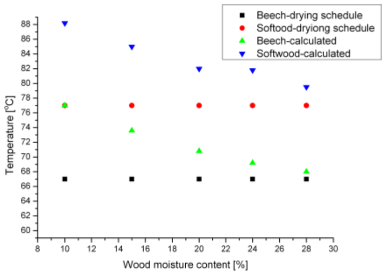

Figure 1. Temperature of air versus wood moisture content

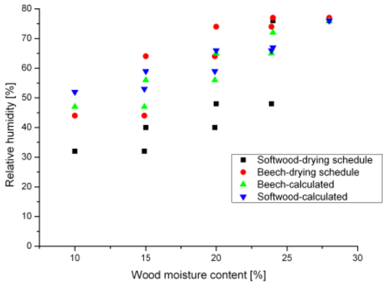

Figure 2. Relative humidity of air versus wood moisture content

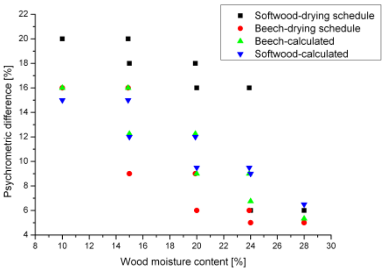

Figure 3. Psychrometric difference versus wood moisture content

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**Session 1**

# Wood anatomical changes on thermally modified surfaces of Norway spruce and Scots pine

**Michael ALTGEN[1], Jukka ALA-VIIKAIRI[2], Antti HUKKA[2], Timo TETRI[2], Holger MILITZ[1]**

[1]Georg-August University of Göttingen, Germany
[2]International ThermoWood Association, Snellmaninkatu 13, FIN-00170 Helsinki, Finland
hmilitz@gwdg.de

**Keywords:** thermally modified wood, anatomical changes, crack formation, microscopy, wetting and drying cycles

Thermal modification of wood leads to improvements in durability and dimensional stability and enables the use of non-durable wood species for various exterior applications. The changes in wood properties caused by the thermal modification can first and foremost be explained by chemical alterations of the wood. However, anatomical changes of the wood structure during the thermal modification process are likely to contribute to property changes as well, as reported in recent investigations (Boonstra *et al.* 2006b, a; Awoyemi&Jones 2011). Pit deaspiration in association with the damages to the ray parenchyma leads to an open and permeable structure and might therefore have an effect on the penetration of coating or adhesive systems into the wood (Boonstra *et al.* 2006a; Awoyemi&Jones 2011). Broken cells perpendicular to the fibre direction lead to abrupt failures in a bending test as shown by Boonstra *et al.* (2006a). In this study, we would like to test the hypothesis that small cracks on the wood surface of thermally modified wood are a potential starting point for the formation of severe cracks during a repeated wetting and drying of the wood during outdoor exposure.

The investigation was done within the framework of a joint project between the International ThermoWood Association (ITWA) and the University of Göttingen. Different lots of Norway spruce (*Picea abies* Karst.) and Scots pine (*Pinus sylvestris* L.) have been selected from the regular production of ThermoWood. From each lot, samples have been collected after different production steps, i.e. after the conventional kiln-drying, during the thermal modification, after the thermal modification and after the planing step. Anatomical evaluation was done at macroscopic and microscopic scale by means of stereoscopic evaluation, light microscopy and scanning electron microscopy. Finally, the material was subjected to repeated wetting and drying cycles and the formation of macroscopic cracks was evaluated after each cycle.

The results give insight into factors that might influence the formation of cracks during the thermal modification process of wood. Furthermore, they provide a potential explanation for the sensitivity of thermally modified wood to the formation of macroscopic cracks during repeated wetting and drying.

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**Session 1**

# Experimental and numerical detections of cracks appear in green wood during drying process

**Rostand MOUTOU PITTI[1,2,3], Frédéric DUBOIS[4], Eric FOURNELY[1,2],
Jean-François DESTREBECQ[1,2]**

[1]Clermont Université, Université Blaise Pascal, Institut Pascal, UMR 6602, BP 10448,
Clermont-Ferrand, France
[2]CNRS, UMR 6602, Institut Pascal, F-63171, Aubière, France
[3]CENAREST, IRT, BP 14070, Libreville, Gabon
[4]GEMH, Département Génie Civil et Durabilité, Université de Limoges, 19300, Egletons, France
rostand.moutou_pitti@univ-bpclermont.fr

**Keywords:** Green wood, experimental characterization, finite element analysis, drying process

In the structures live duration studies, the presence of cracks and their propagation is a very important criterion. This fact is more marked in timber structure submitted to moisture variation due to the heterogeneous aspect, the orthotropic character and the viscoelastic behaviour of wood material. This paper proposes experimental and numerical approaches for the crack appearing prediction in green wood slice during a natural drying phase. In order to take into account stress relaxation coupled with hydrothermal effects, an ageing elastic behaviour [1], including shrinkage–swelling effects with inhomogeneous properties in the radial and transversal directions, is considered.

The experimental protocol is based on a green wood slice in Douglas fir with a thickness of 30 mm, figure 1 (a). Sample is initially conditioned in water in order to ensure its saturation; all details of the experiment being recalled by Dubois et al [1] and Moutou Pitti [2]. In order to identify each black mark, figure 1 proposes a notification by the intersection between radiuses and rings. The radial strain is show by figure 1 (a), where $\Delta r_{1-2}$ is the length between $M_1$ and $M_2$. The tangential strain is defined by figure 1 (b), where $\Delta\theta_{1-2}$ is the distance between $M_1$ and $M_2$ that $r$ is their symmetric axis. $r_d$ is the distance between the referential origin and the projection of the two points on $r$. For example, the mark $M_i^j$ traduce the point at the intersection between the radius $R_i$ and the ring $C_j$. Let us note that radial and tangential strains are defined at the middle point between $M_1$ and $M_2$. The tangential strain is calculated at the point $M_{2-3}^2$ placed in the ring n°2 and between radiuses 2 and 3.

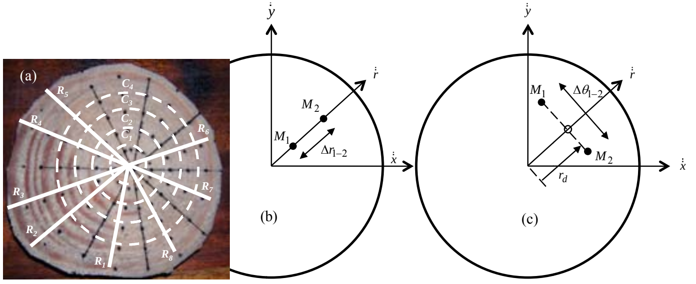

Figure 1. (a) Real green wood slice. (b) Radial representation. (c) Tangential representation

Figure 2 shows the comparison of tangential strains between experimental and numerical results. We observe that the values are important around the moisture content of 8%. This fact justify the crack appear introduced according to the Tsai-Wu's criterion posted on figure 3.

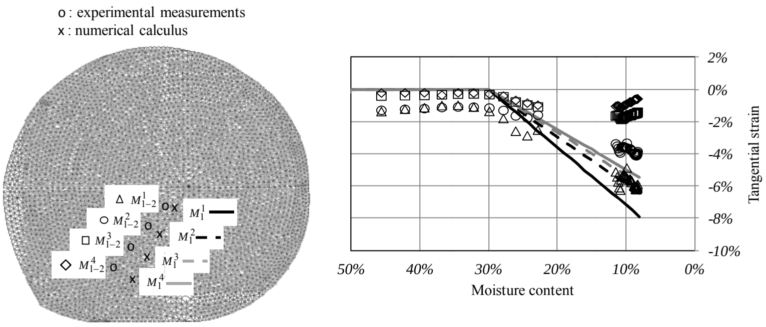

Figure 2. Experimental and numerical radial strains along radius n°3

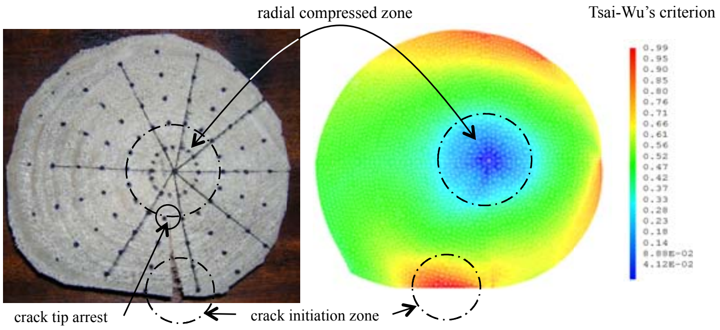

Figure 3. Experimental and numerical radial strains along radius n°3

Finally, this work should be completed by a fracture process study. In this case, a coupling between fracture mechanics approaches, viscoelastic and mechano-sorptive behaviour could be proposed by coupling finite element developments with digital image correlation measurements.

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**Session 1**

# About some chemical changes to wood due to densification

**Jakub SANDAK, Anna SANDAK, Dusan PAULINY, MariaPaola RIGGIO,
Ilaria SANTONI**

IVALSA/CNR, Trees and Timber Institute, via Biasi 75, 38010 San Michele All'Adige, Italy
sandak@ivalsa.cnr.it

**Keywords:** FT-NIR, wood densification, wood chemistry

Wood densification is an old process applied to wood in order to improve selected properties. An apparent reduction of the sample dimensions and in consequence its increased density is evident effect of wood densification. Deformation of cell wall, dislocation of molecules inside of the tissues, colour change and degradation of some chemical components is also taking role on the molecular level. The quality and quantity of such changes depends on the intensity of densification, wood species, moisture content, temperature, time of treatment, among others. The goal of this work was to employ FT-NIR spectroscopy for non-destructive and rapid characterization of changes to wood due to densification process in open system.
Beech (*Fagus silvatica*) was selected as a wood species for investigation. Small blocks (100mm x 80mm, width and length respectively) were cut-out from one board, taking care to assure three grain orientations of 0, 45 and 90°. The initial thicknesses of the samples were 6, 7.5 and 9mm. The densification was performed in a hydraulic press equipped with the hot plates. The densification cycle followed the literature references and was composed of pre-heating (plasticization); 10min, densification; 10min, post treatment; 5min and cooling; 5min. The hot press temperature was set to 180°C.

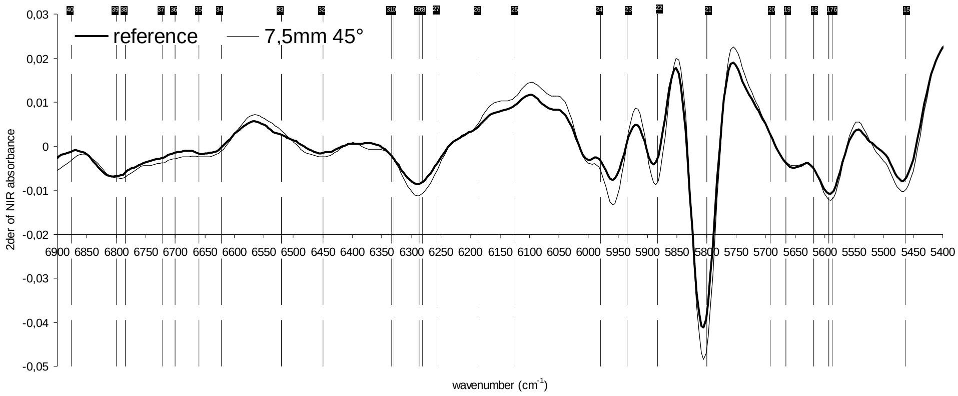

Figure 1. FT-NIR spectra of densified and reference wood (beech, initial thickness 7,5mm, grain angle 45°)

Near infrared spectra were measured by means of VECTOR 22n FT-NIR spectrometer (Bruker Optics) equipped with fibber optic probe. The surface of the sample was refreshed before measurement by sanding. At least three measurements were performed on each sample and were subsequently averaged. Second derivative (S-G with 21 smoothing points) and vector normalization were used for spectra pre-processing. The spectra analyses indicate that there are significant dissimilarities between densified and not treated reference spectra (Figure 1). Most considerable differences were noticed for the vibration bands (in $cm^{-1}$) related to:

- cellulose; 4401, 6790, 7003, 7321, 8160
- hemicelluloses; 4686, 5245, 5800, 5950, 7300
- lignin; 4686, 5935, 5963, 6874, 6944, 7092
- and also of extractives; 4686, 5995, 7092

Such changes are related to molecular reorganization of woody molecules caused by densification (pressing) besides to the effect of thermal treatment. It was also observed that the grain orientation has not a major effect on the NIR spectra, even if some of the mechanical properties were affected.

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**Session 2**

# Reducing set recovery of densified wood with heat treatment

**Miha HUMAR[1], Frederick A. KAMKE[2], Andreja KUTNAR[3]**

[1]University of Ljubljana, Biotechnical Faculty, Department of Wood Science and Technology,
Rožna dolina, C. VIII/34, SI-1000 Ljubljana, Slovenia
[2]Department of Wood Science and Engineering, Oregon State University, 119 Richardson Hall,
Corvallis, OR, USA
[3]Andrej Marušič Institute, University of Primorska, Muzejski trg 2, SI-6000 Koper, Slovenia;
ILTRA d.o.o., Celovška cesta 268, SI-1000 Ljubljana; Slovenia
miha.humar@bf.uni-lj.si

**Keywords:** densification, hydro-thermal compression, dimensional stability

Wood is one of the most important construction materials. Wood has several advantages, but on the other hand consumers are facing several issues as well. Predominately, insufficient mechanical properties, dimensional stability and insufficient decay resistance are the most important drawbacks. In order to overcome these issues, wood was densified, and in a separate step heat-treated. It was presumed, that densification will improve the mechanical properties, and heat treatment will reduce set recovery and improve fungicidal properties.

In order to elucidate that issues, beech wood (*Fagus sylvatica*) was conditioned in an environmental controlled room (20 °C, 65 % relative humidity) and afterwards densified by the Viscoelastic Thermal Compression (VTC) process at 170 °C. The densification process was performed in a pressurized vessel that is equipped with a heated hydraulic press at temperature of 170 °C. The specimens were compressed to two different target thicknesses resulting in two different degrees of densification (Table 1). In the next step, test specimens (30 mm × 10 mm) were heat treated according to the process developed by [1]. Prior to heat treatment, all the specimens were oven-dried at 103°C and then placed in a vacuum pressure chamber (Kambič, SI Semič), where a 92.5 % vacuum was achieved. The heat treatment process consisted of the three main steps. In the first step, the specimens were heated, which took about 2 hours depending on the target modification temperature. In the second step, the specimens were held at the desired modification temperature for three hours. The modification temperatures were 190°C, 200°C, 210°C, and 220°C. Before the last step, cooling, the specimens were again exposed to vacuum for 15 minutes and cooled down to 100°C. After the thermal treatment, specimens were oven-dried again, and mass loss as a result of the heat treatment was determined. Furthermore, set recovery as affected by temperature of thermal treatment was determined. Specimens (30 mm × 10 mm) were first dried in a convection oven over night at 103°C to establish an initial oven-dry weight and dimensions and then soaked in water for 24 h, and again oven-dried.

Table 1. Characteristics of the VTC and control specimens

| | Initial thickness (mm) | Initial density (D1) at MC=0% ($g\ cm^{-3}$) | Density after compression (D2) at MC=0% ($g\ cm^{-3}$) | Density ratio (D2-D1)/D1 |
|---|---|---|---|---|
| Beech | | | | |
| VTC_A | 6 (n=6) | 0.677 | 1.198 | 0.433 |
| VTC_B | 6 (n=5) | 0.683 | 1.075 | 0.365 |
| control | 6 (n=6) | 0.680 | - | - |

The procedure was repeated for a total of five wet/ dry cycles. After each cycle the percentage of set recovery was determined.

Thermal treatment had the same effect on control and VTC wood specimens. It caused mass loss of control and VTC wood specimens; with increased temperature of the thermal treatment the mass loss increased from approximately 3 % at 190°C to 12 % at 220°C. Furthermore, it was found that thermal treatment considerably reduced the set recovery of VTC wood specimens. In specimens that were not thermally treated the set recovery after 5 water soaking – drying cycle was high; in VTC_A specimens the determined set recovery was 40 %. In lower density VTC specimens, VTC_B, the set recovery was lower, about 25 % (Figure 1). Thermal treatment reduced the set recovery in both groups of VTC specimens. With increasing temperature of the thermal treatment the set recovery was reduced. Thermal treatment at 220°C completely eliminated the set recovery of compression deformation (Figure 1).

It can be concluded that thermal treatment of denisified wood can lead to complete fixation of the compressive deformation. Therefore, the thermal treatment after the VTC process should be considered as a successful method to eliminate the critical drawback of VTC wood, recovery of compressive deformation.

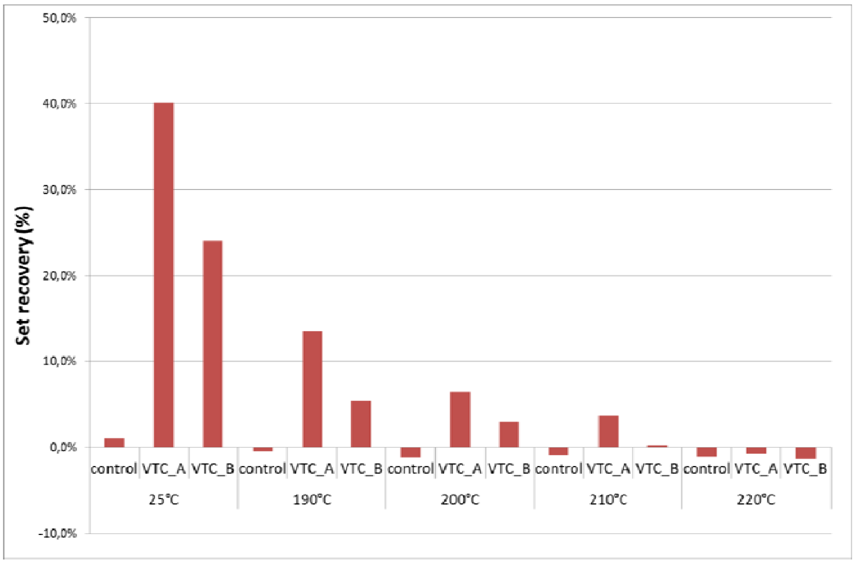

Figure 1. Set recovery of heat treated VTC and control specimens after fifth water soaking cycle

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**Session 3**

# Studies on post-welding heat-treated vibrational welded wood

**Jussi RUPONEN[1], Martin RHÊME[2], Silvia FERRARI[3], Lauri RAUTKARI[1], Mark HUGHES[1]**

[1]Aalto University School of Chemical Technology, Department of Forest Products Technology, Espoo, Finland
[2]Bern University of Applied Sciences, Material and Wood Technologies, Biel, Switzerland
[3]IVALSA-CNR, Trees and Timber Institute, San Michele all'Adige (TN), Italy
jussi.ruponen@aalto.fi

**Keywords:** birch, heat treatment, moisture tolerance, thermal modification, welding of wood

As is commonly known, one of the properties of wood material is to shrink and swell according to the changes in moisture conditions. Due to shrinking and swelling we can say that the dimensional stability of wood is often insufficient. However, the thermal modification, i.e., the heat-treatment of wood is at present a common method for improving, for instance, the dimensional stability of wood when exposed to moist conditions. The key issues behind the improved dimensional stability are the chemical changes of cellulose and hemicelluloses due to thermal treatment. These changes lead to lessened chances for hydrogen bonding of water molecules, i.e. lessened chances for adsorption and therefore cell wall thickening. However, the dimensional stability of compressed wood is more complex. Normally a piece of compressed wood swells back to near its original dimensions when immersed in water. This phenomenon (the spring-back effect) can, however, be reduced with thermal modification [1], and actually, it is suggested that thermal modification releases the internal stresses of wood and finally, compressed wood may be dimensionally stabilised through certain thermal treatments [2].

According to Omrani et al. [3], rotationally welded wooden dowel joints have rather high tolerance against moist conditions. On the contrary, according to Mansouri et al. [4] linear vibration welded wood joints suffer from sensitivity to water. As their studies included various methods of manufacturing linear vibration welded wood joints they were able to improve the resistance to water by optimising the process parameters [4]. According to several studies [5, 6, 7, 8 & 9], also self-bonded plywood (i.e. plywood produced without external adhesives) suffers from delamination when immersed in water. However, there have been promising results to suppress this behaviour through post-manufacture thermal modification and a considerable enhancement in bond stability of self-bonded plywood in moist conditions is reported [8 & 9]. As the similarity in the bond formation between self-bonded plywood and friction welding of wood derives from similarity in processing conditions (pressure, moisture, temperature) and compounds in wood only, it could be possible to successfully utilise thermal modification for linear vibration welded wood as well. The aim of this study is to assess the applicability of post-manufacture thermal modification of linear friction welded wood joints to enhance the moisture tolerance of the joints. Some of the results have already been promising [10].

The experimental test series was performed to study if the water resistance of linear vibration welded wood joints could be improved through thermal modification. Moreover, changes in mechanical properties were observed. The specimens were manufactured at the Bern University of Applied Sciences facilities in Biel, Switzerland, with a Branson M-DT24L linear friction welding machine. Knotless birch (*Betula pendula*, L.) was the raw material for this experiment. The specimens were post-manufacture thermally treated at two facilities: in vacuum atmosphere at IVALSA-CNR Trees and Timber Institute in Italy, and in vapour atmosphere at Aalto University, Finland. The preliminary results are presented at COST Action FP 0904 workshop, in Iasi, Romania, on April 9th -11th 2013.

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**Session 3**

# Continuous Wood Densification Process of Circular Profiles

**Jörg WEHSENER[1], Peer HALLER[1], Jens HARTIG[1], Tom-Egmont WERNER[2]**

[1]Technische Universität Dresden, Germany, Institute of Steel and Timber Structures
[2]STM GmbH Lunzenau, Germany
joerg.wehsener@tu-dresden.de

**Keywords:** trunk densification, cylindrical profiles, fibre reinforcement

The objective of the investigations was a continuous process for manufacturing of hollow timber profiles. The developed and patented process is based on a process developed by Pfleumer in 1923 and applies densification of wood perpendicular to the grain. Therefore, the wood (dried or fresh sawed) is plasticised at temperatures above 80°C and pushed through a tapering mould by a piston.
The tapering of the circumference leads to increasing lateral pressure and a tangential densification of the material. The necessary high deformability of the wood originates from on its cellular and polymeric structure.
First experimental investigations were performed in a simple biaxial device to determine material and process parameters for densification. The varied properties were moisture and temperature of the wood, the pressure in longitudinal and transverse direction as well as the orientation of the annual rings.
In a second step, conic moulds for the production of hollow cylinders were developed with a diameter at entry of 100 mm and 70 mm at exit. By this mould, round sticks with diameters of 15 mm and lengths of 50 mm were densified up to about 40 %. The compression force for pine (*Pinus Silvestris L.*) was 20 kN at a moisture content of 6 % and a temperature of 120 $^{o}$C.
The density distributions before and after the process were determined to evaluate the densification and deformation behaviour. The measurement of the density profiles was performed based on gamma rays (radiation source Am$^{241}$, Raytest Isotopenmessgerät GmbH).

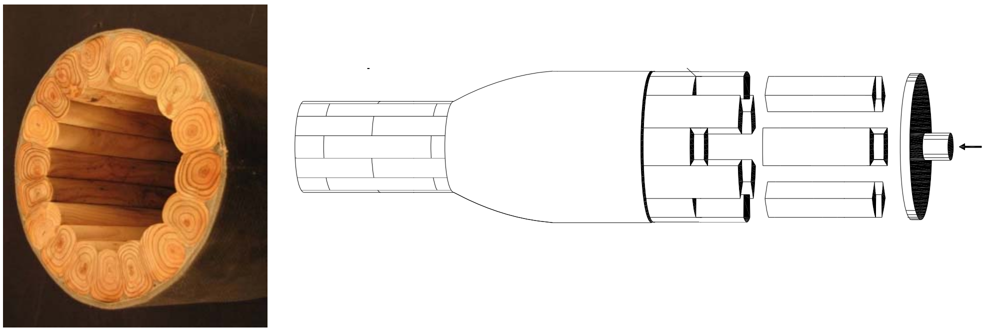

Figure 1. Densified and glass fibre reinforced wood profile (left) and principle process (right)

As a last step, tubes with diameters of 220 mm and length of 1500 mm for upscaling to structural dimensions were produced, needed, e.g., for building constructions. Therefore, sticks with diameters of 45 mm were pushed through a heated conic mould with an entry diameter of 314 mm and an exit diameter corresponding to the resulting tube. Endless profiles can be achieved by staggering of the sticks. To avoid rotation of the cross sections in the process, steel rails were arranged in the mould.

Right after leaving the mould, fibre reinforcement was wrapped around the tubes. The glass fibres were oriented tangentially to the tube. The total weight per unit area was 500 g/m². The fibres were embedded in polyester resin, which increases the protection and durability of the wood. Moreover, the fibre reinforcement suppresses recovery of the wood due to the shape-memory effect, increases dimensional stability and leads to a fixation of the tube cross sections during drying of the wood.

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**Session 3**

# Shape stability of THM-processed laminated veneer products glued with bio-based adhesive systems

**Lars BLOMQVIST, Jimmy JOHANSSON, Dick SANDBERG**

Linnaeus University, SE-351 95 VÄXJÖ, Sweden
lars.blomqvist@lnu.se

**Keywords:** bio-based adhesive, lignin, lignosulfonates, tannin, laminated veneers

There are several methods of producing laminated wood such aslaminated veneers or boards for structural purposes, plane and cross-wise laminated veneers (i.e., plywood), continuous laminated shapes, and veneers that are shaped and laminatedagainst a mould (essentially for furniture and interior purposes)[1]. This paper is about the shape stability ofplane and cross-wise laminated veneers that are glued with bio-based adhesives.

Increasing environmental concernsand the rising cost of petroleum-based (synthetic) adhesives has made bio-based adhesives of interest. Some bio-based adhesives, such as tannin and lignin,can be extracted from wood itself. Tannins are more reactive than many other adhesives, including phenol, but it is also more expensive. Tannin has been used as an adhesive for wood composite production, such as particleboard and medium density fibre board production as well as laminate and finger joint bonding. Lignin is available in large quantities from the pulp industry, because it is a by-product ofthe industry's processes. Lignin is less expensive than tannin, but it is not as reactive as tannin. Nevertheless, the lignosulfonates that come from the sulfite pulping of wood have been found to be more reactive thanlignin [2]. Research shows that it is possible to bond veneers together with bio-based adhesive in the form of lignosulfonates and tannin-type compounds [3]. A drawback of many bio-based adhesives istheir lowreactivity during hardening, resulting in long pressing times or the need forelevated temperatures. High pressures and temperatureschange the conditions ofthe THMprocess andthus increase the risk of unacceptable distortion or cracking.

The aim of the present work was to investigate the shape stability of THM-processed laminated veneer products glued withthree bio-based adhesive systems:lignin, tannin, ora mixture of lignin and tannin.

Three veneerswere bonded together into a flat constructionwith the middle veneer oriented crosswise. The basic demands of the laminate were that it would be shape stable and useful in climates with high humidity without delaminating in the bond line. The strength of the bond line was not tested in this work. Quarter-sawn veneers of Scotch pine (*Pinussilvestris* L.) with a size of 150x150x2.6 mm were used. The veneers were conditioned at a relative humidity (RH) of 20 % and a temperature of 20°C. The veneers' thicknessesweremade uniform by sanding. Used veneers have straight fibres. Lignosulfonates(BorregaardLignoTech, Norway) and tannin (Kremer Pigmente, Germany) were tested, and aurea formaldehyde adhesive (Casco Adhesives Inc., Sweden) was usedas a reference. Three groups with bio-based adhesive were tested: lignosulfonates, tannin, and a mixture of lignosulfonates and tannin.All three had aglue spread of approximately 129 g/m2. The glue spread of the reference was 170 g/m2.

The pressing were applied by a press with press plates that underwent resistive heating. The contact pressure was 0.5 MPa, and the temperature and the pressing time varied. The shape of the individual samples and the condition of the bond line were determined immediatelyafter pressing and at different phases of RH cycling.

The results from the investigation show that it is possible to bond veneers together with bio-based adhesive in the form of lignosulfonates and tannin-type compounds to form a stable laminated veneer product. Future studies can refine thetime and temperature for optimum bonding.

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**Session 3**

# Effect of thermal-vacuum treatment on bond shear strength. A comparison among Norway spruce, White ash and Turkey oak wood

**Luigi TODARO[1], Silvia FERRARI[2], Paola CETERA[1], Ottaviano ALLEGRETTI[2], Nicola MORETTI[1], Achille PELLERANO[3]**

[1]University of Basilicata, V.le Ateneo Lucano 10, 85100 Potenza, Italy
[2]CNR-IVALSA, via Biasi 75, 38010 San Michele a/A. Trento, Italy
[3]University of Bari, Via Amendola 165/A - 70126 Bari, Italy
luigi.todaro@unibas.it

**Keywords:** shear strength, steaming, thermo-vacuum process

Spruce (from Nord Europe) and white ash (from USA) woods are largely used for building and indoor applications. Due to a broad quantity of research, these species provide some basis for comparison with the results of other woods less investigated *e.g.,* T.oak. This last species is widely distributed in South-Eastern Europe, but actually arouses indifference in rich wood markets due to a less dimensional stability, elevated internal tensions and a low durability.

More than 70% of wood products use adhesive (2), so hydrothermal treated wood as well needs to be glued for certain application. The modification of wood by steaming and heat can affect the bonding performance of wood. The aim of the present work was to study the influence of thermal-vacuum treatment on mass loss and shear strength, both for wood and bondline, of the three species above-named.

Green T. oak logs were initially steamed at 110°C for 24 hours and then cut into boards 32 mm thick. Steamed boards were then loaded into the dedicated machine in order to perform drying and thermal modification under low pressure conditions (200-250 mbar). The thermo-vacuum process (2) is characterized by a convection heat transfer and can be defined as an open system because all the volatile products of the process are continuously removed from the kiln by means of a pump which keeps the set pressure. T. oak wood was dried until the 0% of moisture content and then it was thermally treated at 160°C for three hours. Instead, Spruce and Ash were thermally treated, at 180°C for two hours, but without steaming.

Three boards for each glued specimens were assembled according to DIN EN 204-205 and the specification of the adhesive schedule. For the bonded wood, 30 samples for each of 6 treatments and sequence were used for a total of 540 samples. In addition, other 180 samples were also used for the strength determination of non-bonded wood specimens. The adhesive used is a water-thinned and solvent free that, with a specific additive, can be converted in D4 (DIN EN 204). The adhesive characteristics and the conditioning sequence are shown in table 1.

Table 1. Adhesive specification and type of test

| Adhesive | Density (23 °C) ($g/cm^3$) (ISO8962) | Viscosity mPa.s (ISO2555) | Rupture load (DIN68602/EN204) | pH |
|---|---|---|---|---|
| Polyvinyl acetate | 1 | 9000-15000 | 1400 kg/ $cm^2$ | 2.8-3.6 |
| Conditioning | Kind and duration | | | |
| Sequence 1 | 7 days in standard atmosphere | | | |
| Sequence 3 | 7 days in standard atmosphere and 4 days in cold water (20°C) | | | |
| Sequence 5 | 7 days in stand atm., 6 hours in hot water (60°C), 2 hours in cold water (20 °C) | | | |

Table 2 shows that the mass loss were increased with treatment especially in T. oak, probably due to the steaming effect. The results of non-bonded wood shear strength highlighted a significantly decreasing for T. oak (-32.3%) and spruce (-13.7), but not for ash (-1.8%).

Table 2. Mass loss and shear strength, ± standard deviation, of non-bonded wood specimens. The different letters indicate significantly differences between the 6 treatments at $P<0.05$. Letter D and relative values means decreasing compared to control (Ctrl)

| Treatments | Mass loss (%) | Strenght ($N/mm^2$) | sign | Decreasing (%) |
|---|---|---|---|---|
| Ctrl spruce | | 14.6±1.2 | *a* | |
| Treated spruce | 1.28±0.02 | 12.6±2.3 | *a* | -13.7 |
| Ctrl ash | | 21.1±5.4 | *c* | |
| Treated ash | 1.76±0.12 | 20.7±3.6 | *c* | -1.8 |
| Ctrl T. oak | | 29.7±3.6 | *d* | |
| Treated T. oak | 2.25±0.59 | 20.1±2.5 | *c* | -32.3 |

Table 3 shows (sequence 1) that thermal treatment reduces the tendency of wood, apart white ash, to be wet by adhesive. This event may result in a weak bondline resistance for polyvinyl acetate adhesive, especially if the specimens were submerged for many times in cold water (sequence 3). Nevertheless, an inversion of D value was depicted in the sequence 5. In fact, all the values of shear strength of the thermally treated specimens highlighted a resistance higher that control samples.

Table 3. Shear strength, ± standard deviation, of bonded wood specimens. The different letters indicate significantly differences between the 6 treatments at $P<0.05$. Letter D and relative values means decreasing compared to control (Ctrl)

| Treatments | Seq 1 ($N/mm^2$) | | D (%) | Seq 3 ($N/mm^2$) | | D (%) | Seq 5 ($N/mm^2$) | | D (%) |
|---|---|---|---|---|---|---|---|---|---|
| Ctrl spruce | 13.8±1.6 | *a* | | 4.0±0.9 | *ab* | | 3.8±0.9 | *a* | |
| Treated spruce | 10.4±1.5 | *a* | -24.2 | 3.3±0.6 | *a* | -16.3 | 4.0±1.3 | *a* | 4.4 |
| Ctrl ash | 22.1±3.8 | *b* | | 5.0±1.5 | *c* | | 5.0±1.2 | *b* | |
| Treated ash | 21.0±3.3 | *b* | -5.0 | 4.4±0.1 | *bc* | -10.7 | 5.6±0.8 | *b* | 11.8 |
| Ctrl T. oak | 29.0±1.7 | *c* | | 3.8±2.2 | *ab* | | 1.2±1.3 | *c* | |
| Treated T. oak | 21.1±2.9 | *b* | -27.3 | 3.5±1.2 | *a* | -7.9 | 1.9±1.4 | *c* | 57.6 |

Adhesive can improve wood utilization and allow the use of low quality timber like T. Oak, but adequate product and strategy should be used. Otherwise, a great quantity of specimens could be weakly bonded. Future work will seek the use of other adhesives for wood difficult to be treated but with a great potential like T. oak.

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**Session 3**

# Set-recovery of densified and thermally modified wood under repeated soaking-drying-cycles

**Kristiina LAINE, Lauri RAUTKARI, Mark HUGHES**

Aalto University, Department of Forest Products Technology, Espoo, Finland
kristiina.laine@aalto.fi

**Keywords:** densification, thermal modification (TM), set-recovery, drying-soaking-cycles

The density and thus selected properties of solid wood can be enhanced by compressing its porous structure under controlled conditions. One challenge in wood compression is to obtain a permanent deformation; that is, to eliminate the set-recovery phenomenon. In this study, Scots pine sap wood samples were treated in three different ways (further described in Hill et al. 2012) [1]: One set of samples was thermally modified (TM) another was densified and the third set densified and subsequently TM. In addition, a set of control samples was left untreated. Densification was performed in an open press at 150°C for conditioned (RH 65%, 20°C) samples with an original thickness of 12 mm to a target thickness of 5 mm and TM at 200°C for 3h with steam injection. After treatment, all samples were re-conditioned (RH 65%, 20°C) and six specimens (10 mm x 10 mm) cut from each treatment for the oven-drying-soaking-cycles. The specimens were oven-dried at 103°C for 24h and subsequently soaked in distilled water at 20°C using a net fabric to hold the samples down without blocking the surfaces. The first soaking was for 13 days and the other three for 6 days. It must be noted, that during soaking, the densified and densified + TM specimens always sank during soaking (because of their higher density), whereas only half of the undensified and TM specimens sank at the end of the first (longer) soaking period and at the end of last soaking period. This suggests, that even during 13 days of soaking, the specimens were not fully saturated but also, that repeated cycling might increase the saturation.

The results in Figure 1 clearly show that set-recovery of densified wood can be reduced by TM. Also, the undensified TM specimens exhibited slightly lower thickness swelling than the untreated specimens, which verifies the hydrophobising effect of TM. With the densified specimens, cycling seems to increase the recovery since both the oven-dry thickness and soaked thickness increase slightly after every cycle (even though soaking time was reduced after first cycle). However, without TM full recovery of the densified specimens is not observed, which suggests that some part of the deformation is permanent.

The moisture uptake (measured in grams) is presented in Figure 2. TM in this case seems to slightly reduce the water intake for undensified samples, however, with combined densification and TM the moisture uptake is clearly diminished. This could mean that with the combined action of densification + TM there is some change in the cellular level that could reduce the water uptake. Also, it is interesting to see that even though the average thickness of the densified specimens is not fully recovered (as seen in Figure 1), the water uptake is approximately the same as for untreated or TM (as seen in Figure 2). This means, that water is penetrating the cell walls of densified wood but not recovering it. Perhaps there is cell wall breakage or some change in the wood chemical structure that inhibits the recovery. Wood densification is often performed on the assumption that the cell walls do not suffer from any major damage. These results might indicate contradictory evidence for this particular densifying process. Thus, further analysis is still required to understand related phenomenon properly.

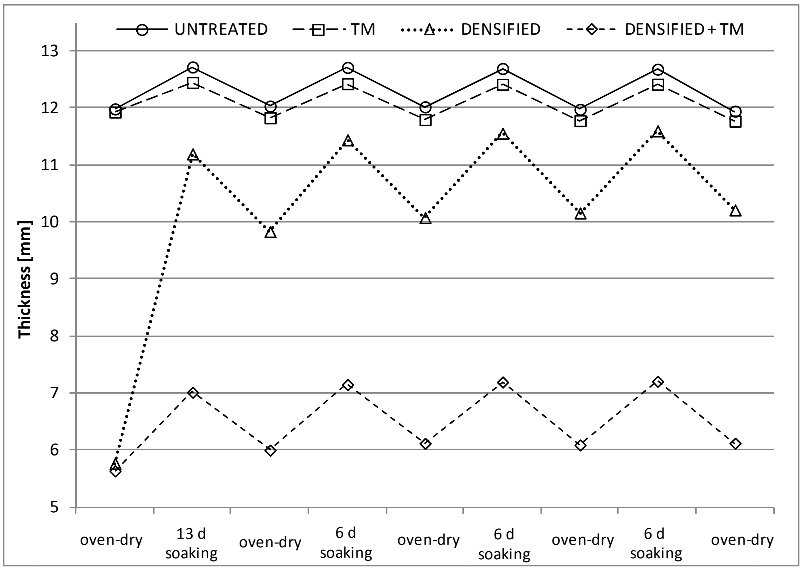

Figure 1. Average (n=6) thickness of differently treated samples during four drying-soaking-cycles.

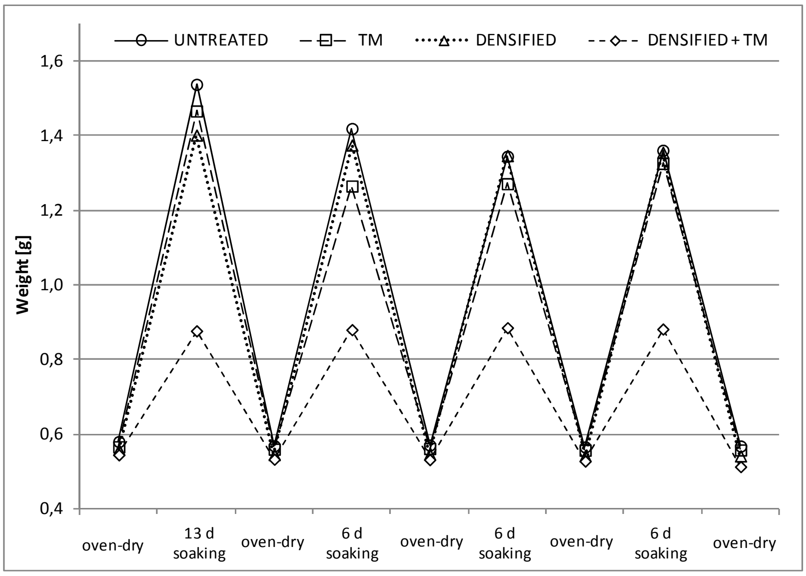

Figure 2. Average (n=6) weight of differently treated samples during four drying-soaking-cycles.

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**Session 4**

# Determination of Thermo-modified Oak Wood Emissivity Properties in the Infrared Spectral Wavelength Range 7.5–13 µm

**Miljenko Klarić[1], Stjepan Pervan[1], Silvana Prekrat[1], Aleš Straže[2], Željko Gorišek[2]**

[1]University of Zagreb, Faculty of Forestry, Croatia
[2] University of Ljubljana, Biotechnical Faculty, Slovenia
mklaric@sumfak.hr

**Keywords:** infrared thermography, emissivity, oak wood, LWIR spectral waveband.

Infrared (IR) thermography is a widely applicable non-destructive testing (NDT) method for measuring the temperature distribution of an observed object surface. Through thermographic analysis, the IR radiation intensity of the electromagnetic spectrum is detected and recorded on the obtained thermograms. IR thermography has its advantages and disadvantages but it is a promising technique, which is easily applicable in the field and has no harmful impacts on the operator or equipment. In wood science and related fields, IR thermography can be applied in production quality control, monitoring of manufacturing equipment and plants for hydrothermal wood processing, wood properties characterization, defects detection (particularly holes, rot and drying cracks), heat flux in wood, and more [1].

During the thermographic analysis, temperature is not measured directly, but indirectly. Therefore, many factors can affect the accuracy of temperature measurement during quantitative thermography analysis, and one such factor is the emissivity of the observed object. Emissivity of the material is the ability of the surface to emit radiant energy expressed as the ratio of the radiant energy emitted by a surface to that emitted by a blackbody at the same temperature and under the same conditions. Emissivity depends on wavelength, temperature, object surface (opacity, roughness, orientation), the material from which the object is made, angle of measurement. Although important, those associated parameters often are not entirely specified along with the emissivity value in the literature. Therefore the experiment for establishing thermo-modified oak wood emissivity values within spectral range 7.5 – 13 µm was conducted. Also the emissivity of innate oak wood was determined for the comparison.

For the experiment, an FLIR infrared system with the following characteristics was used. Imaging and optical data were as follows : field of view 25° x 25°, minimum focus distance 0.1 m, focal length 10 mm, instantaneous field of view (spatial resolution) 2.42 mrad, F-number 1.5, NETD 100 mK, image frequency 9 Hz. Detector type: FPA, uncooled microbolometer, with spectral range 7.5–13 µm (LWIR, ISO–MIR), IRR 180 x 180 pixels.

Oak wood (*Quercus robur* L.) specimens used in the experiment were radial texture elements clear of defects, with approximately the same three ring width. There were 60 innate oak wood specimens and 60 thermo-modified oak wood specimens with the final dimensions 270 mm (length) by 65 mm (width) by 25 mm (thickness). Innate oak wood elements were kiln dried and then four sides planed. The moisture content of the specimens was 9 %. Thermo-modified wood elements were kiln dried, then thermally treated and afterwards four sides planed. The moisture content of the specimens was 5 %. The moisture content of specimens was determined according to EN 13183-1:2002/AC:2003 Moisture content of a piece of sawn timber – Part 1: Determination by oven dry method. A polyvinyl chloride material with known emissivity in long wavelength infrared (LWIR) spectral range was applied to the surface of all specimens. The emissivity of the polyvinyl chloride material was verified and the oak wood specimens were heated to 50 °C.

During the experiment, the ambient characteristics were as follows: air temperature 21 °C, relative humidity 60 %, reflected apparent temperature 20.2 °C, the distance of oak wood specimens surface from the lens was 120 mm, angle of measurement in relation to the object normal was 15°, airflow and dotted reflections were inhibited. Emissivity values were determined via FLIR software with an area box tool with following dimensions 70×30 pixels.

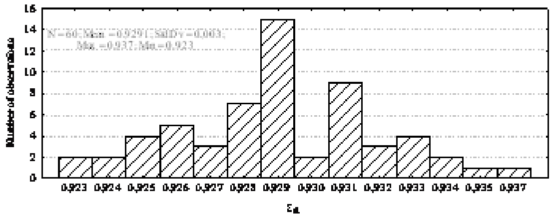

Figure 1. Innate oak wood emissivity values

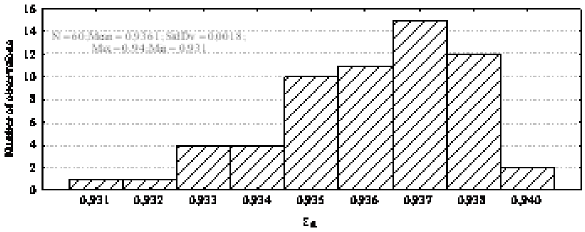

Figure 2. Thermo-modified oak wood emissivity values

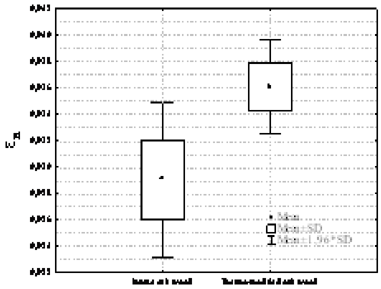

Figure 3. Innate oak wood vs. thermo-modified oak wood emissivity values

There was a significant difference in the results for innate and thermo-modified oak wood emissivity values (**two-sample t**(df) = -15.6, **p** = 0.00).

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**Session 4**

# Characterisation of chemical and physical properties of thermo-modified wood by FT-NIR spectroscopy

**Aleš STRAŽE[1], Željko GORIŠEK[1], Stjepan PERVAN[2], Anna SANDAK[3], Jakub SANDAK[3]**

[1]University of Ljubljana, Biotechnical Faculty, C. VIII/34, SI 1000 Ljubljana, Slovenia
[2]University of Zagreb, Faculty of Forestry, Svetošimunska 25, HR-10000 Zagreb, Croatia
[3]IVALSA/CNR, Via Biasi 75, San Michele All'Adige, I-38010 Trento, Italy
ales.straze@bf.uni-lj.si

**Keywords:** thermal modification, FT-NIR spectroscopy, ashwood, chemical properties, physical properties

The use of near-infrared spectroscopy as a method to determine chemical changes during thermal treatment of wood and its reliability to predict some physical properties of both, treated and untreated material was researched. Therefore, the ashwood (*Fraxinus excelsior* L.) was treated under steam atmosphere with pre-drying, heating at 230 °C for 6 hours, followed by conditioning and cooling. Clear wood specimens (7 × 10 × 170 mm) were made afterwards in a series from innate-(C) and heat-treated wood (HT), for preconditioning of the material (T = 20 °C) in a wide climate range (0 to 97% RH; n = 8), to achieve different equilibrium moisture contents. Three point standard bending tests were carried out afterwards (EN 310), to determine material's stiffness. All the equilibrated specimens were further 3-point measured on the radial plane by fibre-optical probe and FT-NIR spectrometer (VECTOR 22-N; Bruker Optics GmbH), in the spectral range between 4,000 and 12,000 $cm^{-1}$. OPUS 7.0 package was used for spectra pre-processing, which included computation of the second derivatives (Savitzky-Golay), and in some cases the spectra smoothing. Prediction models based on the partial least squares (PLS) regression algorithm, were developed for quantification of the prior determined physical and mechanical properties of wood. The optimization procedure was applied in order to obtain the best performing models. Drying by vacuum drier (T = 50 °C) was the final experimental procedure, to achieve the oven dry state, needed for gravimetrical determination of equilibrium moisture content (EMC).

The most significant influence of heat treatment on ashwood was confirmed at reduction of equilibrium moisture content (ΔEMC = - 50%), and also at wood density (- 10%) and stiffness (- 7%) (Tab. 1).

Table 1. Impact of thermal treatment on average equilibrium moisture content (EMC), wood density and modulus of elasticity (MOE) of ashwood (RH – relative humidity)

| | Untreated wood | | | Heat treated wood | | |
|---|---|---|---|---|---|---|
| RH [%] | EMC [%] | Density [kg/m$^3$] | MOE [GPa] | EMC [%] | Density [kg/m$^3$] | MOE [MPa] |
| 0 | 0.0 | 628 | 9.92 | 0.0 | 579 | 9.51 |
| 20 | 2.6 | 625 | 11.67 | 1.7 | 582 | 9.88 |
| 33 | 4.4 | 647 | 11.32 | 2.7 | 590 | 10.44 |
| 44 | 5.6 | 630 | 11.08 | 3.1 | 592 | 10.50 |
| 65 | 7.0 | 658 | 10.91 | 4.1 | 616 | 10.72 |
| 75 | 9.8 | 710 | 9.83 | 4.8 | 619 | 10.23 |
| 87 | 14.5 | 665 | 9.65 | 5.8 | 615 | 10.41 |
| 97 | 17.8 | 692 | 9.58 | 6.5 | 626 | 10.81 |

The most significant changes in FT-NIR spectra due to heat treatment of ashwood on the equilibrium moisture content (EMC), and consequently on sorption and chemical properties, were found in the regions from 4770 to 5236 $cm^{-1}$, from 5800 to 6126 $cm^{-1}$, and from 6660 to 7321 $cm^{-1}$. Significant changes in spectra were confirmed also at wave numbers 4404 $cm^{-1}$, 5219 $cm^{-1}$, 5618 $cm^{-1}$, 6281-6287 $cm^{-1}$ and at 7418 $cm^{-1}$. These changes can be generally ascribed to significant differences in chemical properties of hemicelluloses, amorphous and semi-crystalline cellulose, and even to the lignin [1-3]. The heat treated wood has generally less significant changes (response) at these spectra bands, whereas at many other bands of HT wood the changes of spectra due to the influence of moisture content almost disappeared.

Values estimated by PLS models were regressed against reference values of determined physical and mechanical properties, and are presented in Table 2. High prediction capability of PLS models was confirmed for all tested variables, the moisture content (MC), wood density and modulus of elasticity (MOE).

Table 2. Validation parameters for PLS regression prediction models developed for innate and heat-treated ashwood ($R^2$ – coefficient of determination; RMSECV - root mean square error of the cross-validation; RPD - residual prediction deviation)

| Variable | Rank | $R^2$ | RMSECV | RPD | Region [$cm^{-1}$] | Preprocessing |
|---|---|---|---|---|---|---|
| Density | 7 | 78.5 | 33.3 | 2.2 | 9403.7 - 6098.1;<br>4601.6 - 4246.7 | Constant offset elimination |
| MOE | 6 | 62.1 | 1930 | 1.6 | 6800.1 - 6098.1;<br>4601.6 - 4424.1 | Constant offset elimination |
| MC | 7 | 97.9 | 0.65 | 7.0 | 5450.1 - 4246.7 | Mult. scattering correction |

The FT-NIR spectroscopy showed high potential for predicting of tested physical properties of innate and heat-treated ashwood. Therefore the method demonstrated high potential for quality assessment of this material. Additionally, the analysis FT-NIR spectra cleared up spectral regions and bands, where significant changes are appearing due to changed water sorption.

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**Session 4**

# Bonding performance of densified VTC beech bonded with liquefied wood

**Andreja KUTNAR[1], Aleš UGOVŠEK[2], Frederick A. KAMKE[3], Milan SERNEK[2]**

[1]Andrej Marušič Institute, University of Primorska, Muzejski trg 2, SI-6000 Koper, Slovenia;
ILTRA d.o.o., Celovška cesta 268, SI-1000 Ljubljana; Slovenia
[2] University of Ljubljana, Biotechnical Faculty, Department of Wood Science and Technology,
Rožna dolina, C. VIII/34, SI-1000 Ljubljana, Slovenia
[3] Department of Wood Science and Engineering, Oregon State University, 119 Richardson Hall,
Corvallis, OR, USA
andreja.kutnar@upr.si

**Keywords:** adhesive, contact angle, densification, liquefied wood, shear strength

Viscoelastic thermal compressed wood (VTC wood) is produced with so-called VTC process. This is a method that uses steam, heat, and mechanical compression to increase the density of wood, without causing fractures in the cell wall. The strength and stiffness of the wood material are increased [1, 2], the morphology of the VTC wood changes significantly and depends strongly upon the degree of densification [5], whereas the surface of VTC wood exhibits hydrophobic behaviour [3, 6]. VTC wood is intended to be used as a component in an engineered composite material which requires adhesive bonding. So far only synthetic adhesives were used to test bonding performance of the VTC wood [2-4]. One naturally-based material that has the potential to replace synthetic adhesives is liquefied wood (LW). Liquefaction of wood is a process in which wood particles are dissolved in organic solvents (phenol, polyhydric alcohols, cyclic carbonates, ionic liquids), with or without a catalyst, at moderate temperatures (from 100 °C to 250 °C). In this research, LW was used for bonding of VTC beech wood (*Fagus sylvatica* L.) and shear strength of bonded specimens was evaluated. VTC beech specimens with two different degrees of densification were analysed with microscopy and sessile drop method (contact angle) for surface energy using water and LW.

Densification strongly influenced morphology of beech wood as seen in Figure 1 in undensified state (B:c; 0.68 g/cm$^3$). Only a few pores remained visible after lower degree of densification (B:a; 1.08 g/cm$^3$), whereas almost completely closed structure was obtained after higher degree of densification (B:b; 1.20 g/cm$^3$). The arrows in B:a and B:b indicate the direction of compression. In VTC specimens B:b unusual deformation of rays was observed. The narrow rays appear in a wavy pattern and the thick ray has an uneven thickness, which might be correlated to the distribution of pores that were completely collapsed. No cell wall fractures were observed after densification.

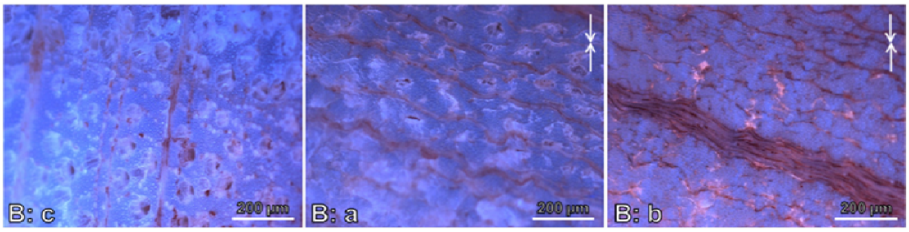

Figure 1. Cross-section view, as observed with UV light, of undensified beech (B:c) and VTC beech wood of lower (B:a) and higher (B:b) degree of densification in completely deformed state

The contact angle of water was significantly different between control and VTC beech specimens (Figure 2). The contact angle of LW did not show significant difference between control and VTC specimens which could be due to lower surface tension of LW [7].

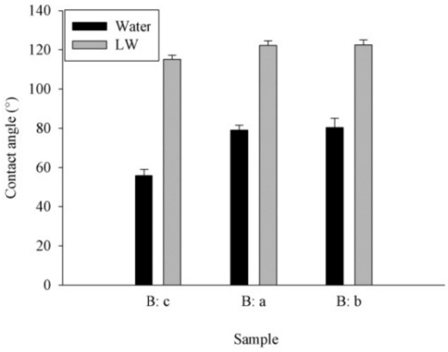

Figure 2. Average (*n = 8-10*) advancing contact angle of water and LW in wood samples of different degree of densification

According to EN 12765 (2001) none of the assemblies bonded with LW achieved standard requirements for non-structural internal applications (≥ 10 MPa). The shear strength of control beech wood assemblies (6.4 ($\sigma = 1.4$) MPa) bonded with LW was statistically greater than shear strength of the highest density VTC wood (1.7 ($\sigma = 1.0$) MPa), whereas the lower degree of densification did not significantly affect the shear strength (4.9 ($\sigma = 1.9$) MPa). The reason for a decrease of shear strength was ascribed to presumably insufficient penetration in the case of VTC beech wood and specific bond line characteristics when bonding wood with LW [8].

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**Session 4**

# Baltic Sea network for efficient and sustainable wood processing based on wood modification processes

**Alexander PFRIEM, Mario ZANDER, Lothar CLAUDER**

EberswaldeUniversity for Sustainable Development - University of Applied Sciences,
Faculty of Wood Science and Technology, Friedrich-Ebert-Straße 28, 16225 Eberswalde, Germany
Alexander.Pfriem@hnee.de

**Keywords:** Baltic Sea Region, Network, Shaping Processes, Wood Modification

Construction using renewable raw materials, combined with high-tech and energy-efficient production processes, enables the development of highly sustainable products. This issue has high potential for innovation and is being taken up by the Eberswalde University for Sustainable Development. The University has created a network for efficient and sustainable wood processing, which is based on wood modification and transformation processes which is an outcome of COST Action activities. These processes are associated with the field of energy and resource efficiency, with aims for sustainable and responsible dealing of renewable raw materials.

Partners from several countries in the Baltic Sea Region, such as Latvia, Estonia, Poland, Sweden and Germany, have joined forces in this network to develop multilateral education and R & D collaborations. They aim to work on current problems in combined modification and transformation processes, the procedural description and calculation and optimization of the processes, as well as to work together on the development of the product design. The website www.wood-modification.net gives a first impression of the network's working methods.

In the long term, within the framework's developed scientific and technical solutions, the goal of collaboration is to lead to practical implementations in modern applications and products. Such processes are handled in current research projects at the Eberswalde University for Sustainable Development in the Faculty of Wood Science and Technology, for example: a research project of the simultaneous shaping and fixation of form using thin layers of wood. By targeted material change, the formability of veneers is improved for the coating of surfaces or for the production of molded articles. The plasticization of the veneers to improve the formability as well as the fixation of form is both realized using an identical chemical substance. Forming, fixation of form and hardening are performed in one operation. Chemical, physical and structural studies are performed on this process, cause-effect relationships are derived, and their general validity and applicability to other types of wood are estimated. Other approaches work to achieve specific changes in material through the use of chemical or thermal modification processes. Emissions are considerably reduced in thermally modified or native woods by chemical techniques.

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**Session 4**

# Fatigue behaviour of VTC and untreated beech wood

**Georg BEHR[1], Holger MILITZ[1], Frederick A. KAMKE[2], Andreja KUTNAR[3]**

[1]Georg-August-Universität Göttingen, Wood Biology and Wood Products, Göttingen, Germany
[2]Department of Wood Science and Engineering, Oregon State University, Oregon, USA
[3]University of Primorska, Andrej Marusic Institute, Koper, Slovenia
georg.behr@forst.uni-goettingen.de

**Keywords:** cyclic creep, densification, 3-point bending, fatigue

Wood as a structural material like metals and plastics breaks down under cyclic deflection or loading. The fatigue properties for wood have been studied in the past in detail [1]. However, none of the studies focused on fatigue properties of densified wood. Therefore the aim of this study was to examine the fatigue behaviour of Viscoelastic Thermal Compressed wood (VTC wood). Since the compression in the VTC process leading to increased density of wood is not causing fractures in the cell wall; the strength and stiffness of the wood material is increased [2, 3]. However, as stated above the performance of the VTC wood under cyclic deflection was till now unknown.

The densification of beech (*Fagus sylvatica* L.) was performed in a pressurized vessel that is equipped with a heated hydraulic press at temperature of 170 °C. The VTC process consisted of the three main phases. The initial phase was steaming of the specimen at 860 kN $m^{-2}$. The specimens were steamed without compression for three minutes. After this treatment, the steam pressure was released, and the specimen was able to vent for 100 s without compression (phase 2). During the venting period the specimen lost moisture. Phase 3 began with an application of a compressive force. During phase 3 the platen temperature was raised from 170 °C to 200 °C and specimens were exposed to 200 °C for 300 seconds. In the last step the specimens were cooled under compression to 100 °C, which lasted 360 seconds. The characteristics of the VTC test specimens are shown in Table 1.

Table 1. Characteristics of the test specimens

| Initial thickness (mm) | Initial density (D1) at MC=0% (g $cm^{-3}$) | Density after compression (D2) at MC=0% (g $cm^{-3}$) | Density ratio (D2-D1)/D1 |
|---|---|---|---|
| 6 | 0.519 | 0.781 | 0.336 |

The tests were carried out at 20 °C and 65 % relative humidity (RH) by the use of the dynamic fatigue test rig by DHM embedded systems, Clausthal-Zellerfeld Germany on densified and undensified control specimens (150 mm (L), 10 mm (T), and 6 mm 3.8 mm (R)) that were conditioned in a controlled environment room (20 °C, 65 % RH) prior to measurements. To determine the force of cyclic loading, 3-point bending tests were performed on VTC and control specimens. Since the thickness of the control (6 mm) and VTC (3.8 mm) wood differed, the span in 3-point bending test and further on in fatigue measurements was different for the VTC wood and control specimens (50 mm and 90 mm respectively). Determined average maximum bending force, $F_{max}$, of VTC wood was 405 N and of control wood 311 N. The force of cyclic loading was selected to be applied at two levels during two separate tests, namely 60 % and 75 % of $F_{max}$. Due to different $F_{max}$, the force applied to the specimens during fatigue measurements was different for VTC wood and control specimens. The tests were performed with the SysCon Easytest software by DHM embedded systems operating a

Festool 4 kN pneumatic piston at a frequency of 14 Hz. The cyclic loading was applied for 50400 cycles (1 h) or until the failure of the specimens occurred. During cyclic loading the maximum and minimum deformation of the specimens were measured. The measurement enabled the analysis of the development of the deformation in the specimens under cyclic loading, the so-called cyclic creep (Figure 1).

The results showed that the cyclic loading at 60 % of $F_{max}$ did not lead to failure of control or VTC wood specimens after 50400 cycles. The cyclic creep rate increased gradually in control and VTC wood specimens, however the absolute deformation was higher in control specimens. Increasing the loading force to 75 % resulted in rapid increase of cyclic creep rate in control specimens, leading to failure already after 5000 cycles. In the VTC specimens initial rapid increase of cyclic creep rate was followed by gradual increase and further rapid increase before failure, which was caused after 27700 cycles. As shown in Figure 1 the cyclic creep was lower in the VTC wood specimens, while critical deflection at failure was lower in VTC wood specimens. Nevertheless, it can be concluded that the VTC densification process improves the performance of wood under cyclic loading.

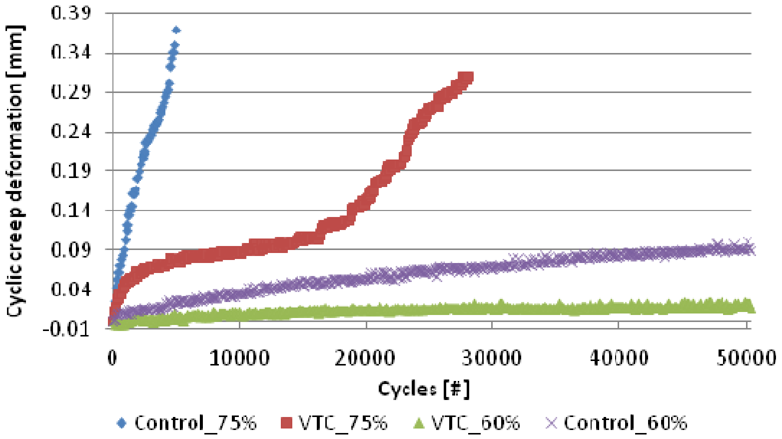

Figure 1. Cyclic creep of control and VTC specimens during cyclic loading at 60 % and 75 % of $F_{max}$.

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**Session 4**

# Gluing ability of Pinus Pinaster depending on its moisture content

**Anne LAVALETTE[1,2], Gérard ELBEZ[1], Alain COINTE[1], Régis POMMIER[1], Michel DANIS[1]**

[1]Université Bordeaux, I2M, UMR 5295, F-33400 Talence, France
[2]Chantier Naval Dubourdieu 1800, 33470 GujanMestras, France
Anne.lavalette@u-bordeaux1.fr

**Keywords:** Green gluing, resin penetration, microscopy

The general objective of this project is to realize an innovative composite material to be used in a plating of a ship. This material should answer to the mechanical characteristics required by the shipbuilding sector and its process should permit to adjust the panel to the desired shape. Traditional wood panels in shipbuilding industry are made from dried veneers, but a study on Polyurethane adhesive revealed that wood panels could be green glued (i.e. wood moisture content higher than the fiber saturated point) [1].Green wood specific properties (such as bending ability) are interesting to realize shaped product, which is a key point in this project. Vacuum formed plywood gets several techniques benefits, such as no dimensional or shape limitations, variable thicknesses allowed in one product, same pressure repartition all over the molded plywood. These benefits lead us to use this process for this work.

We investigated the technical feasibility of producing plywood by green-gluing technology. Several theories explain the bond performance [2]. We ordered these models in three phases during the gluing process, as shown in the following figure. The effect of several parameters influencing the glue line quality is observed for each gluing step. We studied factors such as wood moisture content, glue rate per square meter, viscosity and pressure. The Polyurethane impregnation in wet wood is observed, as well as chemical and mechanical phenomena.

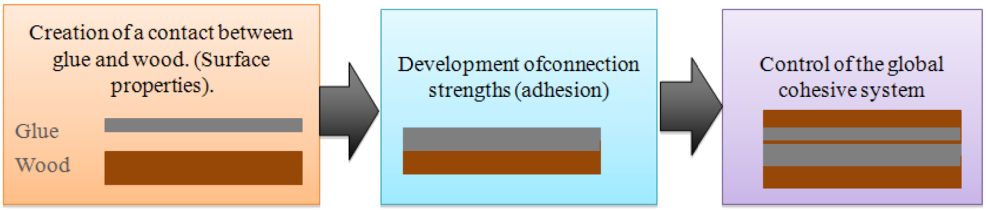

Figure 1. Gluing steps characterizing adherence

The first part of this study is focused on the wood surface characterization (wettability, porosity). Then, researches on the bond line performance are conducted by experimental design method. Shear tests are carried out in order to determine the panels mechanical properties and failure type.In addition to the mechanical tests, microscopic observations provide a better understanding of the internal 3D microstructure of the green-glued plywood. Confocal laser scanning microscopy enables to visualize the internal part of the tested material. Glue penetration depth in wood, pore space saturation and bond line thickness is observed.

First results suggest that wood moisture content and glue spread rate per square meter play an important role on the joint behavior. Experimental design analysis defines a parametric equation using these two factors to determine the ultimate shear stress. A correlation between the glue line

morphology and its mechanical properties is also observed. These analyses allow fixing the panels manufacturing parameters.
To conclude, the document aims at validating the process according to the shipbuilding requirements. Results on traditional shear and bending tests show the potential of this process.

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**Acknowledgments:** The authors are grateful to ANRT (National Agency for Research and Technology) for funding the project and to the Cluster ABOVE for supplying wood raw material. Furthermore, we are thankful to Aalto School of Chemical Technology (Forest Product Technology) in Finland and its team, namely prof. Mark Hughes for their cooperation to this project.

Session 4

# Influence of hot-pressing regimes at MDF production

**Viktor SAVOV, Julia MIHAILOVA, Cvetelin EVSTATIEV**

Department of Wood Mechanical Technology, University of Forestry, Sofia, Bulgaria
victor_savov@abv.bg

**Keywords:** MDF, hot-pressing, duration, temperature

The hot-pressing is main technological stage in order engineering of fiberboards' properties. At MDF production the applied pressure after first stage of hot-pressing has limited significance. That defines as main factors in process to be considered temperature and duration of pressing. The last should be determined in order of wood-species, resin type, wood-mat characteristics and other.

In the presented report is conducted empirical research on influence of temperature and duration of second stage of hot-pressing on properties of MDF produced on the base of Phenol-Formaldehyde resin from hardwood raw material. For the purpose are produced boards at laboratory conditions at alternation of temperature in range from 150° to 200° C and alternation of duration from 45 s to 90 s per millimeter of board thickness. The results for MDF properties are processed with use of regression analyze methods and are deduced dependences for influence of treated factors at the aforementioned alternation range.

On fig.1 is shown the alternation of bending strength at studied range of factors values.

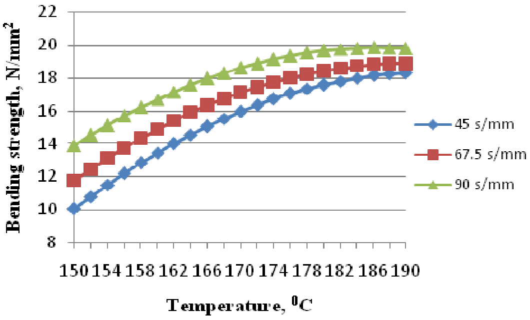

Figure 1. Variation of bending strength, depending on pressing temperature at different levels of pressing duration

With increasing duration of heat treatment and temperature the bending strength of board is improved. The relative bigger increase of strength is observed up to 170$^0$ C then its gradient decrease. At temperatures above 186$^0$ C and duration of pressing from 90 s/mm is observed slight decrease in strength. This may be due to "caramelization" of some wood elements or to initial destruction processes at phenol-formaldehyde resin which could lead to fragility of material. For more complete study of the processes it should be rise diapason of temperature variation. Much more significant is the effect of heat duration at hot-pressing when temperature of plates is 150$^0$ C in comparison when is 190$^0$ C. In the last case even at 70 s/mm are achieved values of studied property closed to maximal ones, fig.2.

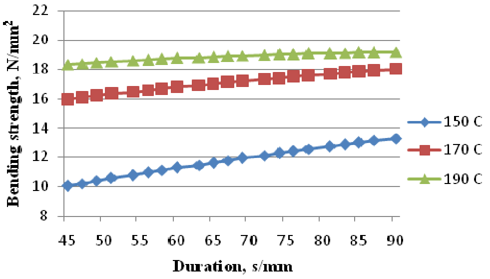

Figure 2. Variation of bending strength, depending on pressing duration at different levels of pressing temperature

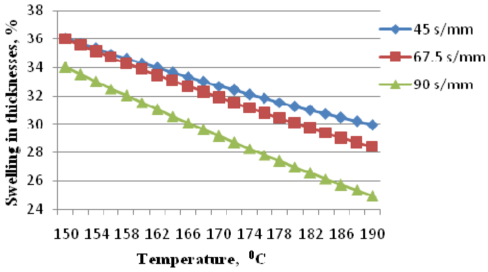

Figure 3. Variation of swelling in thicknesses, depending on pressing temperature at different levels of pressing duration

The dependence of swelling in thicknesses from duration and temperature of pressing is from second degree. This property is improved, decrease its values, at increscent of factor’s levels. Much more significantly is improvement at temperature level of 190$^0$ C and duration of process up to 55 s/mm, fig. 3. At temperature of 150$^0$ C the decrease of swelling in thicknesses in function of treat duration is relatively smaller, fig. 4.

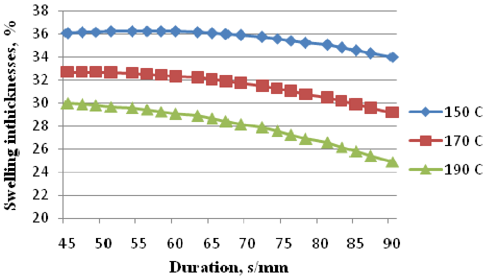

Figure 4. Variation of swelling in thicknesses, depending on pressing duration at different levels of pressing temperature

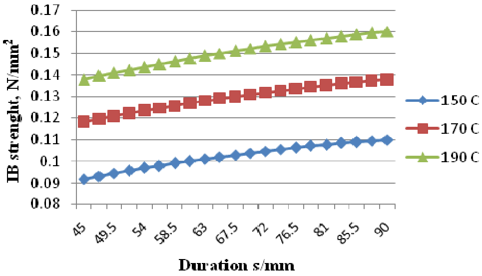

Figure 5. Variation IB strength, depending on pressing duration at different levels of pressing temperature

With increase of hot-pressing duration at temperature of treatment IB (internal bond) strength of boards is improving. Again this tendency is stronger expressed when duration is rising at 190$^0$ C.

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**Session 4**

# Thermo-Hydric behaviour of dowelled and bolted steel-to-timber connections exposed to fire

**Abdoulaye SAMAKE, Patricio PALMILI, Mustapha TAAZOUNT, Philippe AUDEBERT, Maxime AUDEBERT, Abdelhamid BOUCHAÏR**

Institut Pascal, Université Blaise Pascal, Polytech'Clermont-Ferrand, 24 avenue des Landais, BP 206,
63174 AUBIERE, France
mustapha.taazount@univ-bpclermont.fr

**Keywords:** Wood, steel-to-timber connections, fire exposure, numerical modelling

Wood, with its vision of combustible material, is underused in the field of the civil constructions. These observations remain ideas because current Structural Fire Design Standards allow by calculation the requirements of fire resistance ensuring the evacuation of people. The EuroCodes [1,10] provide numerical procedures to determine the fire resistance of member-to-member and steel-to-member connections but tend to be very conservative in some cases [2,3].

The aim of this study is to develop numerical FEM model supported by experimental observations to optimize the calculation methods and reduce the cost of tests [4,5,6,7,8]. Indeed, when timber burns at high temperature ( >90°C) , the moisture content moves in its internal volume and turns into saturated water vapor [8,9,11]. During this hydric transformation an endothermic chemical reaction occurs and helps to slow down the combustion of timber.

This exploration is dedicated to the study of heat transfer in steel-to-timber connections (dowels and bolts) in a fire exposure. The study was performed experimentally by testing specimens of connections in an oven with gas burner (Fig.1) under the action of an ISO 834 fire temperature-time curve (Fig.2). The proposed numerical model is based on finite element modeling and the phenomenon of the thermodynamics phase change of the water contained is taken into account through a numerical value pic of specific heat coefficient which value was adjusted through experimental observations.

Figure 1. Oven with gas burner.

Figure 2. burned specimen

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