



UNIVERSITÀ  
DEGLI STUDI  
FIRENZE  
**DAGRI**  
DEPARTMENT OF  
AGRICULTURE, FOOD,  
ENVIRONMENT AND FORESTRY



 **Consiglio Nazionale delle Ricerche**  
CNR - Istituto per la BioEconomia



# BOOK OF ABSTRACTS

15.04.24 | 16.04.24 • FLORENCE, IT

## ECWM11

*The 11<sup>th</sup> European Conference  
on Wood Modification*  
15/16 April 2024 Florence, ITA



Start-up supporting the Conference



**The 11<sup>th</sup> European Conference on Wood Modification**

**BOOK OF ABSTRACTS**

Florence, Italy, 15-16 April 2024

**Editors:** Paola Cetera, Ignazia Cuccui, Giacomo Goli, Dennis Jones, Holger Militz, Francesco Negro, Luigi Todaro

**Scientific Committee**

Dr. Julia Milne e Carmo (Carmo Group, Portugal)  
Dr. Ottaviano Allegretti (CNR IBE, Italy)  
Pr. Giacomo Goli (Firenze University, Italy)  
Pr. Callum Hill (JCH Industrial Ecology Ltd., UK)  
Dr. Dennis Jones (Luleå University of Technology, Sweden)  
Pr. Holger Militz (Göttingen University, Germany)  
Dr. Marina van de Zee (SHR, The Netherlands)  
Pr. Philippe Gérardin (Lorraine University, France)  
Pr. Joris Van Acker (Ghent University, Belgium)

**Organizing committee**

Pr. Giacomo Goli, Firenze University, Italy – Chair  
Dr. Michele Brunetti, CNR IBE, Italy  
Dr. Paola Cetera, Sassari University, Italy  
Dr. Elena Conti, CATAS, Italy  
Dr. Ignazia Cuccui, CNR IBE, Italy  
Dr. Fabio De Francesco, CNR IBE, Italy  
Dr. Valentina Lo Giudice, Basilicata University, Italy  
Dr. Paola Mazzanti, Firenze University, Italy  
Pr. Francesco Negro, Torino University, Italy  
Dr. Sabrina Palanti, CNR IBE, Italy  
Pr. Luigi Todaro, Basilicata University, Italy  
Pr. Gianluca Tondi, University of Padova, Italy

## **PREFACE**

Dear ECWM participants,

The 11<sup>th</sup> European Conference on Wood Modification, being held in Florence this year, will bring together more than 150 participants from more than 25 countries, demonstrating the vitality of the sector. A total of 96 contributions will be presented during the conference, divided into 49 oral presentations and 46 posters. Thanks to a strong response from the industrial sector, an "Industrial" session has been included this year to show that wood modification is far from being at the research stage. This will give researchers a view of the commercial reality, as well as providing industrialists a view of future opportunities. A special session on "new trends" has also been introduced. The vitality of the sector is also demonstrated by the strong response from industrial sponsors, whom both the Organising and Scientific Committees would like to thank for their help in supporting the conference. This book of abstracts is published in electronic format to provide a trace of the complete set of papers presented for the 11<sup>th</sup> European Conference on Wood Modification, but this year the publication of the proceedings will be attempted in a new format. The proceedings will be published in the Springer Proceedings in Materials series and will be indexed by Scopus. This will give authors the opportunity to have their work recognised in terms of bibliometric indexes, as well as giving the sector a better understanding of the impact of the conference on the scientific world, as the knowledge produced will be tracked. Each author is strongly encouraged to participate with a final paper in the indexed proceedings.

We hope that you will enjoy the programme, full of very interesting presentations, as well as your stay in Florence, and we wish you a fruitful scientific exchange.

Giacomo Goli - Chair of the Organising Committee

Holger Militz - Chair of the Scientific Committee



# MONDAY 15TH APRIL 2024

- 08:15 Registration and welcome coffee
- 08:50 Welcome from Organizing and Scientific Committee
- 09:10 **Session 1 INDUSTRIAL**  
Chair: *Julia Carmo*
- 09:10 1,01 What we know and what we still don't know about industrial TM plants producers in Italy  
**Ottaviano Allegretti**
- 09:30 1,02 Certification of thermally modified timber – the experience and view of an industrialist **Bror Moldrup**
- 09:40 1,03 The thermally treated wood in the world with termo vuoto method  
**Alessio Lucarelli, Umberto Pagnozzi**
- 09:50 1,04 Testing and approval of modified wood within NTR labelling system  
**Ramunas Digaitis, Niels Morsing, Jonas Stenbæk, Fredrik Westin**
- 10:00 1,05 STYL+WOOD® system for the thermal modification of wood  
**Michele Bigon, Sonia Marchiori**
- 10:10 1,06 Commercial wood products achieved by industrial thermal treatment process  
*Paola Cetera, Alessandro Porcu*
- 10:20 Coffee
- 11:20 **Session 2 MODIFICATION WITH CHEMICALS**  
Chair: *Philippe Gerardin*
- 11:20 2,01 Studying the impact of a silicone oil treatment on the elasto-mechanical properties of wood *Lukas Emmerich, Kilian Erdelen, Holger Militz*
- 11:30 2,02 Modifying wood with a bio-based thermosetting resin – different approaches to curing and drying **Christoph Hötte, Holger Militz**
- 11:40 2,03 Hydrophobisation of beech wood scantlings with radiation-curing epoxidised vegetable oils for use as dimensionally stable components in exterior applications  
**Christiane Swaboda, Roger Scheffler**
- 11:50 2,04 Dimensional stability of Scots pine sapwood modified by tannin-based formulas  
*Sheikh Ali Ahmed, Gianluca Tondi, Filippo Rizzo, Reeta-Maria Stöd, Reza Hosseinpourpia*
- 12:00 2,05 Improving fire resistance of wood through a combined chemical and thermo-mechanical treatment  
**Črt Svajger, Alexander Scharf, Chia-feng Lin, Olov Karlsson, Dennis Jones, Miha Humar, Dick Sandberg**
- 12:10 2,06 Development of novel guitar fretboards by thermal modification and impregnation with PF-resin of beech (*Fagus sylvatica*) and maple (*Acer* spp.) wood  
**Christina Zwanger, Marcus Müller**
- 12:20 2,07 A study of the influence of the curing conditions on Scots pine treated with SorCA coupled with catalysts  
**Adèle Chabert, Katarzyna Kurkowiak, Holger Militz**



- 12:30 2.08 Wood modification by different chestnuts tannin – furfuryl alcohol resins and effect on conferred wood durability  
**Christine Gerardin Charbonnier**, Joao Vitor Dorini, Pedro Gonzales de Cademartori, Philippe Gerardin
- 12:40 LUNCH
- 14:00 **Session 3 POSTERS 1**  
Chair: Joris van Acker
- 14:00 3.01 Heat treatment of *Cryptomeria japonica* from Azores  
Yurlet Mercado, Lina Nunes, **Bruno Esteves**, Luísa Paula Cruz Lopes
- 14:03 3.02 Effects of QUV accelerated weathering on surface hardness of thermally modified woods (*Fagus Sylvatica* L. and *Pinus nigra*)  
Holta Cota, Entela Lato, Leonidha Peri, **Hektor Thoma**, Doklea Quku, Dritan Ajdinaj, Erald Kola, Marco Togni, Giacomo Goli, Ottaviano Allegretti
- 14:06 3.03 Effect of thermal post-treatment on the properties of densified *Ceiba pentrandia* Wood  
Larissa Mesquita do Vale, **Claudio Del Menezzi**
- 14:09 3.04 Analysis of thermally modified Norway spruce shingles after eight years of use  
**Boštjan Lesar**, Davor Kržišnik, Miha Humar
- 14:12 3.05 Physical properties of thermally modified *Gmelina arborea* wood modified under different process conditions  
Samuel Olaniran, **Holger Militz**
- 14:15 3.06 Effect of thermal treatment on the interaction of wood with liquid water  
Dace Cirule, **Edgars Kuka**, Ingeborga Andersone, Bruno Andersons
- 14:18 3.07 Direct evaluation of the effect of thermal treatment on the parallel compression strength of wood  
Rossana Rosa, Isabella de Sá, Bento Viana, Paula Dornelles, Lucia Garcia, **Claudio Del Menezzi**, Annie Cavalcante
- 14:21 3.08 Increasing opportunities for Maillard-type reactions in wood through the addition of glucose and citric acid to bicine and tricine modification  
**Domen Borko**, Alexander Scharf, Chia-feng Lin, Olov Karlsson, Dennis Jones, Miha Humar, Dick Sandberg
- 14:24 3.09 Thermally modified wood in wood-PLA composites for 3D printing  
**Daša Krapež Tomec**, Mirko Kariž, Manja Kitek Kuzman
- 14:27 3.10 Plywood panels made of alternate layers of thermally densified and non-densified alder and birch veneers  
**Pavlo Bekhta**, Tomáš Pipiška, Vladimír Gryc, Pavel Král, Jozef Ráhel', Jan Vaněrek, Ján Sedliačik
- 14:30 3.11 Improving the commercial value of some Canadian West Coast species through thermal modification  
Yaohui Liu, **Gregory Smith**, Philip D. Evans, and Stavros Avramidis
- 14:33 3.12 Durability of thermal modified wood of *Pinus pinaster*, *Pinus radiata* and *Pinus sylvestris* from Galicia, Spain  
**David Lorenzo**
- 14:36 3.13 Effect of thermal modification on the color of *Hymenaea* spp. and *Ficus* sp. wood  
**Kamilyly da Silva Pereira**, Anna Clara Oliveira Rupf, Paulo Henrique dos Santos Silveiras, Djeison Batista, Victor Fassina Brocco, Saulo Lima
- 14:39 3.14 The main challenges in bonding heat-treated wood  
**Milan Sernek**
- 14:42 3.15 Surface properties of thermally modified beech wood after radio-frequency discharge plasma treatment  
**Ján Sedliačik**, Pavlo Bekhta, Igor Novák, Angela Kleinová, Ján Matyašovský, Peter Jurkovič
- 14:45 3.16 Effect of paraffin-thermal modification on water absorption and dimensional stability of Louro-preto wood (*Nectandra dioica*)  
**Saulo Lima**, Anna Clara Oliveira Rupf, Kamilyly da Silva Pereira, Paulo Silveiras, Djeison Batista, Fernando Andrade
- 14:48 3.17 Study of the machinability of thermally and chemically modified wood for art objects  
**Leila Rostom**, Jérémie Damay, Philippe Gerardin, Michael Jousserand
- 14:51 3.18 Moisture diffusion characteristics of thermally modified beech wood  
**Aleš Straže**, Primož Tomec, Zeljko Gorisek, Jure Žigon
- 14:54 3.19 Exploring the mechano-sorptive behavior of thermally modified wood  
Claude Feldman Pambou Nziengui, **Giacomo Goli**, Rostand Moutou Pitti
- 14:57 3.20 Temperature and moisture content effects on wood compressive properties  
**Hussein Daher**, Sabine Caré, Gilles Forêt, Loïc Payet
- 15:00 3.21 Moisture content distribution of densified wood and the impact of various heat post-treatments on Brinell hardness and set recovery  
Elena Jäger, **Guillaume Andre**, Thomas Volkmer
- 15:03 3.22 Correlation between color and biodeterioration of short-rotation thermally modified teak wood  
**Anna Clara Oliveira Rupf**, Kamilyly da Silva Pereira, Saulo José da Costa Lima, Paulo Henrique dos Santos Silveiras, Jessica Sabrina da Silva Ferreira, Jaqueline Rocha de Medeiros, Adriano Ribeiro de Mendonça, Juarez Benigno Paes, Djeison Batista
- 15:15 COFFEE
- 16:15 **Session 4 MODIFICATION WITH CHEMICALS**  
Chair: Holger Militz
- 16:15 4.01 Acetylation of European hornbeam wood (*Carpinus betulus* L.) – An 8-year-long study  
**Robert Nemeth**, Fanni Fodor
- 16:25 4.02 Solvent-exchange acetylation of simulated green Scots pine wood  
**Mikko Valkonen**, Md Tipu Sultan, Lauri Rautkari
- 16:35 4.03 Mechanical properties and biological durability of wood modified with PEG and various carboxylic acids  
**Melissa Christ**, Nicole Flaig, Marcus Müller
- 16:45 4.04 Novel wood modification through the use of heterocyclic organic compounds  
**Alexander Scharf**, Henric Dernegård, Johan Oja, Dick Sandberg, Dennis Jones
- 16:55 4.05 Combining kraft lignin-glyoxal and organic phase-change materials for a modified wood with thermal-energy storage capacity  
Chia-feng Lin, **Olov Karlsson**, Dennis Jones, Dick Sandberg
- 17:05 4.06 Compatibility of lignocellulosic materials to form thermoplastic film by a single esterification reaction: wood and natural fibers  
**Prabu Satria Sejati**, Laura Roche, Jennifer Afrim, Vincent Mariani, Frédéric Fradet, Philippe Gerardin, Firmin Obounou Akong, Firmin Obounou Akong

- 17:15 4,07 Furfurylated wood : using Pyrolysis-GC/MS to characterize polymer-wood bonds existence  
*David Hentges, Philippe Gerardin, **Stephane Dumarcay***
- 17:25 4,08 Mould growth, fungal growth and strength of wood treated with maleic anhydride combined with sodium hypophosphite  
*Injeong Kim, Lone Ross, Gry Alfredsen, Olov Karlsson, **Dennis Jones**, George I. Mantanis, Dick Sandberg*
- 17:35 4,09 Effect of lactic acid impregnation on some physical properties of wood  
**Miklós Bak**, Robert Nemeth, Mátyás Báder
- 17:45 4,10 Relevant bonding aspects of acetylated beech (*Fagus sylvatica* L.) LVL for load-bearing construction in exterior use  
**Maik Slabohm**, Jan-Oliver Haase, Holger Militz
- 17:55 End of day 1
- 20:00 **CONFERENCE BANQUET at Palazzo Budini Gattai**  
*P.za della SS. Annunziata, 1, 50122 Firenze FI*



## TUESDAY 16TH APRIL 2024

- 08:15 *Arrival and welcome coffee*
- 09:00 **Session 5 ANALYSIS**  
*Chair: Marina van der Zee*
- 09:00 5,01 VOCs emission from thermally treated poplar solid wood and plywood  
*Corrado Cremonini, **Francesco Negro**, Roberto Zanuttini*
- 09:10 5,02 Physical, mechanical and biological tests of solid wood and bio-composites with bioPCM and thermal characteristics of small-scale models in three European countries  
**Sabrina Palanti**, Giovanni Aminti, Andrea Atena, Paolo Burato, Michele Brunetti, Gaye Köse Demirel, Özge Nur Erdeyer, Fabio De Francesco, Mohamed Jebrane, Meysam Nazari, Michela Nocetti, Güliz Öztürk, Benedetto Pizzo, Thomas Schnabel, Federico Stefani, Ali Temiz, Nasko Terziev, Jakub Grzybek
- 09:20 5,03 Comprehensive multi-scale investigation of heat treated wood at room or elevated temperature: summary of our decade's researches  
**Siqun Wang**, Dong Xing, Xinzhou Wang, Deliang Xu, Yujie Meng, Jian Li, Timothy Young
- 09:30 5,04 Resistance of thermally and chemically modified timber against soft rot and findings to improve the lab test  
**Wolfram Scheiding**, Kordula Jacobs, Christian Brischke, Susanne Bollmus
- 09:40 5,05 The chemical interactions between phenolic resin and wood studied by liquid-state NMR spectroscopy  
**Carlo Kupfernagel**, Daniel Yelle, Morwenna Spear, Graham Ormondroyd, Andrew Pitman
- 09:50 5,06 Decay and termite resistance on sapwood, transition wood, and heartwood of short rotation teak wood by chemical and thermal modification  
**Resa Martha**, Beatrice George, Istie Sekartining Rahayu, Philippe Gerardin, Wayan Darmawan
- 10:00 5,07 The Influence of moisture content and thermal modification on the non-linearity in mode I fracture of spruce wood  
*Miran Merhar, **Rostand Moutou Pitti***
- 10:10 **COFFEE**
- 11:00 **Session 6 THERMAL MODIFICATION**  
*Chair: Giacomo Goli*
- 11:00 6,01 Influence of thermal modification on fatigue life of Norway spruce wood  
**Miha Humar**, Boštjan Lesar, Davor Kržišnik, Gorazd Fajdiga
- 11:10 6,02 Detection of the aromatic profile of different thermally modified wood species  
**Valentina Lo Giudice**, Angelo Rita, Luigi Todaro
- 11:20 6,03 Wood modification methods and fire resistance of façades/cladding  
**Joris Van Acker**, Liselotte De Ligne, Bogdan Parakhonskiy, Andre Skirtach, Jan Van den Bulcke, Marcy Durimel
- 11:30 6,04 Comparison of major wood heat treatment technologies paves the way for a generalized mass loss kinetic model  
**Bertrand Marcon**, Giacomo Goli
- 11:40 6,05 Natural weathering of thermally modified wood cladding treated with fire retardants at different exposure levels  
**Inge Wuijters**, Imke De Windt, Kurt De Proft, Lieven De Boever

- 11:55 **Session 7 DENSIFICATION AND MINERALISATION**  
*Chair: Dennis Jones*
- 11:55 7,01 Frictional behaviour of modified-in-surface hardwoods preliminary obtained through strong tribological transformation  
**Pierre-Henri Cornuault**, Stani Carbillet, Luc Carpentier
- 12:05 7,02 Removal of non-cellulosic wood constituents and subsequent densification for improved mechanics of wood  
**Matthias Jakob**, Ulrich Müller, **Wolfgang Gindl-Altmutter**
- 12:15 7,03 Bending performance of thermo-hydro-mechanically treated Scots pine (*Pinus sylvestris* L.) at elevated temperature  
**Lei Han**, Dick Sandberg, Andreja Kutnar
- 12:25 7,04 Wood modification via geopolymer impregnation: Effects on decay, mechanical properties and fire retardancy  
**Aitor Barbero Lopez**, Paivo Kinnunen, Antti Haapala
- 12:35 7,05 Wood modification by bio-inspired hydroxyapatite mineralization  
**Matic Šitar**, Boštjan Lesar, Andreja Pondelak
- 12:45 7,06 An innovative process of mineralisation with magnesium compounds improves fire properties of wood  
**Andreja Pondelak**, Andrijana Sever Škapin, Nataša Knez
- 13:00 **LUNCH**
- 14:00 **Session 8 POSTERS 2**  
*Chair: Callum Hill*
- 14:00 8,01 Modification of wood by fast Pyrolysis Bio-Oil – results from the screening test  
**Anna Sandak**, Jakub Sandak, Faksawat Poohphajai, Rene Herrera Diaz, Ana Gubenšek, Karen Butina Ogorelec, Wojciech Pajerski, Lex Kiezebrink, Klaas Jan Swager, Hans Heeres, Bert van de Beld
- 14:03 8,02 Anatomical variations between natural and delignified wood: a case of study of some Italian “minor” wood species  
**Francesco Bolognesi**, Alessandra Bianco, Francesca Romana Lamastra, Marco Togni
- 14:06 8,03 Improving the energy storage properties of wood by using lauric acid  
**Ahmet Can**
- 14:09 8,04 Evaluation of treatments for preventing resin exudation through coatings  
*Dennis Jones, Aubin Vieillescazes, Micael Öhman, Olov Karlsson, Rostand Moutou Pitti*
- 14:12 8,05 Preliminary evaluation of wood impregnated with oak bark-derived residuals  
**Rene Herrera Diaz**, Mariem Zouari, Faksawat Poohphajai, Jakub Sandak, Anna Sandak
- 14:15 8,06 Optical properties of spectrally irradiated wood  
**Hiroyuki Sugimoto**, Kai Maruyama, Masatoshi Sugimori
- 14:18 8,07 Exploring the potential of carbon nanodots as an UV protection reagent for wood  
*Sarah Jue, Chia-feng Lin, Alexander Scharf, Dennis Jones, Rostand Moutou Pitti, Dick Sandberg*
- 14:21 8,08 Identifying influential factors affecting wettability patterns on wood surfaces through multilevel analysis  
*Valentina Lo Giudice, Petar Antov, Lubos Kristak, Nicola Moretti, Angelo Rita, Luigi Todaro*
- 14:24 8,09 Dimensional stability and sorption properties of acetylated and non-acetylated birch plywood as a function of the face veneer grain angle  
**Jure Žigon**, Yue Wang, Tianxiang Wang, Aleš Straže, Magnus Wållinder
- 14:27 8,10 Upgrading sawdust from wood bark to produce new thermoplastic materials  
**Firmin Oboutou Akong**, Célia Pinto, Ania Belarbi, Prabu Sejati Satria, Philippe Gerardin
- 14:30 8,11 Micromorphological and chemical changes of densified ash wood (*Fraxinus americana*)  
**Alexandra Guevara Castillo**, José Antonio Silva Guzmán, Francisco Javier Fuentes Talavera, Raúl Rodríguez Anda
- 14:33 8,12 Development of innovative methods for assembling lignocellulosic materials for the manufacture of glasses  
**Adrien Magne**, Juliette De Nas De Tourris, Jennifer Afrim, Teldja Benzid, Prabu Satria Sejati, Firmin Obounou Akong, Robin Féron, Philippe Gerardin
- 14:36 8,13 Exploring the solid wood modification with preserved hierarchical structure via non-cellulosic substances removal  
**Yi Hien Chin**, Pascal Biwole, Joseph Gril, Christophe Vial, Rostand Moutou Pitti, Salah-Eddine Ouldboukhitine, Nicolas Labonne, Yoshiki Horikawa
- 14:39 8,14 Malic acid/glycerol polyester treated beech boards: curing kinetics and density distribution  
**Emmanuel Fredon**, Romain Rémond, Adèle Chabert
- 14:42 8,15 Implementing fire retardants into a biobased adhesive system for wood-based composites  
**Luka Kopač**, Alexander Scharf, Dennis Jones, Dick Sandberg, Sergej Medved
- 14:45 8,16 Laser incising – a philosophical shift: from timber treatment to wood modification  
**Morwenna Spear**, Paul Mason, Geraint Williams, Graham Ormondroyd
- 14:48 8,17 X-ray CT scanning as a method for quantifying mineralization in spruce and beech woodblock  
**Marcy Durimel**, Liselotte De Ligne, Bogdan Parakhonskiy, Jan Van den Bulcke, Andre Skirtach, Joris Van Acker
- 14:51 8,18 Wood surface modification using metal and ceramics to make wood fire and termite resistant  
**Laurence Podgorski**, Alain Denoirjean
- 14:54 8,19 Production and application of chemically modified cellulose nanofibrils  
**Primož Oven**, Ida Poljanšek, Vesna Žepič, Jaka Levanič, Urša Osolnik, Viljem Vek
- 14:57 8,2 Effects of microwave treatment on the improvement in the retention of a preservative product in two Portuguese wood species  
**Fernando Mascarenhas**, André Dias, Alfredo Dias, André Christoforo, Rogério Simões
- 15:00 8,21 Wood modification as an opportunity for local wood species in musical instrument making  
**Mario Zauer**, Tobias Dietrich, Herwig Hackenberg, André Wagenführ
- 15:03 8,22 Maximum compressibility along the grain of different wood species  
*Mátyás Báder, Miklós Szauer, Robert Nemeth*
- 15:06 8,23 Studies on the durability of the reaction to fire performance of melamine formaldehyde resin and phosphorus polyol treated wood  
**Muting Wu**, Lukas Emmerich, Holger Militz
- 15:09 8,24 Effect of aspen face veneer thickness on the fire performance of post-manufacture fire-retardant treated birch plywood  
**Percy Festus Alao**, Anti Rohumaa, Karl Harold Dembovski, Jussi Ruponen, Jaan Kers

15:15

COFFEE

16:15

## Session 9 NEW TRENDS

Chair: *Ottaviano Allegretti*

16:15

Ultrafast self-propelling directionally water transporting wood via constructing multi-hierarchical structures on cell wall

**Yanjun Xie**

16:25

Delignified wood as substrate for nanostructured composites with extended range of functionalities

**Lars Berglund**

16:35

Optical Wood with switchable solar transmittance for all-round thermal management

**Daxin Liang, Yanjun Xie**

16:45

Functional transparent wood through incorporation of modified antimony-doped tin oxide nanoparticles

**Zhe Qiu**

16:55

Enhancing building energy efficiency: impregnation of wood with phase change materials

**Jakub Grzybek, Thomas Schnabel**

17:05

Optical smart transparent wood via based on phase-change copolymer

**Yonggui Wang**

17:15

Thermoplastic from wood: dream or reality?

**Philippe Gerardin, Prabu Satria Sejati, Frédéric Fradet, Firmin Obounou Akong, Firmin Obounou Akong**

17:30

CLOSING REMARKS

Announcement of ECWM12

Announcement of PhD best oral and best poster prize

18:00

CLOSE OF CONFERENCE





# **DAY 1**

## **SESSION ONE**

### **INDUSTRIAL**

## Oral 1.01 - What we Know and What we Still don't Know About Industrial TM Plants Producers in Italy

Ottaviano Allegretti<sup>1</sup>

<sup>1</sup>CNR-IBE Istituto per la Bioeconomia of the National Research Council of Italy, via Biasi, 75 38098 San Michele all'Adige TN, Italy. E: ottaviano.allegretti@cnr.it

**Keywords:** accelerated laboratory tests, advertising hype, comparative data, durability, energy efficiency, field tests, wood modification technologies

### ABSTRACT

It is known that Italy, despite being a strong importer of raw wood, has a significant tradition in the production and export of machinery for wood processing throughout its various transformation phases. Since around 2000, some of the many Italian producers of wood dryers have begun to show interest in developing their own technologies for wood modification, mostly through thermal processes, often integrated into the drying process. Behind many brands of plant producers there are typical small Italian family-run businesses, now led by the second or third generation, mostly located in the northeast of the country. Their interest in the development of wood modification technologies is a testament to their innovation efforts and reflects these companies' ability to adapt to market needs but also the need to find new market niches to withstand global competition.

The Wood Drying Laboratory of CNR-IBE in San Michele all'Adige, established in the 1960s by Prof. Cividini and now directed by the authors in the third generation, has always had a traditional role as a scientific partner with companies and associations in the sector. Here, in the 1960s, in collaboration with Maspell, the first patents for vacuum wood drying were developed. Fifty years later, in the same laboratory and still in collaboration with WDE Maspell, the first tests of vacuum heat treatment (thermovacuum) were carried out using a prototype still used in various research programs, including the European project ECOINNOVATION TW4NEWOOD, completed in 2018, and the current Horizon 2023 project BIOBUILD focused on innovative biobased building materials with thermal energy storage function.

Recently, CNR IBE, together with other European scientific partners, cooperated with BIGonDry srl, another dryer manufacturer, for the development and optimization of the thermal treatment system called Styl+wood.

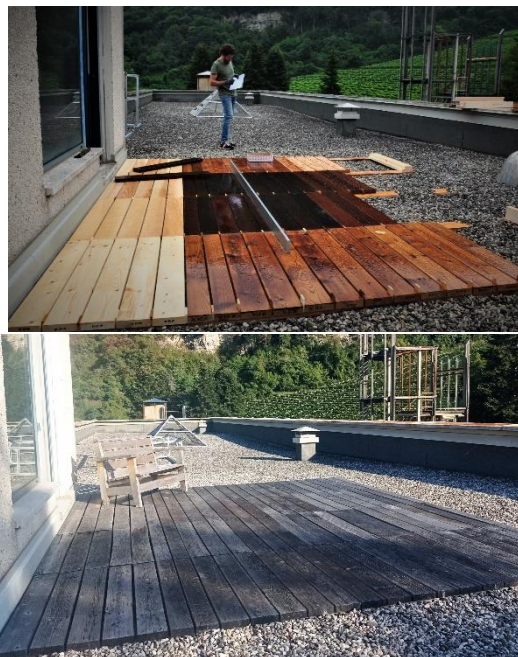
In 2011, CNR IBE, together with Federlegno-Arredo, organized a national workshop on thermally modified wood. In addition to an informative intent, there was also an attempt to bring together the various players in the sector and coordinate the establishment of an Italian brand that would regulate production, as had already happened in other European countries. The failure of the initiative is a testament to the poor ability of individual small Italian companies to collaborate, due to the high internal competitiveness and their very low propensity to invest in research and development.

The current result of this fragmentation and DIY trend has over time led to consequences such as the disappearance of some plant manufacturing companies and the abandonment of others in dealing with modified wood, also following some incidents such as fire cases or forced shutdowns of plants due to pollution problems. It is also relevant that in Italy, alongside a significant turnover in the sale of plants, there is a negligible production and sale of TMW, where the domestic consumption is mostly supported by imports and small-volume productions for niche markets such as saunas or outdoor furniture. In these cases,

the interest is driven by a limited number of wood species such as thermo-treated ash as a substitute for more expensive or restricted tropical woods.

In this context, CNR IBE has had a privileged panoramic view of the Italian TMW industry and has been able to directly access comparative data on the qualitative performance of the product from various competitors, with some results already published and others discussed here. Many characteristics of TMW are widely expected and entirely comparable among different competitors. In general, a direct correlation between the mass loss (ML) and physical and mechanical characteristics was always observed. In general, under the same process parameters, the Styl+wood treatment produces higher ML than the Thermovacuum treatment. An exception is the conferred durability, which surprisingly is better in the former case. For example, for ash and beech (natural durability class = 5), the Thermovacuum treatment brings durability to class 3, while the Styl+wood treatment improves durability up to class 1. These are data obtained from accelerated laboratory tests. Currently, further investigations are underway through standard and non-standard long-term field tests.

However, on other decisive aspects regarding the qualities of a production system, we do not have certain direct data beyond the advertising hype of some. Among such aspects are certainly those concerning pollutant emissions, energy efficiency, and the quality of materials and equipment of the plants. These are all aspects on which the differences between different production systems probably lie, and potential customers have difficulty navigating, also because needs vary from country to country based on different standards and legal obligations.



**Non standard comparative weathering deck in terrace of the CNR IBE in San Michele all'Adige (TN), new and after 3 years. Comparison among different wood species not treated and treated at different intensity level with Styl+wood and Thermo vacuum methods.**

## **Oral 1.02 - Certification of Thermally Modified Timber - the Experience and View of an Industrialist**

Bror Moldrup<sup>1</sup>

<sup>1</sup>COO, IWT-Moldrup, Lerbaek Moelle, 7100 Vejle, Denmark. E: bm@moldrup.com

**Keywords:** EN113-2:2020, EN350:2016, TMT certification, TMT product control, TMT and use classes.

### **ABSTRACT**

Quality control and certification of thermally modified timber (TMT) has undergone a number of changes after the product was introduced on the industrial market in the 1990'ies. But the standards and certification still leave a lot of questions, misunderstandings and faults to be resolved before it will provide a sound and true basis for product and quality assessment. The paper deals with these issues based on theoretical and practical experience by an equipment/process supplier (Moldrup-SSP) as well as plant operator. Even the latest version of EN113-2 from 2020 and EN350 from 2016 have major flaws and fail to address the basic question of how to provide quality assessment and certification for durability based on the use areas of TMT. Treated product control as mostly done today may not offer a good picture of the quality of the finished product when bought by the final user and may result in disappointed expectations that will have an unnecessary negative impact on the product in the long term.



## Ora 1.03 - Thermally Treated Wood in the World with TermoVuoto Method

Alessio Lucarelli<sup>1</sup>, Umberto Pagnozzi<sup>1</sup>

<sup>1</sup>WDE Maspell, Strada di Maratta, 7 int. 1, 05035 Narni (TR), Italy E: alessio.lucarelli@wde-maspell.it; umberto.pagnozzi@wde-maspell.it

**Keywords:** closed system, thermally treated wood, vacuum

### ABSTRACT

Thermal treatment (or heat treatment, or thermal modification) of the wood can be obtained throughout different technologies:

- Cells for thermal treatment in a vacuum environment;
- Cells for thermal treatment at ambient pressure, in a superheated steam environment;
- Cells for thermal treatment saturated with nitrogen at atmospheric pressure;
- Autoclaves for heat treatment under pressure, with saturated or superheated steam;

Although the result at the end of the process is intended to be the same, the above technologies show some differences that characterize the end-product: the peculiarities of the vacuum technology have been briefly mentioned in this document only aiming to describe some of these differences.

#### **Applications of Thermally Treated Wood.**

The possible applications of heat-treated wood are many and the searches for new applications are constantly increasing. We list some of the most common or innovative uses:

- Building Industry

Outdoor: facades, wall claddings, panelling;

Indoor: flooring, ceiling, panelling, frames, doors, windows;

- Furniture industry

Indoor furniture and accessories

Outdoor furniture

- Urban furniture

Benches, tables, chairs, etc...

Walkways, platforms and beach patios

- Saunas, flooring and pool furniture
- Decking for boats and piers
- Flooring for trailer decks
- Musical instruments
- Furnishing accessories and objects

#### **Vacuum Thermally Treated Wood: A “Green” Process For A “Green” Product.**

Thermally treated wood under vacuum is a truly eco-friendly material, due to the following reasons, concerning either the production process and the final product itself:

- Possible power supply of the system from renewable energy sources (solar panels, accumulators);
- Energy saving: the plant is a closed system, without energy dispersion. Moreover, the air-to-air exchange cooling system, not using water spray, allows to eliminate both air pollution and water consumption;
- Wood thermal treatment process involves no chemical additives \*;
- Process gases are condensed and stored for disposal as “non-hazardous liquid industrial waste”;

- Thermo-treated wood is “environmentally friendly” throughout its life: once its life cycle is over, it is totally recyclable \*;
- V.O.C. (Volatile Organic Compound) – A study, carried out by CNR-IVALSA in 2015, (Sánchez del Pulgar *et al.* 2015) shows that “(...) the wood treated in vacuum seems to have a VOC emission close to that of the untreated (wood) samples (...)”. The practical result, unique in the market, is that the wood thermally treated through vacuum technology is also suitable for indoor uses.
- The use of thermally treated wood contributes to the reduction of deforestation and imports of “exotic” woods \*. \* True, regardless the technology used.

### Geography of The Markets of Thermally Modified Wood

Use of thermally treated wood is diffused worldwide, mainly in the following regions:

- North Europe (where the thermally treated wood as known today was originated)
- Central – Western Europe - North and South America
- South-East Asia

Thermally treated wood is growing also in some areas of Africa and Oceania.

Furthermore, the “Treated” species in the world are many: in the table “A” we list the most common:

**Table 1: Most common thermally treated wood species**

HARDWOOD	SOFTWOOD	EXOTIC
Red Oak	Sylvan Pine	Ayous
Ash	Yellow Pine	Iroko
Hemlock	Radiata Pine	Movingui
Larch	Spruce	Frake
Beech	Alder	Mahogany
Poplar	Larch	

### Thermally Treated Wood - Market Expected Growth

Research, conducted in 2022 by Bosson Research through face-to-face interviews with 13 of the world's leading manufacturers of heat-treated wood, predicts a market growth rate (CAGR) of around 40% by 2028, at a rate of 7% per year.

### REFERENCES

Bosson research (2022) - Global Thermally Modified Wood Boards Market Research Report (Status and Outlook).

Sánchez del Pulgar, J., Santoni, I., Romano, A., Cappellin, L., Cuccui I., Biasioli, F. & Allegretti, O. (2014) Rapid assessment by PTR-ToF-MS of the effect on volatile compound emission of different heat treatments on larch and spruce. In proceeding of COST FP0904, final conference. May 19 -21 Skelleftea, Sweden.

## Oral 1.04 - Testing and Approval of Modified Wood within NTR Labelling System

Ramunas Digaitis<sup>1</sup>, Niels Morsing<sup>1</sup> Jonas Stenbæk<sup>1</sup>, Fredrik Westin<sup>2</sup>

<sup>1</sup>Wood and Biomaterials, Danish Technological Institute, Gregersensvej 1, DK-2630 Taastrup, Denmark. E: rdi@teknologisk.dk; nmo@teknologisk.dk; jos@teknologisk.dk

<sup>2</sup>Swedish Wood Preserving Association, Box 502, SE-10130 Stockholm, Sweden. E: fredrik.westin@traskydd.com

**Keywords:** certification, modified wood, NTR, NWPC

### ABSTRACT

NTR is a trusted quality labelling system used for the classification and quality control of wood products that have been treated with preservatives or have been modified. NTR was introduced in 1969 at the initiative of the Nordic Ministerial Council. It has become the largest wood quality system for durability in Europe, with the majority of wood products in the Nordic countries bearing the NTR label, Figure 1. The NTR classes align with the European standardisation system and its use classes, making it easier for end-users to select the appropriate wood for their specific needs. In practice, the NTR classes are the ones known to the trade and end-users on the market.



**The Wood Durability Quality System**

**Figure 1. NTR label**

The NTR system encompasses all wood materials and new technologies, allowing modified wood products such as chemical modified wood, heat-treated wood, silica-impregnated wood, and linseed oil-impregnated wood to compete on an equal footing. The Nordic Wood Preservation Council (NWPC) manages and develops the NTR standards, which are openly available on the NWPC website: [www.nwpc.eu](http://www.nwpc.eu). The conditions for approval of modified wood are outlined in NWPC Document No. 2 Part 4:2017, which specifies the mandatory tests for each protection class. The conditions are in principle based on EN 599 introducing additional requirements with respect to the mode of action of the modified wood. If chemical agents are used, for instance that are either filling the lumen and/ or cell wall or even reacting with cell wall components, the agents must not have any biocidal effect on the wood. The modification procedure, i.e. both possible agents and the process of modification, must be described in full detail as well as the possible mode of action when applying for an approval. The standard is applicable to industrial treatment of any wood species suitable for the treatment as long as the requirements are fulfilled. The rigorous approval process consists of field tests, which among other things assess the wood products resistance to rot. The moisture exclusion efficiency, MEE, is used to quantify the effectiveness of a modification method. All classes require testing according to EN 15083-1 and when in ground contact also EN 15083-2 including ageing after EN 73 and EN 84 separately. The MLP (mass loss

percentage) is determined and requirements for MLP are included. The evaluation can also be done based on the x-value comparing the weight loss with the weight loss of the reference samples. Furthermore, efficacy testing in field (EN 330, EN 252, EN 12037 and EN 275) is included depending on the use class. Independent institutes accredited for the methods carry out the efficacy tests, physical tests as well as chemical analyses.

The NTR approval system is managed by an independent technical group consisting of experts from the Nordic technical institutes. Products that pass the tests earn the right to have the NTR label, which serves as a guarantee of a long service life, durability and quality. A NWPC-approval is normally valid for five years. Application for renewal shall contain updated field test results. The NWPC Technical Expert Group can withdraw an approval if the modification's biological efficacy fails in practical use, following consultation with the producer.

Requirements for quality control of modified wood produced to comply with the requirements for the wood preservation classes A mod, AB mod, B mod, M mod are defined in NWPC Document No. 3. Part 4:2017. The quality control of modified wood consists of factory production control as well as a third-party control. The aim of the thirdparty control is to ensure that the factory production control is carried out and to check that the quality of the modified wood complies with the requirements in NWPC Document No 1, Part 4. The third-party control is carried out through at least two unannounced visits during one calendar year. The comprehensive approval process and quality control measures serve to ensure and enhance consumer confidence in wood, thereby safeguarding the esteemed reputation of timber as a high-performance construction material.

## REFERENCES

NTR Document no. 1, part 4: 2017. Nordic wood protection classes and product requirements for industrially protected wood. Part 4: Modified wood

NWPC Document no. 3. Part 4:2017. Nordic requirements for quality control of industrially protected wood. Part 4: Modified wood



## Oral 1.05 - STYL<sup>+</sup>WOOD<sup>®</sup> System for the Thermal Modification of Wood

Michele Bigon<sup>1</sup> and Sonia Marchiori<sup>1</sup>

<sup>1</sup>BIGonDRY S.r.l., Via G. Falcone, 30 31037 Castione (TV), ITALY. E: sonia.marchiori@bigondry.com

**Keywords:** semi dry process, SME enterprise, thermal modification, standard production, wood characterisation

### ABSTRACT

BIGonDRY s.r.l, is a SME enterprise located in Veneto, north-east of Italy, operating for more than 30 years in the field of kiln drying manufacturing. The Company, has gained great reputation in the thorny sector of hardwood wooden floors and decking developing personalized solutions in function of the different wooden species and end-use requirements. In the last decade, part of this background has gradually moved towards an integrated approach between drying and thermal modification developing a proprietary system and process for the thermal modification of wood named the STYL<sup>+</sup>WOOD<sup>®</sup> system. It is a semi-dry process in a close system, where the oxygen concentration is controlled by means a slight overpressure given by the natural production of gases from the wood (water vapour and gases from pyrolysis) and from the fire combustion. The temperature difference between air and wood core are used by the system to control the process parameters. As a result, the STYL<sup>+</sup>WOOD<sup>®</sup> system uses a very little quantity of steam allowing to reach an eco-friendly approach, optimizing process technology developed on aspects related to costs, environmental impact, safety and to standardize the production of thermally modified wood STYL<sup>+</sup>WOOD<sup>®</sup> with proven characteristics of durability, stability and appearance.

In partnership with CNR-IBE, STYL<sup>+</sup>WOOD<sup>®</sup> process has been optimised in function of wood species and different final end-uses and the STYL<sup>+</sup>WOOD<sup>®</sup> product has been extensively characterised. The results have been condensed in product sheets for 9 wood species treated with different processes with different modification intensity. A standard production and quality control protocol was developed and adopted by the factory on voluntary base to guarantee the quality and performance results as indicated by the product sheets.

Some extraordinary results, such as the improvement of durability (standard test EN 113) from 5 to class 1 or 2 achieved for treated ash, poplar and beech are an evidence of the quality of the STYL<sup>+</sup>WOOD<sup>®</sup>, confirmed by customers all over the world and the use of STYL<sup>+</sup>WOOD<sup>®</sup> for wild range of outdoor and indoor products.



Figure 1: Saunas Carmenta



Figure 2: Segheria Vallesacra SRL-Poplar THW



Figure 3: City Design-Casteo TWS Model bench

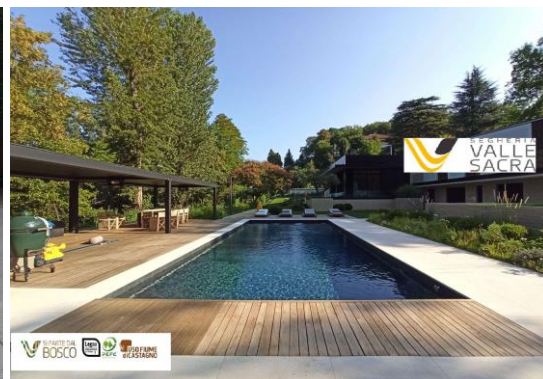


Figure 4: Segheria Vallesacra SRL-Ash THW

## CREDITS

Financial supported by Veneto Region in the framework of POR FESR STY+LWOOD project 2014/2020

CNR-IBE, National Research Council-Istituto per la Bioeconomia, San Michele all'Adige (TN) Italy  
Swedish University of Agricultural Sciences, Department of Forest Biomaterials and Technology.  
Uppsala, Sweden

Carmenta Saune – Veneto

Segheria Vallesacra Srl – Piemonte

City Design – Veneto

## Oral 1.06 - Commercial Wood Products Achieved by Industrial Thermal Treatment Process

Alessandro Porcu<sup>1</sup> and Paola Cetera<sup>2</sup>

<sup>1</sup>BASCHILD S.r.l. Via degli Assini, 24048 Treviolo (BG), Italy. E: a.porcu@baschild.com

<sup>2</sup>Department of Agricultural Sciences, University of Sassari, Viale Italia 39/a, 07100 Sassari, Sardinia, Italy. E: pcetera@uniss.it

**Keywords:** Baschild company, decking, thermal treatment, wood, wood panelling

### ABSTRACT

Thermal treatment of sawn timber is carried out under carefully controlled conditions at high temperatures (Bourgois 1989). In the processing process, only heat, water vapor and fresh water are used without the use of any chemical preparations or additives, which makes it completely ecologically friendly and harmless to the environment. Silvabp and Evolen, are two European manufacturers that use high temperature of treatment to increase some properties of wood and preserve the material without using any chemical additive or fossil fuels. Sivalbp's Eco Thermo or thermostabilization is a process that consists in heating slowly the wood up to 200 °C, alternating rise of temperature with rehumidification phase of the pine wood. Sivalbp wood boards are treated in the factory on the production site in Thônes (France) according to specifications in compliance with the requirements of the French standard NF B 50-105-3. During the thermal process, it uses exclusively wood supplies from sustainably managed French forests, certified PEFC (PEFC/10-31-1593) & FSC® (FSC-C118242). By selecting only the best wood species, such as western red cedar, larch, spruce, Nordic pine or Douglas fir, which are all different wood species, each with its own characteristics. The final product of Sivalbp is a range of wood cladding (Figure 1) called Elegance, New Age, Vintage, Montagne and Colors are attested with the CTB B+ for preventive treatment. The latter certification, delivered by the FCBA (French Institute of Technology for Forest-based and Furniture Sectors) guarantees the performance of the treatments used to durably protect cladding woods.



**Figure 1: Exterior and interior wood cladding by Sivalbp Company, France.**

Evolen company is located in Novska in the Republic of Croatia and uses thermo-treated ash wood to produce decking, decking panels and facade wooden (Figure 2) treated at 210 °C with the highest resistance class and different dimensions.



**Figure 2: Different thermally treated wood products by Evolen Company, Croatia.**

The positive effect of the high temperature on some wood properties, e.i. colour, resistance (Candelier *et al.* 2016), confirm that today in Europe the thermal treatment is an important industrial process for commercial products wood.

### REFERENCES

Bourgois, J., Bartholin, M. C., & Guyonnet, R. (1989). Thermal treatment of wood: analysis of the obtained product. *Wood Science and Technology*, **23**(4), 303-310.

Candelier, K., Thevenon, M. F., Petrissans, A., Dumarcay, S., Gerardin, P., & Petrissans, M. (2016). Control of wood thermal treatment and its effects on decay resistance: a review. *Annals of Forest Science*, **73**, 571-583.

<https://www.sivalbp.fr/en/>

<https://evolen.hr/en/>



# **DAY 1**

## **SESSION TWO**

### **MODIFICATION WITH CHEMICALS**

## Oral 2.01 - Studying the Impact of a Silicone Oil Treatment on the Elastomechanical Properties of Wood

Lukas Emmerich<sup>1</sup>, Kilian Erdelen<sup>2</sup> and Holger Militz<sup>2</sup>

<sup>1</sup>Landesbetrieb Wald und Holz NRW, Zentrum für Wald und Holzwirtschaft, Team Holzwirtschaft, Carlsauerstraße 91A, 59939 Olsberg, Germany. E: lukas.emmerich@wald-und-holz.nrw.de

<sup>2</sup>Wood Biology and Wood Products, Georg-August-Universität Göttingen, Büsgenweg 4, 37077 Göttingen, Germany. E: kilian.erdelen@stud.uni-goettingen.de; holger.militz@uni-goettingen.de

**Keywords:** chemical impregnation modification, mechanical properties, polysiloxane, silicone oil treatment, softwood

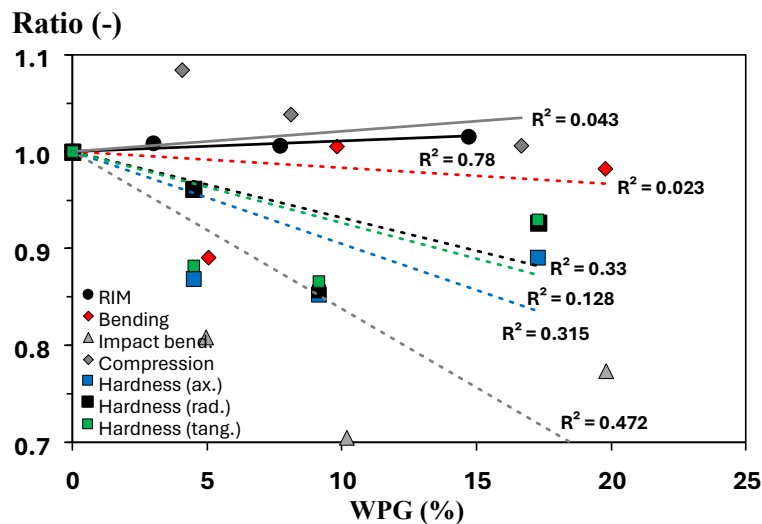
### ABSTRACT

For centuries, wood is used as a manifold building material and by that exposed to a multitude of environments. Specifically in outdoor applications, extremely harsh conditions impact on wooden constructions and surfaces, which stands opposed to a low natural durability and UV sensitivity of this sustainable, carbon-storing material. The latter applies to most native softwood and hardwood species in Europe, which suffer under a low durability against attack and impacts of biotic and abiotic factors. For this reason, a preventive timber treatment may be obligatory in outdoor applications of wood, in order to extend service lives and guarantee a sustainable utilization of the resource wood. Nowadays, the big bulk of wood is treated with biocides to increase its material resistance in outdoor exposure. Basically, such biocidal preservatives are applied by vacuum pressure impregnation processes in industrial-sized autoclaves and fixed by a subsequent drying under ambient conditions. Albeit its simplicity and efficacy related to treatment processes and protective effects, health and disposal issues altered the choice of a wood preservative from an economic and efficacy perspective to a more ecology-driven decision (Schultz *et al.* 2007). Thus, besides biocidal treatments, further attention was put to non-biocidal wood preservation like ‘wood modification’ (Hill 2006).

Although chemical wood modifications generate high-performing wooden products, such technologies do (1) require specific and new equipment for an implementation on industrial scale and (2) may severely decrease the resistance of wood towards dynamic, mechanical loads (‘embrittlement’). By that fact, the utilization of chemically modified wood in load-bearing constructions is highly restricted. In order to overcome such process and performance related hurdles, a wood modification approach on the basis of a cell wall penetrating, heavy-metal free silicone oil compound has recently been studied. Emmerich *et al.* (2022) showed that this technological approach does (1) improve the biological durability and water-related properties of wood and (2) enables to be implemented in impregnation and drying plants (fixation at < 70 °C), which at present exist and operate in the wood preservative and wood processing industry.

To define potential applications of silicone oil treated wood, the present study analyzed the impact of such treatment on the elasto-mechanical properties. The silicone oil (Siligen®MIH) applied to Scots pine (*Pinus sylvestris* L.) sapwood specimens was provided by Archroma LLC (Reinach, Switzerland). Differently concentrated solutions (2.5, 5 and 10 %) of the silicone emulsion were introduced into wood by a vacuum pressure impregnation (full volume treatment) and subsequently dried and fixed at 70 °C. As a consequence of the treatment, a positive weight gain (4.3 – 17.8 %) and cell wall bulking (0.9 – 2.5 %) were measured. The equilibrium moisture content (EMC<sub>material</sub> at 20 °C/ 65 % RH) decreased by the modification and with increasing chemical retention. Depending on the latter, Fig. 1

illustrates the impact of the silicone treatment on static and dynamic elasto-mechanical properties.



**Figure 1: Impact of a polysiloxane treatment and its retention on selected elasto-mechanical properties. Corresponding properties (RIM = Resistance to impact milling (%), Bending = 3 point bending strength N/mm<sup>2</sup>, Impact bending = Impact bending strength (kJ/m<sup>2</sup>), Compression = Compression strength N/mm<sup>2</sup> and Hardness = Brinell hardness N/mm<sup>2</sup>) are illustrated in relation to corresponding untreated control specimens (ratio < 1 = decrease; ratio > 1 = increase).**

While compression strength, bending strength and the structural integrity (RIM) were almost unaffected by the silicone treatment, surface hardness (here: Brinell hardness) and impact bending strength experienced a slight decrease. Nevertheless, the impact on the dynamic strength properties was negligible compared to those of chemical modifications with polymerizing chemicals (Zelinka *et al.* 2022). In combination with the treatment process related simplicity, the latter differentiates the treatment of choice from existing chemical modifications and may establish new areas of application for modified wood. Finally, this paper will review the new wood modification technology of silicones and discuss the challenges of the latter in comparison to other technologies.

## REFERENCES

- Emmerich, L., Militz, H., Vila, M. (2022). A novel wood preservation technology improving durability and water-related properties. Proceedings IRG Annual Meeting, IRG/WP 22- 40926.
- Hill, C.A.S. (2006). Wood Modification - Chemical, thermal and other processes. John Wiley & Sons Ltd., West Sussex, United Kingdom.
- Schultz, T.P., Nicholas, D. D. Preston A.F. (2007). A brief review of the past, present and future of wood preservation. *Pest Management Science* 63(8), 784-788.
- Zelinka, S.L., Altgen, M., Emmerich, L., Guigo, N., Keplinger, T., Kymäläinen, M., Thybring, E.E., Thygesen, L.G., (2022). Review of Wood Modification and Wood Functionalization Technologies. *Forests* 13, 1004

## Oral 2.02 - Modifying Wood with a Bio-Based Thermosetting Resin – Different Approaches to Curing and Drying

Christoph Hötte<sup>1</sup> and Holger Militz<sup>1</sup>

<sup>1</sup>Wood Biology and Wood Products, Georg-August-Universität Göttingen, Büsgenweg 4, 37077 Göttingen, Germany. E: choette@uni-goettingen.de; hmilitz@gwdg.de

**Keywords:** chemical distribution, curing, drying quality, modification quality, up-scaling

### ABSTRACT

#### Background

Modification processes based on thermosetting resins, usually consist of (1) impregnating the wood with the chemicals dissolved in water and (2) a curing step, which involves the reaction of the chemicals and removing the excess water – i.e., re-drying the wood. Since almost all wood modification processes rely on full cell impregnation, the first step is largely uncritical for the modification result. The curing step, on the other hand, is crucial for the subsequent product properties. Several studies have shown the effect of curing conditions on the modification quality, the chemical distribution within the wood and the drying quality of modified wood (Krause 2006, Klüppel and Mai 2013, Behr *et al.* 2018). Problems and challenges of the curing step increase in the context of up-scaling of modification processes on wood in larger dimensions. This presentation presents an overview of different approaches for the curing of wood in semi-industrial scale modified with a bio-based thermosetting resin.

#### Thermosetting resins for wood modification

Thermosetting resins can be roughly divided into formaldehyde-based resins and formaldehyde-free resins. Among the latter are polycondensation resins based on polycarboxylic acids and polyols, the best researched of which is based on impregnation with sorbitol and citric acid (SorCA). For modification, both chemicals are dissolved in water and impregnated into the wood using a vacuum pressure process. The subsequent reaction mechanism is based on the co-polymerisation of both chemicals for which a temperature of 140 °C is required (Mubarok *et al.* 2020). SorCA treated wood exhibits increased dimensional stability and improved durability against white and brown rot fungi (Kurkowiak *et al.* 2021).

#### Requirements for the curing process

In summary three main requirements must be met for a successful curing process:

- **High modification quality**, as expressed by Weight Percent Gain (WPG) and Cell Wall Bulking (CWB). Moreover, a good fixation of the modification chemicals is of high importance for the long-term improvement of product properties such as dimensional stability and durability.
- **Uniform chemical distribution on the cross-section.** Former research has shown that migration of chemicals into the outer zones of the cross section can occur during curing (Kurkowiak *et al.* 2022). This results in insufficiently modified inner areas as well as highly inhomogeneous product properties.
- **Low occurrence of drying defects.** The combination of high initial moisture contents (frequently > 100 % after impregnation) with the temperature required for curing (140 °C) poses a high risk of drying defects (checking and changes in dimension, such as cup, twist, or bow) to the wood.



### Possible approaches for a curing process

In the process of upscaling the modification technology for SorCA from laboratory scale to quasi-industrial scale a curing process is needed, that simultaneously fulfills the abovementioned requirements. During curing, various parameters can be regulated in order to control the quality of the modified product. While only the temperature and the total curing time are usually controlled on a laboratory scale, the relative humidity or the process pressure can be controlled with the help of specialized equipment. It is also possible to divide the process into different phases and adjust the individual parameters during these phases, very similar to customized plans for wood drying.

The different process parameters show different effects on modification quality, chemical distribution and drying quality. Curing and drying velocity, for example, are both temperature dependent and the chemical reaction rate as well as the drying rate increase with rising temperature. At this point, a conflict of objectives develops: while high temperatures and sudden heating have a positive impact on WPG and chemical fixation, they simultaneously lead to an increased occurrence of drying defects like cracking or warping. Longer process times at high temperatures will result in more complete polycondensation of the polyester, but may also have a negative impact on wood quality. Furthermore, the chemical distribution can be influenced by controlling the holding temperature and holding time and the heating and cooling rates. Similarly, the properties of the modified product can be controlled to a large extent by adjusting the relative humidity during the process.

Our presentation will compare different processes for wood impregnated with aqueous formulations of sorbitol and citric acid with regard to the previously mentioned process requirements. In particular, it focuses on challenges that increasingly arise during the upscaling of the modification to industrial scale.

### REFERENCES

- Behr, G., Gellerich, A., Bollmus, S., Brinker, S., Militz, H. (2018) The influence of curing conditions on properties of melamine modified wood. *European Journal of Wood and Wood products*, 76, 1263-1272.
- Klüppel, A., Mai, C. (2013) The influence of curing conditions on the chemical distribution in wood modified with thermosetting resins. *Wood Science and technology*, 47,643–658.
- Krause, A. (2006) Holzmodifizierung mit N-Methylolvernetzern. Dissertation, GeorgAugust-Universität Göttingen, Germany.
- Kurkowiak, K., Emmerich, L., Militz, H. (2021) Biological durability of sorbitol and citric acid (SorCA) modified wood. The International Research Group on Wood Preservation. Document No. IRG/WP/22-40928.
- Kurkowiak, K., Mayer, A.K., Emmerich, L., Militz, H. (2022) Investigations of the chemical distribution in sorbitol and citric acid (SorCA) treated wood—Development of a quality control method on the basis of electromagnetic radiation. *Forests*, 13, p. 151.
- Mubarok, M., Militz, H., Dumarçay, S., and Gérardin, P. (2020). Beech wood modification based on in situ esterification with sorbitol and citric acid. *Wood Science and technology*, 54, 479–502.

## Oral 2.03 - Hydrophobisation of Beech Wood Scantlings with Radiationcuring Epoxidised Vegetable Oils for Use as Dimensionally Stable Components in Exterior Applications

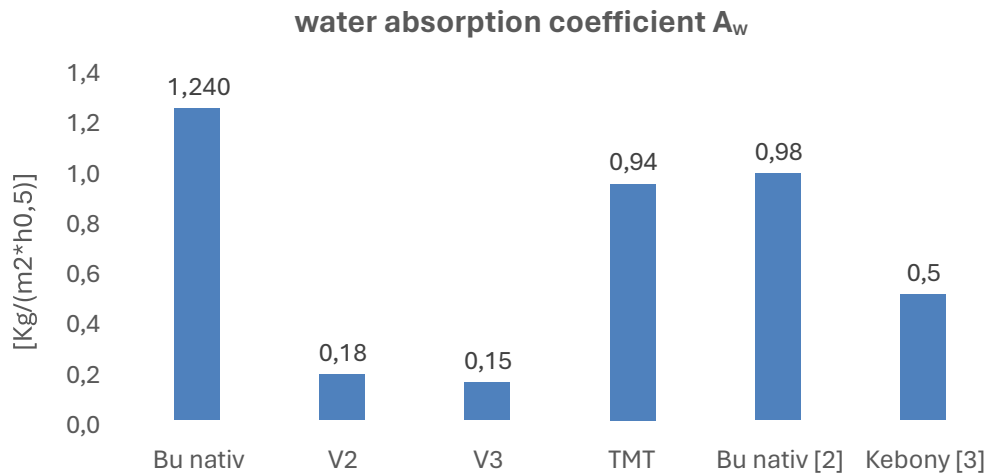
Christiane Swaboda<sup>1</sup> and Roger Scheffler<sup>1</sup>

<sup>1</sup>Institut für Holztechnologie Dresden, gemeinnützige GmbH, Zellescher Weg 24, 01217 Dresden, Germany. E: christiane.swaboda@ihd-dresden.de; roger.scheffler@ihd-dresden.de

**Keywords:** beech wood, building elements, dimensional stability, durability, epoxidized plant oils, hydrophobisation

### ABSTRACT

The work investigates the influence of a treatment of copper beech (*Fagus sylvatica* L.) lamellae with curing epoxidized vegetable oils on wood properties to improve their hygroscopic properties for a use as building elements (such as windows, exterior doors, façade claddings) or for outdoor applications. The requirements and regulations on environmental protection make the use of domestic resources more attractive: a decreasing number of wood species, especially tropical woods, many of them well suited for window construction, are allowed to be imported into the European Union [1]. On the other hand, the supply of beech wood will continue to increase in the years to come due to forest conversion in Germany. However, load-bearing components or components exposed to weathering made of untreated beech wood (not durable according to DIN EN 350 (2016)) are not permitted. The moisture-dependent properties (swelling/shrinking, deformation, cracking, biological durability) with their effects on use and the service life are problematic. The criterion of the lowest possible care and maintenance effort is given outstanding importance (user perspective). Building elements with frame constructions made of wood are compete strongly with products made of other materials or combinations of materials such as plastics or aluminium. Therefore, a tempering process for copper beech was developed at the IHD, which is based on the use of radiation-curing epoxidized vegetable oils. By optimising the formulation, it was possible to generate variants that guarantee optimum curing with a low  $\gamma$ -radiation dose to ensure a significant improvement of the material properties compared to natural copper beech wood, while at the same time reducing the tendency to embrittlement. Although this type of tempering is not a wood modification in the strict sense, which means that no direct modification of the wood cell wall is achieved, material properties are achieved that allow use in window and door construction and are comparable to those of already approved modified woods such as thermowood or Kebony. Not only changes in hygric and mechanical properties but also their influence on further processability and application of the treated wood as window scantlings were investigated. Thus, in addition to reduced swelling and shrinkage properties, an increase in strength values and an improvement in the durability classes against basidiomycetes from DC 5 to DC 1 - 2 as well as a delay in the growth of blue stain fungi are achieved. With regard to the reproducibility of the results on a larger scale, a quality control of the loading degree as well as the degree of curing is necessary, as some material properties, e.g. the water absorption, strongly depend on the oil distribution and the degree of curing. Nevertheless, at lower degrees of curing, lower linear shrinkage measures were found, which can be attributed to an adsorbing effect of the oil polymer by the uncross linked hydroxyl groups formed during the epoxy ring opening reaction.



**Figure 1: Capillary water absorption coefficients (mean values from all anatomical Directions) of different formulations in comparison with thermos wood (TMT) and Kebony.**

**Table 1: time to reach equ. moisture after 3 cycles of water increase (85%20°C) and water decrease (35% 20°C)**

		<b>Beech</b>	<b>V2<sup>a</sup></b>	<b>V3<sup>b</sup></b>
	65% --> 35%	32,50	92,40	93,90
1. cycle	35%--> 85%	65,00	204,61	194,20
2. cycle	35%--> 85%	62,53	137,20	144,66
3. Cycle	35%--> 85%	58,54	129,60	127,50
1. cycle	85% --> 35%	50,40	138,00	139,50
2. cycle	85% --> 35%	48,87	133,60	126,14
3. Cycle	85% --> 35%	46,14	119,40	126,70

<sup>a</sup> epoxidised linseed oil treated, <sup>b</sup> epoxidized linseed oil with reactive thinner

## REFERENCES

Koch, G.; Haag, V.: Auswirkungen der neuen CITES - Listungen wichtiger Wirtschaftsbaumarten für die Holzverwendung und den Holzhandel. Thünen Institut für Holzforschung. 2017. [https://www.thuenen.de/media/ti/Infrastruktur/Thuenen-Kompetenzzentrum\\_Holzherkuenfte/Expertise\\_CITES/Expertise\\_Koch\\_CITES-Hoelzer\\_2017.pdf](https://www.thuenen.de/media/ti/Infrastruktur/Thuenen-Kompetenzzentrum_Holzherkuenfte/Expertise_CITES/Expertise_Koch_CITES-Hoelzer_2017.pdf). Zugriff: 22.11.2018

Klepel, C.: Charakterisierung ausgewählter Vergütungseigenschaften von Rotbuchenholz nach Behandlung mit strahlenpolymerisierbaren Lipiden hinsichtlich der Verwendung für Bauelemente. Masterarbeit, TU Dresden Fakultät Umweltwissenschaften, 2017

Wagenführ, R. 2012, Holzatlas, Hanser Verlag, 7. vollständig überarbeitete Auflage 2021, ISBN 978-3-446-46838-2

## Oral 2.04 - Dimensional stability of Scots Pine Sapwood Modified by Tannin-based Formulas

Sheikh Ali Ahmed<sup>1</sup>, Gianluca Tondi<sup>2</sup>, Filippo Rizzo<sup>3</sup>, Reeta-Maria Stöd<sup>4</sup> and Reza Hosseinpourpia<sup>1,5</sup>

<sup>1</sup>Department of Forestry and Wood Technology, Faculty of Technology, Linnaeus University, Georg Lückligs Plats 1, 351 95 Växjö, Sweden. E: sheikh.ahmed@lnu.se

<sup>2</sup>Università degli studi di Padova - University of Padua, TESAF Department, Viale dell'Università 16, 35020 - Legnaro (PD) – Italy. E: gianluca.tondi@unipd.it

<sup>3</sup>WP Innovation, Stora Enso Wood Products GmbH, Bahnhofstraße 31, AT-3370 Ybbs, Austria. E: filippo.rizzo@storaenso.com

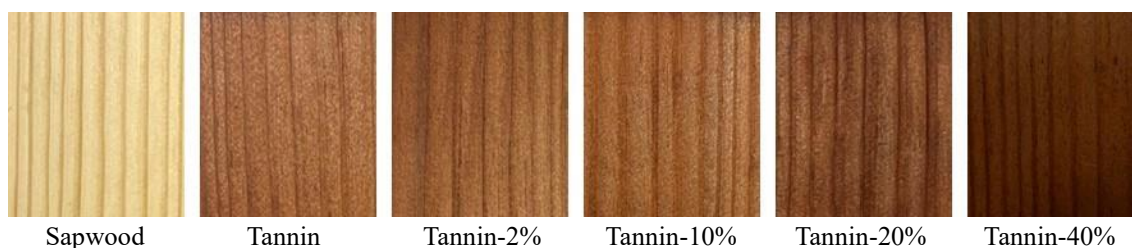
<sup>4</sup>Stora Enso Oyj, Wood Products, PO Box 309, FI-00101 Helsinki. E: reetamaria.stod@storaenso.com

<sup>5</sup>College of Forest Resources and Environmental Science, Michigan Technological University, Houghton, Michigan 49931, United States. E: reza.hosseinpourpia@lnu.se

**Keywords:** bulking, cross-linker, impregnation modification, leaching

### ABSTRACT

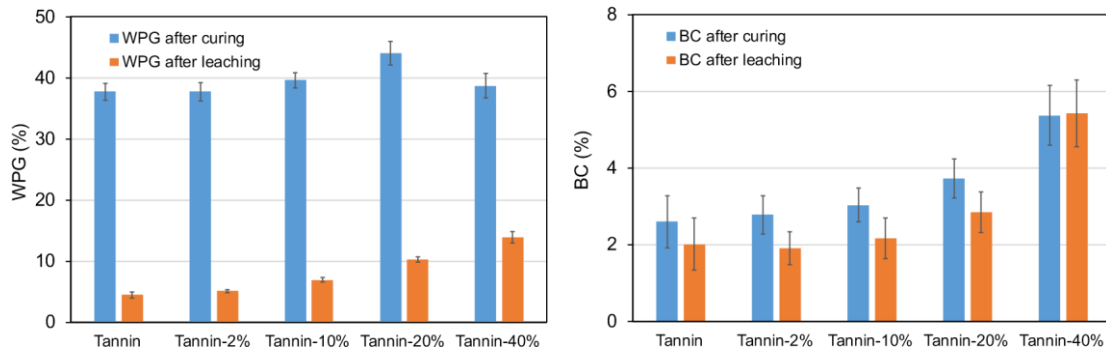
Tannins are polyphenolic compounds extracted from various tree species and used in various applications such as adhesives, composites, pharmaceuticals, medicines, and food and beverage production (Mubarak *et al.* 2023). However, tannins, especially condensed tannins, have limited reactivity with wood. Consequently, to effectively modify wood, cross-linkers and other reactive chemicals or additives must be used. In this work, we have introduced a bio-based cross-linker in a 20% tannin (Quebracho) aqueous solution. Five levels of cross-linker (0, 2, 10, 20 and 40% of total tannin solid) were used to modify Scots pine (*Pinus sylvestris* L.) sapwood with dimensions of 25 × 25 × 10 mm<sup>3</sup> (radial × tangential × longitudinal). Full-cell impregnation was applied with a 60 min vacuum and pressure for 60 min. Samples were kept at room condition for 24 h, stepwise dried to avoid drying defects and cured at 140 °C for 10 h. Modified wood samples are shown in Figure 1. Weight percentage gain (WPG) and bulking coefficient (BC) after water leaching for 5 days were measured according to Donath *et al.* (2004).



**Figure 1: Modified wood with different concentrations of cross-linker based on total tannin solid**

Experimental results showed that with an increase in cross-linker level, there is an increase in WPG and BC (Figure 2), which might be due to the improving fixation level of tannin in wood. However, not all tannin was fixed in wood structure. Due to leaching, there was thus a considerable reduction of WPG to 88, 87, 82, 77 and 64% for 0, 2, 10, 20 and 40% cross-linker levels, respectively. Similarly, BC also reduced, and it was 22, 32, 29, 24 and 0% respectively. With the high level of cross-linker i.e. 40%, there was no observed reduction of BC after leaching. The increased volume of wood samples following tannin treatment is

indicative of a cell bulking process resulting from the incorporation of chemicals into the wood cells. Less reduction of BC after leaching also indicates the fixation of tannin to the cells. This bulking phenomenon serves to reduce the cell's capacity to undergo shrinkage and swelling, thereby enhancing the dimensional stability of the wood samples.



**Figure 2: Weight percentage gain (WPG) and bulking coefficient (BC) of the tannin-based formulations after curing and after leaching for five days in water.**

The results of this study clearly demonstrate the promising potential of tannin in enhancing the physical characteristics of pine sapwood. By controlling the concentration of cross-linker, it is possible to produce a bio-based formula with high weight percent gain and leaching resistance. Consequently, biological, and mechanical properties further need to be investigated to evaluate its suitability in the wood products industry.

## REFERENCES

Donath, S., Miltz, H. and Mai, C. (2004). Wood modification with alkoxy silanes. *Wood Science and Technology*, 38:555-566.

Mubarok, M., Azadeh, E., Obounou Akong, F., Dumarçay, S., Gérardin, P. and GérardinCharbonnier, C. (2023). Effect of tannins addition on thermal stability of furfurylated wood. *Polymers*, 15, 2044.





were first dried and conditioned to 12% moisture content and then thermo-mechanically compressed in the radial directions, reducing their thickness from 22 mm to 11 mm. Pressing temperature was varied between 150 and 210 °C and pressing time between 5 and 60 minutes. In this approach, the curing of the chemicals took place during densification. Density profiles were acquired with an X-ray densitometer before and after densification. Hardness, set-recovery, and time to ignition were tested.

## REFERENCES

Gan, W., Chen, C., Wang, Z., Song, J., Kuang, Y., He, S., Mi, R., Sunderland, P.B. and Hu, L. (2019). Dense, self-formed char layer enables a fire-retardant wood structural material. *Advanced Functional Materials*, **29**(14), Article ID: 1807444.

Lin, C.-f., Karlsson, O., Das, O., Mensah, R.A., Mantanis, G.I., Jones, D., Antzugin, O.N., Försth, M. and Sandberg, D. (2023). High leach-resistant fire-retardant modified pine wood (*Pinus sylvestris* L.) by in situ phosphorylation and carbamylation. *ACS Omega*, **8** (12), 11381–11396.

Navi, P. and Sandberg, D. (2012). Thermo-hydro-mechanical processing of wood. EPFL Press, Lausanne, Switzerland.

## Oral 2.06 - Development of Novel Guitar Fretboards by Thermal Modification and Impregnation with PF-Resin of Beech (*Fagus sylvatica* L.) and Maple (*Acer ssp.*) Wood

Christina Zwanger<sup>1</sup> and Marcus Müller<sup>1</sup>

<sup>1</sup>Material development and processing, University of Applied Forest Sciences Rottenburg, Schadenweilerhof, 72108 Rottenburg, Germany. E: mueller@hs-rottenburg.de; zwanger@hs-rottenburg.de

**Keywords:** brinell hardness, CIE L\*a\*b, dimensional stability, PF-resin impregnation, thermal modification, wood modification

### ABSTRACT

Tropical woods like ebony (*Diospyrus ssp.*) or rosewood (*Dalbergia ssp.*) are typical tonewoods, which are characterized by high dimensional stability, durability, hardness, density and a dark wood colour. These properties are good prerequisites for using them as tonewood in instruments. CITES (Convention of International Trade in Endangered Species) restricts the trade of ebony and rosewood, which are traditionally used for the production of guitars fretboards. Furthermore, there is an increasing demand for guitars built without tropical woods. Therefore, it is necessary to search for alternatives. Most native woods in Europe do not meet the requirements that are important for usage in guitar fretboards. These requirements are a sufficiently high hardness and density, dimensional stability and a dark wood colour.

In order to achieve these properties a two-step modification was applied. In a first step wood samples of beech (*Fagus sylvatica*) and maple (*Acer ssp.*) were heat-treated at 220 °C for 4h under nitrogenous atmosphere, followed by an impregnation with a 15% (solid content) solution of phenolic resin (PF) and demineralized water. After treatment, the wood was dried stepwise closing with a curing step. The effect of the two-step treatment on swelling and shrinking behaviour was investigated according to DIN 52 184 (1979). Brinell-hardness was examined following DIN EN 1534 (2019). For documenting colour changes, L\*a\*b-values according to CIE L\*a\*b (Comission Internationale d'Eclairage) standard following DIN EN 927-6 were measured.

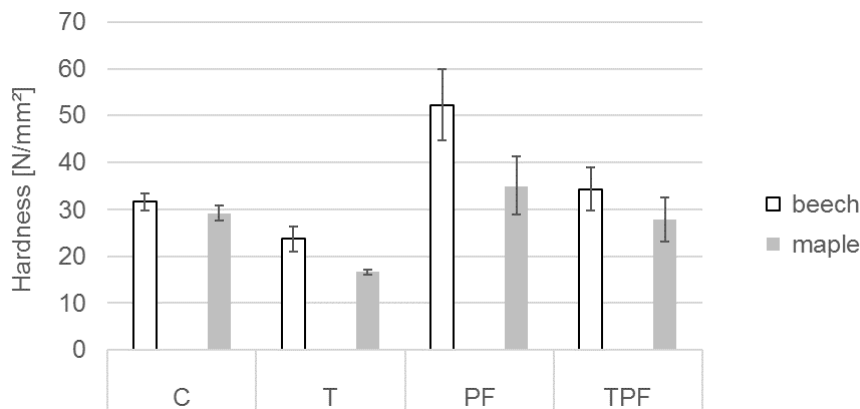
Thermally treatment of beech and maple wood at 220 °C for 4 hours changed the light wood colour to dark brown/black, similar to that of rosewood (Figure 1). Impregnation of the thermally treated wood with a 15% solution of PF resin resulted in an increase of hardness of 8% for beech and a slight decrease of 5% for maple (Figure 2). Density could be increased by 1.6% for beech and 11.6% for maple wood. The ASE (Anti-SwellingEfficiency) values of TPF (thermally and PF-resin impregnated)-beech (51% in 10<sup>th</sup> cycle) and TPF-maple (52% 10<sup>th</sup> cycle) were higher than for the thermally treated (T) and the resin impregnated (PF) wood. The ASE values of TPF treatment remain stable over ten cycles (Table 1).

**Table 1: Mean values (SD) of the Anti-swell-efficiency (ASE) for thermal treatment 220 °C for 4h (T), impregnation with 15% PF-resin (PF), first thermal treatment 220 °C for 4h, followed by impregnation with 15% PF-resin (TPF) of the first, fifth and tenth cycle.**

Wood species	Label	ASE [%] 1 <sup>st</sup> cycle	ASE [%] 5 <sup>th</sup> cycle	ASE [%] 10 <sup>th</sup> cycle
beech	T	51,36 (2,45)	38,77 (3,35)	32,18 (6,38)
	PF	38,89 (3,68)	43,09 (3,29)	43,75 (3,52)
	TPF	53,20 (4,00)	50,50 (3,58)	50,77 (3,78)
maple	T	48,10 (0,99)	40,97 (1,65)	38,01 (2,22)
	PF	39,46 (1,89)	35,90 (1,48)	37,95 (1,53)
	TPF	51,73 (2,03)	51,44 (1,45)	52,09 (1,53)



**Figure 1: Change of wood colour after a two-step modification (thermal modification and impregnation with PF-resin) and comparison to the wood colour of rosewood**



**Figure 2: Brinell hardness of beech and maple specimens treated under the following conditions: (C) control, (T) thermal treatment 220°C for 4h, (PF) impregnation with 15% PF-resin, (TPF) first thermal treatment 220°C for 4h, followed by impregnation with 15% PF-resin**

## Oral 2.07 - A Study of the Influence of the Curing Conditions on Scots Pine Treated with SorCA Coupled with Catalysts

Adèle Chabert<sup>1</sup>, Katarzyna Kurkowiak<sup>1</sup>, Holger Militz<sup>1</sup>

<sup>1</sup>Wood Biology and Wood Products, Georg-August-Universität Göttingen, Büsgenweg 4, 37077 Göttingen, Germany. E: hmilitz@gwdg.de

**Keywords:** catalysts, curing conditions, SorCA

### ABSTRACT

Wood modification through the utilization of polyesters necessitates a curing phase, during which the wood undergoes a dual treatment encompassing both chemical and thermal processes. Over the past two decades, a multitude of treatments involving various polyesters has been the subject of scholarly investigation. These treatments have resulted in the acquisition of new wood properties characterized by elevated durability and enhanced dimensional stability (L'Hostis 2017; Chabert, *et al.* 2022; Kurkowiak *et al.* 2022; Essoua Essoua *et al.* 2016; Larnøy *et al.* 2018). Nonetheless, a discernible alteration in the wood's mechanical characteristics, particularly its brittleness, has been observed.

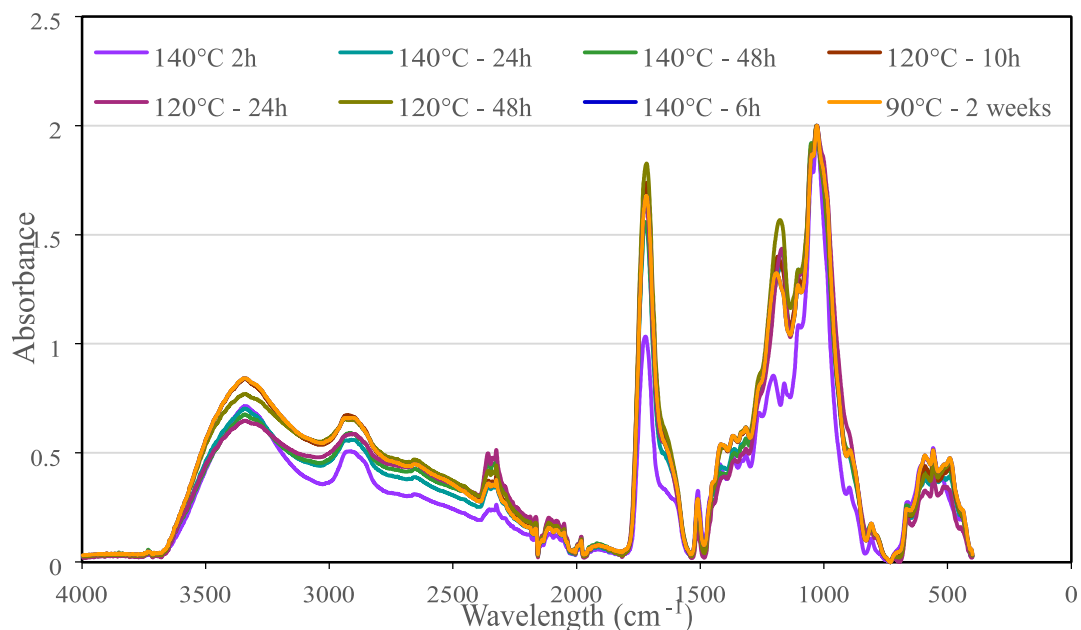
The curing procedure is postulated to exert a notable influence on a range of wood properties, highlights the transformations undergone by the wood material during the curing phase (Chabert, Kurkowiak and Militz 2023), particularly in terms of its mechanical attributes. Although many of these induced modifications enhance the material's overall quality, mitigating the impact of the curing process has the potential to expand the scope of applications for SorCA-treated wood. To assess this prospect, various curing conditions spanning from 140 °C for a duration of 2 hours to 90 °C over a two-week period have been investigated and are presented herein. Furthermore, the results discussed in this context pertain to SorCA treatment coupled with phosphoric acid (H<sub>3</sub>PO<sub>4</sub>). In this work, an emphasis on the dimensional stability, chemical analysis, sorption properties as well as some decay testing has been given.

The tests presented in the following were done on Scots Pine (*Pinus sylvestris*). In this work five treatment were studied, SorCA and a combination of SorCA and catalysts. To assess the impact of SorCA treatment on wood, a comprehensive methodology was employed, focusing on dimensional stability, chemical composition (FTIR analysis), and water sorption properties. These samples were then conditioned to a specific moisture content to establish a consistent baseline.

Fourier-transform infrared spectroscopy (FTIR) was employed to analyze the chemical composition of the wood before and after SorCA and catalysts treatment. This technique allowed for the identification of changes in functional groups and chemical bonds, providing insights into the chemical alterations induced by SorCA.

The combination of these methodologies provided a comprehensive understanding of how SorCA treatment influences the dimensional stability, chemical composition, and water sorption characteristics of wood, offering valuable insights into the suitability of SorCA as a wood treatment method.

The Following graph shows the spectrum obtained through FTIR-ATR. As such, the only observation that can be done is the significantly lower peak at 1725cm<sup>-1</sup> of the treatment at 140 °C for 2h. This peak can be associated to the C=O and C-O bonds of esters, possibly indicating a lower amount of those hence a less advanced polymerisation.



**Figure 1: FTIR-ATR Spectrum of Wood SorCA treated with H<sub>3</sub>PO<sub>4</sub>**

## REFERENCES

- Chabert, A., Kurkowiak, K., and Miltz, H. (2023). Preliminary Analysis of the Curing Conditions of SorCA Wood Treated with Catalysts. In Proceedings of the 19th Meeting of the Northern European Network for Wood Science and Engineering (WSE), 16–18. Ås, Norway.
- Chabert, A., Fredon, E., and Rémond, R. (2022). Improving the Stability of Beech Wood with Polyester Treatment Based on Malic Acid. *Holzforschung* 76 (3): 268–75. <https://doi.org/10.1515/hf-2021-0030>.
- Essoua Essoua, G. G., Blanchet, P., Landry, V., and Beauregard, R. (2016). Pine Wood Treated with a Citric Acid and Glycerol Mixture: Biomaterial Performance Improved by a Bio-Byproduct. *BioResources* 11 (2): 3049–72. <https://doi.org/10.15376/biores.11.2.3049-3072>.
- Kurkowiak, K., Mayer, A.K., Emmerich, L., and Miltz H. (2022). Investigations of the Chemical Distribution in Sorbitol and Citric Acid (SorCA) Treated Wood—Development of a Quality Control Method on the Basis of Electromagnetic Radiation. *Forests* 13 (2): 151. <https://doi.org/10.3390/f13020151>.
- Larnøy, E., Karaca, A., Gobakken, L. R., and Hill, C. A. S. (2018). Polyesterification of Wood Using Sorbitol and Citric Acid under Aqueous Conditions. *International Wood Products Journal* 9 (2): 66–73. <https://doi.org/10.1080/20426445.2018.1475918>.
- L’Hostis, Clément. (2017). Développement de Nouveaux Traitements Non-Biocides de Protection Du Bois Basés Sur La Formation in Situ de Polyesters Bio-Sourcés’. Thesis, Université de Lorraine. <http://www.theses.fr/2017LORR0319>.

## Oral 2.08 - Wood Modification by Different Chestnuts Tannin - Furfuryl Alcohol Resins and Effect on Conferred Wood Durability

Christine Gerardin-Charbonnier<sup>1</sup>, Vitor Joao Dorini<sup>1,2</sup>, Pedro H. Gonzalez de Cademartori<sup>2</sup>, Philippe Gerardin<sup>1</sup>

<sup>1</sup>Laboratoire d'Etudes et de Recherche sur le Matériau bois, Université de Lorraine, France. E: christine.gerardin@univ-lorraine.fr

<sup>2</sup>Department of Forest Engineering and Technology, Federal University of Paraná, Curitiba, Brazil

**Keywords:** chemical modification, chestnuts tannin, decay, durability, furfuryl alcohol, wood

### ABSTRACT

The aim of this study is to design an original wood-based composite by combining wood furfurylation with hydrolysable tannins to obtain the same polymer as that used for the production of tannin-furan foams (Azadeh *et al* 2022). The use of tannins was investigated not only as crosslinking agents but also as co-monomers to reduce the amount of furfuryl alcohol needed for good material protection. Tannins are also believed to confer antioxidant properties via phenolic functions, which improve wood durability during its degradation by fungi known to produce several radical intermediates to depolymerize wood components. Due to their antioxidant properties, addition of polyphenols in furfuryl alcohol resin may be an interesting approach to confer durability on species sensitive to biological attack and degradation because they can interfere with the biochemical mechanisms used by fungi to degrade wood. Condensed or hydrolysable tannins are molecules easily extracted from lignocellulosic biomass powder. They represent up to 30% by mass of the dry material found in the bark or wood of various conifers and hardwoods (mimosa, acacia, pine, quebracho, chestnut, oak, among others). Their chemical structure enables polymerization and easy functionalization.

On the model of composites obtained with mixture of furfuryl alcohol and condensed tannins, we develop formulations with hydrolysable tannins extracted from Chestnut. These tannins were used directly after extraction or after chemical modification by acylation with fatty alkyl chain to improve their hydrophobicity in order to limit water sorption and reduce fungal colonization. Different formulations were investigated to modify wood and their effect on wood decay resistance evaluated.

### REFERENCES

Azadeh, E., Abdullah, U. H., Ali, N. B. M., Pizzi, A., Gerardin-Charbonnier, C., Gerardin, P., Samium W.S., Ashari S.E., (2022). Development of Water Repellent, Non-Friable TanninFuranic-Fatty Acids Biofoams. *Polymers*, 14, 5025



# **DAY 1**

## **SESSION THREE**

### **POSTERS 1**

**Poster 3.01 - Heat Treatment of *Cryptomeria japonica* from Azores**Yurlet Mercado<sup>1</sup>, Lina Nunes<sup>2</sup>, Luisa Cruz-Lopes<sup>1</sup> and Bruno Esteves<sup>1</sup><sup>1</sup>CERNAS Research Centre, Polytechnic Institute of Viseu, 3504-510 Viseu, Portugal; E: bruno@estgv.ipv.pt; lvalente@estgv.ipv.pt<sup>2</sup>Structures Department, National Laboratory for Civil Engineering, Av. do Brasil, 101, 1700-066 Lisbon, Portugal CE3C, Centre for Ecology, Evolution and Environmental Changes & CHANGE, Global Change and Sustainability Institute, University of the Azores, 9700-042 Angra do Heroísmo, Portugal. E: linanunes@lnec.pt**Keywords:** cryptomeria, dimensional stability, heat-treatment, termites**ABSTRACT**

*Cryptomeria japonica* is a species originated from Japan that has been introduced to the Azores archipelago (Portugal) on the mid-19th century and has been thriven since then. The main problem of this wood is its high dimensional instability and therefore heattreatment might be a valuable treatment to improve this wood properties. Chemical composition, swelling, bending strength, MOE and subterranean termite resistance were determined. Extractive composition of untreated and heat-treated cryptomeria wood is presented in Table 1. Untreated cryptomeria extractives are mainly composed of ethanol extractives (1.3%) followed by water (0.99%) and dichloromethane (0.6%). With heat treatment there is a small increase in ethanol extractives similar to what was found before for other species like heat-treated Paulownia wood (Esteves *et al.* 2021). This increase, however, was not observed on some thermally modified tropical hardwoods like afrormosia (*Pericopsis elata*) and duka (*Tapirira guianensis*) probably do to the already higher ethanol extractives content in untreated wood (Esteves *et al.* 2022).

**Table 1: Chemical composition of untreated and treated wood.**

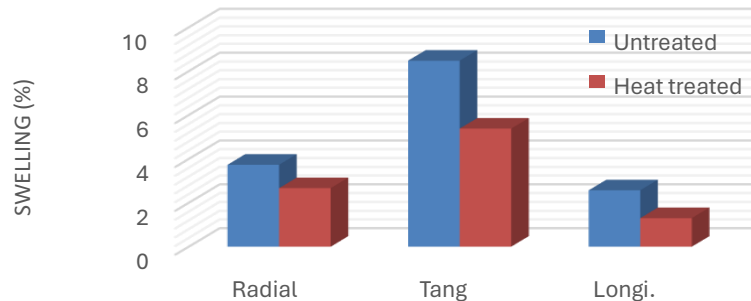
Sample	Extractives (%)											
	Dic		Ethanol			Water			Total			
Untreated	0.60	±	0.00	1.30	±	0.43	0.99	±	0.11	2.89	±	0.00
Heat-treated	0.55	±	0.04	2.05	±	0.13	0.99	±	0.24	3.59	±	0.00

The percentage of lignin increases from 32.5% to around 38.9% probably due to the higher degradation of hemicelluloses that decrease from 22.9% to 15.5% but also to the condensation reactions between lignin and degradation compounds from polysaccharides. The natural durability of *C. japonica* against subterranean termites has been shown to vary significantly even within trees of the same plantation though always susceptible to some level of attack. In the present tests conducted according to the method described in EN117 (2012), all samples, untreated or heat treated showed high susceptibility to subterranean termites (Table 2).

**Table 2: Untreated and treated wood resistance to termite (EN 117, 2012).**

Material	Survival rate [%]	Moisture content [%]	Mass loss [%]	Attack level
Untreated	40.60	56.89	8.82	3.8
Treated	31.30	64.42	10.48	4.0

As expected, the swelling decreased with the treatment and the decrease was higher for tangential direction followed by radial and longitudinal directions (Figure 1). The improvement was around 37% for the tangential direction changing from 8% to around 5% swelling.



**Figure 1: Swelling of untreated and heat-treated wood.**

Mechanical strength is known to decrease due to heat-treatment, especially bending strength. MOE has been known to increase in the beginning of the treatment, decreasing afterwards for more severe treatments. A similar pattern was observed here, where bending strength decreased from 52.6 MPa to 42.3 MPa corresponding to a 19.6% decrease in relation to untreated wood while MOE increased from 7268 MPa to 10836 MPa (Table 3).

**Table 3. MOE and Bending Strength**

Sample	MOE (MPa)		Bending strength (MPa)	
	Average	Std. Dev.	Average	Std. Dev.
Untreated	7268	1194	52.6	2.4
Heat-treated	10836	771	42.5	6.0

Results show that cryptomeria chemical composition changes with heat-treatment, increasing lignin and ethanol extractives, improving dimensional stability, apparently with no significant difference on the resistance to subterranean termites and with relatively low mechanical degradation.

## REFERENCES

Cheng, S.-S., Lin, C.-Y., Chung, M.-J., and Chang, S.-T. (2012). Chemical Composition and Antitermitic Activity against *Coptotermes formosanus* Shiraki of *Cryptomeria japonica* Leaf Essential Oil: *Chemistry & Biodiversity*, **9**(2), 352–358. DOI: 10.1002/cbdv.201100243.

European Standard EN117. (2012). Wood preservatives. Determination of toxic values against *Reticulitermes* species (European termites) (Laboratory method) CEN, Brussels.

Esteves, B., Ayata, U., Cruz-Lopes, L., Brás, I., Ferreira, J., and Domingos, I. (2022). Changes in the content and composition of the extractives in thermally modified tropical hardwoods: *Maderas-Cienc Tecnol*, **24**(22), 1–14. DOI: <http://dx.doi.org/10.4067/s0718221x2022000100422>.

Esteves, B., Ferreira, H., Viana, H., Ferreira, J., Domingos, I., Cruz-Lopes, L., Jones, D., and Nunes, L. (2021). Termite resistance, chemical and mechanical characterization of *Paulownia tomentosa* wood before and after heat treatment: *Forests*, MDPI, **12**(8), 1114. DOI:10.3390/f12081114.

## Poster 3.02 - Effects of QUV Accelerated Weathering on Surface Hardness of Thermally Modified Woods (*Fagus Sylvatica* L. and *Pinus nigra* J.F. Arnold)

Holta Cota<sup>1</sup>, Entela Lato<sup>1</sup>, Leonidha Peri<sup>1</sup>, Hektor Thoma<sup>1</sup>, Doklea Quku<sup>1</sup>, Dritan Ajdinaj<sup>1</sup>, Erald Kola<sup>1</sup>, Ottaviano Allegretti<sup>2</sup>, Marco Togni<sup>3</sup>, Giacomo Goli<sup>3</sup>

<sup>1</sup>Agricultural University of Tirana, Faculty of Forest Sciences, Department of Wood Industry, Tirana, Albania E: hcota@ubt.edu.al; E: elato@ubt.edu.al; E: hektor.thoma@ubt.edu.al E: dquku@ubt.edu.al; E: leonidha.peri@ubt.edu.al; E: d.ajdinaj@ubt.edu.al; E: ekola@ubt.edu.al

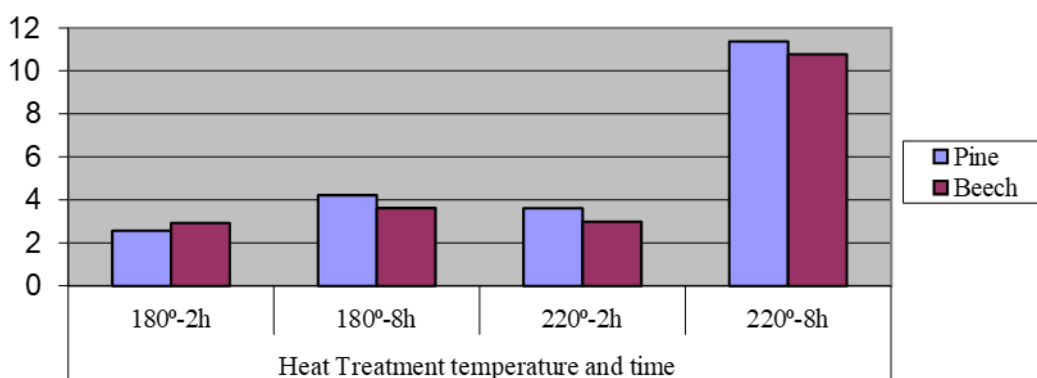
<sup>2</sup>Istituto per la BioEconomia, IBE, CNR, San michele all'Adige TN, Italy. E: ottaviano.allegretti@ibe.cnr.it

<sup>3</sup>Università degli Studi di Firenze, DAGRI, Firenze, Italy. E: marco.togni@unifi.it, E: giacomo.goli@unifi.it

**Keywords:** accelerated weathering, beech, hardness, mass loss, pine, thermal modification

### ABSTRACT

Wood is an organic material used for structural, decorative and aesthetic applications. However, when it is used in external environment exposed to atmospheric agents a lot of its properties change. Thermal modification has become a conventional answer to improve some of the wood properties that makes the material suitable for external use. The aim of this study was to determine the effect of both thermal modification and QUV accelerated weathering on the surface hardness of different woods species such as beech (*Fagus Sylvatica* L.) and pine (*Pinus nigra*). Five groups of 20 samples each were prepared for the modification process. The samples were modified at high temperatures (180 and 220 °C) with two different periods (2h and 8h). After that, the modified samples were exposed to the accelerated weathering conditions for two different times durations (120 and 240 hour) according to the ASTM G 154 standard. The hardness measurements of radial surface were made on natural (untreated), modified and accelerated weathered samples, according to the respective standards. The highest percentage of mass loss is detected at treatment with the higher temperature and the longer time. The hardness values are increased for samples modified at lower temperature and time (180 °C for 2 h), and decreased for treatment with higher temperature and longer time (the highest decrease is detected for treatment 220 °C for 8 h). The hardness of heat-treated wood samples exposed in QUV accelerated weathering decreased compared to the control samples.



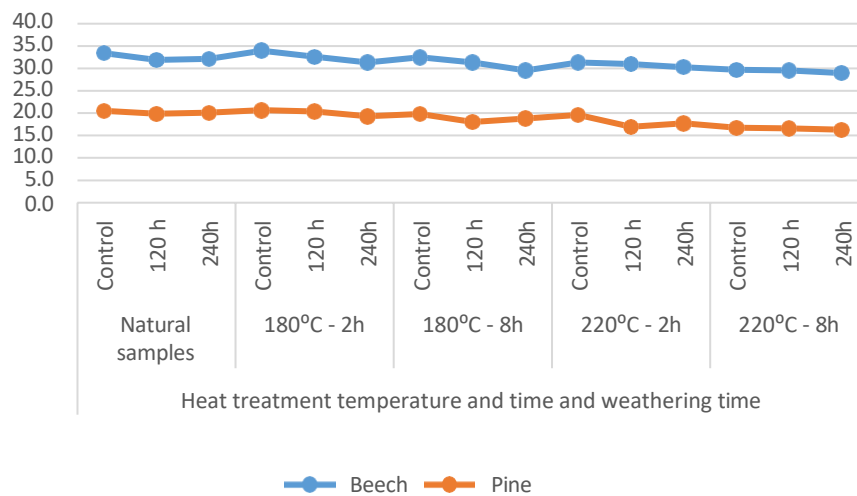
**Figure 1: The average mass loss according heat modification regimes (%)**



**Figure 2: Thermally modified samples**



**Figure 3: QUV treatment**



**Figure 4: The hardness values (N/mm<sup>2</sup>) of modified wood after accelerated weathering treatment**

## REFERENCES

- Allegretti, O., Brunetti, M., Cuccui, I., Ferrari, S., Nocetti, M., Terziev, N. (2012). Thermo-Vacuum modification of spruce (*Picea Abies* Karst.) and fir (*Abies Alba* Mill.) wood. *BioResources* 7(3), 3656-3669
- Bekhta, P., Niemz, P. (2003). Effect of high temperature on the change in color, dimensional stability and mechanical properties of Spruce wood. *Holzforschung* 57: 539-546. - doi: 10.1515/HF.2003.080
- Cheng, L., Di, Y., Zhao, P., Dai, J., Yang, Zh., Chang, Y. (2021) Effects of accelerated weathering test on properties of Larch wood, *BioResources* 16(4), 7400-7415
- Esteves, B.M., Pereira, H.M. (2009). Wood modification by heat treatment: a review. *BioResources* 4: 370404.
- Hill, C. Wood Modification—Chemical, Thermal and Other Processes; Stevens, C.V., Ed.; John Wiley & Sons: West Sussex, UK, 2006; ISBN 9780470021729.

Godinho, D., Araujo, S., Quilho, T., Diamantion, T., Gominho, J. (2021). Thermally Modified wood exposed to different weathering conditions: A review. *Forests* 12, 1400. <https://doi.org/10.3390/f12101400>

Goli, G., Negro, F., Emmerich, L., Militz, H. (2023). Thermal and chemical modification of wood – a combined approach for exclusive, high- demanding performance products. *Wood Material Science & Engineering*, Volume 18, Number 1, pg 58-66(9)



## Poster 3.03 - Effect of Thermal Post-Treatment on the Properties of Densified *Ceiba pentandra* Wood

Larissa do Vale<sup>1</sup> and Cláudio Del Menezzi<sup>1</sup>

<sup>1</sup>Department of Forest Engineering, Faculty of Technology, University of Brasília, Campus Asa Norte, 70910-900, Brasília, DF, Brazil. E: cmenezzi@unb.br

**Keywords:** kapok wood, thermo-mechanical treatment, tropical wood

### ABSTRACT

*Ceiba pentandra* (kapok wood) is a tropical wood species presenting low density, easy machinability, low natural durability and is mainly used for aeromodelling because of the very high strength/weight ratio. Regardless, since this wood species is very disponsible on the e market it is important to promote its utilization for others ends. In this context, the modification of wood using thermal, chemical or mechanical processes can play an important role to achieve this goal. In recent years, research efforts have focused on thermal modification combined with mechanical compression, which is known as thermomechanical modification. In these processes the wood is thermally treated and at same time is compressed, leading to a density improvement. The main advantage is to minimize the usual drawback of thermal modification: the reduction in mechanical properties. As the wood is densified, the mechanical properties usually do not reduce. On the other hand, the wood treated this way houses high level of compression stresses, which can be released when it gets in contact with water, improving significantly the thickness swelling. Despite of these findings, several studies have evaluated the utilization of densification process to improve properties of tropical woods (Costa and Del Menezzi, 2017; Freitas *et al.* 2016; Arruda and Del Menezzi, 2015).

In this context, the aim of this study was to evaluate the effect of a thermal post-treatment on the properties of *Ceiba pentandra* wood previously densified. The densification treatment (DT) was performed using a hydraulic press with controlled temperature and pressure (Ribeiro and Del Menezzi, 2019). Forty boards ( $\rho=0.34 \text{ g/cm}^3$ ) measuring 400 mm x 200 mm x 35 mm (l x w x t) were densified at 180 °C following four schedules: two duration times (5 and 10 minutes) and two pressures (1.07 MPa and 2.15 MPa). Densification rate (DR) and compaction rate (CR) were evaluated after densification. Afterwards, half of the samples were submitted to a thermal post-treatment (PT) for 25 minutes at 180°C under a pressure only enough to provide contact with the heated plates of the hydraulic press, as proposed by Del Menezzi and Tomaselli (2005). ASTM D143 (2014) standard was observed to determine parallel and perpendicular compression strength ( $f_{c,0}$ ;  $f_{c,90}$ ), parallel Janka hardness ( $f_{H,0}$ ), bending strength ( $f_m$ ) and bending stiffness ( $E_M$ ). Stress-wave dynamic modulus of elasticity ( $E_d$ ) was also evaluated. Thickness swelling (TS), water absorption (WA) and equilibrium moisture content (EMC) was also assessed.

It was observed that density of densified material was improved up to 67% ( $0.57 \text{ g/cm}^3$ ) and DR varied from 20.2% to 68.2% while CR from 20.4% and 43.6% depending on pressure applied. This way, all mechanical properties were significantly improved in comparison with undensified material but the most benefited property was  $f_{H,0}$  which value was 115.6% higher (Table 1). On the other hand, the TS and WA after 2 and 24 hours of water immersion were degraded in comparison with undensified material. Nevertheless, EMC was significantly reduced after densification.

It was observed that PT applied was effective in yield further improvement of the mechanical properties, except  $E_M$  which was negatively affected (Table 1). Regarding

dimensional stability properties, TS after 2 hours benefited from PT only for the material densified under higher pressure (2.15 MPa). However, it was observed that DT reduced significantly EMC of the material treated under all tested conditions.

**Table 1: Mechanical properties of untreated, densified and post-treated Ceiba pentandra wood.**

Property [MPa]	Situation	T1	T2	T3	T4	Untreated
f <sub>c,90</sub>	AD <sup>a</sup>	6.03	6.12	7.12	8.21	4.99
	APT <sup>b</sup>	7.94*	6.78	9.28*	11.93*	
f <sub>c,0</sub>	AD	28.62	29.19	36.81	36.19	21.25
	APT	30.20*	32.25*	43.25*	44.77*	
f <sub>H,0</sub>	AD	18.73	19.81	25.79	29.88	13.86
	APT	22.91*	22.76*	33.61*	33.57*	
f <sub>m</sub>	AD	53.13	49.71	67.41	65.76	38.12
	APT	48.17*	56.41*	68.57*	75.44*	
E <sub>M</sub>	AD	6278.7	5803.1	7383.9	6494.8	4987.6
	APT	4674.5*	5265.1	5622.1*	5923.7*	
E <sub>d</sub>	AD	5880.6	5252.4	8377.3	8862.4	5154.5
	APT	6543.7*	6732.2*	9468.8*	10429.2*	

<sup>a</sup>After densification, <sup>b</sup>After post-treatment; \*AD x APT value significantly different at 5% level; T1: 5°/1.07 MPa; T2: 10°/1.07 MPa; T3: 5°/2.15 MPa; T4: 10°/2.15 MPa.

It can be concluded that the densification processes improved significantly all mechanical properties, but dimensional stability properties were not improved. The proposed thermal post-treatment yielded further improvement of the mechanical properties, while it was ineffective in reducing the dimensional stability of the densified boards.

## REFERENCES

- Arruda, L. M. and Del Menezzi, C. H. S. (2016). Properties of a laminated wood composite produced with thermomechanically treated veneers. *Advances in Materials Science and Engineering*, 2016, p. 1-9.
- Costa, M. A. and Del Menezzi, C. H. S. (2017). Effect of thermo-mechanical treatment on properties of parica plywoods (*Schizolobium amazonicum* Huber ex Ducke). *Revista Árvore*, 41(1), 1-8.
- Del Menezzi, C. H. S. and Tomaselli, I. (2006). Contact thermal post-treatment of oriented strandboard: a preliminary study. *European Journal of Wood and Wood Products*, 64(3), 2122-217.
- Freitas, A. C., Gonçalves, J. C. and Del Menezzi, C. H. S. (2016). Tratamento termomecânico e seus efeitos nas propriedades de Simarouba amara (Aubl.). *Floresta e Ambiente*, 23(4), 565-572
- Ribeiro, M. N. F. and Del Menezzi, C. H. S. (2019). Effect of the temperature and pressure on properties of densified medium density fiberboards. *Wood Research*, 64(4), 613-624.

## Poster 3.04 - Analysis of Thermally Modified Norway spruce Shingles after Eight Years of Use

Boštjan Lesar<sup>1</sup>, Davor Kržišnik<sup>1</sup>, Miha Humar<sup>1</sup>

<sup>1</sup>University of Ljubljana, Biotechnical Faculty, Department of Wood Science and Technology, Jamnikarjeva 101, SI1000, Ljubljana, Slovenia. E: miha.humar@bf.uni-lj.si; davor.krzisnik@bf.uni-lj.si; bostjan.lesar@bf.uni-lj.si

**Keywords:** exposure, Norway spruce, protection, resistance, shingle, thermally modified wood

### ABSTRACT

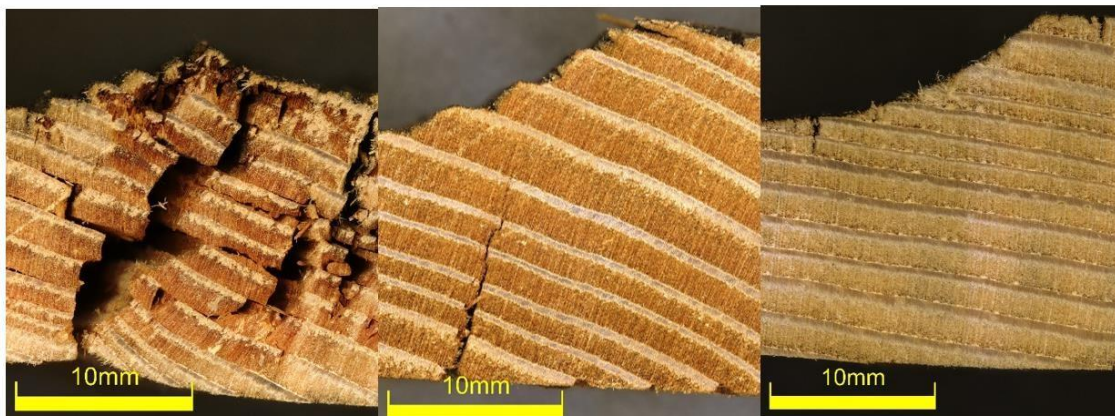
In the past, wooden roofing was one of the most widespread roofing materials in presentday Slovenia, along with thatch (Šarf 1976). Later, however, they were largely replaced by tiled roofs. Today, wooden roofing tiles are used mainly in the Triglav National Park, in the pasture areas of Gorenjska and Koroška, and for covering cultural and historical buildings. Wood-based roofing was used throughout the Alpine area (Palanti *et al.* 2014). In the 20th century, wooden roofing was often seen as a sign of poverty and a backward economy and was quickly replaced by tile and later by cement-asbestos roofs (Šarf 1976). It has always been a challenge to select wood of sufficient quality for this type of use and, moreover, to ensure its technical suitability. Shingles were made from spruce (*Picea abies*) heartwood, sometimes also from the heartwood of larch (*Larix decidua*). However, in some southern Slovenian regions (Notranjska and Kočevsko), fir wood (*Abies alba*) was also used for this purpose, mainly because of its availability (Šarf 1976). In recent decades, a number of wood preservation, conservation and modification techniques have been developed to prolong the service life of the wood. These techniques make it possible to manufacture products that meet the requirements for the use of wood in exterior applications, even from non-durable wood. One of these is thermal treatment. Due to changing lifestyles and climate change, wooden roofs today often fail earlier than planned, so that the use of wooden roofs often does not pay off (Humar *et al.* 2021). The aim of the study was to evaluate the suitability of the thermal modification process for the production of wooden roof tiles.

The shingles were made of thermally modified (TM) Norway spruce (*Picea abies*). Modification was carried out using the Silvapro method at 230 °C for three hours under semi-anoxic conditions (Rep *et al.* 2012). Untreated spruce shingles and spruce shingles impregnated with a copper-based preservative solution (Silvanolin, Silvaproduct) were used as references. The shingles were in use for 7 years on the model object at the Department of Wood Science and Technology, Ljubljana, Slovenia. Thermally modified shingles and reference shingles were exposed on the south side, while copper treated shingles were exposed on the north side with inclination of 45%. Moisture content monitoring was performed on parallel shingles using Scanntronik equipment during the exposure.

After seven years in use, we have isolated nine TM, seven untreated and four impregnated shingles. After conditioning in the laboratory for two weeks, scans were taken of the top and bottom surfaces. Then each shingle was cut into one 3 cm and nine 5 cm pieces. Then each piece of shingle was scanned from the axial planes. The top and axial surfaces of the shingles were then analysed with a digital microscope (Olympus DSX1000) at 1x and 10x magnification. The surface roughness of the shingles was observed with the Olympus Lext OLS5000. We analysed the exposed part of the shingle and the unexposed ones.

Later, the contact angle of water on the shingle surface was determined using a tensiometer (Theta Lite). The compressive strength of the shingle pieces was determined with a

Zwick/Roell Z250. The loading rate was set at 2 mm/min. The shingles made of the reference spruce wood were already severely degraded after seven years. The modified and impregnated spruce, on the other hand, was better preserved. The compressive strength of the modified and impregnated spruce wood was comparable to the unexposed control samples. On the other hand, the compressive strength of the untreated spruce wood decreased significantly. The special characteristic of TM spruce wood is the brittleness and abrasion of the early wood, which is reflected in a strongly grooved surface. The least rough and consequently most hydrophobic shingles were those impregnated with Silvanolin, while on the other hand TM spruce wood had the least hydrophobicity, which was also the most rough. With thermally modified wood we can guarantee a reasonable and similar durability as with biocide-treated wood. In general, we can state that thermal modification is a suitable method for treating spruce shingles to be used in Central Europe.



**Figure 1: Cross-section of exposed samples of unprotected spruce (left), TM spruce (centre) and impregnated spruce (right) at 1x no magnification**

### ACKNOWLEDGEMENTS

The authors acknowledge the financial support of Slovenian Research Agency (ARRS) within research programme P4-0015 (Wood and lignocellulosic composites), and the Infrastructure Centre (IC LES PST 0481-09). The Ministry of Agriculture, Forestry and Food, under the V42017 project, also supported part of the published research.

### REFERENCES

- Humar, M., Lesar, B., Kržišnik, D. (2021) Vpliv podnebnih sprememb na dinamiko glivnega razkroja lesa v Sloveniji. *Acta Silvae et Ligni* 125:53–59. <https://doi.org/10.20315/asetl.125.5>
- Rep, G., Pohleven, F., & Kosmerl, S. (2012). Development of the industrial kiln for thermal wood modification by a procedure with an initial vacuum and commercialisation of modified Silvapro wood. In M. H. and M. P. D. Jones, H. Miltz, M. Petrič, F. Pohleven (Ed.), *Proceedings of the 6th European Conference on Wood Modification* (pp. 11–17). University of Ljubljana.
- Palanti, S., Macchioni, N., Paoli, R., Feci, E. and Scarpino, F. (2014) A case study: The evaluation of biological decay of a historical hayloft in Rendena Valley, Trento, Italy. *Int Biodeterior Biodegradation* 86:179–187. <https://doi.org/10.1016/j.ibiod.2013.06.026>
- Šarf F (1976) Lesene strehe v Sloveniji. *Slovenski etnograf* 53–74

## Poster 3.05 - Physical Properties of Thermally Modified *Gmelina arborea* Wood Modified under Different Process Conditions

Samuel Olaniran<sup>1</sup> and Holger Militz<sup>1</sup>

<sup>1</sup>Wood Biology and Wood Products, Georg-August-Universität Göttingen, Büsgenweg 4, 37077 Göttingen, Germany. E-Mail: samuel.olaniran@uni-goettingen.de; holger.militz@uni-goettingen.de

**Keywords:** closed process, corrected mass loss, fast-growing, *Gmelina* wood, open process, physical properties, volumetric swelling

### ABSTRACT

This study investigated the physical properties of *Gmelina arborea* wood following thermal modification using the process conditions similar to ThermoWood® and Firmolin® processes, which are hereafter referred to as open and closed processes respectively. These processes are used in Europe among many other thermal modification processes. The major aim of examining these processes on *gmelina* wood is to find a suitable processing condition that will offer acceptable reduction of moisture ingress, deliver good quality of thermally modified wood, while enhancing other important technological properties based on target end uses. Although *Gmelina arborea* is a fastgrowing wood species, it is dimensionally unstable and non-durable. Furthermore, *gmelina* is a difficult-to-treat wood species, and extremely refractory to treatment with chemicals as revealed by outcome of impregnation tests of this species from different countries. To preserve the aesthetic value, and prevent excessive mechanical damage due to popularly used pre-treatment methods such as incising; thermal modification was chosen as a possible option for modification of *gmelina* wood. For this study, thermal treatment using the open process covered three temperature regimes; 180 °C, 200 °C, and 220 °C. Modification in the closed process was done under temperature regimes including 160, 170 and 180 °C, and under low pressure, as use of high pressure have been reported to cause more severe degradation to wood polymers. The outcome of the above modification methods showed that corrected mass loss of samples modified under both processes increased with increasing temperature; but higher losses was recorded in the open process. Further results showed that, in both processes, mass loss significantly influenced the final moisture content, while density was significantly influenced by mass loss through the open process. Volumetric swelling was also reduced from 9.86% to 3.8% for samples modified at 220 °C in the open process, while similar volumetric swelling of 5.41% and 5.26% was recorded for samples modified at 180 °C in open and low-pressure closed process respectively. From these results, thermal treatment of *gmelina* wood showed improved resistance to moisture ingress in the open process at higher temperatures between 200 to 220 °C, but its effect on density loss was significant, and this may further negatively influence its final quality and strength properties.

## Poster 3.06 - Effect of Thermal Treatment on the Interaction of Wood with Liquid Water

Dace Cirule<sup>1</sup>, Edgars Kuka<sup>1</sup>, Ingeborga Andersone<sup>1</sup>, Bruno Andersons<sup>1</sup>

<sup>1</sup>Laboratory of Wood Biodegradation and Protection, Latvian State Institute of Wood Chemistry, Dzērbenes 27, Riga, Latvia. E: dace.cirule@kki.lv

**Keywords:** drying, interaction with liquid water, permeability, thermally modified wood, wettability

### ABSTRACT

Among the extensively investigated properties of thermally modified (TM) wood, the aspects related to changes in the interaction of wood with liquid water as a result of thermal treatment are less well studied. However, such knowledge is important for the proper use of materials in places where they may be exposed to liquid water or for treatments that expose them to liquid water, e.g. impregnation. In the study, the trends of potential wetting and drying of TM birch and TM pine wood compared to their unmodified counterparts were evaluated. Wood wettability and permeability were assessed by determining constant wetting rate angle (CWRA) (Nussbaum 1999) and capillary water absorption coefficients  $A_w$  ( $\text{kg/m}^2 \text{ h}^{1/2}$ ) in transversal directions (through the radial and tangential surfaces), respectively. Implication of wood wettability and permeability on material performance during outdoor exploitation was compared by assessing moisture distribution after a rainy period in cross-sections of boards exposed outdoors on weathering rack. Drying was characterized by rates of water release through the surfaces of the side edges from impregnated laboratory specimens (20 x 20 x 100 mm) and boards (20 x 100 x 700 mm) with sealed end-grains.

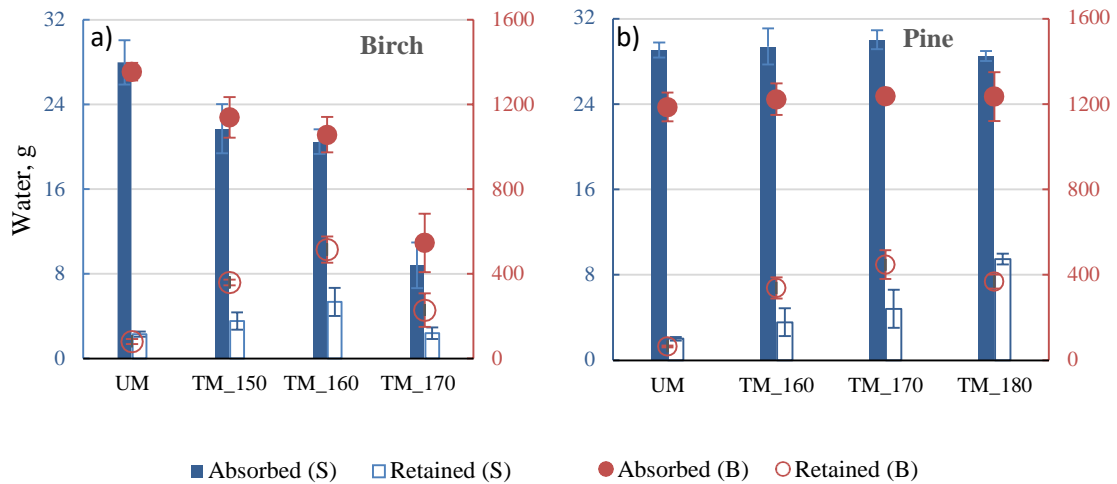
Decreased surface wettability of TM wood of both species was detected by CWRA implying that liquid water penetration into the wood could be hindered. However, other studies have found that the changes in wood permeability due to thermal treatment depend on the wood type and that increased capillary water absorption is characteristic of TM pine sapwood (Johansson *et al.* 2006, Scheiding *et al.* 2016). The results of the present study were consistent with these findings and showed reduced capillary water absorption of TM birch while increased water uptake of TM pine, with the changes through the tangential surface being particularly pronounced. The opposite trend in the change in water absorption due to thermal treatment of birch and pine showed excellent agreement with the moisture distribution pattern in the boards when increased water penetration into the TM pine boards was detected after a prolonged rain event (Table 1). The most likely cause for the increased water permeability of TM pine could be the micro-defects in the wood caused by the thermal treatment, as observed in microscopic investigations of TM wood (Altgen *et al.* 2015).

**Table 1: Moisture content (%) in cross-section of unmodified (UM) and thermally modified (TM) birch and pine boards depending on the distance from the surface.**

Distance, mm	Birch_UM	Birch_TM	Pine_UM	Pine_TM
0-5	42.4 (6.0)	40.2 (6.9)	53.6 (4.8)	77.9 (10.7)
6-10	28.2 (1.8)	22.7 (5.7)	49.7 (8.0)	71.6 (10.4)
11-15	25.3 (1.1)	17.4 (1.7)	40.0 (5.2)	61.5 (9.3)
16-20	25.2 (0.7)	17.8 (1.1)	25.9 (1.0)	40.8 (8.8)



Regarding water release, significantly delayed drying was observed in TM wood of both species compared to the unmodified wood with the drying rate slowing down with increasing temperature of thermal treatment (Fig. 1).



**Figure 1: Amount of water absorbed in impregnation and retained after conditioning in unmodified (UM) and thermally modified (TM) birch (a) and pine (b) wood: S – laboratory specimens, conditioned for 180 h (primary axes); B – boards, conditioned for two weeks (secondary axes).**

The results indicate that, particularly for TM pine, exposure of unprotected TM wood to liquid water can lead to a significantly prolonged state of increased moisture content due to slowed release of water during the drying phase, which in the case of TM pine is coupled with increased wood permeability.

## REFERENCES

- Altgen, M., Adamopoulos, S., Miltz, H. (2015). Wood defects during industrial-scale production of thermally modified Norway spruce and Scots pine. *Wood Material Science and Engineering*, **12**(1), 14-23.
- Johansson, D, Sehlstedt-Persson, M., Morén, T. (2006). Effect of heat treatment on capillary water absorption of heat-treated pine, spruce and birch. In: *Wood Structure and Properties '06*. Kurjatko, S, Kúdela, J., Lagaña, R. (Ed.), Arbora Publishers, Zvolen, Slovakia, pp. 251-255.
- Nussbaum, R.M. (1999). Natural surface inactivation of Scots pine and Norway spruce evaluated by contact angle measurements. *Holz als Roh- und Werkstoff*, **57**, 419-424.
- Scheiding, W., Direske, M., Zauer, M. (2016). Water absorption of untreated and thermally modified sapwood and heartwood of *Pinus sylvestris* L. *European Journal of Wood and Wood Products*, **74**, 585-589.

## Poster 3.07 - Direct Evaluation of the Effect of Thermal Treatment on the Parallel Compression Strength of Wood

Rossana da Rosa<sup>1</sup>, Annie Cavalcante<sup>1</sup>, Isabella de Sá<sup>1</sup>, Bento Viana<sup>1</sup>, Paula Dornelles<sup>1</sup>, Lucia Garcia<sup>2</sup> and Cláudio Del Menezzi<sup>1</sup>

<sup>1</sup>Department of Forest Engineering, Faculty of Technology, University of Brasilia, Brasilia, 70.910-900 DF, Brazil. E: ro.cortelini@hotmail.com

<sup>2</sup>Forest Products Laboratory, Brazilian Forestry Service, Brasilia, 70.818-900 DF, Brazil. E: lucia.garcia@mma.gov.br

**Keywords:** heat treatment, mechanical properties, wood chemistry, wood modification

### ABSTRACT

Due to the hygroscopicity, wood presents some degree of dimensional instability, which limits its applications, especially in structural uses. A viable and well-studied solution to counteract this problem is submitting the material to thermal modification treatments. Although thermal treatment can improve the physical and biological properties of wood, several studies have reported a reduction in mechanical properties (Ferreira *et al.* 2019; Gaff *et al.* 2019; Korkut 2008; Korkut and Hiziroglu 2009), which imposes a barrier to the use of this type of treatment for wood that will be subjected to high mechanical stress. Ipê wood (*Handroanthus* spp.) is known for its high mechanical strength, poor dimensional stability, and high natural durability. Therefore, this study aims to evaluate the effect of thermal treatment on the parallel compression strength of Ipê wood and the chemical and physical changes. For this purpose, the thermal treatment was performed using wood specimens of 150 mm x 50 mm x 50 mm (l x w x t) and an average moisture content of 10%. The set temperatures and residence time in each stage were determined considering the preestablished drying parameters and the high density of Ipê wood. The treatment was divided into three phases: (I) 50 °C for three hours; (II) 120°C for 48 hours and finally (III) 180 °C for 48 hours. The equilibrium moisture content (EMC), density, dimensional stability, and compressive strength ( $f_{c,0}$ ) were determined before and after the thermal treatment. X-ray densitometry (GreCon DAX-6000) was used to evaluate the longitudinal density profile of the untreated and treated wood samples. Dimensional stability and parallel compression strength ( $f_{c,0}$ ) of the samples were determined according to the ABNT 7190:1997 standard. The chemical characterization was performed before and after the thermal treatment the following chemical components were quantified following TAPPI methodology: ash, extractives, lignin, holocellulose,  $\alpha$ -cellulose, and hemicellulose by the difference between the holocellulose and  $\alpha$ -cellulose content. The results of this study indicated that the thermal treatment of Ipê wood resulted in a significant weight loss (WL), averaging a 15.7% difference. The WL is most likely caused by the degradation of wood chemical components, such as the degradation of hemicellulose. The main changes in mass were observed in phases II (150 °C) and III (180 °C) of the treatment, which were performed at the highest temperatures. The Table 1 presents the results of density, EMC, and compression strength. The EMC of Ipê wood were statistically different ( $p$ -value >0.0001) before treatment were 10.1% and 2.7% after heat treatment. This indicates that the hemicellulose was heavily degraded since they are the most sensitive wood polymer to thermal degradation and their degradation directly affects wood water adsorption ability.

**Table 1. Heat treatment effects on properties of Ipê wood (*Handroanthus* sp.)**

Ipê wood	EMC [%]	Density [kg/m <sup>3</sup> ]	fc,0 [MPa]
Non treated	10.1 <sub>(0.18)*</sub>	1016.2 <sub>(72.67)ns</sub>	102.0 <sub>(5.53)*</sub>
Heat treated	2.7 <sub>(0.44)*</sub>	961.0 <sub>(76.85)ns</sub>	124.1 <sub>(11.62)*</sub>
<i>p</i> -value	<0.0001	0.161	<0.001

Note: values between parentheses indicate the standard deviation; \*, <sup>ns</sup> significant and no-significant in *T*test at 0.05 level.

For the results of  $f_{c,0}$ , the t-test showed a significant difference before and after the thermal treatment ( $p$ -value=.000664). Before the treatment, the Ipê samples presented an average compressive strength of 102 MPa and 124 MPa afterwards, which means 22% increase. Studies have shown that thermal treatments with maximum temperatures of 190°C positively impact mechanical properties (Boonstra *et al.* 2007; Brito *et al.* 2008; Herrera Díaz *et al.* 2019). In this temperature range, the degradation of low molecular weight sugars in wood (hemicellulose and cellulose) and a proportional increase in the amount of crystalline cellulose and lignin occurs due to the degradation of components that are more fragile at temperature, such as hemicellulose and the non-crystalline fraction of cellulose. From the results obtained, it is evident that heat treatment has a positive effect on the mechanical properties of wood. The thermal treatment makes the wood less hygroscopic and more resistant to dimensional changes.

## REFERENCES

- Boonstra, M.J., Van Acker, J., Tjeerdsma, B.F. and Kegel, E.V. (2007). Strength properties of thermally modified softwoods and its relation to polymeric structural wood constituents. *Annals of Forest Science*, **64**(7), 679–690.
- Brito, J.O., Silva, F.G., Leão, M.M. and Almeida, G. (2008). Chemical composition changes in eucalyptus and pinus woods submitted to heat treatment. *Bioresource Technology*, **99**(18), 8545–8548.
- Ferreira, M.D., Melo, R.R. de, Zaque, L.A.M. and Stangerlin, D. M. (2019). Propriedades Físicas e Mecânicas da Madeira de Angelim-Pedra submetida a Tratamento Térmico. *Tecnologia Em Metalurgia Materiais e Mineração*, **16**(1), 3–7.
- Gaff, M., Kačík, F., & Gašparík, M. (2019). Impact of thermal modification on the chemical changes and impact bending strength of European oak and Norway spruce wood. *Composite Structures*, **216**(February), 80–88.
- Herrera-Díaz, R., Sepúlveda-Villarreal, V., Torres-Mella, J., Salvo-Sepúlveda, L., Llano-Ponte, R., Salinas-Lira, C., Peredo, M.A. and Ananías, R.A. (2019). Influence of the wood quality and treatment temperature on the physical and mechanical properties of thermally modified radiata pine. *European Journal of Wood and Wood Products*, **77**(4), 661–671.
- Korkut, S. (2008). The effects of heat treatment on some technological properties in Uludağ fir (*Abies bornmuelleriana* Mattf.) wood. *Building and Environment*, **43**(4), 422–428.
- Korkut, S. and Hiziroglu, S. (2009). Effect of heat treatment on mechanical properties of hazelnut wood (*Corylus colurna* L.). *Materials and Design*, **30**(5), 1853–1858.

## Poster 3.08 - Increasing Opportunities for Maillard-type Reactions in Wood through the Addition of Glucose and Citric Acid to Bicine and Tricine Modification

Domen Borko<sup>1,2</sup>, Alexander Scharf<sup>1</sup>, Chia-feng Lin<sup>1</sup>, Olov Karlsson<sup>1</sup>, Dennis Jones<sup>1</sup>, Miha Humar<sup>2</sup> and Dick Sandberg<sup>1</sup>

<sup>1</sup>Wood Science and Engineering, Luleå University of Technology, Forskargatan 1, 931 87 Skellefteå, Sweden. E: domen.borko13@gmail.com, alexander.scharf@ltu.se, chiafeng.lin@ltu.se, olov.karlsson@ltu.se, dennis.jones@ltu.se, dick.sandberg@ltu.se

<sup>2</sup>Biotechnical Faculty, University of Ljubljana, Jamnikarjeva 101, 1000 Ljubljana, Slovenia. E: miha.humar@bf.uni-lj.si

**Keywords:** fire-retardants, impregnation, Maillard reaction, sugars, wood modification

### ABSTRACT

Previously, the use of bicine and tricine as part of an enhanced thermal-modification process was considered (Jones *et al.* 2022). Results suggested there were indications of improving properties and evidence of some degree of bonding, potentially *via* a Maillardtype reaction. However, the thermal stability of the selected compounds resulted in the need for a reduced thermal-modification temperature, which would have expected impacts on the effectiveness of the thermal modification on its own.

In these continued studies, the combined effects of chemical treatment and the thermal modification are investigated in terms of the use of bicine [2-(Bis (2-hydroxyethyl) amino) acetic acid] and tricine [N-(2-Hydroxy-1,1-bis (hydroxymethyl)ethyl) glycine] (Figure 1), in addition to citric acid and glucose. The presence of these chemicals was considered to assess the combined effects and possible interaction between the chemicals and wood respectively, either via the self-polymerisation of citric acid (Mihulja *et al.* 2021), the esterification of citric acid and glucose (He *et al.* 2016) or a combined Maillardtype reaction.

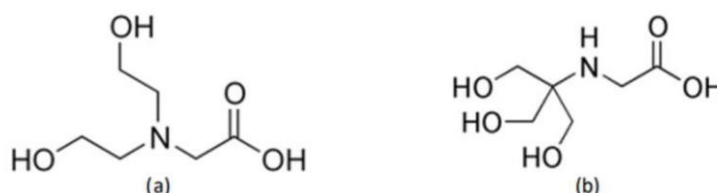
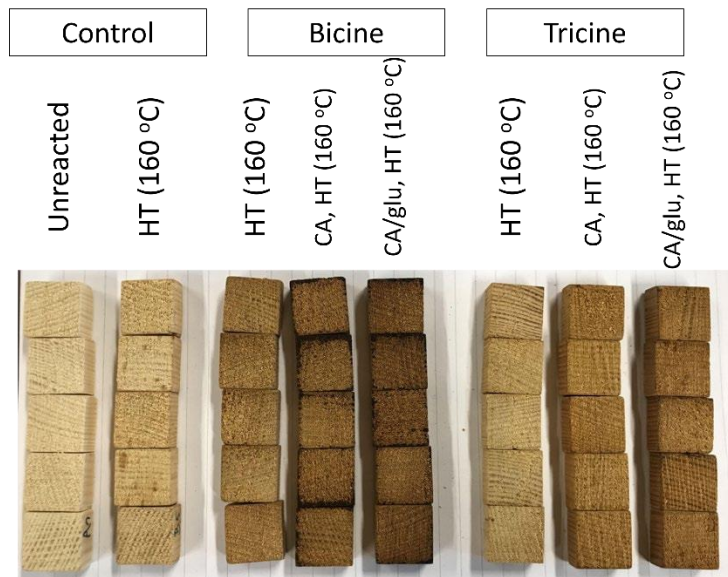


Figure 1: Chemical structure of (a) bicine and (b) tricine.

Scots pine (*Pinus sylvestris* L.) sapwood specimens were pressure impregnated with a range of solutions comprising citric acid, glucose, bicine and tricine in various combinations, followed by drying at 60 °C, followed by thermal treatment at 160 °C for various durations. Visual inspection of samples after reaction (Figure 2) suggests some degree of Maillard reaction has occurred, evidenced by the darker colour of the bicine- and tricine-treated specimens, with further colour enhancement noted due to the presence of citric acid and citric acid/glucose, respectively. After impregnation, significant volume increases were noted, which in several cases, further increased after thermal treatment at 160 °C, which could be evidence of Maillard reactions or a polymeric network occupying a greater volume in the cell walls and lumen than the non-polymerised material.



**Figure 2: Potential Maillard reaction due to increased colouring of bicine and tricine respectively with the addition of citric acid (CA) and glucose (glu).**

Initial results would seem to suggest:

- citric acid alone contributes to a better modification,
- tricine provides better bonding in the wood,
- heat treatment above 160 °C is not suitable if all three reagents are not used, and
- the Maillard reaction takes place only in the case of all three reagents and is enhanced with increased temperature.

Further studies are ongoing and will be presented at the conference.

## REFERENCES

- He, X., Xiao, Z., Feng, X., Sui, S., Wang, Q. and Xie, Y. (2021). Modification of poplar wood with glucose crosslinked with citric acid and 1,3-dimethylol-4,5-dihydroxy ethyleneurea *Holzforschung*, **70**(1), 47-53.
- Jones, D., Kržišnik, D., Hočevar, M., Zagar, A., Humar, M., Popescu, C.-M., Popescu, M.-C., Brischke, C., Nunes, L., Curling, S., Ormondroyd, G., Scharf, A., Parracha, J.L., and Sandberg, D. (2022). Evaluation of the effect of a combined chemical and thermal modification of wood through the use of bicine and tricine. *Forests*, **13**, Article ID: 834.
- Mihulja, G., Živkovic, V., Poljak, D., Šefc, B. and Sedlar, T. (2021). Influence of citric acid on the bond strength of beech wood. *Polymers*, **13**, Article ID: 2801.

## Poster 3.09 - Thermally Modified Wood in Wood-PLA Composites for 3D Printing

Daša Krapež Tomec<sup>1</sup>, Manja Kitek Kuzman<sup>1</sup>, Mirko Kariž<sup>1</sup>

<sup>1</sup>Department of Wood Science and Technology, Biotechnical Faculty, Jamnikarjeva 101, 1000 Ljubljana, Slovenia. E: dasa.krapez.tomec@bf.uni-lj.si; manja.kitek.kuzman@bf.uni-lj.si; mirko.kariz@bf.uni-lj.si

**Keywords:** 3D printing, thermally modified wood, wood-plastic composites

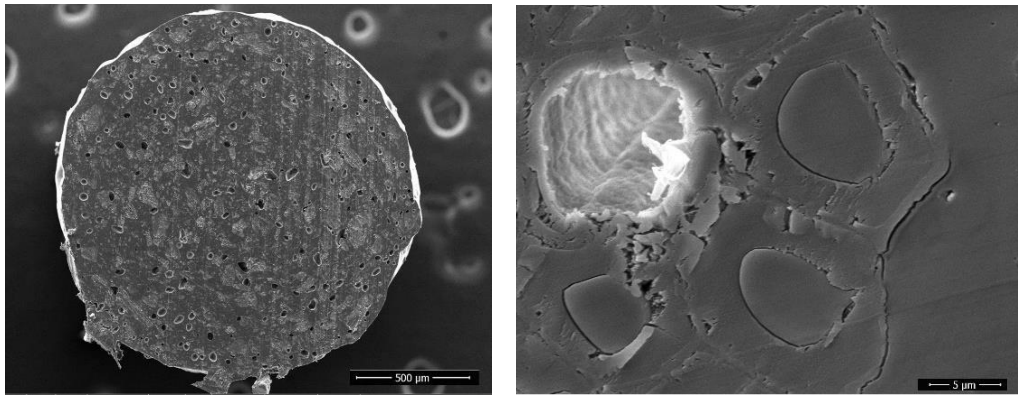
### ABSTRACT

Additive technologies, often referred to as 3D printing, have made great development in recent years, both in the field of the technology itself and in printing materials. There has been an emphasis on developing materials that are derived from natural sources, that emit as few pollutants as possible during the manufacturing process, and that offer the possibility of using residual materials and recycling them at the end of their service life. Biodegradable polymers such as polylactic acid (PLA) are gaining attention as an alternative to petroleum-based products. PLA is a thermoplastic aliphatic polyester derived from renewable sources and has high strength, good optical transparency and high elastic modulus compared to other synthetic polymers. However, it also has disadvantages such as brittleness, low thermal stability, and low crystallization ability (Sin, L. Tin *et al.* 2012).

To overcome its drawbacks, the use of natural fibers and particles in wood-plastic composites has increased in recent decades, including materials for 3D printing (Mazzanti *et al.* 2019). Natural fibers/particles are used as reinforcements in polymer composites to replace synthetic fibers due to their mechanical, and acoustic, combined with their low density, lower environmental impact, and potential alternative end-of-life uses (Ayrilmis *et al.* 2019). These particles often come from wood residues from other wood processing sectors, making them costeffective and adding value to the material.

Adhesion between particles and matrix plays a crucial role in shaping the strength and toughness of composites. The effective stress transfer between the particles and the matrix determines the strength, while the adhesion determines toughness and brittleness. For the appropriate properties of wood-plastic composites, it is also necessary to ensure the compatibility of wood particles and polymer. Higher adhesion leads to lower porosity of the material, better intermolecular diffusion, and thus better mechanical properties. Thermal modification (TM) of wood is one of the possible modifications by which the number of OH groups is reduced - the polarity of the surface of the particles is reduced by the decomposition of hemicelluloses. The reduced polarity of the surface allows better compatibility with the non-polar surface of the polymers and improve the properties of the wood-plastic composite despite the poorer mechanical properties of the thermally modified particles (Kaboarani and Blanchet 2014).

Our research has found greater penetration of the polymer into the cells of TM wood through electron micrographs, indicating better compatibility of the modified wood with the polymer. The same was observed in CT scans, electron micrographs (Figure 1), pycnometry and surface roughness measurements.



**Figure 1: Cross-section of PLA-TM wood filament and of lumens filled with PLA.**

Due to better mixing and lower interfacial energy of TM wood and polymer, TM particles tend to aggregate less than particles from untreated wood. Moreover, from grinding TM wood, smaller particles are obtained due to its brittle nature, and the power/energy consumption of grinding TM wood is much lower compared to non-treated. This translates into better processability, extrusion with less clogging and more uniform filaments because of the smaller wood particles. In summary, the use of TM wood results in lower porosity of the material, better intermolecular diffusion, and thus better mechanical properties.

The use of residues from thermally modified wood is one of the options to increase compatibility with polymers. The effective use of wood throughout its value chain in forest management, the various utilization cycles, and end-of-life disposal can lead to a more sustainable development. This is especially true for cascading wood use – the sequential use of a given resource for different purposes (Höglmeier *et al.* 2017). The concept of using TM wood residues for 3D printing originates from this very idea.

## REFERENCES

- Ayrilmis, N., Kariž, M., and Kitek Kuzman, M. (2019). “Effect of Wood Flour Content on Surface Properties of 3D Printed Materials Produced from Wood Flour/PLA Filament.” *International Journal of Polymer Analysis and Characterization* 24(7): 659–66.
- Höglmeier, K., Weber-Blaschke, G., and Richter, K. (2017). “Potentials for Cascading of Recovered Wood from Building Deconstruction—A Case Study for South-East Germany.” *Resources, Conservation and Recycling* 117: 304–14. <https://www.sciencedirect.com/science/article/pii/S0921344915001779>.
- Kaboorani, A., and Blanchet, P. 2014. “Determining the Linear Viscoelastic Region of Sugar Maple Wood by Dynamic Mechanical Analysis.” *BioResources* 9(3):4392–4409. <https://www.scopus.com/inward/record.uri?eid=2-s2.0-84930626340&doi=10.15376%2Fbiores.9.3.4392-4409&partnerID=40&md5=be2fd1e7eec4cf62333d8f0a59d7115f>.
- Mazzanti, V., Malagutti, L. and Mollica, F. (2019). “FDM 3D Printing of Polymers Containing Natural Fillers: A Review of Their Mechanical Properties.” *Polymers* 11(7): 1094.
- Sin, L. Tin., Rahmat, A. Razak, and Rahman, W. Aizan Wan Abdul. (2012). *Poly(lactic Acid): PLA Biopolymer Technology and Applications*. Oxford: William Andrew.



## Poster 3.10 - Plywood Panels Made of Alternate Layers of Thermally Densified and Non-densified Alder and Birch Veneers

Pavlo Bekhta<sup>1,2,4</sup>, Tomáš Pipiška<sup>2</sup>, Vladimír Gryc<sup>2</sup>, Pavel Král<sup>2</sup>, Jozef Ráhel<sup>2</sup>, Jan Vaněrek<sup>3</sup> and Ján Sedliáčik<sup>4</sup>

<sup>1</sup>Department of Wood-Based Composites, Cellulose, and Paper, Ukrainian National Forestry University, Gen. Chuprynky 103, 79057 Lviv, Ukraine. E: bekhta@nltu.edu.ua

<sup>2</sup>Department of Wood Science and Technology, Mendel University in Brno, Zemědělská 3, Brno 613 00 Czech Republic. E: tomas.pipiska@mendelu.cz; vladimir.gryc@mendelu.cz; pavel.kral@mendelu.cz; jozef.rahel@mendelu.cz

<sup>3</sup>Faculty of Civil Engineering, Institute of Technology of Building Materials and Components, Brno University of Technology, Brno 602 00 Czech Republic. E: vanerek.j@fce.vutbr.cz

<sup>4</sup>Department of Furniture and Wood Products, Technical University in Zvolen, T.G. Masaryka 24, 960 01 Zvolen, Slovakia. E: jan.sedliacik@tuzvo.sk

**Keywords:** birch, black alder, plywood properties, thermal densification, wood veneers

### ABSTRACT

There is no information in the literature about the manufacture of plywood panels by combining thermally densified and non-densified veneers of different high-grade and low-grade hardwood species in one plywood panel. It is hypothesized that mixing veneers from high-grade wood species and low-grade species may improve the properties of the resulting mixed-species plywood panels because of the maximizing the advantages and overcoming the disadvantages of the two resources. Therefore, the aim of this work was to evaluate the effect of combining different high-grade (birch (*Betula verrucosa* Ehrh.) (B), having relatively better structural properties) and low-grade (black alder (*Alnus glutinosa* L.) (A), having lower properties and cost) wood species and different lay-up schemes on the mixed-species plywood properties made from alternate layers of densified (D) and non-densified (N) veneers. The veneer assembly for the production of plywood samples was formed from either birch or black alder veneers only, or from birch veneer combined with black alder veneer, non-densified and densified veneers in one package. There were a total of 16 different veneer panel set options (Table 1). Moisture content (MC), density, bending strength (MOR), modulus of elasticity (MOE), shear strength, thickness swelling (TS), water absorption (WA) and surface roughness of plywood samples were evaluated in accordance with European standards.

The results showed that the type of construction, the wood species and the applied thermal densification of the veneer affect the examined physical and mechanical properties. According to the ANOVA analysis, the WA, MOR, MOE and shear strength of plywood were more sensitive to mixing of wood species in one panel than mixing of densified or non-densified veneers. Moreover, the results indicate great potential of black alder wood in plywood manufacture. Alder veneer can be used to form the inner layers of plywood panels without reducing the shear strength. The B<sup>D</sup>-A<sup>N</sup>-B<sup>D</sup>-A<sup>N</sup>-B<sup>D</sup> and B<sup>D</sup>-A<sup>N</sup>-A<sup>N</sup>-A<sup>N</sup>-B<sup>D</sup> were determined to be the most reasonable lay-up schemes when the shear strength, MOR and MOE values of mixed-species plywood panels manufactured were examined. The values of plywood thicknesses for all types of panels were in the range 6.7-7.4 mm and they do not beyond tolerances for unsanded panels in accordance with standard EN 315.

**Table 1: Configuration of five-layer plywood panels.**

Code of panels	Type of panels	Veneer assembly patterns by wood species	Veneer assembly patterns by type of veneer treatment
I	B <sup>N</sup> -B <sup>N</sup> -B <sup>N</sup> -B <sup>N</sup> -B <sup>N</sup>	B - B - B - B - B	N - N - N - N - N
II	B <sup>N</sup> -A <sup>N</sup> -B <sup>N</sup> -A <sup>N</sup> -B <sup>N</sup>	B - A - B - A - B	N - N - N - N - N
III	B <sup>N</sup> -A <sup>N</sup> -A <sup>N</sup> -A <sup>N</sup> -B <sup>N</sup>	B - A - A - A - B	N - N - N - N - N
IV	A <sup>N</sup> -B <sup>N</sup> -B <sup>N</sup> -B <sup>N</sup> -A <sup>N</sup>	A - B - B - B - A	N - N - N - N - N
V	A <sup>N</sup> -B <sup>N</sup> -A <sup>N</sup> -B <sup>N</sup> -A <sup>N</sup>	A - B - A - B - A	N - N - N - N - N
VI	A <sup>N</sup> -A <sup>N</sup> -A <sup>N</sup> -A <sup>N</sup> -A <sup>N</sup>	A - A - A - A - A	N - N - N - N - N
VII	B <sup>D</sup> -A <sup>N</sup> -A <sup>N</sup> -A <sup>N</sup> -B <sup>D</sup>	B - A - A - A - B	D - N - N - N - D
VIII	A <sup>D</sup> -B <sup>N</sup> -B <sup>N</sup> -B <sup>N</sup> -A <sup>D</sup>	A - B - B - B - A	D - N - N - N - D
IX	B <sup>D</sup> -A <sup>N</sup> -B <sup>D</sup> -A <sup>N</sup> -B <sup>D</sup>	B - A - B - A - B	D - N - D - N - D
X	A <sup>D</sup> -B <sup>N</sup> -A <sup>D</sup> -B <sup>N</sup> -A <sup>D</sup>	A - B - A - B - A	D - N - D - N - D
XI	B <sup>D</sup> -B <sup>D</sup> -B <sup>D</sup> -B <sup>D</sup> -B <sup>D</sup>	B - B - B - B - B	D - D - D - D - D
XII	B <sup>D</sup> -A <sup>D</sup> -B <sup>D</sup> -A <sup>D</sup> -B <sup>D</sup>	B - A - B - A - B	D - D - D - D - D
XIII	B <sup>D</sup> -A <sup>D</sup> -A <sup>D</sup> -A <sup>D</sup> -B <sup>D</sup>	B - A - A - A - B	D - D - D - D - D
XIV	A <sup>D</sup> -B <sup>D</sup> -B <sup>D</sup> -B <sup>D</sup> -A <sup>D</sup>	A - B - B - B - A	D - D - D - D - D
XV	A <sup>D</sup> -B <sup>D</sup> -A <sup>D</sup> -B <sup>D</sup> -A <sup>D</sup>	A - B - A - B - A	D - D - D - D - D
XVI	A <sup>D</sup> -A <sup>D</sup> -A <sup>D</sup> -A <sup>D</sup> -A <sup>D</sup>	A - A - A - A - A	D - D - D - D - D

*B* – birch veneer; *A* – alder veneer; *N* – non-densified veneer; *D* – densified veneer.

Increasing the proportion of thermally densified veneer in one panel leads to a lower thickness and WA, but higher density, MOR, MOE, shear strength and TS of plywood panels. It is important to note that positive effects can be achieved not only by increasing the proportion of densified veneers in one panel but also with a change of construction by mixing wood species. It was shown that non-treated alder veneer, despite somewhat lower strength properties than birch veneer, could be successfully used with proper lay-up schemes in the veneer-based products industry.

Plywood with outer layers of alder veneer has lower bending strength than plywood with outer layers of birch veneer. Alder plywood with inner layers of birch veneer or adjacent birch and alder veneers in the core has a lower MOR and MOE, but higher shear strength, than birch plywood with inner layers of alder veneer or adjacent alder and birch veneers in the core. It was found that plywood panels manufactured from mixing species offer higher bending properties when compared to panels manufactured from alder veneers alone. Moreover, the surface roughness of plywood panels with outer layers of birch veneer is lower than panels with outer layers of alder veneer.

For single-species plywood panels, the birch plywood performed the best in terms of MOR, MOE, shear strength and WA, while the alder plywood performed the worst. The negative influence of alder veneer was attenuated by combining the densified and nondensified veneer as well as alder and birch veneers in one panel. The mixed-species plywood panels allow the increased use of lower cost, low-grade, and low-density alder wood veneers as core veneers in panels to reduce product cost and increase the mechanical properties of predominately low-density alder wood plywood.

## ACKNOWLEDGEMENTS

This work was supported by the EU through the Recovery and Resilience Plan for Slovakia under project No. 09I03-03-V01-00124 and from the European Union's Horizon 2020 research and innovation program under grant agreement No. 952314, and by the project VEGA 1/0264/22.

## Poster 3.11 - Improving the Commercial Value of Some Canadian West Coast Species through Thermal Modification

Yaohui Liu<sup>1</sup>, Gregory Smith<sup>1</sup>, Philip D. Evans<sup>1</sup>, and Stavros Avramidis<sup>1</sup>

<sup>1</sup>Wood Science Department, University of British Columbia, Vancouver, Canada E:  
yaohui@student.ubc.ca; stavros.avramidis@ubc.ca

**Keywords:** canadian west coast species, durability, mechanical properties, physical properties, thermal modification

### ABSTRACT

Wood is one of the oldest construction materials with a superior strength-to-weight ratio, renewability, and versatility. Nevertheless, wood has some unwanted characteristics, such as hygroscopicity, dimensional inconsistency, and biodegradability (Dogu *et al.* 2016; Sandberg and Kuntar 2016). Thermal modification with increasing market acceptance improves some wood properties, producing a new material that does not present an environmental hazard at the end of its life cycle compared to untreated wood (Moore and Cown 2015; Fernandes *et al.* 2017). Local softwoods (Western hemlock, Douglas-fir, and red cedar) and hardwoods (red alder and paper birch) are locally abundant species with relative applications of variable extent. Thermal modification may quickly broaden their application range, bringing extra benefits to product manufacturers and users. This study concentrates on thermal treatment and its effects on the properties of the aforementioned west coast species. Kiln-dried timbers underwent thermal modification at two temperature levels and fixed residence time. Selected physical properties such as density, water absorption, anti-swelling effectiveness, permeability, color change, mechanical properties such as Janka hardness and dynamic modulus of elasticity, and natural durability such as termite resistance and weathering were evaluated and statistically compared.

**Table 1: Categorization of wood species based on thermal treatment levels**

Wood type	Species	Treatment temperature	Number of timbers
Softwood	Western hemlock	50 °C (Control)	13
		190 °C (Mild)	13
		212 °C (Aggressive)	13
	Douglas-fir	50 °C (Control)	13
		190 °C (Mild)	13
		212 °C (Aggressive)	13
	Western red cedar	50 °C (Control)	13
		190 °C (Mild)	13
		212 °C (Aggressive)	13
Hardwood	Red alder	50 °C (Control)	13
		190 °C (Mild)	13
		212 °C (Aggressive)	13
	Paper birch	50 °C (Control)	13
		190 °C (Mild)	13
		212 °C (Aggressive)	13
Total	Five	Three	195

In terms of durability, for the termite resistance, by analyzing weight losses in soil blocks after termite attacks, we have discovered that hardwood and softwood exhibit contrasting

levels of vulnerability, with hardwood experiencing a 69% weight loss and softwood a 33% weight loss on average after five months. Within softwood, Western red cedar stands out as the most termite resistant with a weight loss of just 17%, compared to Douglas-fir (52%) and Western hemlock (30%), which are not remarkably different. Additionally, weight loss was insignificantly different for paper birch (70%) and red alder (68%). Moreover, the mild treatment (190 °C) rendered wood more susceptible to termite attacks (57% weight loss), but the intensive treatment (212 °C) increased the termite resistance of the wood (40% weight loss). The control, mild-treated, and intensive-treated groups collectively demonstrated mass loss of 45%, 57%, and 40%, respectively. For the weathering resistance, results revealed surface checks significantly improved for untreated and mildly treated wood, while samples treated at 212 °C had satisfactory weathering resistance in terms of the surface checks. Color tests in three different layers demonstrated negligible changes in all five species (in terms of the  $L$ ,  $a$ , and  $b$  factors). However, the  $L$  factor significantly decreases as high-temperature treatment is applied to the samples.

Regarding physical properties, thermal treatment at 190 °C significantly reduced water absorption after 2 hours in Douglas-fir, paper birch, and western hemlock. Thermal treatment did not significantly change basic density in five species. Equilibrium moisture content ( $M_e$ ) at relative humidity 20 °C, and relative humidity at 65% significantly decreased following mild and intensive thermal treatments. In contrast,  $M_e$  content at a temperature of 30 °C and relative humidity 40% showed a slight decrease following the mild and intensive treatments. This trend also occurred for  $M_e$  at a temperature of 50 °C and relative humidity of 40%. However,  $M_e$  significantly decreased for the treated samples when exposed to a temperature of 30 °C and relative humidity of 80%.

Similarly,  $M_e$  was significantly reduced for the treated samples when samples were subject to a temperature of 50 °C and relative humidity of 80%. Dimensional stability remarkably improved following heat treatment for five species. Red alder and red cedar were the most and least permeable species in this study. Thermal treatment did not significantly affect the permeability of wood.

An interesting result of this research was hardness in the longitudinal direction did not significantly decrease following mild and intensive heat treatments. Likewise, hardness in transverse direction was not significantly affected by heat treatment. Paper birch and red cedar showed the greatest and smallest hardness values in both longitudinal and transverse directions. Stiffness (MOE) did not show a significant reduction for thermally treated wood. Collectively, thermal treatment greatly impacts BC coastal species' physical and mechanical properties, improving their weathering resistance.

## REFERENCES

- Dogu, D., Bakir, D., Tuncer, F. D., Tirak Hizal, K., Unsal, O., and Candan, Z. (2016). Microscopic investigation of defects in thermally compressed poplar wood panels. *Maderas Ciencia y Tecnologia*, **18**(2): 337–348.
- Fernandes, C., Gaspar, M.J., Pires, J., Silva, M.E., Carvalho, A., Brito, J.L., Lousada, J.L. (2017). Within and between-tree variation of wood density components in *Pinus sylvestris* at five sites in Portugal. *European Journal of Wood and Wood Products*, **75**(4): 511–526.
- Moore, J.R. and Cown, D.J. (2015). Wood quality variability—what is it, what are the consequences and what we can do about it? *New Zealand Journal of Forestry*, **59**(4): 3–9.
- Sandberg, D., and Kuntar, A. (2016). Thermally modified timber: Recent developments in Europe and North America. *Wood and Fiber Science*, **48** (2015 Convention, Special Issue): 28–39.

## Poster 3.12 - Durability of Thermal Modified Wood of *Pinus pinaster*, *Pinus radiata* and *Pinus sylvestris* from Galicia, Spain

David Lorenzo<sup>1</sup>, Josu Arancón<sup>2</sup>, Jorge Crespo<sup>3</sup>

<sup>1</sup>Engineering for Rural and Civil Development. University of Santiago de Compostela, 27002 Campus s/n Lugo, Spain, E: davidlorenzofouz@gmail.com

<sup>2</sup>TECNALIA R&I, Área Anardi 5, 20730 Azpeitia Guipuzcoa, Spain, E: josu.arancon@tecnalia.com

<sup>3</sup>SAVIA-FINSA, Nacional 550, Km 57, 15707 Santiago de Compostela, Spain, E: j.crespo@finsa.es

**Keywords:** modified durability, pine, pinaster, radiata, sylvestris, thermal

### ABSTRACT

This article provides the results of a research about the properties of thermal modified wood of maritime pine (*Pinus pinaster* Ait.), radiata pine (*Pinus radiata* D. Don) and Scots pine (*Pinus sylvestris* L.) from Galicia, Spain, thermal treated in the industrial vacuum-heat autoclave plant of FINSA group in Galicia, Spain. These three pine wood species are used in different solid products but due the low natural durability the application in outdoor applications is limited. The properties of durability of thermal modified wood of these three pine species provide very interesting data improving considerably with thermal treatment at 212 °C reaching a durability class 2 very important for using wood products in indoor and outdoor applications as facade, claddings carpentries, furniture, etc., where wood destroying fungi attacks can occur in the wood products. The improvement in durability against wood destroying fungi allows using these thermal modified wood species without preservatives, using a local wood species with low natural durability against wood destroying fungi, with competitive prices and allowing their recycling at the end of their service life, confirming the suitability of the thermal modification in local wood species with low natural durability.

### REFERENCES

CTE (2009): Spanish Technical Code of Edification.

EN 350 (2016): "Durability of wood and wood-based products - Testing and classification of the durability to biological agents of wood and wood-based materials".

EN 335 (2013): "Durability of wood and wood based products. Use classes: definitions, application to solid wood and wood-based products".

EN 460 (1995): "Durability of wood and wood-based products- Guide to the durability requirements for wood".

EN 113-2 (2020): "Durability of wood and wood based products-Test method against wood destroying basidiomycetes-Part 2: Assessment of inherent or enhanced durability".

EN 84 (1997): "Wood preservatives. Accelerated ageing of treated wood prior to biological testing. Leaching procedure".

Hill, C. (2006) Wood modification. Chemical thermal and other processes. John Wiley and Sons.

Norm FD P 20-651 2011: "Durability of wood products and works".

Jämsä, S., Viitaniemi, P. (2001) Heat treatment of wood- Better durability without chemicals. In: Review on heat treatments of wood. Proceedings of the special on heat treatments. 09.02.2001 in Antibes. 64p.

Kumar, S (1994) Chemical modification of wood. *Wood and Fiber Science*, 26(2) 270-280.

Militz, H., Beckers, E. P. J. and Homan, W. J. (1997) Modification of solid wood: Research and practical potential, Intern. Res. Group on Wood Preserv. Document. No.: IRG/WP 97-40098

Militz, H., Tjeerdsma, B. (2001) Heat treatment of wood by the “PLATO –process”. European Commission Directorate-General for Research, EUR 19885. Proceedings of the Special Seminar held in Antibes/France, 9 February 2001, 23-33.

Rapp AO, Sailer M (2001) Oil heat treatment of wood in Germany- State of the art. In: Review on heat treatments of wood. Proceedings of the special seminar on heat treatments. 09.02.2001 in Antibes. 64p.

Repellin, V. and Guyonet, R. (2003). Evaluation of heat treated beech by non-destructive testing. In: Proceedings of the First European Conference on Wood Modification. Ghent, Belgium, pp. 73-82.

Tjeerdsma BF, Boonstra M, Militz (1998) Thermal modified of non-durable wood species. 2 Improved wood properties of thermally treated wood. Stockholm: Intern. Res. Group Wood Pres., Doc. No.: IRG/WP 98-40124,10 p.

UNI-CEN/TS 15679 (2008) “Thermal modified timber. Definitions and characteristics”.

Welzbacher CR, Rapp AO (2002) Comparison of thermally modified originating from four industrial scale processes – durability. Stockholm: Intern. Res. Group Wood Pres., Doc. No.: IRG/WP 02-40229, 13 p.

### Poster 3.13 - Effect of Thermal Modification on the Color of *Hymenaea* spp. and *Ficus* sp. Wood

Kamilly da Silva Pereira<sup>1</sup>, Anna Clara Oliveira Rupf<sup>1</sup>, Paulo Henrique dos Santos Silveiras<sup>1</sup>, Saulo José da Costa Lima<sup>1</sup>, Djeison Cesar Batista<sup>1</sup>, Victor Fassina Brocco<sup>2</sup>

<sup>1</sup>Department of Forest and Wood Sciences, Federal University of Espírito Santo, Jerônimo Monteiro, Brazil. E: kamillysilv4p@gmail.com; annac.rupf@gmail.com; paulo.silveiras@edu.ufes.br; saulo.j.lima@edu.ufes.br; djeison.batista@ufes.br

<sup>2</sup>Center For Higher Studies of Itacoatiara, University of the State of Amazonas, Itacoatiara, Brazil. E: vfbrocco@uea.edu.br

**Keywords:** caxinguba, CIELAB, colorimetry, jatobá, Munsell Color Chart, open system, thermal modification

#### ABSTRACT

Thermal modification is a process that aims to improve some properties of wood, such as greater biological resistance, greater dimensional stability, and color change, enhancing the value of the product, in addition to being an environmental-friendly method that does not use toxic chemicals. This process is still poorly understood in Brazilian Amazon species, especially when it refers to the effect of combining different temperatures and times (Ronsoni, 2015). Therefore, there is a need to carry out research that evaluates the colorimetric changes of Amazonian species subjected to the effect of thermal modification. This work aimed to evaluate the effect of thermal modification on the colorimetric properties of *Hymenaea* spp. and *Ficus* sp. The wood sampled from sawmills in the municipality of Itacoatiara, Amazonas. The schedules were carried out in an oven with forced air circulation, applying a temperature of 180 °C for 150 minutes. The mass loss was calculated by the variation between the initial (before thermal modification) and final (after thermal modification) oven-dried mass. The wood color was analyzed using the Munsell Color Soil Charts (MUNSELL COLOR, 2000), where we obtained the values of hue (tonality), brightness, and chroma (saturation). These data were converted to CIEL\*a\*b\* coordinates following the conversion table proposed by Vodyanitskii and Kirillova (2016). The color differences for the L\*, a\*, and b\* coordinates were calculated, in addition to the total color variation ( $\Delta E$ ; Equation 1).

$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2} \quad (1)$$

Where:  $\Delta E$ , total color variation;  $\Delta L^2$ , variation in luminosity;  $\Delta a^2$ , variation of parameter a\* (colorimetric parameter of the red-green color axis); and  $\Delta b^2$ , variation of the parameter b\* (colorimetric parameter of the yellow-blue color axis).

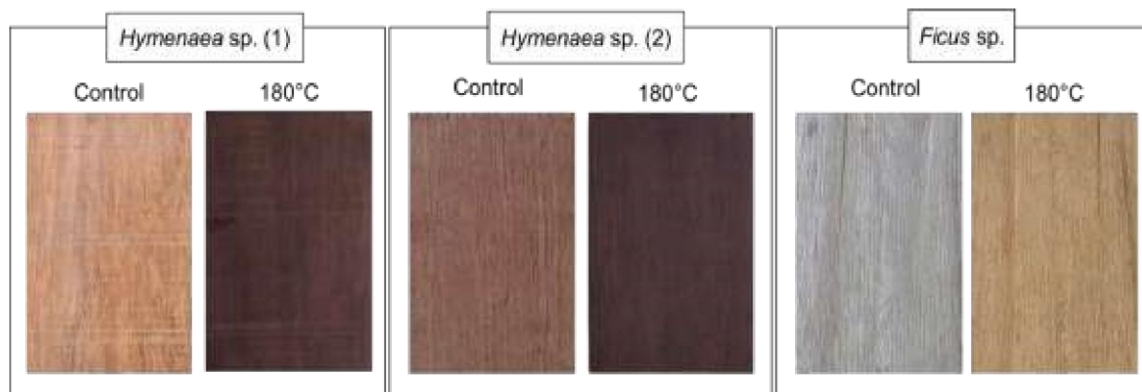
Mass loss was low for *Hymenaea* sp. 1 (0.77) *Hymenaea* sp. 2 (0.72) and *Ficus* sp. (0.23), at 180 °C the main chemical constituents degraded were hemicelluloses, as well as the volatilization of some extractives (Czajkowski *et al.* 2020). There was a gradual reduction in L\* values in all species, indicating the darkening of the wood as a result of thermal modification (Figure 1). For *Ficus* sp., there was not much reduction in the values, however, there was a slight reduction in luminosity (L\*) when compared to the control. In species of *Hymenaea* spp., the a\* and b\* coordinates, the patterns were similar to the L\* coordinate, therefore, there was a decrease in values. The opposite occurred for *Ficus* sp.,



where the values for these coordinates increased (Table 1). Thermal modification at 180 °C significantly changed the wood color of all species.

**Table 1: Conversion of Munsell Color Chart values to colorimetric coordinates of the CIE-L\*a\*b\* system.**

Species	Treatment	L*	$\Delta L^*$	a*	$\Delta a^*$	b*	$\Delta b^*$	$\Delta E^*$
<i>Hymenaea</i> sp. 1	Control	61,7	-----	12,5	-----	34,4	-----	-----
	180 °C	30,8	-30,9	6,9	-5,6	10,1	-24,3	39,71
<i>Hymenaea</i> sp. 2	Control	51,6	-----	11,2	-----	20,1	-----	-----
	180 °C	30,8	-20,8	4,4	-6,8	4,3	-15,8	26,99
<i>Ficus</i> sp.	Control	81,4	-----	-0,4	-----	15	-----	-----
	180 °C	71,6	-9,8	1,2	1,6	27,8	12,8	16,20



**Figure 1: Color changes in thermally modified wood.**

## REFERENCES

Czajkowski Łukasz; Olek, Wiesław.; Weres, Jerzy. (2020). Effects of heat treatment on thermal properties of European beech wood. *European Journal of Wood and Wood Products*, v. 78, p.301312.

MUNSELL COLOR. (2000). Munsell Soil color charts. Baltimore: Koelmorgen.

Ronsoni, Taisa. (2015). Influência do tratamento térmico nas propriedades tecnológicas da madeira de três espécies amazônicas. Trabalho de Conclusão de Curso (Graduação em Engenharia Florestal) – Universidade Federal de Mato Grosso, Instituto de Ciências Agrárias e Ambientais, Sinop.

Vodyanitskii, Yu N.; Kirillova, N. P. (2016). Conversão de coordenadas de cores Munsell para o sistema Cie-L\* a\* b\*: Tabelas e exemplos de cálculo. *Boletim de Ciência do Solo da Universidade de Moscou*, v. 71, n. 4, pág. 139-146.

## Poster 3.14 - The Main Challenges in Bonding Heat-Treated Wood

Milan Šernek<sup>1</sup>

<sup>1</sup>University of Ljubljana, Biotechnical Faculty, Department of Wood Science and Technology, Jamnikarjeva 101, 1000 Ljubljana, Slovenia. E: milan.sernek@bf.uni-lj.si

**Keywords:** adhesive, bonding, heat-treated wood, shear strength, wettability

### ABSTRACT

Heat treatment of wood is often used to improve the dimensional stability of wood and increase its resistance to decay. However, it also has some unfavourable effects, such as reduced strength and toughness. In addition, the chemical, physical and structural changes of wood after heat treatment can affect the bonding process with various adhesives. Decreases in the pH of the heat-treated wood surface can delay (PF) or accelerate (UF, MF and MUF) the cure of adhesives. Heat-treated wood is less hygroscopic and more hydrophobic. This can reduce the wettability of the wood surface with water-based adhesives (PVAc, UF, MF, MUF, PF) and alter the penetration of the adhesives into the porous wood structure. In addition, the decreased equilibrium moisture content of heat-treated wood can affect the cure of adhesives that require water (hydroxyl groups) for the curing reaction (PUR), resulting in slower or incomplete cure. Heat-treated wood usually absorbs water at a slower rate, so the hardening of adhesives that cure by water removal (PVAc) takes longer times.

Even if the adhesive system cures sufficiently, the shear strength of the bonded joint is usually reduced due to the lower strength of the heat-treated wood. This contribution reviews the results obtained over the last two decades related to the bonding of heat-treated wood with the most common wood adhesives.

### REFERENCES

- Šernek, M., Kamke, F.A., Glasser, W.G. (2004). Comparative analysis of inactivated wood surfaces. *Holzforschung*. 58(1), 22-31.
- Šernek, M., Boonstra, M., Pizzi, A., Despres, A., Gerardin, P. (2008). Bonding performance of heat treated wood with structural adhesives. *European Journal of Wood and Wood products*. 66(3), 173-180.
- Kariž, M., Kitek Kuzman, M., Šernek, M. (2013). The effect of the heat treatment of spruce wood on the curing of melamine-urea-formaldehyde and polyurethane adhesives. *Journal of Adhesion Science and Technology*. 27(17), 1911-1920.

## Poster 3.15 - Surface Properties of Thermally Modified Beech Wood After Radio-Frequency Discharge Plasma Treatment

Ján Sedliačik<sup>1</sup>, Pavlo Bekhta<sup>1</sup>, Igor Novák<sup>2</sup>, Angela Kleinová<sup>2</sup>, Ján Matyašovský<sup>3</sup> and Peter Jurkovič<sup>3</sup>

<sup>1</sup>Technical University in Zvolen, T.G. Masaryka 24, 960 01 Zvolen, Slovakia E: sedliacik@tuzvo.sk; bekhta@nltu.edu.ua

<sup>2</sup>Slovak Academy of Sciences, Polymer Institute, Dúbravská cesta 9, 845 41 Bratislava, Slovakia. E: igor.novak@savba.sk; angela.kleinova@savba.sk

<sup>3</sup>VIPO, a.s., Gen. Svobodu1069/4, 958 01 Partizánske, Slovakia, E: jmatyasovsky@vipo.sk; pjurkovic@vipo.sk

**Keywords:** beech wood, contact angle, FTIR, thermal modification, plasma treatment

### ABSTRACT

Heat treatment is widely used to improve the properties of wood, in particular its colour. However, this treatment causes changes in the surface properties of wood, the surface becomes hydrophobic, which can cause serious problems when gluing or coating. In this study, the radio-frequency discharge (RFD) plasma was used to increase the hydrophilicity of the steam-modified wood due to the formation of various polar groups (e.g., hydroxyl, carbonyl, carboxyl, etc.). The increased surface polarity improves the wettability and hydrophilicity due to oxidation reactions. Beech wood (*Fagus sylvatica* L.) was thermally modified with saturated water steam at a temperature of  $135 \pm 2$  °C during a period of 9 hours. The RFD plasma treatment of native and thermally modified wood was performed in a laboratory plasma reactor working at a reduced pressure of 100 Pa, consisting of two 240 mm brass parallel circular electrodes with a symmetrical arrangement, 10 mm thick, between which RFD plasma was created. The wood samples were treated at a power of 400 W during 60 s.

The FTIR spectrum of wood is basically a mixed spectrum of cellulose and lignin with characteristic peaks of both OH bonds and in the fingerprint wave numbers, which are particularly low for C–O–C, CH<sub>2</sub> and COO bonds, typical for polysaccharides. Spectra of beech wood samples were recorded by the ATR (Attenuated Total Reflection) technique in the mid-infrared region (4000-650 cm<sup>-1</sup>) using a germanium crystal on a NICOLET 8700™ instrument. From each sample, spectra were measured from five different points; the resolution was set to 4 cm<sup>-1</sup> and the number of scans ranged from 64 to 200. The biggest differences in FTIR spectra were observed in the case of steamed wood before and after plasma treatment, as well as in the case of the spectra of native beech wood and steamed wood both treated with plasma. A visual comparison of the FTIR spectra of beech wood samples (native wood, plasma-treated native wood, steamed wood, and plasmatreated steamed wood) found that there are few differences between the individual spectra in the whole infrared range (Figures 1 and 2).

The wettability of native and steam-modified beech wood before/after plasma treatment was evaluated by the determination of contact angle (CA,  $\theta$ ) with a water-free 99.5% glycerol as the testing liquid. The 5  $\mu$ l drops of testing liquid were placed on the wood surface with a micropipette. Glycerol CA measurements were taken using a professional Surface Energy Evaluation System device coupled to a web camera and the required PC software. The measurements of CA were repeated 8 times, and the arithmetic mean and standard deviation of the measurements were analysed (Table 1).

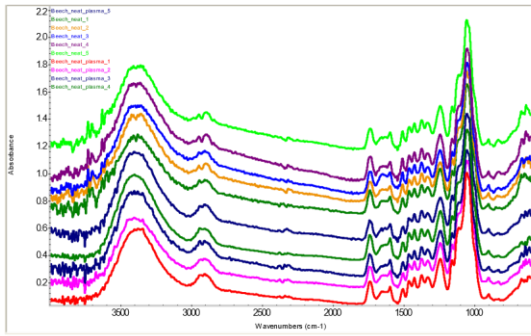


Figure 1: Spectra of beech\_native vs beech\_native\_plasma in the whole IR range.

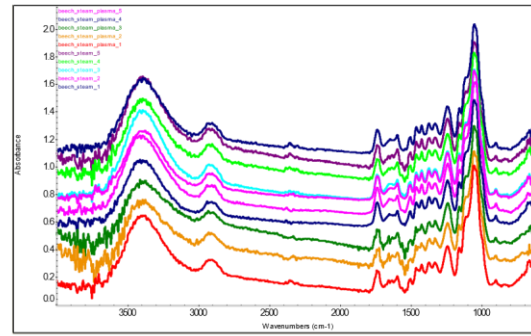


Figure 2: Spectra of beech\_steamed vs beech\_steamed\_plasma in the whole IR range.

The CAs of glycerol on steam-modified beech wood are higher in comparison with native wood. With the time duration after plasma treatment, the CA values decreased in all cases. After plasma treatment, the CA values were lower than those of non-treated wood.

Table 1: Glycerol contact angles of native and water steam-modified beech wood treated subsequently by radio-frequency discharge plasma.

Sample	CA [°] 0 s	CA [°] after 60 s	CA [°] after 120 s
Beech wood – native	61.5 (2.8)	51.5 (2.2)	26.2 (1.8)
Beech wood – native + plasma	46.6 (2.2)	36.1 (2.2)	18.8 (1.6)
Beech wood – steam-modified	65.5 (2.6)	54.8 (2.2)	32.2 (2.0)
Beech wood – steam-modified + plasma	48.2 (2.0)	38.8 (2.0)	26.2 (2.5)

*Standard deviations are in parentheses*

A test for determining the tensile strength of lap joints was carried out for native and steam-modified beech wood before/after plasma treatment (Table 2). Adhesive strength was determined according to EN 204 durability class D4 under conditioning sequences: dry (7 days in standard atmosphere); boiled (7 days in standard atmosphere, 6 hours in boiling and 2 hours in cold water), and soaked (7 days in standard atmosphere, 4 days in cold water). It was found that the strength of samples prepared from native and steammodified beech wood is equal. Plasma-treated samples have a higher tensile strength when compared to samples without plasma treatment, approximately up to 10%.

Table 2: Tensile strength properties of lap joints after D4 according to EN 205.

Sample	dry [MPa]	boiled [MPa]	soaked [MPa]
Beech wood – native	15.5 (2.21)	3.1 (0.63)	3.6 (0.53)
Beech wood – native + plasma	15.8 (2.64)	3.6 (0.86)	3.8 (0.85)
Beech wood – steam-modified	15.7 (2.76)	2.9 (0.64)	3.7 (0.59)
Beech wood – steam-modified + plasma	15.7 (2.24)	3.4 (0.66)	4.0 (0.65)

*Standard deviations are in parentheses*

The radio-frequency discharge plasma treatment proved its efficiency, plasma-treated samples reached significantly lower contact angles in comparison with non-treated samples. Better hydrophilicity was confirmed by the higher tensile strength properties of lap joints after different conditioning sequences.

## ACKNOWLEDGEMENTS

This work was supported by the Slovak Research and Development Agency under the contracts APVV-20-0159 and APVV-21-0051; also by the EU through the Recovery and

Resilience Plan for Slovakia under project No. 09I03-03-V01-00124; and also by the project VEGA 1/0264/22.

### Poster 3.16 - Effect of Paraffin-Thermal Modification on Water Absorption and Dimensional Stability of Louro-Preto Wood (*Nectandra dioica*)

Saulo José da Costa Lima<sup>1</sup>, Anna Clara Oliveira Rupf<sup>1</sup>, Kamilly da Silva Pereira<sup>1</sup>, Paulo Henrique dos Santos Silves<sup>1</sup>, Djeison Cesar Batista<sup>1</sup>, Fernando Wallase Carvalho Andrade<sup>2</sup>

<sup>1</sup>Department of Forest and Wood Sciences, Federal University of Espírito Santo, Jerônimo Monteiro, Brazil. E: saulo.j.lima@edu.ufes.br; annac.rupf@gmail.com; kamillysilv4p@gmail.com; paulo.silves@edu.ufes.br; djeison.batista@ufes.br

<sup>2</sup>Institute of Biodiversity and Forests, Federal University of Western Pará, Santarém, Brazil. E: fernando.andrade@ufopa.edu.br

**Keywords:** hygroscopicity, swelling, thermally modified wood, tropical wood, waterwood relations.

#### ABSTRACT

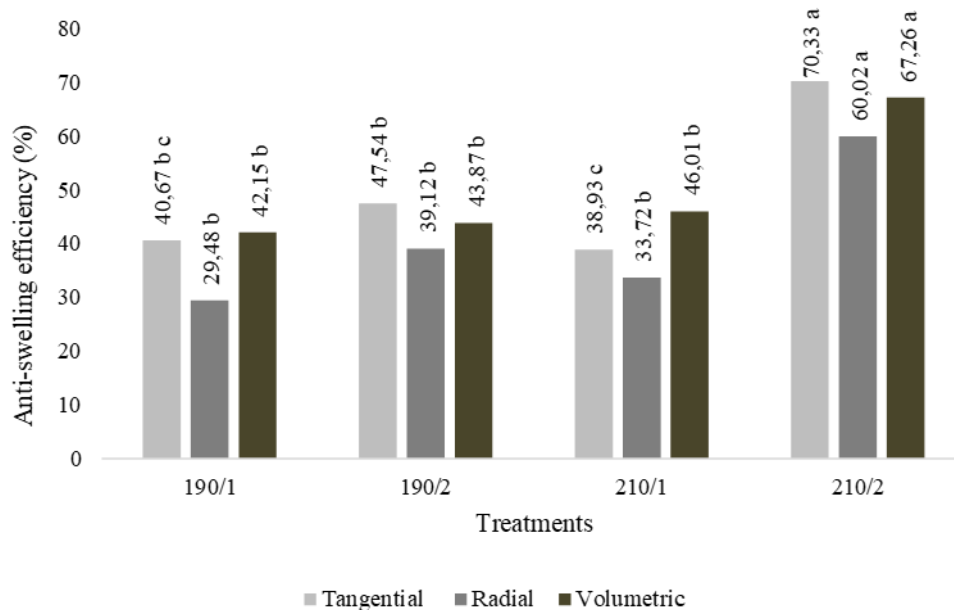
The diffusion of thermal modification as a process that aims to improve undesirable characteristics of wood with low commercial value is important. However, better results can be obtained by combining processes and adding hydrophobic agents (Reinprecht & Repák 2019). The objective of this study was to evaluate the effect of paraffin-thermal modification on the hygroscopicity and dimensional stability of *Nectandra dioica* wood. The wood was thermally modified in paraffin at two temperatures (190 °C and 210 °C), for 150 minutes (Table 1).

**Table 1: Treatments applied to *Nectandra dioica* wood.**

Treatment	Maximum temperature (°C)	Modification time [minutes]	Paraffin
190/1	190	150	No
190/2	190	150	Yes
210/1	210	150	No
210/2	210	150	Yes
Untreated	-	-	-

The entire process is divided into five stages (Reinprecht & Repák 2019): i) the wood and granulated paraffin were arranged in a stainless steel tray and exposed to a temperature of 100 °C for 60 minutes, causing the paraffin to melt; ii) the wood was kept immersed in liquid paraffin for 60 minutes at 100 °C; iii) the temperature was raised until the target temperature (190 °C or 210 °C); iv) the target temperature was kept for 150 minutes; v) the wood was removed from the paraffin solution and placed on a steel screen for 60 minutes at 100 °C, allowing the unabsorbed paraffin to drain. After this process, the oven was turned off and cooled naturally, allowing the paraffin to solidify. Later, the samples were subjected to three immersion-drying cycles to evaluate the water absorption rate and the anti-swelling efficiency. It consisted in immersion in distilled water for 24 hours and, subsequently, dried at 40 °C for 24 hours to prevent cracking, followed by drying at 100 °C for another 24 hours. Dimensional stability increased proportionally to the intensity of the treatment, increasing with the addition of the paraffin solution (Figure 1). The opposite behavior was observed for water absorption by wood, recording rates of up to 0.77% in the most severe treatment

(Table 2). In general, the best results were at 210 °C with paraffin immersion. Paraffin-thermal modification is recommended, as it will result in some advantages in terms of dimensional stability and water repellency, making it an option for wood in various applications.



**Figure 1: Linear and volumetric anti-swelling efficiency averages.**

**Table 2: Means of water absorption rate (WA) and anisotropy coefficient ( $\beta_t/\beta_r$ ).**

Treatments	WA [%]	$\beta_t/\beta_r$
190/1	2,24 c (0,21)	1,95 bc (0,21)
190/2	0,96 a (0,13)	1,81 ab (0,36)
210/1	1,46 b (0,37)	1,78 ab (0,19)
210/2	0,77 a (0,26)	1,57 a (0,50)
Ctrl	2,49 c (0,23)	2,11 c (0,24)

Means followed in a column by the same lowercase letter do not differ statistically from each other (Tukey test,  $p > 0.05$ ); Values in parentheses correspond to the standard deviation.

## REFERENCES

Reinprecht, L., Repák, M. (2019). The impact of paraffin-thermal modification of beech wood on its biological, physical and mechanical properties. *Forests*, v. 10, n. 12, p. 1–15



## Poster 3.17 - Study of the Machinability of Thermally and Chemically Modified Wood for Art Objects

Leila Rostom<sup>1</sup>, Jérémie Damay<sup>1</sup>, Philippe Gérardin<sup>1</sup>, Michael Jousserand<sup>2</sup>

<sup>1</sup>LERMAB, Université de Lorraine, Faculté des Sciences et Technologies, BP 70 239, F-54506 Vandœuvre-lès-Nancy, France. E: leila.rostom@univ-lorraine.fr E: philippe.gerardin@univlorraine.fr E: jeremie.damay@cirad.fr

<sup>2</sup>Buffet Crampon, Research and Development, 5 rue Maurice Berteaux, 78711 Mantes-la-Ville, France. E: michael.jousserand@buffetcrampon.com

**Keywords:** furfurylation, thermal treatment, wood modification, wood machining

### ABSTRACT

It is now widely known that wood is a hygroscopic material that interacts and evolves with the changes in the environmental conditions (humidity, temperature, UV light). Therefore, wood is subject to dimensional changes and deformations due to humidity, which can make it unsuitable for some applications that require high dimensional stability such as for flooring, decking, railroad ties, music instruments, and handles for tools. To meet the needs for more stable and durable wood materials, the research on wood, since the 20<sup>th</sup> century, enabled to develop new preservation methods through wood preservatives (pesticides) or wood modifications (thermal or chemical modification) in order to use the modified wood material in challenging environments (Zelinka *et al.* 2022).

As environmental concerns grow, the use of wood as a renewable resource is greatly promoted, and the need to avoid environmental pollution due to certain chemicals that are present in wood preservatives, leads us to develop more environmental friendly processes to enhance the properties of wood. Different treatments can be considered (Gérardin 2016, Sandberg *et al.* 2017, Zelinka *et al.* 2022) and a very interesting way to enhance wood properties is by thermal modification and chemical modification using esterification with polyglycerol solutions or furfurylation with furfuryl alcohol solutions for instance (Sejati *et al.* 2017, Martha *et al.* 2021, Mubarak *et al.* 2019). To continue the research efforts in this subject, we propose to explore the thermal modification as well as the chemical densification of wood by furfurylation of European wood species and to study the machining of these modified woods in order to assess their behaviour and performances.

Three wood species were selected for this study (Alder, Maple and Hornbeam) and impregnated under vacuum-pressure with a furfuryl alcohol (FA) solution containing tartaric acid (5%) and water (10%). Another group of samples was subjected to a thermal treatment at three different temperatures (160, 180 and 200 °C) before the impregnation with the FA solution. Several properties of the modified wood materials were characterized, such as solution uptake, weight percent gain, density, equilibrium moisture content, swelling, dimensional stability and surface hardness.

Results show an overall better improvement of the dimensional stability, with a lower moisture content, a higher density and Brinell hardness for both treatment protocols (with or without pre-heat treatment). However, Brinell hardness and anti-swelling efficiency are greater for the samples subjected to a pre-heat treatment before FA impregnation. Therefore, one could suppose that the machining performances of the former modification should be better than for the samples subjected to the FA impregnation only. However, results obtained during the machining of these modified wood samples show that the samples with pre-heat treatment are not resistant enough and tend to crack more than the FA only impregnated

wood samples. These observations lead to suppose that even though the hardness and density of modified wood is greatly increased with pre-heat treatment, it does not make it suitable for machining. This could be due to the decrease of the mechanical properties and more specifically the elasticity (Modulus of Elasticity) and bending strength (Modulus of Rupture) of the modified wood, which is known to be greatly decreased due to thermal treatments (Esteves and Peireira 2009).

Wood modification by furfurylation only appears to be a more suitable option for the machining of these chemically modified European wood materials. In addition, the chemically modified Hornbeam species shows the best machinability.

## REFERENCES

- Esteves, B. M. and Pereira, H. M. (2009). Wood modification by heat treatment: A review. *BioResources*, 4(1), 370-404.
- Gérardin, P. (2016). New alternatives for wood preservation based on thermal and chemical modification of wood- a review. *Annals of Forest Science*, 73 (3), pp.559-570.
- Martha R., Mubarak, M., Batubara, I., Rahayu, I.S., Setiono, L., Darmawan, W., Obounou Akong, F., George, B., Gérardin, C., Gérardin, P. (2021). Effect of furfurylation treatment on technological properties of short rotation teak wood. *Journal of Materials Research and Technology* 12:16891699.
- Mubarak, M., Dumarçay, S., Militz, H., Candelier, K., Thévenon, M.F., Gérardin, P. (2019) Comparison of different treatments based on glycerol or polyglycerol additives to improve properties of thermally modified wood. *European Journal of Wood and Wood Products* 77:799810.
- Sandberg, D., Kutnar, A., Mantanis, G. (2017). Wood modification technologies – a review. *iForest* 10: 895-908.
- Sejati, P.S., Imbert, A., Gérardin-Charbonnier, C., Dumarçay, S., Fredon, E., Masson, E., Nandika, D., Priadi, T., Gérardin, P. (2017). Tartaric acid catalyzed furfurylation of beech wood. *Wood Science Technology* 51:379-394.
- Zelinka, S. L., Altgen, M., Emmerich, L., Guigo, N., Keplinger, T., Kymäläinen, M., Thybring, E. E., & Thygesen, L. G. (2022). Review of Wood Modification and Wood Functionalization Technologies. *Forests*, 13(7), [1004].

## Poster 3.18 - Moisture Diffusion Characteristics of Thermally Modified Beech Wood

Aleš Straže<sup>1</sup>, Primož Tomec<sup>1</sup>, Gorišek Željko<sup>1</sup> and Jure Žigon<sup>1</sup>

<sup>1</sup> Wood Science and Technology Department, Ljubljana, University of Ljubljana, Biotechnical Faculty, Jamnikarjeva 101, 1000 Ljubljana, Slovenia. E: ales.straze@bf.uni-lj.si; E: primoz.tomec@gmail.com; E: zeljko.gorisek@bf.uni-lj.si; E: jure.zigon@bf.uni-lj.si

**Keywords:** beech, moisture diffusivity, thermal modification, wood

### ABSTRACT

Thermal modification (HT) is a promising technology for improving some of the physical properties of wood, such as hygroscopicity and dimensional stability, which, together with the chemical and structural changes in the material, also improves its natural durability and allows outdoor use (Turner *et al.* 2010). In outdoor applications, heat-treated wood is constantly exposed to changing climatic conditions, which requires a good knowledge of moisture diffusion in the material.

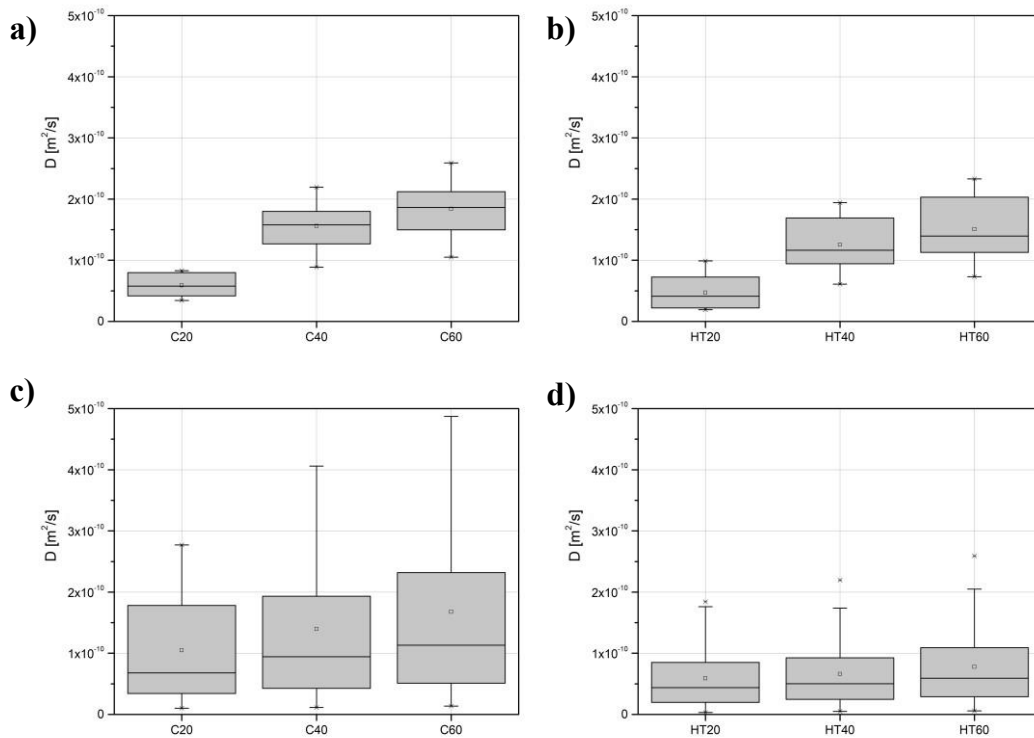
In this study, the behaviour of thermally modified beech wood under fluctuating climatic conditions was investigated. Beech wood was heat treated in an unsaturated steam atmosphere in a standard one-day procedure consisting of four phases: predrying, heating (up to 235 °C), conditioning, and cooling. The samples were oriented specimens without growth anomalies ( $n = 7$ ) with dimensions of 20 mm × 100 mm (width × length) and different thicknesses (5, 10, 15, and 20 mm). Water vapour diffusivity ( $D$ ) was determined at three temperatures (20, 40, and 60 °C) by the non-stationary method, determining the 2<sup>nd</sup> Fick's law and Nevman's equation (Choong and Skaar, 1972), and the equilibrium rate of the specimens from a dry (RH = 33%) to a humid climate (RH = 87%) was studied (Fig. 1a). In addition, the evolution of the moisture gradient in the specimens with dimensions 20 mm × 20 mm × 350 mm was studied, and the diffusion coefficient was determined using the finite difference method (Fig 1b).



**Figure 1: Determination of water vapour diffusivity of thermally modified beech wood by the nonstationary experiment (a) and finite difference method (b).**

The equilibrium moisture content (EMC) of thermally modified beech wood (HT) was lower than that of natural wood (C) in all climates studied. Both methods confirmed a lower diffusivity for water vapour in thermally modified beech wood at all temperatures studied (Fig. 2). Using the non-stationary method, we determined an average water vapour diffusion ratio over the temperature range (20-60 °C) of thermally modified and natural wood of about 0.6 ( $D_{HT20}/D_{C20} = 0.4$ ;  $D_{HT40}/D_{C40} = 0.8$ ;  $D_{HT60}/D_{C60} = 0.6$ ). Using the finite difference method, we measured lower water vapour diffusivity over the temperature range studied, which also confirms the effect of heat treatment on reducing moisture diffusion in wood.

However, the average ratio of water vapour diffusivity is in perfect agreement and is about 0.6 ( $D_{HT20}/D_{C20} = 0.3$ ;  $D_{HT40}/D_{C40} = 0.8$ ;  $D_{HT60}/D_{C60} = 0.7$ ).



**Figure 2: Water vapour diffusivity of thermally modified beech wood by the non-stationary experiment (control (a), thermally modified (b)) and finite difference method (control (c), thermally modified (d)).**

The lower moisture diffusivity of thermally modified beech wood is influenced by chemical and structural changes (Olek *et al.*, 2016), although the lower density of thermally modified wood does not appear to play a significant role. Additional importance is given to the lower hygroscopicity of thermally modified wood, which is due to the partial degradation of hemicelluloses, which increases the possibilities of using this wood under fluctuating climate.

## REFERENCES

- Choong, E.T., and Skaar C. (1972). Diffusivity and surface emissivity in wood drying. *Wood Fibre*, **4**, 80-86
- Olek, W., Rémond, R., Weres, J., Perré, P. (2016). Non-Fickian moisture diffusion in thermally modified beech wood analyzed by the inverse method. *International Journal of Thermal Sciences*, **109**, 291-298.
- Turner, I., Rousset, P., Rémond, R., Perré, P. (2010). An experimental and theoretical investigation of the thermal treatment of wood (*Fagus sylvatica* L.) in the range 200–260°C. *International Journal of Heat and Mass Transfer*, **53**(4), 715-725.

## Poster 3.19 - Exploring the Mechano-Sorptive Behavior of Thermally Modified Wood

Claude Feldman Pambou Nziengui<sup>1</sup>, Goli Giacomo<sup>2</sup> and Rostand Moutou Pitti<sup>3</sup>

<sup>1</sup>URMM, Ecole Polytechnique de Masuku (EPM), Université des Sciences et Techniques de Masuku (USTM), BP 941, Franceville, Gabon. E: pclaudefeldman@gmail.com

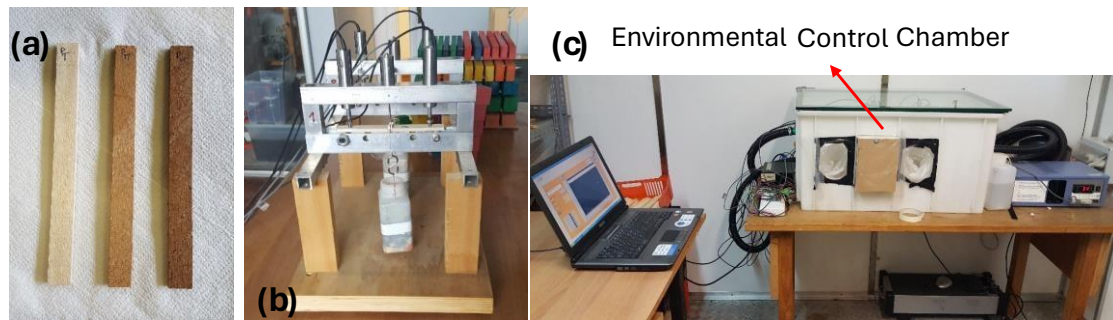
<sup>2</sup>DAGRI – Dipartimento di Scienze e Tecnologia Agrarie, Alimentari, Ambientali e Forestali, Università degli di Firenze, Italy. E: giacomo.goli@unifi.it

<sup>3</sup>Institut Pascal, Clermont Auvergne INP, CNRSS, Université Clermont Auvergne, BP 63000 Clermont Ferrand, France. E: rostand.moutou\_pitti@uca.fr

**Keywords:** creep behavior, impact of relative humidity, mecano-sorptive test, poplar wood

### ABSTRACT

Mechano-Sorptive Testing in a Climate-Controlled Chamber involved conducting 3-point bending tests on both unmodified and thermally modified wood samples, as depicted in Figure 1. To ensure a straight grain, the specimens were obtained through a split-cutting method and sized at 15×5×160 mm<sup>3</sup> (width×height×length). This specific geometric configuration, with a reduced thickness, was chosen to facilitate the rapid diffusion of moisture within the sample. Subsequently, the samples were loaded at 10% of their respective breaking loads, following the approach of Saifouni *et al.* (2016). The elastic modulus and bending strength were determined using a separate set of samples sourced from the same board.



**Figure 1:** Experimental devices used: (a) typical samples of thermally modified poplar wood tested; (b) typical 3-points bending test used for mechano-sorptive test; (c) box of environmental control chamber.

The tests were conducted within a controlled climate chamber capable of adjusting relative humidity within the range of 35% to 80%. In our study, three humidity levels were specifically targeted (45%, 55%, and 75%). Initially, the humidity was set at 55%, and the sample was subjected to loading under these conditions. After 19 hours of creep, the relative humidity was then elevated to 75%, which was maintained for 5 hours before returning to 55%. Subsequently, another 19-hour period lapsed, following which a second humidity cycle was initiated, reducing the environment's humidity to 45% over 5 hours. Finally, the chamber's humidity was restored to 55%. The load was removed after 19 hours following the conclusion of the second cycle, marking the commencement of a new test.

Fig. 2 shows typical curves of mechano-sorptive tests obtained for the unmodified (PT10) and thermally modified wood (PM8). The curve shows that for PT10 its initial relative creep ( $u_0^{PT10}$ ) is more important compared to this obtained on PM8 sample ( $u_0^{PM8}$ ). This result shows that thermally modified wood exhibits greater mechanical characteristics than the non-modified ones. A reduction of elastic deformation of about 30% is obtained because of thermal modification. A lower tendency to viscous deformation of thermally modified sample can also be observed (3.1% for PM8 and 4% for PT10 of the total deformation).

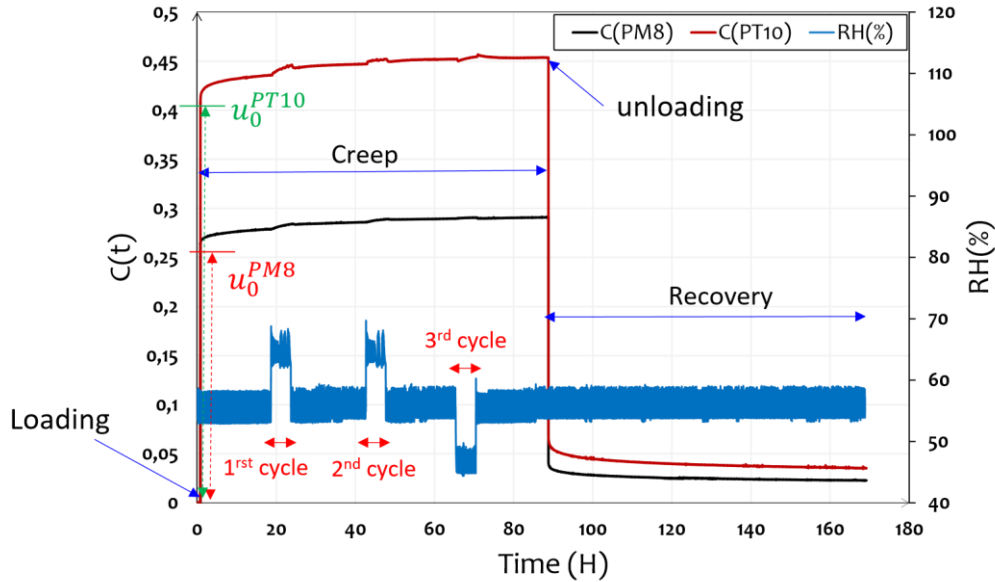


Figure 1: Behavior of thermally modified poplar wood (PM8) and unmodified poplar wood (PT10) under mechano-sorptive tests

## REFERENCES

Saifouni, O., Destrebecq, J.F., Froidevaux, J. and Navi, P., (2016). Experimental study of the mechanosorptive behaviour of softwood in relaxation. *Wood science and technology*, 50, pp.789-805.

## ACKNOWLEDGMENTS

The authors would like to acknowledge the GESAAF (University of Florence) for the provision of the material used for the tests carried out and the COST Action FP1407 for the financial support during the STSM.

## Poster 3.20 - Temperature and Moisture Content Effects on Wood Compressive Properties

Hussein Daher<sup>1,2</sup>, Sabine Caré<sup>2</sup>, Gilles Forêt<sup>2</sup> and Loïc Payet<sup>1</sup>

<sup>1</sup>CSTB, 84 Avenue Jean Jaurès, 77420 Champs-sur-Marne, France. E: hussein.daher@cstb.fr; loic.payet@cstb.fr

<sup>2</sup>Navier Laboratory, Gustave Eiffel University, ENPC, CNRS (UMR 8205), F-77455 Marne-la-Valée, France. E: hussein.daher@enpc.fr ; sabine.care@univ-eiffel.fr ; gilles.foret@enpc.fr

**Keywords:** drying temperatures, humidity, mechanical properties, timber design, wood

### ABSTRACT

Wood material can absorb or release water depending on the humidity of the surrounding air. This can affect its mechanical properties, especially its stiffness or its strength at room temperature (Gerhards 1982, Guitard 1987, Li 2023, Wood Handbook 1987). In the case of timber design to contribute to climate change, few pieces of information are given to the effect of temperature, for example when structures are subjected to fire. The current challenge is to better understand the residual properties in the sections of non-carbonated wood structural elements that can be subjected to temperatures up to 150 °C. Wood properties may be modified and residual mechanical properties have to be evaluated to ensure the security of the occupants.

Building standards suggest that the compressive strength of wood, at 100 °C is 25% lower than the one at 20 °C (Eurocode 5, 2005). But, they do not specifically mention how wood moisture content plays a role. Experiments carried out by CSTB in 2020 (Manthey 2020) showed that within the temperature range of 20-100 °C, moisture content had an important impact on strength. At 100 °C, when wood is completely dry, the compressive strength remains the same as at 20 °C. If wood material has around 10% moisture content, then its compressive strength drops to 60% of its initial strength.

This paper aims to better investigate how temperature and moisture content affect the mechanical properties of wood.

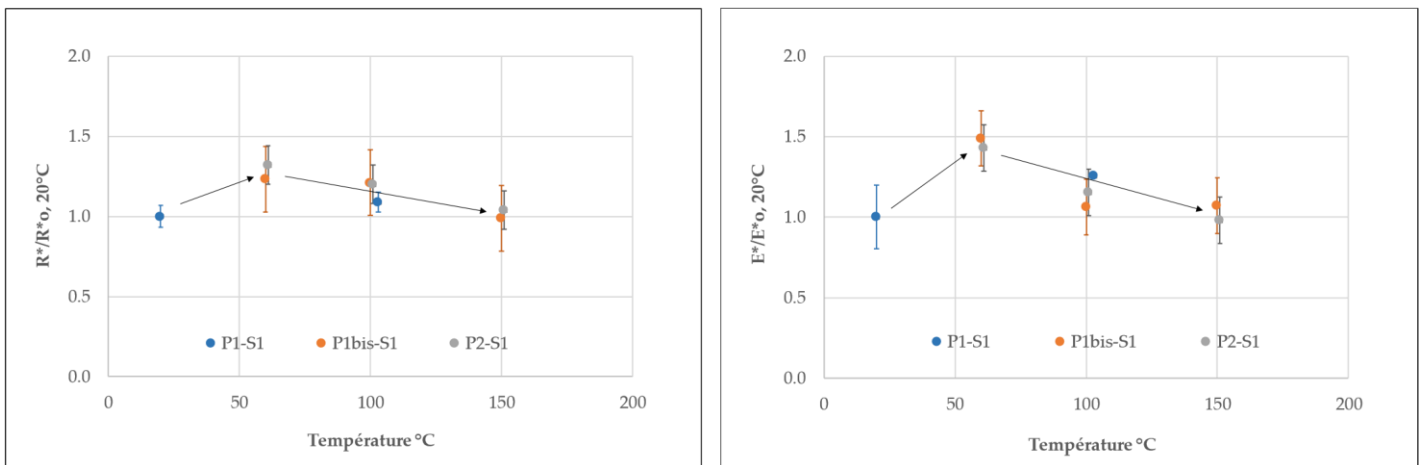
The experiments involved using spruce wood to examine how variability of solid wood and glued laminated timber (GL24H) respond under varying levels of moisture and temperature. So, several heat protocols are considered (Table 1) to evaluate the effect of temperature and moisture content on timber design: P<sub>1</sub> at 20 °C with various moisture content; P1bis and P2 at three temperatures (60, 100 and 150 °C) with a different heating duration in an oven. Moreover, all results are discussed taking into account the size of samples (S<sub>0</sub>, S<sub>1</sub>, S<sub>2</sub> and S<sub>3</sub>).



**Table 1: Dimensions of samples (from solid wood and Glued Laminated Timber) and number of tests at different temperatures according to three protocols**

Section	Dimensions [mm <sup>3</sup> ]	Wood	P <sub>1</sub>				
			20 °C	Dry	60 °C	100 °C	150 °C
Section S0	35x35x80	Solid Wood	5 X 5	1	-	-	-
Section S1	50x50x100	GL24H	5	2	5 X 2	5 X 2	5 X 2
Section S2	97x92x200	GL24H	5	4	5 X 2	5 X 2	5 X 2
Section S3	187x198x400	GL24H	5	4	5 X 2	5 X 2	5 X 2

Figure 1 provides a representation of how the mechanical properties of the glued laminated timber, such as strength and longitudinal modulus change with temperature (ranging from 20 °C to 150 °C). It was observed that changes in temperature and moisture content have contrasting effects, on strength and stiffness of the wood. All results are discussed taking into account the chemical changes and induced cracking with temperature and moisture content. In particular, chemical changes on the surface of samples in relation to their color after heating and possibly inner chemical modifications will be considered.



**Figure 1: Compressive strength and Young modulus parallel to the grain for samples subjected to different temperatures (20-150 °C) and under various moisture conditions (for three protocols and for the section S1, see text). Mechanical properties are normalized to sample density (R\* and E\*) and the mechanicals results at 20 °C (before heating treatments at 60, 100 and 150 °C)**

## REFERENCES

- Eurocode 5, (2005). Conception et calcul des structures en bois. *NF EN 1995-1-1,2*.
- Gerhards, C.C. (1982). Effect of moisture content and temperature on the mechanical properties of wood, *Wood Fiber (1):4-36*.
- Guitard, D., (1987). Mécanique du matériau bois et composites. *Cépaduès*.

Li, W., Liu, C., Wang, X., Shi, J., Mei, C., Van den Bulcke, J. and Van Acker, J. (2023). Understanding the impact of wood type and moisture content on the bonding strength of glued wood. *Wood material Science & Engineering*. 18:1, 303-313.

Manthey M., (2020). Test report n° EEM/EA2R 20 26087828. *CSTB, Etude de la résistance en compression en fonction de la température du matériau bois – épicéa*, France

Wood Handbook, (1987). Wood as an engineering material, *The Laboratory N°72, Department of Agriculture*, United States.

## Poster 3.21 - Moisture Content Distribution of Densified Wood and the Impact of Various Heat Post-Treatments on Brinell Hardness and Set Recovery

Elena Jäger<sup>1</sup>, Guillaume André<sup>1</sup> and Thomas Volkmer<sup>1</sup>

<sup>1</sup>Institute for Materials and Wood Technology, Architecture, Wood and Civil Engineering, Bern University of Applied Sciences, Switzerland. E: elena.jaeger@bfh.ch; guillaume.andre@bfh.ch; thomas.volkmer@bfh.ch

**Keywords:** densification, hardness, heat post-treatment, set recovery

### ABSTRACT

The on going climate change affects many wood species historically used by the industry. Thus, local resilient wood can be used as an alternative, due to sustainable modification to improve the mechanical properties and mitigate raw material unavailability risk. One way to modify wood is through densification. Due to the densification, the mechanical properties of the wood are improved (Niemz and Sonderegger 2003). However, one of the biggest challenge is to ensure the dimensional stability of densified wood. The densified wood has the tendency to recover (so-called set recover) to the original state when is exposed to high humidity and high temperature. Many researchers tried to reduce the set recovery (Inoue *et al.* 1993; Kutnar & Šernek, 2007; Kutnar and Kamke 2012; Sandberg *et al.* 2013; Laine *et al.* 2016; Neyses *et al.* 2020; Pelit and Yorulmaz, 2023), with more or less promising results. This study focuses on the impact of heat posttreatment on the moisture distribution, Brinell hardness and set recovery on densified poplar (*Populus Nigra* L.).

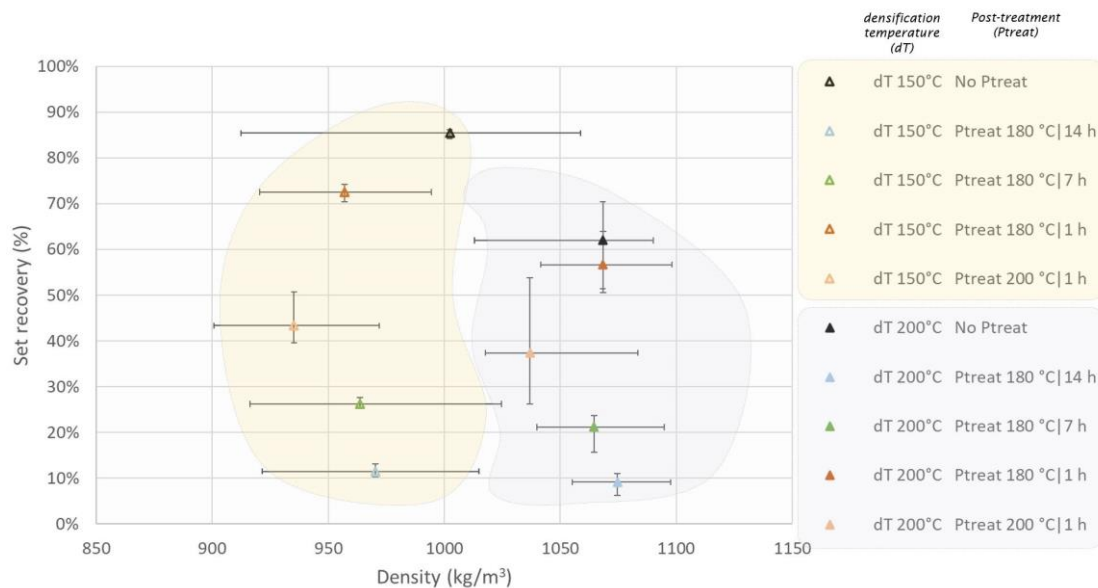
Poplar samples with an average density of 490 kg/m<sup>3</sup> with dimensions 500 mm (L) x 10 mm (R) x 100 mm (T) were compressed with a compression ratio of 60% at 150 °C in a batch press. Samples were stabilized at 20 °C/65% RH prior to the densification. Afterwards, the densified samples were heat post-treated at 180 °C for 7h, 10h, as well as 200 °C for 3h.

The set recovery test was performed according to Inoue *et al.* 1993. Samples with dimensions 10mm (L) x 5 mm (R) x 45 mm (T) intended for the set recovery tests were impregnated with water for 30 min with 8 bars pressure and subsequently left submerged for 210 min at room temperature. Afterwards, specimens were boiled for 30 minutes at 98 °C and finally dried at 103 °C. The set recovery “R” is calculated according to equation (1):

$$R = (TT_{rr} - TT_{cc}) / (TT_0 - TT_{cc}) \times 100\% \quad (1)$$

Where, T<sub>c</sub> is oven-dry thickness after compression [mm], T<sub>0</sub> – oven-dry thickness before compression [mm] and T<sub>r</sub> is oven-dry thickness after the set recovery cycle [mm]. The Brinell hardness test was done according to the standard EN 1534, 2011. Analysis of variance (ANOVA) tests were performed with the significance level set at p < 0.05.

The pre-trial results are presented in Figure 1. The pressing method at 200 °C resulted in a higher density, compared to the pressing method at 150 °C. The reason could be due to the higher temperature which resulted in a higher densification ratio. Independently of the densification method and temperature, the post-treatment reduces the set recovery by the densified samples. The lowest results (< 30%) were reached by the post-treatment at 180 °C for 7h and 14h for both densification methods.



**Figure 1: Dependency between the density, set recovery, pressing method (150 °C and 200 °C) and different post-treatment (\*the bars are minimum and maximum) n= 4**

## REFERENCES

- EN 1534 Wood flooring – Determination of resistance to indentation – Test method, (2011).
- Inoue, M., Norimoto, M., Tanahashi, M., & Rowell, R. M. (1993). Steam or heat fixation of compressed wood. *Wood and Fiber Science*, 25(3), 224–235.
- Kutnar, A., & Kamke, F. A. (2012). Influence of temperature and steam environment on set recovery of compressive deformation of wood. In *Wood Science and Technology* (Vol. 46, Issue 5, pp. 953–964). <https://doi.org/10.1007/s00226-011-0456-5>
- Kutnar, A., & Šernek, M. (2007). *Densification of wood*. 82.
- Laine, K., Segerholm, K., Rautkari, L., Wa, M., & Hughes, M. (2016). *Wood densification and thermal modification: hardness*, 883–894. <https://doi.org/10.1007/s00226-016-0835-z>
- Neyses, B., Karlsson, O., & Sandberg, D. (2020). The effect of ionic liquid and superbase pretreatment on the spring-back, set-recovery and Brinell hardness of surface-densified Scots pine. *Holzforschung*, 74(3), 303–312. <https://doi.org/10.1515/hf-2019-0158>
- Niemz, P., & Sonderegger, W. (2003). Untersuchungen zur Korrelation ausgewählter Holzeigenschaften untereinander und mit der Rohdichte unter Verwendung von 103 Holzarten. *Schweizerische Zeitschrift Für Forstwesen*, 12(154), 489–493. <https://doi.org/10.3188/szf.2018.s0001>
- Pelit, H., & Yorulmaz, R. (2023). Influence of thermal pretreatments on dimensional change and humidity sensitivity of densified spruce and poplar wood. *Maderas-Cienc Tecnol* 26. <https://doi.org/10.4067/S0718-221X2024005XXXXXX>.

## Poster 3.22 - Correlation between Color and Biodeterioration of Short-Rotation Thermally Modified Teak Wood

Anna Clara Oliveira Rupf<sup>1</sup>, Kamilly da Silva Pereira<sup>1</sup>, Saulo José da Costa Lima<sup>1</sup>, Paulo Henrique dos Santos Silveiras<sup>1</sup>, Jessica Sabrina da Silva Ferreira<sup>1</sup>, Jaqueline Rocha de Medeiros<sup>1</sup>, Adriano Ribeiro de Mendonça<sup>1</sup>, Juarez Benigno Paes<sup>1</sup>, Djeison Cesar Batista<sup>1</sup>

<sup>1</sup>Department of Forest and Wood Sciences, Federal University of Espírito Santo, Jerônimo Monteiro, Espírito Santo, Brasil. E: annac.rupf@gmail.com; kamillysilv4p@gmail.com; saulo.j.lima@edu.ufes.br; paulo.silveiras@edu.ufes.br; jessica.s.ferreira@edu.ufes.br; jaquelinerocha256@gmail.com; ribeiroflorestal@yahoo.com.br; juarez.paes@ufes.br; djeison.batista@ufes.br

**Keywords:** CIELAB, colorimetry, *Coniophora puteana*, *Tectona grandis*, thermal modification, *Trametes versicolor*

### ABSTRACT

Short-rotation teak wood (*Tectona grandis* L.f.) is composed of a greater proportion of sapwood, resulting in heterogeneous characteristics for the color and natural durability of the species (Bellon 2013, Menezes 2017). Thermal modification can be an alternative to improve the properties and characteristics of short-rotation teak wood, making it more economically valued. Many studies have been carried out to evaluate the effect of thermal modification on the color and biodeterioration resistance of teak wood (Bellon 2013, Motta *et al.* 2013, Lopes 2012, and Gasparik *et al.* 2019). However, biodeterioration tests are long and expensive, while measurement of colorimetric parameters are fast and cheaper. The main objective of this work was to verify how colorimetric parameters correlate with mass loss, as an exploratory analysis for the future development of models for predicting wood durability based on colorimetry. Untreated (Control) and thermally modified wood were tested in closed system at 160 °C (CS160) and in open system at 185 °C and 210 °C (OS185 and OS210, respectively). The wood used came from the first thinning, carried out when the plantation was around six years old, acquired from a producer in Cáceres, Mato Grosso, Brazil. The biodeterioration test was carried out according to the standard AWPA E-30-16 (2016), with the fungi *Trametes versicolor* (white rot) and *Coniophora puteana* (brown rot), testing ten specimens per treatment and fungus. Colorimetric analyses were performed with the CIELAB system, using a portable spectrophotometer. Measurements were carried out on the tangential and transverse faces of the specimens, before and after exposure to the fungi. The colorimetric coordinates L\*, a\*, and b\* were measured, and the parameters C, h°, and ΔE\* were calculated. The correlation between all colorimetric parameters and mass loss was calculated considering the colorimetric parameters measured on both faces of the specimens, before and after the biodeterioration test. The significance test (Student's t-test) was carried out for the correlation coefficient (r) and a scale proposed by Appolinário (2006) was adopted for its qualitative assessment. The discussion focused on the significant correlation coefficients (p < 0.05) and qualitatively classified, at least, as “strong” (0.60-0.80). Overall, better correlations were found for *T. versicolor* (12 “very strong” and 21 “strong” correlations) than for *C. puteana*, which had no “very strong” and 22 “strong” correlations (Table 1). For *T. versicolor*, luminosity (L\*) was the colorimetric parameter that best correlated with mass loss, with six “very strong” and four “strong” correlations, followed by the hue angle (h°). In general, the best correlations, both before and after fungal exposure occurred in treatment OS185. For *C. puteana*, the colorimetric parameters a\* and

b\* were those that best correlated with mass loss, both with six “strong” correlations. In general, the best correlations, both before and after fungal exposure, occurred in treatment CS160. For all conditions tested, there were no significant correlations for thermally modified wood in open system at 210 °C. There were significant “strong” and “very strong” correlations between the colorimetric parameters L\*, a\*, b\*, C\*, and h° for both fungi.

**Table 1: Number of significant correlation coefficients – r (p < 0.05) and its qualitative classification.**

Colorimetric parameters	<i>Trametes versicolor</i>		<i>Coniophora puteana</i>
	Very strong	Strong	Strong
L*	6	4	2
a*	1	2	6
b*	1	5	6
C*	1	5	5
h°	3	5	3

## REFERENCES

- American Wood Protection Association (2016). AWPA. **E30-16**: Standard method for evaluating natural decay resistance of woods using laboratory decay tests. AWPA Book of Standards, Birmingham.
- Appolinário, F. (2006). *Science methodology: philosophy and practice of research*. Thomson, São Paulo, Brazil.
- Bellon, K. R. R. (2013). Thermal modification of wood from three forest species planted using the VAP Holzsystem® process. Master's thesis, Federal University of Paraná, Curitiba, Brazil.
- Gasparik, M., Gaff, M., Kacik, F., Sikora, A. (2019). Color and chemical changes in teak (*Tectona grandis* L. f.) and meranti (*Shorea* spp.) wood after thermal treatment. *BioResources*, **14**(2), 26672683.
- Lopes, J. O. (2012). Uniformity and color stability of *Tectona grandis* L.f. heat-treated wood. Master's thesis, Federal Rural University of Rio de Janeiro, Seropédica, Brazil.
- Menezes, W.M. (2017). Effect of thermal modification on an industrial scale on the quality of *Tectona grandis* Linn. F. wood. Doctoral thesis, Federal University of Santa Maria, Santa Maria, Brazil.
- Motta, J. P., Oliveira, J. T. S., Paes, J. B., Alves, R. C., Dambroz, G. B. V. (2013). Natural resistance of *Tectona grandis* wood in laboratory test. *Ciência Rural*, **43**(8), 1393-1398.

# **DAY 1**

## **SESSION FOUR**

### **MODIFICATION WITH CHEMICALS**

## Oral 4.01 - Acetylation of European Hornbeam Wood (*Carpinus betulus* L.) – An 8-Year-Long Study

Fanni Fodor<sup>1</sup> and Róbert Németh<sup>1</sup>

<sup>1</sup>Institute of Wood Technology and Technical Sciences, University of Sopron, Bajcsy-Zsilinszky Str 4, 9400 Sopron, Hungary. E: fodor.fanni@uni-sopron.hu; nemeth.robert@uni-sopron.hu

**Keywords:** acetylation, bonding, carpinus, color, durability, hornbeam, microscopy

### ABSTRACT

Many European wood species have low natural durability which makes them unable to be used for exterior applications without additional protection. In this 8-year-long research work, European hornbeam wood (*Carpinus betulus* L.) was industrially acetylated in order to improve its properties and widen its usage.

In a preliminary research (Fodor *et al.* 2017; Pozsgayné Fodor 2015) on acetylated hornbeam, improved dimensional stability, mechanical properties, durability against fungi in laboratory conditions, and aesthetical color was concluded as a result of modification. These promising results supported the continuation of this project, where more concrete and industrial-related research had been carried out.

Thorough literature research was made on the availability and future of European hornbeam wood, from forestry and wood industry point of view. Then, the current state of research was explained regarding the modification of this species (Pozsgayné Fodor 2023).

In order to understand the changes in product-related properties after acetylation, chemical analysis was carried out to determine the chemical components, pH, buffering capacity, and content of organic extractives. These findings were evaluated by Fouriertransform infrared spectroscopy (FTIR) and high-performance liquid chromatography (HPLC) (Fodor *et al.* 2018).

After acetylation, changes in microscopic structure were examined by bright-field microscopy and scanning electron microscopy (Rousek *et al.* 2022).

In a seven-year-long test in soil, acetylated hornbeam showed no signs of fungal decay or insect damage, except for one stake, that was locally attacked by brown rot after 1.5 years. The decayed area was less acetylated compared to the good performing stakes, proven by FTIR analysis (Fodor *et al.* 2022).

Acetylated hornbeam is more hydrophobic, having increased contact angle and lower surface energy. This influences its bonding and coating properties. Compared to untreated hornbeam, the shear strength was reduced in dry conditions, but showed enhanced strength when soaked in cold or boiling water. The same was experienced in the bonding strength results with polyvinyl acetate and polyurethane adhesives (Fodor and Bak 2023). When exposed to ultraviolet light, acetylated hornbeam fades and grays similarly to untreated hornbeam, based on test results of natural irradiation (2 years) and artificial (200 hours) irradiation. Besides getting a patina, it remains intact, without cracking or warping, like untreated hornbeam. For the case when photostability is desired, various coatings were tested, among which three dark pigmented stains showed the best results (Fodor *et al.* 2022, Fodor and Németh 2017).

According to these results, use classes, product groups, market share of acetylated hornbeam were determined, supplemented by SWOT analysis and related literature, in order to give a full picture of it as a product. An outdoor bench was produced and installed in the Botanical Garden of Sopron in 2022 (Pozsgayné Fodor 2023).



In the future, further research is to be carried out on the acetylation of other important, but underutilized wood species, like Turkey oak and Pannonia poplar.

### ACKNOWLEDGEMENTS

This article was made in frame of the project TKP2021-NKTA-43 which has been implemented with the support provided by the Ministry of Innovation and Technology of Hungary (successor: Ministry of Culture and Innovation of Hungary) from the National Research, Development and Innovation Fund, financed under the TKP2021-NKTA funding scheme.

### REFERENCES

- Fodor, F., Bak, M. (2023). Studying the Wettability and Bonding Properties of Acetylated Hornbeam Wood Using PVAc and PUR Adhesives. *Materials*, **16**(5), 2046. <https://doi.org/10.3390/ma16052046>
- Fodor, F., Bak, M., Bidló, A., Bolodár-Varga, B., Németh, R. (2022). Biological Durability of Acetylated Hornbeam Wood with Soil Contact in Hungary. *Forests*, **13**(7), 1003. <https://doi.org/10.3390/f13071003>
- Fodor, F., Bak, M., Németh, R. (2022). Photostability of Oil-Coated and Stain-Coated Acetylated Hornbeam Wood against Natural Weather and Artificial Aging. *Coatings*, **12**(6), 817. <https://doi.org/10.3390/coatings12060817>
- Fodor, F., Lankveld, C., Németh, R. (2017). Testing common hornbeam (*Carpinus betulus* L.) acetylated with the Accoya method under industrial conditions. *iForest - Biogeosciences and Forestry*, **10**(6), 948-954. <https://doi.org/10.3832/ifor2359-010>
- Fodor, F., Németh, R. (2017). Testing the Photostability of Acetylated and Boiled Linseed Oilcoated Common Hornbeam (*Carpinus betulus* L.) *Wood. Acta Sylvatica et Lignaria Hungarica*, **13**(1), 81–94.
- Fodor, F., Németh, R., Lankveld, C., Hofmann, T. (2018). Effect of acetylation on the chemical composition of hornbeam (*Carpinus betulus* L.) in relation with the physical and mechanical properties. *Wood Material Science & Engineering*, **13**(5), 271–278. <https://doi.org/10.1080/17480272.2017.1316773>
- Pozsgayné Fodor, F. (2015). Modification of hornbeam (*Carpinus betulus* L.) by acetylation. Master Thesis, University of West Hungary, Sopron, Hungary. <http://diploma.uni-sopron.hu/310/>
- Pozsgayné Fodor, F. (2023). Acetylation of European hornbeam (*Carpinus betulus* L.) wood for outdoor applications. PhD Thesis, University of Sopron, Sopron, Hungary. <http://doktori.unisopron.hu/852/>
- Rousek, R., Fodor, F., Németh, R. (2022). Microscopic characterization of sound and decayed acetylated hornbeam (*Carpinus betulus* L.). *Wood Material Science & Engineering* (published online). <https://doi.org/10.1080/17480272.2022.2057817>

## Oral 4.02 - Solvent-Exchange Acetylation of Simulated Green Scots Pine Wood

Mikko Valkonen<sup>1</sup>, Md Tipu Sultan<sup>1,2</sup> and Lauri Rautkari<sup>1</sup>

<sup>1</sup>Department of Bioproducts and Biosystems, Aalto University School of Chemical Engineering, Vuorimiehentie 1, 02150 Espoo, Finland / P.O. Box 16300, FI-00076 AALTO. E: mikko.valkonen@aalto.fi; md.sultan@aalto.fi; lauri.rautkari@aalto.fi

<sup>2</sup>Directorate of Secondary and Higher Education, Ministry of Education, 16 Abdul Gani Road, 1000 Dhaka, Dhaka Division, Bangladesh

**Keywords:** acetic acid, acetic anhydride, Scots pine, simulated green wood, solventexchange acetylation

### ABSTRACT

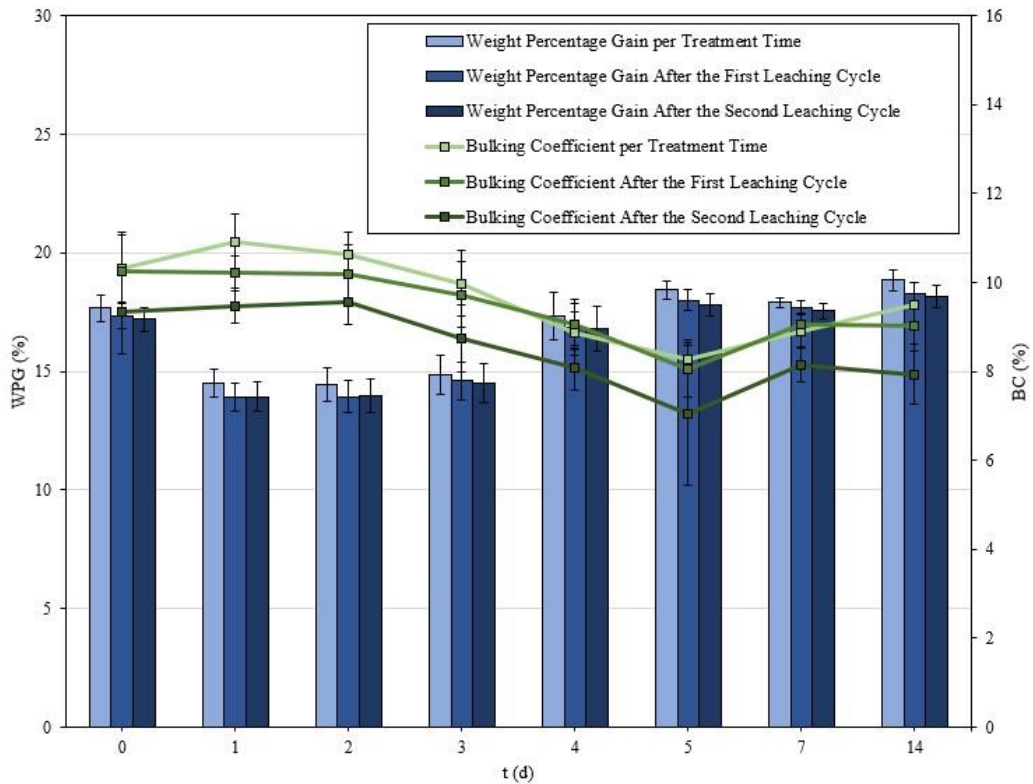
The solvent exchange of acetone Soxhlet-extracted (6 h) Scots pine sapwood was performed by (1) vacuum-impregnating the air-dry and extracted wood with deionised water to simulate green wood, (2) submerging the impregnated wood in 100 % acetic acid for 1, 2, 3, 4, 5, 7 and 14 d and (3) submerging the acetic acid submerged wood to acetic anhydride for the exact durations. The wood specimens were then acetylated with neat acetic anhydride at an elevated temperature of ca. 120 °C for 6 h. Residual acetic anhydride and by-product acetic acid were removed via a 6 h Soxhlet extraction. The average weight percentage gain of a comparative zero submersion time (dry wood) under inert Ar gas to simulate a vacuum was superior to 1, 2, and 3 d submersion times, while the 4, 5, 7 and 14 d submersions were on par or slightly surpassed the Ar reference. Due to diffusion and capillary phenomena, the submersion specimens are believed to have achieved a deeper chemical penetration depth as a function of submersion time.

The bulking coefficient reduced for the submersions until the fifth day, after which it increased. The bulking coefficient of the dry treatment was on par with the 3-d submersion case. The decreasing trend in the bulking coefficient and its reduction due to the leaching can be attributed to the hornification of the cell-wall polysaccharides in the wood. Herein, the polysaccharides come into close contact and at least become irreversibly H-bonded. The solvent exchange causes the wood to become dehydrated, and this is the reason for the occurrence of the hornification. Hornification further leads to the collapse of the cell wall pores. Moreover, the anti-swelling efficiency as a function of an additional antismelling efficiency parameter results could lead one to believe that cross-linking occurs; this cannot be the case as the acetylation reaction cannot induce cross-linking. What is more likely is that “physical cross-linking” in the form of hornification of the cellulose microfibrils, or in general, cell-wall polysaccharides in the wood, occurs.

### AN EXCERPT OF RESULTS AND DISCUSSION

Fig. 1 illustrates the average weight percentage gain (WPG) and bulking coefficient (BC) per submersion treatment time for the initial results after the 6 h reactions, Soxhlet extractions, and those of the two leaching cycles. As observed, the dry under Ar gas treatment without submersion surpasses the 1, 2, and 3 d submersions while the treatments from 4 d onwards are on par with the Ar scenario or slightly above it concerning the WPG. Furthermore, the leaching cycles tend to lower the WPGs to a minor degree. The obtained WPGs are either near or between the 15 to 20 % values above which the acetylated wood would show significant resistance to typical wood-destroying fungi (Bongers and Uphill

2019). As for the BC, there was an initial decrease for the submersions until the 5-d case, after which the parameter slightly increased in value. Moreover, the leaching cycles led to a reduction in almost all the BCs. Thus, the decreasing trend in the BC and the decreasing quantity can be attributed to the hornification phenomenon wherein the polymer structure of the wood stiffens due to drying or dehydration. Consequently, the internal volume of the wood cells shrinks because of the stiffening-induced structural changes in the cells. Therefore, the original water-swollen state of the wood will not be regained if the material is reswollen after drying (Borrega and Kärenlampi 2010, Fernandez Diniz *et al.* 2004).



**Figure 1: The average WPG and BC per submersion treatment time for the initial results, those after the first leaching cycle (first week) and second leaching cycle (second week) [cf., the inset legend for the colour codes]. The sample standard deviations are shown by the error bars. N.B.  $N=6$  for the initial results, whereas  $N=5$  for the leached ones.**

## REFERENCES

- Bongers, F. and Uphill, S.J. (2019). The potential of wood acetylation. In: Proceedings of the 7th International Conference on Hardwood Processing. Delft, The Netherlands, pp. 49-59.
- Borrega, M. and Kärenlampi, P.P. (2010). Hygroscopicity of heat-treated Norway spruce (*Picea abies*) wood. *European Journal of Wood and Wood Products*, **68**, 233-235.
- Fernandez Diniz, J.M.B., Gil, M.H. and Castro, J.A.A.M. (2004). Hornification—its origin and interpretation in wood pulps. *Wood Science and Technology*, **37**, 489-494.

## Oral 4.03 - Mechanical Properties and Biological Durability of Wood Modified with PEG and Various Carboxylic Acids

Melissa Christ<sup>1</sup>, Nicole Flaig<sup>1</sup> and Marcus Müller<sup>1</sup>

<sup>1</sup>Material development and processing, University of Applied Forest Sciences, Schadenweilerohof, 72108 Rottenburg am Neckar, Germany. E: mueller@hs-rottenburg.de

**Keywords:** carboxylic acids, dimensional stability, physical and mechanical properties, polyethylene glycol, wood modification

### ABSTRACT

An innovative modification process was developed from polyethylene glycol (PEG) and various carboxylic acids. In contrast to the low-molecular weight polyols that are currently being used, PEG 400 is a large and flexible polymer (Kiljunen *et al.* 2018). It has only two OH groups, whereas polyols such as sorbitol, glycerol or glucose have more OH groups. Consequently, PEG has less crosslinking possibilities with acids than the polyols. It is possible that the degree of crosslinking is lower and thus the embrittlement.

The water-soluble PEG was fixed in the wood with 1,2,3,4-butanetetracarboxylic acid (BTCA), citric acid (CA) and malic acid (MA). For beech wood (*Fagus sylvatica*), a dimensional stabilization between 31 % and 50 % could be achieved. In terms of mechanical properties, an increase in compression strength (by 12 % to 41 %), modulus of elasticity (by 2 % to 15 %) and hardness (by 6 % to 17 %) was obtained. Tensile strength (between 36 % and 46 %) and impact bending strength (between 43 % and 69 %) decreased significantly, with only a slight change in modulus of rupture (between 2 % and 15 %). Biological durability against brown and white rot (16 weeks incubation time) and soft rot (32 weeks incubation time) increased significantly. The results are shown in Table 1. For beech wood, durability class 1 was achieved by the treatment.

**Table 1: Biological durability of modified beech wood against brown rot, white rot and soft rot fungi.**

	Control	BTCA/PEG	CA/PEG	MA/PEG
Mass loss brown rot [%]	34.53 (5.64)	-0.29 (1.55)	4.62 (3.23)	11.64 (5.36)
Mass loss white rot [%]	29.41 (2.31)	2.88 (1.66)	1.30 (1.05)	2.48 (1.66)
Mass loss soft rot [%]	35.14 (3.18)	2.78 (0.86)	5.13 (0.48)	13.91 (2.78)

### REFERENCES

Kiljunen, S.; Koski, A.; Kunttu, M.; Valkonen, T. (2018): Impregnation of chemicals into wood. Publication number: WO2011-042609A1.

## Oral 4.04 - Novel Wood Modification Through the Use of Heterocyclic Organic Compounds

Alexander Scharf<sup>1</sup>, Henric Dernegård<sup>2</sup>, Johan Oja<sup>3</sup>, Dick Sandberg<sup>1</sup>  
and Dennis Jones<sup>1</sup>

<sup>1</sup>Wood Science and Engineering, Luleå University of Technology, Forskargatan 1, 931 87 Skellefteå, Sweden. E: alexander.scharf@ltu.se; dick.sandberg@ltu.se; dennis.jones@ltu.se

<sup>2</sup>Holmen AB, Strandvägen 1, 11451 Stockholm, Sweden. E: henric.dernegard@holmen.com

<sup>3</sup>Norra Timber, Skeppargatan 1, SE-904 03 Umeå, Sweden. E: johan.oja@norraskog.se

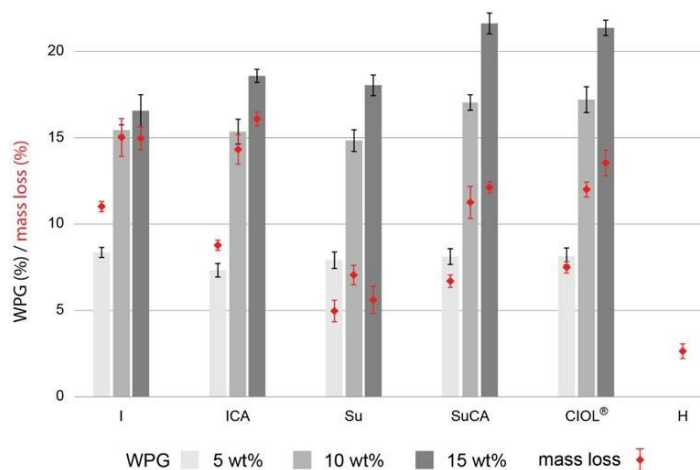
**Keywords:** heterocyclic compounds, properties, thermal treatment, wood modification

### ABSTRACT

Chemical wood modification is based on the introduction of various chemicals into the wood. It exists in different forms with the main difference being the location of chemical deposition and the type of bonding with the cell wall (Hill 2006). Chemicals can react with groups of cell-wall polymers, blocking *e.g.* hydroxyl groups or leading to cross linking by reacting with two hydroxyl groups. The former results in cell-wall bulking while the latter additionally limits the maximum distance between the cell-wall polymers, effectively reducing the maximum swelling, *i.e.* providing anti-swelling efficiency (ASE) (Ohmae *et al.* 2002). Additionally, lumen-filling treatments exist which may or may not react with the cell-wall components. Similar to thermal modification, chemical modification improves water-related properties, biological durability and can affect mechanical properties (Verma *et al.* 2009). Among recent developments in chemical modification has been the polymerization of citric acid and sorbitol in an aqueous solution (Larnøy *et al.* 2018, Mubarok *et al.* 2020) in wood at 140 °C.

Imidazole, a heterocyclic compound comprising two nitrogen and three carbon atoms, possesses acidic and basic properties. It has been used for the extraction of cellulose and hemicelluloses from wheat straw at a temperature of 170 °C (Morais *et al.* 2016). Succinimide, another heterocyclic compound containing nitrogen, exhibits a carbonyl and an amide group. Succinimides have high chemical reactivity due to the presence of both the carbonyl and methylene groups. It has applications in the pharmaceutical, polymer and material industries. To this point, neither chemical appears to have been used for wood modification.

Scots pine (*Pinus sylvestris* L.) sapwood was treated with imidazole (C<sub>3</sub>N<sub>2</sub>H<sub>4</sub>), succinimide (C<sub>4</sub>H<sub>5</sub>NO<sub>2</sub>), with and without citric acid (C<sub>6</sub>H<sub>8</sub>O<sub>7</sub>) at a temperature of 220 °C, which were compared to citric acid/D-sorbitol (C<sub>6</sub>H<sub>14</sub>O<sub>6</sub>) and samples that had undergone thermal treatment at the same temperature. The weight uptakes are shown in Figure 1. Treatments with imidazole exhibited an increased mass loss during heat treatment, which led to the formation of water-soluble degradation products which were leached out over wet-dry cycles. The mass loss during heat treatment of succinimide-containing treatments seemed to be unaffected by the chemical and a large amount of succinimide was leachable. Leaching resistance was highest with a combination of citric acid and sorbitol (as noted with the CIOL®-process). However, in this treatment the share of citric acid was almost twice as high as in the other treatments containing citric acid. ASE after three cycles reached 31% for imidazole-treated specimens and improved to 38% with the addition of citric acid. For succinimide, ASE increased from 17% to 41%. Citric acid/sorbitol (CIOL®) exhibited an ASE of 48%. Mechanical properties were also determined and compared.



**Figure 1: Mean  $\pm$  standard deviation of weight percentage gain (WPG) after pressure impregnation and drying and mass loss due to heat treatment at 220 °C.**

The results within this study show that wood modification with imidazole and succinimide may be utilized as part of a new treatment system. Studies continue to further understand the mechanisms and resulting properties.

## REFERENCES

- Hill, C.A.S. (2006). *Wood Modification: Chemical, Thermal and Other Processes*; 1st ed.; Wiley, Chichester, UK; ISBN 978-0-470-02172-9.
- Larnøy, E., Karaca, A., Gobakken, L.R. and Hill, C.A.S. (2018). Polyesterification of Wood Using Sorbitol and Citric Acid under Aqueous Conditions. *International Wood Products Journal*, **9**, 66–73.
- Morais, A.R.C., Pinto, J.V., Nunes, D., Roseiro, L.B., Oliveira, M.C., Fortunato, E. and BogelŁukasik, R. (2016). Imidazole: Prospect Solvent for Lignocellulosic Biomass Fractionation and Delignification. *ACS Sustainable Chem. Eng.*, **4**, 1643–1652.
- Mubarok, M., Militz, H., Dumarçay, S. and Gérardin, P. (2020). Beech Wood Modification Based on in situ Esterification with Sorbitol and Citric Acid. *Wood Science and Technology*, **54**, 479– 502.
- Ohmae, K., Minato, K. and Norimoto, M. (2002). The Analysis of Dimensional Changes Due to Chemical Treatments and Water Soaking for Hinoki (*Chamaecyparis Obtusa*) Wood. *Holzforschung*, **56**, 98–102.
- Verma, P., Junga, U., Militz, H. and Mai, C. (2009). Protection Mechanisms of DMDHEU Treated Wood against White and Brown Rot Fungi. *Holzforschung*, **63**, 371–378.

## Oral 4.05 - Combining Kraft Lignin-Glyoxal and Organic Phase-Change Materials for a Modified Wood with Thermal-Energy Storage Capacity

Chia-feng Lin<sup>1</sup>, Olov Karlsson<sup>1</sup>, Dennis Jones<sup>1</sup> and Dick Sandberg<sup>1</sup>

<sup>1</sup>Wood Science and Engineering, Luleå University of Technology, Forskargatan 1, 931 87 Skellefteå, Sweden. E: chia-feng.lin@ltu.se; olov.karlsson@ltu.se; dennis.jones@ltu.se and dick.sandberg@ltu.se

**Keywords:** Paraffin, PCM, thermal-regulated wood

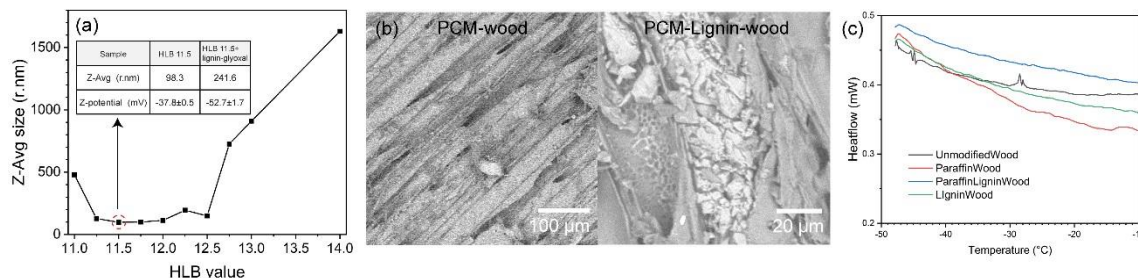
### ABSTRACT

The emission of greenhouse gases during energy production is a significant contributor to climate change. Approximately 20% of the annual total global energy is used for the heating/cooling required to maintain comfortable conditions in constructed buildings (Omrany *et al.* 2016). To reduce this energy consumption, the use of organic phasechange materials (PCMs) has been suggested (Rathore and Shukla 2019). The selection of organic PCMs is based on their melting point, which should be close to the comfortable building temperatures, potentially allowing them to absorb and release large latent heat energy to stabilize the environment temperature. However, PCM leakage from the treated building material during their liquid phase by temperature variations hinders wider applications. An approach to reduce the leakage is to microencapsulate the PCMs, limiting their movement within polymeric shells, formed by the use of a phenolformaldehyde resin (Liu *et al.* 2022). The objective of the study was to use Kraft ligninglyoxal as a polymeric matrix to encapsulate PCMs for alleviating the leakage of PCMs from wood material for the use in timber-building construction.

To prepared the encapsulated PCMs, the paraffin oil was firstly dispersed in water (to obtain micelles) by using the mixture of surfactants Tween 80 and Span 80 (Liu *et al.* 2006). The Z-average radius ( $Z_{avg,r}$ ) of the PCM micelle, correlated to its hydrophiliclipophilic balance (HLB) value, was analysed by dynamic light scattering (DLS), Fig. 1(a). The PCM micelles exhibited the smallest  $Z_{avg,r}$  of 98.3 nm when the HLB value was 11.5. Subsequently, the emulsion was mixed with lignin-glyoxal prepolymer, resulting in an increased  $Z_{avg,r}$  to 241.6 nm. This increase may be attributed to the polymeric lignin that surrounds the PCM nanomicelles, thereby increasing the  $Z_{avg,r}$  value. Additionally, the Z-potential (the higher absolute value indicates that the emulsion is better electrically stabilized) of the PCM emulsion increased upon the additional of lignin-glyoxal prepolymer, possibility due to the steric hindrance associated with the polymeric structure of lignin. This PCM-lignin emulsion was further impregnated into Scots pine (*Pinus sylvestris* L.) sapwood, and its morphology and thermal properties were analysed.

The SEM analysis of pure PCM-modified wood in longitudinal-radial section showed that the PCMs have filled the lumen of the tracheid cells, Fig. 1(b). In contrast, the PCMlignin-glyoxal modified wood showed some aggregation of granules within the lumen, which may be related to the polymerisation of lignin-glyoxal. Further investigation into the microencapsulation of PCMs by lignin-glyoxal is currently on-going. Differential scanning calorimetry (DSC) was conducted to study the thermal properties of the PCMmodified wood, Fig. 1(c). The results revealed no new endothermic or exothermic peaks after the modification. This initial study used paraffin oil as the PCM, which did not exhibit a melting temperature within the investigated temperature range. To further explore the heat storage application of the PCM-modified wood, paraffin oil will be substituted with a paraffin with

specific melting point of PCM such as RT18HC (Rubitherm GmbH, Germany). Additionally, the thermal reliability of the microencapsulation will be tested by the DSC through a thermal cycle test.



**Figure 1: Physiological and chemical analyses of (a) dynamic light scattering (DLS) analysis of the PCM emulsion, (b) electron microscope images of PCM with/without lignin-glyoxal modified wood, and (c) differential scanning calorimetry (DSC) curves of unmodified wood, PCM modified wood, lignin-glyoxal modified wood and PCM-lignin-glyoxal modified wood.**

In conclusion, the DLS results revealed the existence of the PCM micelles under the presence of lignin-glyoxal prepolymer. The PCM-lignin-glyoxal modified wood exhibited distinct morphological differences compared to PCM-modified wood. The DSC results implied that further improvements are required for the thermal energy storage application of PCM-modified wood, possibly by substituting paraffin oil with a material that offers higher thermal energy storage capacities.

## ACKNOWLEDGEMENTS

Support through the project “Luleå University of Technology's initiative “Natural Resources for Sustainability transitions” (SUN), and the Swedish Research Council for Environment, Agricultural Sciences and Spatial Planning (FORMAS), project “Biobased fire protection of wood panel for exterior conditions by using phosphorylated lignin from wheat straw” 2021-00818”, are gratefully acknowledged.

## REFERENCES

- Liu, C., Cao, H., Jin, S., Bao, Y., Cheng, Q., and Rao, Z. (2022). Synthesis and characterization of microencapsulated phase change material with phenol-formaldehyde resin shell for thermal energy storage. *Solar Energy Materials and Solar Cells*, **243**, 111789.
- Liu, W., Sun, D., Li, C., Liu, Q., and Xu, J. (2006). Formation and stability of paraffin oil-in-water nano-emulsions prepared by the emulsion inversion point method. *Journal of Colloid and Interface Science*, **303**(2), 557–563.
- Omrany, H., Ghaffarian Hoseini, A., Ghaffarian Hoseini, A., Raahemifar, K., and Tookey, J. (2016). Application of passive wall systems for improving the energy efficiency in buildings: A comprehensive review. *Renewable and Sustainable Energy Reviews*, **62**, 1252–1269.
- Rathore, P.K.S. and Shukla, S.K. (2019). Potential of macroencapsulated PCM for thermal energy storage in buildings: A comprehensive review. *Construction and Building Materials*, **225**, 723–744.



## Oral 4.06 - Compatibility of Lignocellulosic Materials to Form Thermoplastic Film by a Single Esterification Reaction: Wood and Natural Fibers

Prabu Satria Sejati<sup>1,2</sup>, Laura Roche<sup>1</sup>, Jennifer Afrim<sup>1</sup>, Vincent Mariani<sup>1</sup>, Firmin Obounou Akong<sup>1</sup>, Frédéric Fradet<sup>3</sup>, Philippe Gérardin<sup>1</sup>

<sup>1</sup>LERMAB, INRAE, Université de Lorraine, 54000 Nancy, France. E: philippe.gerardin@univ-lorraine.fr

<sup>2</sup>Research Center for Biomass and Bioproducts, National Research and Innovation Agency (BRIN), 16911 Bogor, Indonesia. E: prabu-satria.sejati@univ-lorraine.fr

<sup>3</sup>PLASTINNOV, IUT de Moselle-Est, Université de Lorraine, 57500 Saint-Avold, France

**Keywords:** cotton, esterification, flax, natural fiber, thermoplastic, wood

### ABSTRACT

Application of natural fibers always facing incompatibility problems due to hydrophilic nature of the fibers which resulted poor interactions with thermoplastic polymer matrix. The objective of this research was to provide thermoplastic properties to the natural fibers by solvent free esterification using trifluoroacetic anhydride (TFAA) as impelling agent and fatty acids as acylating agent, without any plasticizers or thermoplastic polymer. We also want to make these fibers hydrophobic, potentially addressing incompatibility issues at the fiber matrix interface. For comparison, esterification was also performed to spruce and poplar wood. The reactivity of esterification varied depending on the chemical composition of the fibers indicated by different values of WPG. Cotton and flax fiber were chosen for further characterization due to their high content of cellulose which resulted in high ester content. Significant changes in chemical structure were detected using FTIR indicated by substitution of hydroxyl groups with alkyl side chains from fatty acids. This chemical change followed by thermal properties improvement observed by TGA. Thermoplasticity of fatty acids esterified fibers successfully achieved indicated by softening temperature at about 60 and 130 °C observed by DSC and TMA, and disappearance of fibers aspect to homogenous and smooth surface of the film. Significant improvements of surface hydrophobicity were also observed by measuring contact angle of water drops to the surface of the film.

## Oral 4.07 - Furfurylated Wood: Using Pyrolysis-GC/MS to Characterize Polymer-Wood Bonds Existence

Hentges David<sup>1</sup>, Philippe Gérardin<sup>1</sup> and Stéphane Dumarçay<sup>1</sup>

<sup>1</sup>Université de Lorraine, INRAE, LERMAB, F-54000 Nancy, France E:  
david.hentges@univlorraine.fr; philippe.gerardin@univ-lorraine.fr; stephane.dumarcay@univ-lorraine.fr

**Keywords:** covalent bonding, furfurylation, Py-GC/MS

### ABSTRACT

Furfurylation is a chemical modification method that involves introducing furfuryl alcohol into wood by impregnation and polymerizing it to form polyfurfuryl alcohol (PFA). The technique was developed by Goldstein in 1955 to improve the properties of wood in terms of resistance to abrasion, bending and fungal decay.

It has long been suspected that PFA can bind to lignin, either by electrophilic substitution or by the Diels-Alder reaction to form covalent bonds. Nordstierna *et al.* (2008) used <sup>13</sup>C NMR models to show that this type of bonding is possible. According to Shen *et al.* (2021), the use of a model polymerisation mixing dihydroconiferyl alcohol and furfuryl alcohol showed the existence of bonds between the aromatic rings and PFA but, when compared with modified wood, concluded that the bonds were rare and therefore difficult to demonstrate. Zhu *et al.* (2022) carried out the most recent work using Klason lignin furfurylated with PFA using 2D HSQC NMR and <sup>31</sup>P NMR to suggest bonding mainly on the aromatic ring carbons as well as ether bonds with phenolic hydroxyls.

The Py-GCMS study of the furfurylation of wood was carried out in order to demonstrate the presence of covalent bonds between PFA and wood biopolymers. Indeed, demonstrating the existence of such bonds between wood and polymers, or possibly their absence, is important for a better understanding of the performance of modified wood and, more particularly from a fundamental point of view, for highlighting the reaction sites involved in the reaction.

In our work, coniferyl alcohol and syringic aldehyde have been used as models of G and S subunits to reveal these unique structures which are sometimes scarce and not easily detectable via reaction with furfuryl alcohol. In a second step, by comparison with the previous results, pyrograms of furfurylated wood have provided evidence of the presence of covalent binding between PFA and lignin on different reactive sites, confirming the interest of the Py-GC/MS technique for such analysis. We also obtained clues of non reaction with wood polysaccharides.

### REFERENCES

- Goldstein, I. (1955). The impregnation of wood to impart resistance to alkali and acid. *Forest Products Journal* 5, 265-267
- Nordstierna, L., Lande, S., Westin, M., Karlsson, O., Furó, I. (2008). Towards novel wood-based materials: Chemical bonds between lignin-like model molecules and poly(furfuryl alcohol) studied by NMR. *Holzforschung*, 62 (6), 709–713.
- Shen, X., Guo, D., Jiang, P., Li, G., Yang, S., Chu, F. (2021). Reaction mechanisms of furfuryl alcohol polymer with wood cell wall components. *Holzforschung*. 75 (12), 1150-1158 <https://doi.org/10.1515/hf-2020-0271>

Zhu, X., Bruijnaers, B., Lourençon, T. V., Balakshin, M. (2022). Structural Analysis of LigninBased Furan Resin. *Materials*. 15 (1), 350. <https://doi.org/10.3390/ma15010350>.

## Oral 4.08 - Mould Growth, Fungal Growth and Strength of Wood Treated with Maleic Anhydride Combined with Sodium Hypophosphite

Injeong Kim<sup>1</sup>, Lone Ross<sup>2</sup>, Gry Alfredsen<sup>2</sup>, Olov Karlsson<sup>1</sup>, Dennis Jones<sup>1</sup>, George I. Mantanis<sup>3</sup>, and Dick Sandberg<sup>1</sup>

<sup>1</sup>Wood Science and Engineering, Luleå University of Technology, Forskargatan 1, 931 87 Skellefteå, Sweden. E: injeong.kim@ltu.se; olov.karlsson@ltu.se; dennis.jones@ltu.se; dick.sandberg@ltu.se

<sup>2</sup>Division of Forest and Forest Resources, Norwegian Institute of Bioeconomy Research, Norway. E: gry.alfredsen@nibio.no; lone.ross@nibio.no

<sup>3</sup>Department of Forestry, Wood Sciences and Design, Laboratory of Wood Science and Technology, University of Thessaly, Greece. E: mantanis@uth.gr

**Keywords:** durability, wood modification

### ABSTRACT

Many properties of wood depend on the wood-and-moisture relationship. Modification of wood with maleic anhydride (MA) and sodium hypophosphite (SHP) enhances dimensional stability of wood and decreases cell-wall affinity to water (Kim *et al.* 2021 a, b). Thus, it is likely that the modification with MA and SHP influences properties of wood, such as durability and mechanical strength. In this study, the wood treated with MA and SHP were exposed in an outdoor environment to observe discolouration by mould growth, as well as biological deterioration using a modified EN113 test (Bravery test). Furthermore, to study mechanical strength, the three-point bending test was performed.

For testing effectiveness against Basidiomycetes fungi, ten specimens for each treated and reference were subjected to mini-block test conditions, commonly referred to as the Bravery test (Bravery 1979), in this case performed using *Trametes versicolor* and *Rhodonía placenta*. The mould test was performed with a mixture of *Penicillium* (2021-142-A-1 and 2015-21/1/3), *Alternaria alternata* (2006-53), *Sydowia polyspora* (2022-122-2), *Ulocladium atrum* (2006-55), *Cladosporium cladosporioides* (2006-54) and *Aurobasidium pullulans* (2006-52) using the method described by Gobakken and Alfredsen (2016), using five specimens for each treated and reference group, with the extent of mould coverage the specimens being assessed visually and with stereo microscope.

Scots pine (*Pinus sylvestris* L.) sapwood were cut into dimensions (50x50x10 mm for outdoor weathering test and 10x10x200 mm for bending test) and impregnated with 3.5 M MA in acetone, followed by 0.5 M SHP according to Kim *et al.* (2021a). The outdoor weathering test was performed on racks in Skellefteå, Sweden (64.744453°N, 20.955569°E). Half of specimens were facing south at a 45° inclination, while the other half were placed vertically in the northern direction. The growth of the mould and discoloration of surface of specimens were observed and visually assessed.

Ten specimens per group were prepared for the bending test. All specimens were conditioned at 20 °C, 65% relative humidity until equilibrium. A universal testing machine Criterion Model 43 (MTS systems Corporation, France) was used for the static three-point bending test. Modulus of elasticity (MOE) and modulus of rupture (MOR) was measured.



Figure 1: Test site for outdoor weathering test.

Initial results from fungal tests showed *Rhodonina placenta* (brown rot) degraded untreated specimens more than *Trametes versicolor* (white rot). The specimens modified with MA and SHP barely reduced its mass while untreated wood lost 10% and 33% by *Trametes versicolor* and *Rhodonina placenta*, respectively. Grading of mould attack according to the EN15457 standard (CEN 2022) showed the growth rate at earlier times was similar between treated and untreated specimens. However, the mould growth rate on untreated specimens became faster after about 20 days than that on treated specimens resulting in total coverage of mould (rating=4) on specimens after 30 days. The mycelia of discolouring fungi were more visually apparent on the untreated specimens, than on treated specimens due to the darker colour of the wood. Results from longer exposures and weathering and mechanical data will be presented at the conference.

## REFERENCES

Bravery AF (1979) Screening techniques for potential wood preservative chemicals. Proceedings of a special seminar held in association with the 10th annual meeting of the IRG, Peebles, UK. IRG/WP 2138. Swedish Wood Preservation Institute.

CEN (2022) EN 15457 - Paints and varnishes. Laboratory method for testing the efficacy of film preservatives in a coating against fungi. European Committee for Standardization (CEN), Brussels, Belgium.

Gobakken LR, Alfredsen G (2006) Susceptibility of wood substrates to *Aurebasidium pullulans* at different temperatures. IRG 47, Lisbon, Portugal. *The International Research Group on Wood Protection*. Report IRG/WP 16-10863.

Kim I, Karlsson O, Jones D, Mantanis G and Sandberg D (2021a) Dimensional stabilisation of Scots pine (*Pinus sylvestris* L.) sapwood by reaction with maleic anhydride and sodium hypophosphite. *European Journal of Wood and Wood Products*, **79**, 589–596.

Kim I, Thybring EE, Karlsson O, Jones D, Mantanis G and Sandberg D (2021b) Characterisation of moisture in Scots pine (*Pinus sylvestris* L.) sapwood modified with maleic anhydride and sodium hypophosphite. *Forests*, **12**, 1333.

## Oral 4.09 - Effect of Lactic Acid Impregnation on Some Physical Properties of Wood

Miklós Bak<sup>1</sup>, Róbert Németh<sup>1</sup> and Mátyás Báder<sup>1</sup>

<sup>1</sup>Faculty of Wood Engineering and Creative Industries, University of Sopron, Bajcsy-Zsilinszky utca 4, 9400 Sopron, Hungary. E: bak.miklos@uni-sopron.hu; nemeth.robert@uni-sopron.hu; bader.matyas@uni-sopron.hu

**Keywords:** anti-swelling-efficiency, hardness, impregnation, polylactic acid, wood-water relation

### ABSTRACT

There is a growing demand for the use of wood, especially in the use of valuable wood that is resistant to biological pests and has high dimensional stability. Impregnation with biopolymers to be used can significantly improve the targeted wood properties, especially dimensional stability. One of the most produced and processed biopolymers is polylactic acid (PLA), produced from lactic acid polycondensation.

This research deals with the combination of wood and PLA, both of which are environmentally friendly materials issued from natural resources and quite easily recycled and degraded in good environmental conditions. Grafting of lactic acid oligomers and polymers onto wood and polymerization into the wood structure have been studied, considering that lactic acid contains carboxylic end groups, while solid wood cell walls contain hydroxyl groups. The main goal of this work was to increase dimensional stability. European beech (*Fagus sylvatica*) and Scots pine sapwood (*Pinus sylvestris*) was used for the tests. As a starting material of the modification, lactic acid monomer was used. The initial step was a removal of water from the monomer, followed by two steps of oligomerization at elevated temperatures (75 °C, 100 °C and 130 °C respectively), under 150 mbar pressure. Samples were impregnated with the prepared oligomers, followed by a curing step at atmospheric pressure, using three different temperatures (110 °C, 120 °C and 140 °C) to test the effect of curing temperature on the properties of modified wood. Weight percent gain (WPG) of beech and Scots pine samples showed 55-60% and 90-105% respectively. As a result, density increased 15-25% in beech and 35-45% in Scots pine. In general, increasing curing temperature resulted in decreasing density of the modified samples, due to degradation caused by the elevated temperature and the acidity of the treatment medium. Equilibrium moisture content (EMC) decreased by a similar extent (45-55%) in both wood species, depending on the curing temperature. Higher curing temperature resulted in lower EMC. Additionally, the time necessary to reach EMC has tripled in treated samples, compared to the untreated ones. As a result of lower moisture uptake, anti-swelling-efficiency (ASE) decreased remarkably as well. ASE is influenced by the curing temperature as well. Higher curing temperature resulted in higher ASE (Fig. 1). Colour of the samples turned remarkably darker as a result of the treatment, showing total colour change ( $\Delta E^*$ ) of 28-55, depending on wood species and curing temperature.

Surface hardness increased as a result of the treatments (Fig. 2). The improvement was 15-40% in cross section and 20-60% in tangential section, depending on wood species. Beside testing the material properties, scanning electron microscopy was used to investigate the location and distribution of the lactic acid in the cell wall structure. It showed that the impregnation was incomplete, as not all cell lumens were filled by the lactic acid (Fig. 3). However, all cell types (tracheids, fibres, rays, vessels) were penetrated by the lactic acid.

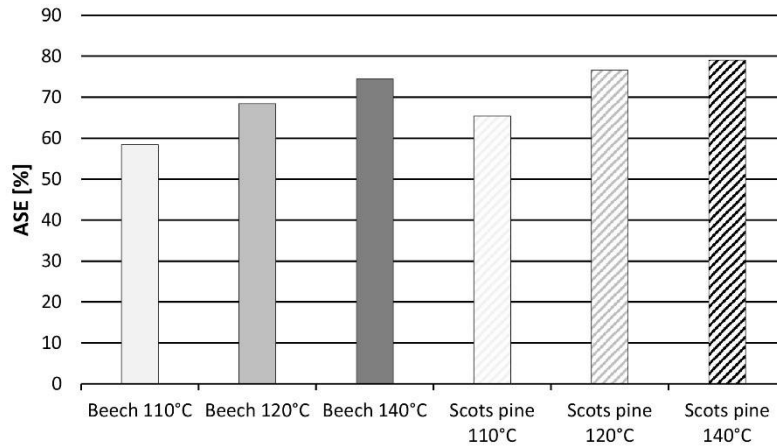


Figure 1: Effect of LA impregnation and curing temperature on ASE of beech and Scots pine wood.

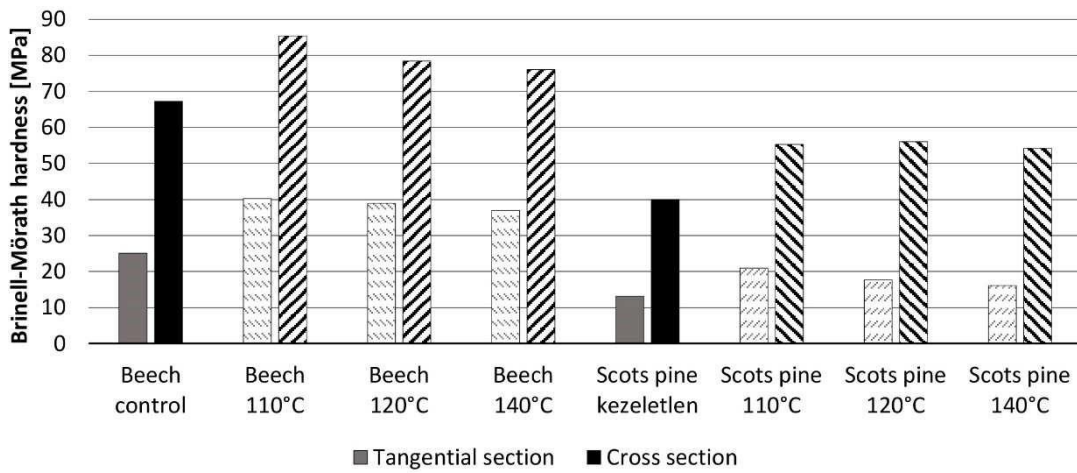


Figure 2: Effect of LA impregnation and curing temperature on surface hardness of beech and Scots pine wood in different anatomical directions.

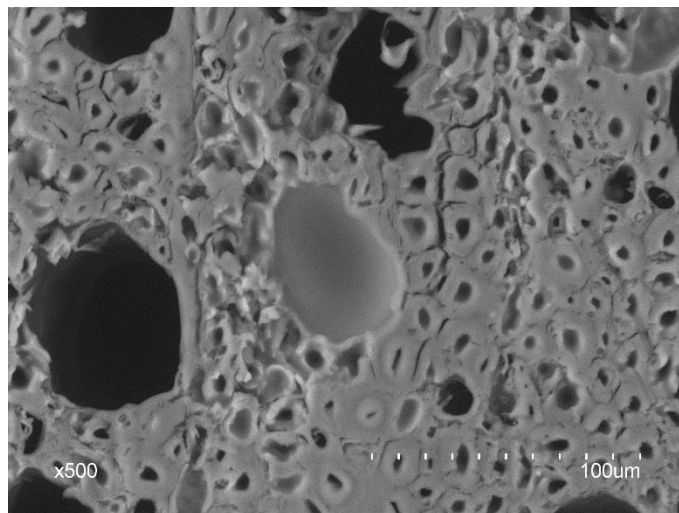


Figure 3: Presence of LA in beech wood cross section, showing saturated, partly saturated and empty cells. Magnification: 500x

## Oral 4.10 - Relevant Bonding Aspects of Acetylated Beech (*Fagus sylvatica* L.) LVL for Load-Bearing Construction in Exterior Use

Maik Slabohm<sup>1</sup>, Jan-Oliver Haase<sup>1</sup>, and Holger Militz<sup>1</sup>

<sup>1</sup>Wood Biology and Wood Products, Burckhardt-Institute, Georg August University of Göttingen, Büsingenweg 4, 37077 Göttingen, Germany E: maik.slabohm@uni-goettingen.de; hmilitz@gwdg.de

**Keywords:** acetylation, alpturm, beech, bonding, laminated veneer lumber

### ABSTRACT

Acetylation with acetic anhydride has attracted much attention during the last decades as it is known to improve the dimensional stability (swelling and shrinking in contact with moisture) and durability of wood against wood destroying fungi. Currently research on acetylated beech laminated veneer lumber (LVL) for load-bearing construction is ongoing. Therefore, we investigated relevant aspects (curing behavior and wood failure percentage (WF)) of bonding acetylated beech LVL. The effects of acetylated wood powder on the curing behavior of phenol-formaldehyde (PF), phenol-resorcinol-formaldehyde (PRF), and polyurethane (PUR) adhesives were investigated. To test the effect of water during curing, the wood powder was conditioned to dry, normal, and wet moisture content (MC). The data revealed that acetylated wood had a minor impact on curing. An additional test demonstrated that the bonding of acetylated beech LVL with PUR, PF, and PRF adhesives was suitable for load-bearing construction.

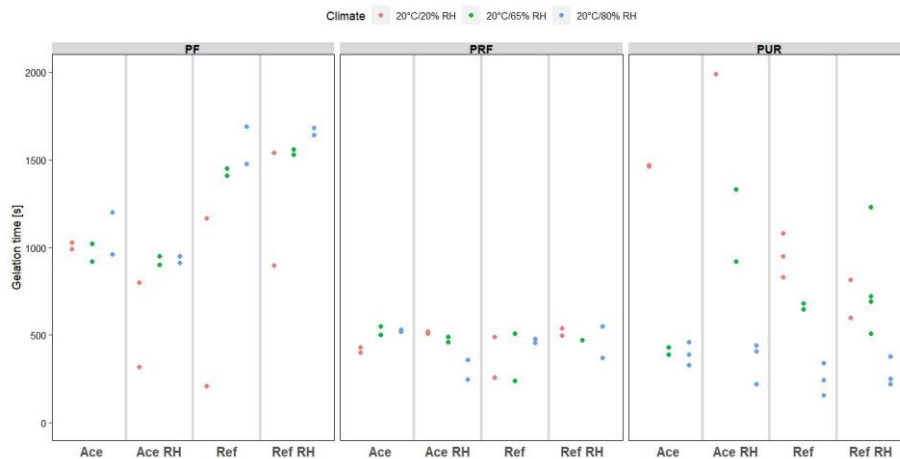
**Table 1: Gelation time set-up of acetylated and untreated wood powders with PF, PRF, and PUR adhesives**

Adhesive	Discolored red-heart	Modification	Climatization
PF	yes	acetylated	20°C/20% RH; 20°C/65% RH; 20°C/80% RH
PF	no	acetylated	20°C/20% RH; 20°C/65% RH; 20°C/80% RH
PF	yes	untreated	20°C/20% RH; 20°C/65% RH; 20°C/80% RH
PF	no	untreated	20°C/20% RH; 20°C/65% RH; 20°C/80% RH
PUR	yes	acetylated	20°C/20% RH; 20°C/65% RH; 20°C/80% RH
PUR	no	acetylated	20°C/20% RH; 20°C/65% RH; 20°C/80% RH
PUR	yes	untreated	20°C/20% RH; 20°C/65% RH; 20°C/80% RH
PUR	no	untreated	20°C/20% RH; 20°C/65% RH; 20°C/80% RH
PRF	yes	acetylated	20°C/20% RH; 20°C/65% RH; 20°C/80% RH
PRF	no	acetylated	20°C/20% RH; 20°C/65% RH; 20°C/80% RH
PRF	yes	untreated	20°C/20% RH; 20°C/65% RH; 20°C/80% RH
PRF	no	untreated	20°C/20% RH; 20°C/65% RH; 20°C/80% RH

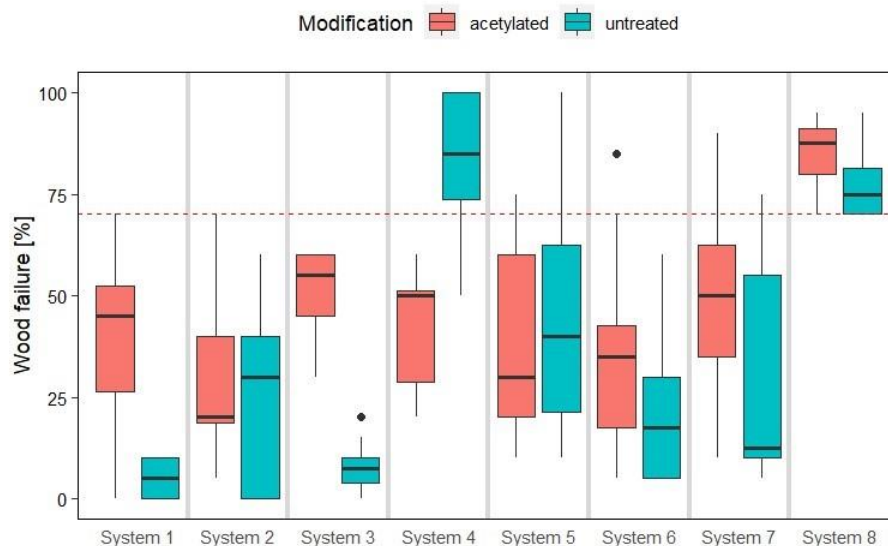


**Table 2: Set-up of selective bonding systems**

Combination	Adhesive	Prepress	Pressing
System 1	1-C PF	-	1.2 N/mm <sup>2</sup> for 20 minutes at 150°C
System 2	1-C PF	0,5 N/mm <sup>2</sup> for 10 minutes at 20-25°C	1.2 N/mm <sup>2</sup> for 20 minutes at 150°C
System 3	2-C PF	0,5 N/mm <sup>2</sup> for 10 minutes at 20-25°C	1.2 N/mm <sup>2</sup> for 20 minutes at 150°C
System 4	1-C PF	0,5 N/mm <sup>2</sup> for 10 minutes at 20-25°C	1.2 N/mm <sup>2</sup> for 20 minutes at 150°C
System 5	1-C PF	0,5 N/mm <sup>2</sup> for 10 minutes at 20-25°C	1.2 N/mm <sup>2</sup> for 20 minutes at 150°C
System 6	1-C PF	0,5 N/mm <sup>2</sup> for 10 minutes at 20-25°C	1.2 N/mm <sup>2</sup> for 20 minutes at 150°C
System 7	1-C PUR	-	1.2 N/mm <sup>2</sup> for 180 minutes at 20-25°C
System 8	2-C PRF	-	1.2 N/mm <sup>2</sup> for 180 minutes at 20-25°C



**Figure 1: Gelation time of acetylated and untreated wood powder combined with PF, PRF, and PUR adhesives**



**Figure 2: Wood failure percentage (WF) of various bonding systems based on EN 14374 (2004). The standard requires that all samples have a WF higher than 75% (marked as red-dotted line).**

## REFERENCES

DIN EN 14374:2005-02, Timber structures - Structural laminated veneer lumber - Requirements, 2004.

EN ISO 9396, Plastics - Phenolic resins - Determination of the gel time of resols under specific conditions using automatic apparatus (ISO 9396:1997), 2001.

Slabohm, M., Mayer, A.K., Militz, H. (2022). Compression of Acetylated Beech (*Fagus sylvatica* L.) Laminated Veneer Lumber (LVL). *Forests* 13, 1122. <https://doi.org/10.3390/f13071122>

Slabohm, M., Militz, H. (2022). Bonding performance of hot-bonded acetylated beech (*Fagus sylvatica* L.) laminated veneer lumber (LVL). *Wood Material Science & Engineering* 1-6. <https://doi.org/10.1080/17480272.2022.2124544>

Slabohm, M., Stolze, H., Militz, H. (2023). Evaluation of wet tensile shear strength and surface properties of finger-jointed acetylated beech (*Fagus sylvatica* L.) laminated veneer lumber. *European Journal of Wood Product*. <https://doi.org/10.1007/s00107-023-01970-3>

## **DAY 2**

### **SESSION FIVE**

#### **ANALYSIS**

## Oral 5.01 - VOCs Emission from Thermally Treated Poplar Solid Wood and Plywood

Corrado Cremonini<sup>1</sup>, Francesco Negro<sup>1</sup>, Roberto Zanuttini<sup>1</sup>

<sup>1</sup>Department of Agricultural, Forest and Food Sciences, University of Torino, Largo Paolo Braccini 2, 10095, Grugliasco, Italy. E: corrado.cremonini@unito.it; roberto.zanuttini@unito.it; francesco.negro@unito.it

**Keywords:** VOCs emission, poplar wood, plywood, thermal treatment

### ABSTRACT

Mainly developed as a method to make non-durable woods suitable for outdoor use, thermal treatment is now applied to wood and wood-based products for indoor use, such as parquet flooring. In these applications, where increased durability is not the main requirement, thermally treated wood is especially used for its improved dimensional stability and for colour variation. However, thermal treatment is known to increase the emission of volatile organic compounds (VOCs) from hardwoods, which is an undesirable side effect in indoor environments.

The aim of this study was to determine the VOCs emission from untreated (air-dried) and thermally treated poplar solid wood and plywood. Poplar (*Populus deltoides*, Lena clone) solid wood and 12 mm, 7 layer poplar plywood (Lena clone veneers bonded with ureamelamine-formaldehyde) were treated at 190 °C for 2 h using a thermo-vacuum process. The treatment temperature was chosen according to a recent study (Zanuttini *et al.* 2020), which proposed 190 °C as an interesting compromise for poplar plywood, taking into account the improvement in dimensional stability and durability, and the loss in mechanical performance. Five specimens (one per type) were randomly drawn from treated and untreated material. VOCs emission was measured 72 hours and 28 days after the thermal treatment according to the ISO 16000-6:2021 standard.

Table 1 shows that total VOCs emission from both thermally treated products was higher than that from untreated material, regardless the time elapsed. This is consistent with the results of studies reporting increased VOCs emission from thermally treated hardwoods (Pohleven *et al.* 2019). With the exception of the untreated material after 28 days, emission from plywood was generally lower than that from solid wood. This can be explained by the fact that the manufacturing of plywood, through hot drying and pressing and the associated vapour release, determines preliminary emission of VOCs.

**Table 1: VOCs emission after 72 and 28 days determined for poplar wood and plywood.**

Type	Treatment	VOCs emission after 72 h [µg/mt]	VOCs emission after 28 days [µg/mt]
Poplar wood	untreated	2,038	294
Poplar wood	190 °C, thermo vacuum	4,637	1,333
Plywood	untreated	950	347
Plywood	190 °C, thermo vacuum	2,710	878

Table 2 shows the emission determined for the different VOCs. Among the various comments, it is worth noting that formaldehyde emission was considerably lower from treated plywood than from untreated plywood. Instead, there was a significant increase in the emission of acetic acid after thermal treatment for both products.

**Table 2: Type (chemical name and Chemical Abstracts Service number) and amount of VOCs emitted after 28 days from poplar wood and plywood subjected to testing.**

VOC	CAS No.	SOLID WOOD		PLYWOOD	
		Untreated [µg/mt]	Thermally treated [µg/mt]	Untreated [µg/mt]	Thermally treated [µg/mt]
Formaldehyde	50-00-0	18	10	115	8
Acetaldehyde	75-07-0	11	36	2	10
Toluene	108-88-3	< 2	< 2	< 2	< 2
Methyl acetate	79-20-9	< 2	58	< 2	15
Acetone	67-64-1	< 2	8	< 2	< 2
Methyl ethyl ketone (MEK)	78-93-3	< 2	11	< 2	< 2
Hexanal	66-25-1	< 2	4	11	< 2
Furfural	98-01-1	0	5	0	2
N, N Dimethylform amide	68-12-2	5	56	3	38
Phenol	108-95-2	3	37	< 2	7
Acetic acid	64-19-7	245	1,108	216	793
Others minor components	NA	12	0	0	5
Total VOC	-	294	1,333	347	878

Clearly, the acceptable limits for indoor VOCs emission depend on the applicable legislation and standards, which vary according to several factors. In general, the focus on indoor air quality has increased steadily in recent years, and thresholds for VOCs emission are being constantly lowered. The thermal treatment of wood products for indoor use needs to successfully meet this trend. The results of this study, which should be further verified by wider sampling and comparisons, can be useful for the optimisation of adequate processes and product applications.

## REFERENCES

- ISO 16000-6:2021. Indoor air - Part 6: Determination of organic compounds (VVOC, VOC, SVOC) in indoor and test chamber air by active sampling on sorbent tubes, thermal desorption and gas chromatography using MS or MS FID.
- Pohleven, J., Burnard, M.D., Kutnar, A. Volatile organic compounds emitted from untreated and thermally modified wood – a review. *Wood and Fiber Science* **51**(3), 2019, 231-254.
- Zanuttini, R., Castro, G., Cremonini, C., Negro, F., Palanti, S. (2020). Thermo-vacuum treatment of Poplar (*Populus* spp.) plywood. *Holzforschung* **74**(1), 60-67.

## Oral 5.02 - Physical, Mechanical and Biological Tests of Solid Wood and Bio-Composites with BioPCM and thermal characteristics of Small-Scale Models in Three European Countries

Giovanni Aminti<sup>1</sup>, Andrea Atena<sup>1</sup>, Paolo Burato<sup>1</sup>, Michele Brunetti<sup>1</sup>, Gaye Köse Demirel<sup>2</sup>, Özge Nur Erdeyer<sup>2</sup>, Fabio De Francesco<sup>1</sup>, Mohamed Jebrane<sup>3</sup>, Jakub Grzybek<sup>4,5</sup>, Meysam Nazari<sup>3</sup>, Michela Nocetti<sup>1</sup>, Güliz Öztürk<sup>2</sup>, Sabrina Palanti<sup>1</sup>, Benedetto Pizzo<sup>1</sup>, Thomas Schnabel<sup>4,6</sup>, Federico Stefani<sup>1</sup>, Ali Temiz<sup>2</sup> and Nasko Terziev<sup>3</sup>

<sup>1</sup>National Research Council Institute of Bioeconomy, Via Madonna del Piano10 Sesto Fiorentino, Italy. E: giovanni.aminti@ibe.cnr.it; andrea.atena@ibe.cnr.it; paolo.burato@ibe.cnr.it; michele.brunetti@ibe.cnr.it; fabio.defrancesco@ibe.cnr.it; michela.nocetti@ibe.cnr.it; sabrina.palanti@ibe.cnr.it; benedetto.pizzo@ibe.cnr.it; federico.stefani@ibe.cnr.it

<sup>2</sup>Karadeniz Technical University, Dept. of Forest Industrial Engineering, 61080, Trabzon, Turkey. E: gkose@ktu.edu.tr; temiz@ktu.edu.tr

<sup>3</sup>Swedish University of Agricultural Sciences, Dept. of Forest Biomaterial and Technology, Box 7008, 756 51 Uppsala, Sweden. E: mohamed.jebrane@slu.se; meysam.nazari@slu.se; nasko.terziev@slu.se

<sup>4</sup>Department of Green Engineering and Circular Design, Salzburg University of Applied Sciences, 5431 Kuchl, Austria. E: jakub.grzybek@fh-salzburg.ac.at; thomas.schnabel@fh-salzburg.ac.at

<sup>5</sup>Faculty of Forestry and Wood Technology, Department of Wood Science and Technology, Mendel University in Brno, 613 00 Brno, Czech, Republic

<sup>6</sup>Faculty for Design of Furniture and Wood Engineering, Transilvania University of Brasov, Romania

**Keywords:** bio-based phase change material, decay fungi, internal bonding, thermal energy storage, termites, wood composite

### ABSTRACT

The study reports the results of the M-ERA Net project BIO-NRG-STORE. The research project is a collaboration of three universities and one research institute from Austria, Sweden, Turkey and Italy. The research includes physical-mechanical and biological tests on solid wood and wood composites, both impregnated with bioPCM (bio-based phase change material). Furthermore, the design of small-scale test models to evaluate the thermal characteristics of the innovation in outdoor exposure is reported.

BioPCM based on fatty acid eutectic mixtures such as capric and stearic acid or ethyl palmitate ester were used to impregnate solid wood or wood particles. The impregnated particles used to prepare composites using starch or epoxidized linseed oil as binders. Following tests were performed according to ISO and European standards to reveal the mechanical and physical behaviour of the materials, i.e., absolute dry density, density at 12% RH, compressive strength, shear strength, internal bonding, modulus of rupture and elasticity (MOR and MOE), hardness, dimensional stability and water vapour permeability. In addition, the biological resistance of the bioPCM-wood composites to decay fungi (EN 113-3), termites (EN 117) and moulds was tested. The assessment against mould growth was carried out according to E24-06 AWPA (American Wood Protection Association Standard, 2008) with some modifications.



**Figure 1: Wood particleboard with encapsulated ethyl palmitate ester as bioPCM processed by the Swedish team for testing the internal bonding (left). A set for testing the susceptibility to mould growth of the bio-composites (right).**

The tested wood composites with bioPCM were resistant to mould growth. The resistance to fungal attack, expressed in mass loss, was only high for the bio-composite containing ethyl palmitate ester. However, the used wood particles and bioPCM composite were resistant to termite attack. The validity of the tests was confirmed by a severe termite attack on the untreated pine control samples. The results of the physical tests showed that the wood composites containing ethyl palmitate ester had a higher vapour permeability than similar products available on the market, while the other (the wood composite impregnated with fatty acid) had a lower permeability.

Small wood building models were produced using engineered wood flooring and wallboards hosting bioPCM. The wallboards are used as interior walls in the structure models to collect data for material performance and energy saving effect. Two identical building models (formed as cubes of 1m<sup>3</sup> inner volume) for each demo site in Sweden (Uppsala), Italy (Florence) and Austria (Kuchl) were designed and produced to demonstrate the project concept. One cube is used as a baseline and compared with the one integrating the novel materials of the concept. The models have integrated heat and ventilation systems to ensure a thermal comfort of 21°C. The models are equipped with temperature sensors to monitor the inner wall surface and indoor air temperatures. The measurements in the cubes are ongoing to collect data for numerical simulations and further improvement of the building structure thermal design by bioPCM containing composite materials.

The study demonstrates the use of bio-based resources with specific functional properties that are further upgraded by bio-manufacturing and bio-technology approaches to develop composite materials with added functionalities. Wood composites with bioPCM can exhibit mechanical properties comparable to those of other commercial wood-based materials. In addition, the study proves that bio-binders and bioPCM in the wood composite are not susceptible to biological attack. The results of the ongoing field test show significant saving of thermal energy by the materials with incorporated bioPCM.

## Oral 5.03 - Comprehensive Multi-Scale Investigation of Heat Treated Wood at Room or Elevated Temperature: Summary of Our Decade's Researches

Siqun Wang<sup>1</sup>, Dong Xing<sup>1,2</sup>, Xinzhou Wang<sup>1,3</sup>, Deliang Xu<sup>1,3</sup>, Yujie Meng<sup>1,4</sup>, Jian Li<sup>5</sup>, Timothy M. Young<sup>1</sup>

<sup>1</sup>Center for Renewable Carbon, University of Tennessee, Knoxville, TN, USA. E: wang@utk.edu

<sup>2</sup>College of Materials Science and Art Design, Inner Mongolia Agriculture University, Hohhot 010018, China. E: xingdong@imau.edu.cn

<sup>3</sup>College of Materials Science and Engineering, Nanjing Forestry University, Nanjing, P.R. People's Republic of China. E: xzwang@njfu.edu.cn

<sup>4</sup>KLA Corporation, Knoxville, TN 37830, USA. E: Yujie.Meng@kla.com

<sup>5</sup>College of Materials Science and Engineering, Northeast Forestry University, Harbin 150040, PR China. E: nefulijian@163.com

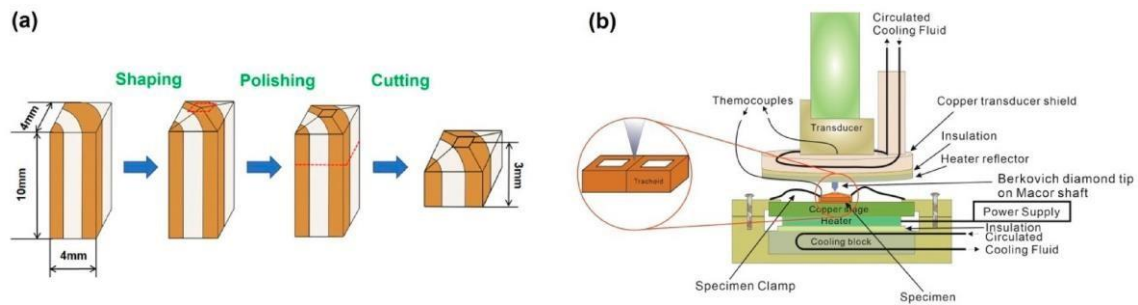
**Keywords:** carbonization, creep, graphene, hardness, modulus, nanoindentation, quasi-static, scanning thermal microscopy

### ABSTRACT

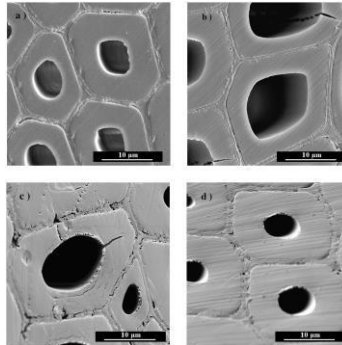
As a renewable resource, wood is an inherently high strength-to-weight ratio material that can directly be used for numerous applications. However, the usefulness of wood is limited by its low resistance to biodeterioration, lack of dimensional stability, and poor durability. Among various techniques for overcoming these problems, heat treatment has been most frequently used in recent years. When heating wood under oxygen free and much higher temperature conditions, the structures and compositions of wood will change to those of charcoal, even graphene, nano carbon tube, diamond et al. The transitional characteristics from wood to carbon materials warrant investigation. What happens if wood structure made of heated wood exposes to high temperature during service? How about under a load for a long period of time at the room or elevated temperature? To answer those questions, our group has conducted a series of researches under microscale and nanoscale in the past decade. In this presentation, we will highlight some of our researches, including:

1. Understanding wood cell wall or polymer change under different temperature if we carbonized *Quercus rubra* wood fiber cell walls (Xu *et al.* 2017);
2. Performing an electron microscopy investigation to understand the relationship between the microstructure and properties of carbonized cellulose and lignin (softwood kraft lignin) relative to the structure of the original biomass components (Meng *et al.* 2021);
3. Conducted *in-situ* measurement of heat-treated wood cell wall at elevated temperature by nanoindentation, showing in Figure 1 (Xing *et al.* 2016);
4. Explaining effects of thermal modification on the physical, chemical and micromechanical properties of Masson pine wood (*Pinus massoniana* Lamb.). (Wang *et al.* 2018);
5. Comparison of the chemical and micromechanical properties of *Larix* spp. after eco-friendly heat treatments measured by *in-situ* Nanoindentation (Figure 2) (Xing *et al.* 2020)
6. Reporting temperature-dependent creep behavior and quasi-static mechanical properties of heat-treated wood (Xing *et al.* 2021);
7. Finally discussing multi-scale investigation of the mechanical properties of Loblolly pine wood at elevated temperature (Wang *et al.* 2022).





**Figure 1: Schematic representation of the elevated temperature nanoindentation: (a) wood sample preparation, (b) nanoindentation test (Wang *et al.* 2022).**



**Figure 2: Microstructure observation of wood by SEM: (a) an untreated sample; (b) a heat-treated sample under a nitrogen atmosphere; (c) a heat-treated sample under an air atmosphere; and (d) a heat-treated sample under an oil atmosphere (Xing *et al.* 2020).**

## REFERENCES

- Meng, Y., Contescu, C.I., Liu, P., Wang, S., Lee, S.-H., Guo, J. and Young, T. M. (2021). Local Structure Investigation of Disordered Carbons from Cellulose and Lignin by Scanning Transmission Electron Microscopy (STEM). *Wood Science and Technology*, **55**, 587-606.
- Wang, X., Chen, X., Xie, X., Wu, Y., Zhao, L., Li, Y. and Wang, S. (2018). Effects of thermal modification on the physical, chemical and micromechanical properties of Masson pine wood (*Pinus massoniana* Lamb.). *Holzforschung*, **72** (12), 1063-1070 (DOI: 10.1515/hf-2017-0205).
- Wang, X., Huang, Y., Lv, C., Wang, J., Wang, S., and Yao, Y. (2022). Multi-scale investigation of the mechanical properties of Loblolly pine wood at elevated temperature. *Wood Material Science & Engineering*, 1-8 (<https://doi.org/10.1080/17480272.2022.2053203>).
- Xing, D., Li, J. and Wang, S. (2020). Comparison of the chemical and micromechanical properties of *Larix* spp. after eco-friendly heat treatments measured by in situ nanoindentation. *Sci Rep*, **10**, 4358 (DOI: <https://doi-org.proxy.lib.utk.edu/10.1038/s41598-020-61314-6>).
- Xing, D., Li, J., Wang, X. and Wang, S. (2016). In situ measurement of heat-treated wood cell wall at elevated temperature by nanoindentation. *Industrial Crops and Products*, **87**, 142-149. Xing, D., Wang, X. and Wang, S. (2021). Temperature-dependent creep behavior and quasi-static mechanical properties of heat-treated wood. *Forests*, **12**, 968.
- Xu, D., Tao, D., Li, Y., Zhang, Y., Zhou, D. and Wang, S. (2017). Transition characteristics of a carbonized wood cell wall investigated by scanning thermal microscopy (SThM). *Wood Science and Technology*, **51**, 831-843 (DOI: 10.1007/s00226-017-0919-4).

## Oral 5.04 - Resistance of Thermally and Chemically Modified Timber Against Soft Rot and Findings to Improve the Lab Test

Wolfram Scheiding<sup>1</sup>, Kordula Jacobs<sup>1</sup>, Christian Brischke<sup>2</sup>, Susanne Bollmus<sup>3</sup>

<sup>1</sup>Institute of Wood Technology Dresden (IHD), Zellescher Weg 24, 01217 Dresden, Germany. E: wolfram.scheiding@ihd-dresden.de, kordula.jacobs@ihd-dresden.de

<sup>2</sup>Thünen Institute of Wood Research, Leuschnerstraße 91, Hamburg-Bergedorf, Germany. E: christian.brischke@thuenen.de

<sup>3</sup>Georg-August-Universität Göttingen, Wood Biology and Wood Products (UGOE), Büsgenweg 4, 37077 Göttingen, Germany. E: sbollmus@gwdg.de

**Keywords:** chemically modified timber, lab test, soft rot, thermally modified timber

### ABSTRACT

The durability classification is an important basis for the service life estimation and provides information to the practice to select appropriate wood products. The extended scope of EN 350:2016 enables to determine not only the natural durability of wood (as before), but also the durability of other materials, e.g. of modified wood. However, a proper sampling and testing of these materials is not possible due to lack of sufficiently accurate and concrete information. In order to overcome the deficits and to improve the standard, the research project DURATEST<sup>1</sup> was initiated, operated by the Institute of Wood Technology Dresden (IHD) and the Georg-August-Universität Göttingen, Wood Biology and Wood Products (UGOE) from 2020-2023 (Scheiding et al. 2020).

Within DURATEST, the durability of natural, preservative-treated and modified timber against wood-destroying basidiomycete and soft rot fungi was investigated in lab and field tests. The samples were taken from different zones of trunks, boards and beams and examined separately. The test results were analysed with statistical methods and critically evaluated (see also Brischke *et al.* 2023 a, b). In addition, suggestions for improving the evaluation of test results were deduced.

The soft rot tests were performed according to CEN/TS 15083-2:2015 (Determination of the natural durability of solid wood against soft rotting micro-fungi). Usually the evaluation is based on mass loss for hardwoods and on MOE loss for softwoods, but the mass loss was determined for all materials. As an example, table 1 shows the test results with the mass loss as measure for resistance. The resulting durability class (DC) of thermally modified Scots pine from MOE loss (4v) was one class worse than from mass loss (3v), but all DC of the CMT variants were equal from both parameters. Generally, the soft rot tests confirmed the previous experiences with modified timber and pointed out the differences between TMT and CMT.

The full paper will compare the results of modified materials with those determined with naturally durable wood species, like larch, oak and black locust, which were investigated in the project as well.

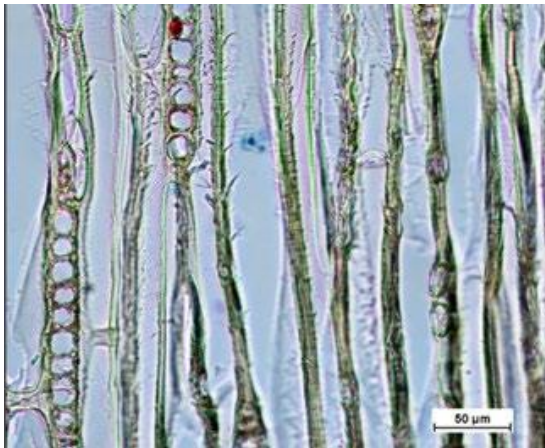
---

<sup>1</sup> “Development of sampling, testing and classification methods for determining the biological durability of wood and wood products”

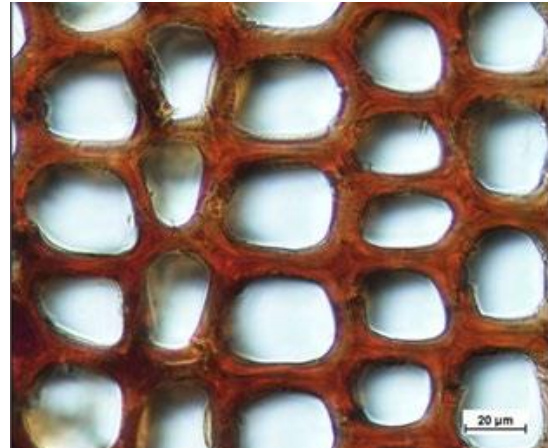
**Table 1: Results of soft rot tests and durability classification based on mass loss**

Material	x-value (mass loss)	Frequency distribution of specimens [%]					DC (EN 350)
		DC 1	DC 2	DC 3	DC 4	DC 5	
Therm. mod. Black alder (board)	0,11	47	43	10	-	-	1-2
Therm. mod. Scots pine (board)	0,19	20	33	47	-	-	2v
Furfuryl. Radiata pine (board)	0,01	100	-	-	-	-	1
Acet. Radiata pine (board)	0,00	100	-	-	-	-	1
Acet. Radiata pine (beam, outer)	0,00	100	-	-	-	-	1
Acet. Radiata pine (beam, inner)	0,00	100	-	-	-	-	1

Additionally to the durability tests, microscopic evaluation of the modified specimens was done. According to CEN/TS 15083-2 this is required with the reference material, to prove a sufficient presence of soft rot. At the CMT specimens, no signs of fungal attack were found (fig. 2 and 3), what confirmed the results and its high resistance.



**Figure 2: Micrograph (longitudinal section) of acetylated Radiata; no signs of attack**



**Figure 3: Micrograph (longitudinal section) of furfurylated Radiata; no signs of attack**

By evaluating the soft rot tests, different suggestions were deduced to improve the test method. These suggestions are provided to the responsible standardisation committees of CEN/TC 38. Currently the work started to transfer CEN/TS 15083-2 to a European standard as part 2 of a revised, two-part EN 807 (EN 807-1 will cover testing of preservatives). The full paper will give more detailed information.

## REFERENCES

- Brischke C, Sievert M, Schilling M, Bollmus S (2023a) Laboratory durability testing of preservative-treated wood products. *Forests* 14:1001, DOI:10.3390/f14051001
- Brischke C, Haase F, Bollmus S, Bächle L (2023b): Statistical analysis of wood durability data and its effect on a standardised classification scheme. *Standards* 3: 210-226
- Scheiding W, Jacobs K, Bollmus S, Brischke C (2020): Durability classification of treated and modified wood – approaching a guideline for sampling, testing, and statistical analysis. The International Research Group on Wood Protection, IRG/WP/20-20676

## Oral 5.05 - The Chemical Interactions between Phenolic Resin and Wood Studied by Liquid-State NMR Spectroscopy

Carlo Kupfernagel<sup>1</sup>, Daniel Yelle<sup>2</sup>, Morwenna J. Spear<sup>1</sup>, Andrew Pitman<sup>3</sup>, Graham A. Ormondroyd<sup>1</sup>

<sup>1</sup>BioComposites Centre, Bangor University, UK. E: crk20scf@bangor.ac.uk

<sup>2</sup>Forest Products Laboratory, Madison, USA

<sup>3</sup>BM Trada, Buckinghamshire, UK

**Keywords:** cell wall solubilisation, PUF resin, solution-state NMR, wood modification

### ABSTRACT

Wood modification with thermosetting resins is an increasingly popular method aiming to expand the number of applications for readily available plantation timber (Hill 2006). Phenol urea formaldehyde (PUF) resin is impregnated into the timber, which is dried, and subsequently heat cured (Kupfernagel *et al.* 2021, 2022). This treatment renders the raw material dimensionally stable and imparts the ability to withstand wood destroying fungi and insects. The mode of action in doing so is typically considered a passive modification, but covalent bonds between the PUF resin and wood might form at low abundance. Since these covalent bonds are rare and structurally similar to linkages that occur in wood itself, a high-resolution method is required to accurately observe the formation of new bonds. In the current study we used liquid-state <sup>1</sup>H-<sup>13</sup>C-heteronuclear single quantum coherence (HSQC) NMR experiments to identify chemical bonds in modified and unmodified wood with high accuracy. This method has been used in the past to detect even rare covalent bonds in modified wood. During the sample preparation, a ball-milled wood powder is dissolved in DMSO-d<sub>6</sub>, which yields a viscous gel that can be handled like a liquid in solution-state NMR experiments (Yelle *et al.* 2008, Kim & Ralph 2010). Lime and poplar samples, that were previously compared in anti-swelling efficiency tests, have been used for the NMR analysis (Kupfernagel *et al.* 2023). While the same kind of covalent bonds are formed in different timbers, their abundance was shown to be significantly different, which could be due differences in the chemical composition of the two wood species.

### REFERENCES

Hill, C. A. S. Wood Modification Chemical, Thermal and Other Processes. Christian V. Stevens (ed.), John Wiley & Sons Ltd, Chapter 2, pp. 19–49. doi:10.1002/0470021748.

Kim, H. and Ralph, J. (2010) Solution-state 2D NMR of ball-milled plant cell wall gels in DMSO-d<sub>6</sub>/pyridine-d<sub>5</sub>, *Organic and Biomolecular Chemistry*, **8** (3), pp. 576–591. doi: 10.1039/b916070a.

Kupfernagel, C., Spear, M., Pitman, A. & Ormondroyd, G. A. (2021) Wood Modification with Phenol-Formaldehyde-Resin and its Influence on the Dimensional Stability of Homegrown and Imported Hardwoods, in *The 17th Annual Meeting of the Northern European Network for Wood Science and Engineering*. Kaunas, Lithuania, pp. 12–15.

Kupfernagel, C., Spear, M., Pitman, A. & Ormondroyd, G. A. (2022) Effekt von Prozessvariablen bei Holzmodifikation mit PF-Harz., in *Holzwerkstoffkolloquium*. Dresden, pp. 101–104.

Kupfernagel, C., Spear, M., Pitman, A. & Ormondroyd, G. A. (2023) Wood modification with phenol urea formaldehyde (PUF) resin: The influence of wood species selection on the dimensional stability., *European Journal of Wood and Wood Products.*, **81**, pp. 5 - 19, <https://doi.org/10.1007/s00107-022-01893-5>

Yelle, D. J., Ralph, J. and Frihart, C. R. (2008) Characterization of nonderivatized plant cell walls using high-resolution solution-state NMR spectroscopy, *Magnetic Resonance in Chemistry*, **46**(6), pp. 508–517. doi: 10.1002/mrc.2201.

## Oral 5.06 - Decay and Termite Resistance on Sapwood, Transition Wood, and Heartwood of Short Rotation Teak Wood by Chemical and Thermal Modification

Resa Martha<sup>1,2</sup>, Béatrice George<sup>1</sup>, Istie S. Rahayu<sup>2</sup>, Wayan Darmawan<sup>2</sup>, Philippe Gérardin<sup>1</sup>

<sup>1</sup>Université de Lorraine, INRAE, LERMAB, 54000 Nancy, France E: resa.martha@univ-lorraine.fr; beatrice.george@univ-lorraine.fr; philippe.gerardin@univ-lorraine.fr

<sup>2</sup>Department of Forest Products, Faculty of Forestry and Environment, IPB University, Bogor 16680, Indonesia. E: istiesr@apps.ipb.ac.id; wayandar@indo.net.id

**Keywords:** chemical and thermal modification, heartwood, sapwood, termite resistance, transition wood

### ABSTRACT

Short rotation teak wood is susceptible to by biodeterioration attack, particularly fungal and subterranean termites. The objective of this work was to investigate the effect of chemical and thermal treatment on decay and termite resistance on sapwood, transition wood, and heartwood of short rotation teak. Furfurylation (FA), thermal treatment, and combination of chemical and thermal treatment using glycerol-maleic anhydride (GMAthermal) were performed on sapwood, heartwood, and their transition (50:50 sapwoodheartwood). Two white rot fungi (*Pycnoporus sanguineus* and *Trametes versicolor*) and two brown rot fungi (*Coniophora puteana* and *Rhodonia placenta*) were used to evaluate the decay resistance of short rotation modified wood. Termite resistance of short rotation teak wood with chemical and thermal modification was investigated in field tests. Small stakes (200 × 20 × 10 mm<sup>3</sup> (L, R, T)) were embedded in-ground for 12 weeks to determine their resistance against subterranean termite at the research field of Faculty of Forestry, IPB University, Indonesia. The mean retention values of FA for sapwood, transition, and heartwood were 244.45 kg m<sup>-3</sup>, 186.29 kg m<sup>-3</sup>, and 182.43 kg m<sup>-3</sup>, respectively. Meanwhile, the retention values of GMA for sapwood, transition, and heartwood were 60.41 kg m<sup>-3</sup>, 39.38 kg m<sup>-3</sup>, and 35.20 kg m<sup>-3</sup>, respectively. The mass change due to FA treatment in sapwood, transition, and heartwood were 30.91%, 23.36%, and 23.16%, respectively. Weight loss due to fungal decay were similar between transition sapwood and transition heartwood after FA and GMA-thermal at 220 °C treatments. The results indicate that short rotation teak board consisting of sapwood and heartwood was homogenized after FA and GMA-thermal at 220 °C treatments. Weight loss of FA and GMA-thermal at 220 °C treatment against termite attacks presented excellent durability against subterranean termites. The FA and GMA treatment could be valuable to protect the short rotation teak wood against decay and termite.

## Oral 5.07 - The Influence of Moisture Content and Thermal Modification on the Non-Linearity in Mode I Fracture of Spruce Wood

Miran Merhar<sup>1</sup> and Rostand Moutou Pitti<sup>2,3</sup>

<sup>1</sup>Department of Wood Science and Technology, Biotechnical Faculty, University of Ljubljana, Slovenia. E: miran.merhar@bf.uni-lj.si

<sup>2</sup>Université Clermont Auvergne, Clermont Auvergne INP, CNRS, Institut Pascal, Clermont Ferrand, France. E: rostand.moutou\_pitti@uca.fr

<sup>3</sup>CENAREST, IRT, BP 14070, Libreville, Gabon. E: rostand.moutou\_pitti@uca.fr

**Keywords:** linear fracture mechanics, strain energy release rate, thermal modification, wood

### ABSTRACT

The use of wood for building purposes is increasing from year to year (Kitek Kuzman *et al.* 2018). However, since wood is biodegradable, it must be protected. One environmentally friendly protection is the thermal modification of wood, which usually leads to a deterioration of the mechanical properties of wood, including fracture toughness. Different approaches are known for determining the fracture toughness (Merhar *et al.* 2013, Moutou Pitti *et al.* 2014, Xavier *et al.* 2014), depending on the type of fracture mechanics. In order to use linear fracture mechanics, the most important conditions (Bucur 2011) must be fulfilled, namely: the necessity of assuming the existence of a crack; the effects of fracture process zone are in the vicinity of the crack tip; and the available energy goes into the creation of a single new fracture zone.

In the case of a larger fracture process zone (FPZ) and other non-linear elements such as fibre bridging, the principles of non-linear fracture mechanics must be applied. Despite the above conditions, linear fracture mechanics can be applied to wood in certain cases. This is the case when both the crack and the ligament are large enough so that the FPZ is small compared to the length of the crack and the length of the ligament.

According to Griffith (Smith *et al.* 2003), the energy required for crack progression, called strain energy release rate, can be given by

$$G_c = \frac{d(F-U)}{dA} \quad (1)$$

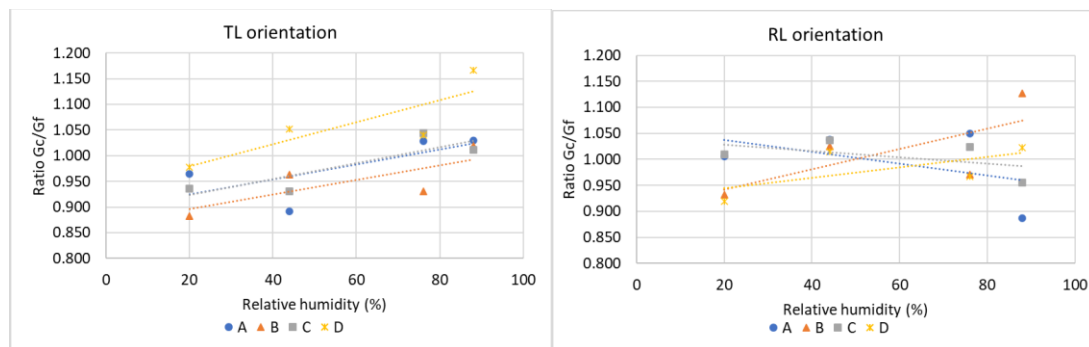
where  $F$  is external work of load,  $U$  strain energy and  $dA$  is the incremental change in crack area. At the same time, the specific fracture energy required for the formation of new surfaces is

$$G_f = \frac{W}{A} \quad (2)$$

where  $W$  is the energy required to create new surfaces and  $A$  is the new surface area created.  $G_f$  is thus determined on the basis of total surface separation when a full load-deformation diagram (including strain softening) is required, while  $G_c$  is based on the energy required for crack progression and full surface separation is not required. If the FPZ has a minimum size, linear fracture mechanics can be used. In this case  $G_f$  and  $G_c$  are equal.

This paper investigates the effects of thermal modification and moisture content of wood on the degree of nonlinearity of spruce wood in fracture mode I. Spruce wood was thermally modified at three different levels of thermal modification. Samples of unmodified and modified wood were exposed to different moisture contents. Double cantilever beam (DCB) specimens were made and then tested in fracture mode I. From the force and crack mouth opening displacement measurements,  $G_c$  and  $G_f$  were determined for all specimens in TL and RL orientation.

The measurements showed that both  $G_c$  and  $G_f$  increase with the humidity of the sample, while they decrease with the degree of thermal modification. The ratio between  $G_c$  and  $G_f$  increases with moisture in TL samples (Figure 1), while it increases in some cases and decreases in others in RL samples. The study shows that the degree of non-linearity varies with both the moisture content of the wood and the degree of thermal modification.



**Figure 1. Ratio of  $G_c/G_f$  for various relative humidities, orientations and thermal modifications (A-unmodified; B-modified at 180 °C; C - modified at 200 °C; D - modified at 230 °C)**

## REFERENCES

- Bucar, B. (2010). *Delamination in Wood, Wood Products and Wood-Based Composites*. Springer Dordrecht.
- Kitek Kuzman, M., Klarić, S., Pirc Barčič, A., Vlosky, R.P., Janakieska, M.M. and Grošelj, P. (2018). Architect perceptions of engineered wood products: an exploratory study of selected countries in Central and Southeast Europe. *Construction and Building Materials*, 179, 360–370. doi:10.1016/j.conbuildmat.2018.05.164
- Merhar, M., Bucar, D.G. and Bucar, B. (2013). Mode I critical stress intensity factor of beech wood (*fagus sylvatica*) in a TL configuration: a comparison of different methods. *Drvna Industrija*, 64 (3), 221–229. doi:10.5552/drind.2013.1253
- Moutou Pitti, R., Badulescu, C. and Grédiac, M. (2014). Characterization of a cracked specimen with full-field measurements: direct determination of the crack tip and energy release rate calculation. *International Journal of Fracture*, 187 (1), 109–121. doi:10.1007/s10704-013-9921-5
- Smith, I., Landis, E. and Gong, M. (2003). *Fatigue and fracture of wood*. Chichester, New York: Wiley.



Xavier, J., Oliveira, M., Monteiro, P., Morais, J.J.L. and de Moura, M.F.S.F. (2014). Direct evaluation of cohesive law in mode I of pinus pinaster by digital image correlation. *ExperimentalMechanics*, 54 (5), 829–840. doi:10.1007/s11340-013-9838-y

## **DAY 2**

### **SESSION SIX**

#### **THERMAL MODIFICATION**

## Oral 6.01 - Influence of Thermal Modification on Fatigue Life of Norway Spruce Wood

Miha Humar<sup>1</sup>, Davor Kržišnik<sup>1</sup>, Boštjan Lesar<sup>1</sup>, Gorazd Fajdiga<sup>1</sup>

<sup>1</sup>University of Ljubljana, Biotechnical Faculty, Department of Wood Science and Technology, Jamnikarjeva 101, SI1000, Ljubljana, Slovenia. E: miha.humar@bf.uni-lj.si; davor.krzisnik@bf.uni-lj.si; bostjan.lesar@bf.uni-lj.si; gorazd.fajdiga@bf.uni-lj.si

**Keywords:** fatigue, Norway spruce, performance, thermally modified wood

### ABSTRACT

Thermal modification is one of the most important modification processes. It has been applied to several wood species, including Norway spruce (*Picea abies*). In general higher modification, temperatures result in increased durability and decreased mechanical properties. Therefore thermally modified wood is not to be used for load bearing applications (Tjeerdsma *et al.* 1998). Most current mechanical studies of thermally modified wood have been performed with static tests: bending, compression, and tension. To our best knowledge, we could not identify many dynamic tests performed on thermally modified wood, except for impact bending tests and HEMI (High Energy Multiple Impact) tests.

Dynamic mechanical properties of materials are also becoming increasingly important in the design of modern structures. In cases where wood elements are frequently exposed to adverse environmental conditions (e.g., wind) or dynamic environments (e.g., vibration or repeated mechanical loading), failure of the member may occur due to material fatigue. Repeated dynamic loading causes permanent deformation, cracking, and fracture in the material, even at lower stresses than static loading. The dynamic strength of the material can be 20% to 60% lower than the static strength due to fatigue under dynamic loading. The objective of the respective manuscript is to determine the fatigue life of thermally modified wood (Clorius, 2001).

Norway spruce planks were modified according to the Silvapro procedure at 210 °C and 230 °C for three h in semi-anoxic conditions (Rep *et al.* 2012). Mass loss after modification was determined on parallel specimens. Afterwards, planks were processed into specimens for fatigue ( $1.0 \times 1.0 \times 15.0$  cm<sup>3</sup>), density and porosity assessment ( $1.0 \times 1.0 \times 1.0$  cm<sup>3</sup>) and DVS analysis. This study performed static and dynamic tests in the commercial test facility DMA Electroforce 3310 Series III. Three-point bending tests were performed. The magnitude of the fatigue load was determined according to the static breaking force, namely, 70%, 80%, and 90% of the maximum static force was chosen for fatigue testing. The results are expressed as the number of load cycles N until the occurrence of fracture and can be presented in the so-called Wöhler curve or the curve of dynamic strength of the material. Before the fatigue testing, the modulus of elasticity was determined for each specimen. In addition, minimum and maximum deformation was recorded as a function of the number of fatigue cycles. The experiment was performed in 15 replicates.

The thermal modification resulted in mass loss between 5.3% (210 °C) and 11.5% (230 °C), representing two distinctive steps. Respective mass losses are in line with the Silvapro process. The mass loss reflects in density as well. The density of wood modified at higher temperature (230 °C; 412 kg/m<sup>3</sup>) compared to the density of reference specimens (456 kg/m<sup>3</sup>) (Table 1). Thermal modification improves the sorption properties of wood as resolved by the DVS study. As expected, thermal modification influences mechanical properties. The modulus of rupture decreased from 84 N/mm<sup>2</sup> measured at control wood to 78 N/mm<sup>2</sup> (210 °C) and 69 N/mm<sup>2</sup> determined at wood modified at the highest temperature (230 °C). Respective values align with the literature data (Humar *et al.* 2017).

However, thermal modification considerably affects the fatigue life of the wood. In general, it can be concluded that thermally modified wood's fatigue life decreases with increasing modification temperature. The effect of thermal modification on the fatigue life of the wood is less prominent at lower load magnitudes. During fatigue testing performed at 70% of maximum load, the fatigue life was reduced by 37% from 1.949.677 cycles at control specimens to 1.222.231 cycles determined at wood modified at 230 °C. On the other hand, fatigue life determined at 90% load decreased by 66% at specimens modified at the highest temperature (Table 1).

The results of the respective test indicate that the modification has a considerable effect not only on static mechanical properties but on the fatigue life of the wood. The fatigue life of wood decreased with increasing modification temperatures. This should be considered when using thermally modified wood for any kind of load-bearing applications.

**Table1: Results of the fatigue testing and basic properties of the wood used (n = 15)**

Modification temperature	No. of cycles at respective load			Max load (N)	Density (kg/m <sup>3</sup> )
	70% of max load	80% of max load	90% of max load		
Control	1.949.677	887.912	296.451	466	456
210°C	1.908.873	680.772	212.327	432	427
230°C	1.222.231	449.986	101.246	383	412

## ACKNOWLEDGEMENTS

The authors acknowledge the financial support of the Slovenian Research Agency (ARIS) within research programme P4-0015 (Wood and lignocellulosic composites), and the Infrastructure Centre (IC LES PST 0481-09). The Ministry of Agriculture, Forestry and Food, supported part of the published research under the V4-2017 project.

## REFERENCES

- Clorius, C. O. (2001). *Fatigue in Wood: An investigation in tension perpendicular to the grain*. Technical University of Denmark.
- Humar, M., Kržišnik, D., Lesar, B., Thaler, N., Ugovšek, A., Zupančič, K., & Žlahtič, M. (2017). Thermal modification of wax-impregnated wood to enhance its physical, mechanical, and biological properties. *Holzforschung*, 71(1), 57–64. <https://doi.org/10.1515/hf-2016-0063>
- Rep, G., Pohleven, F., & Kosmerl, S. (2012). Development of the industrial kiln for thermal wood modification by a procedure with an initial vacuum and commercialisation of modified Silvapro wood. In M. H. and M. P. D. Jones, H. Militz, M. Petrič, F. Pohleven (Ed.), *Proceedings of the 6th European Conference on Wood Modification* (pp. 11–17). University of Ljubljana.
- Tjeerdsma, B. F., Boonstra, M., Pizzi, A., Tekely, P., & Militz, H. (1998). Characterisation of thermally modified wood: Molecular reasons for wood performance improvement. *Holz Als Roh - Und Werkstoff*, 56(3), 149–153. <https://doi.org/10.1007/s001070050287>.

## Oral 6.02 - Detection of the Aromatic Profile of Different Thermally Modified Wood Species

Valentina Lo Giudice<sup>1</sup>, Angelo Rita<sup>2</sup>, Luigi Todaro<sup>1</sup>

<sup>1</sup>School of agricultural, forestry, food and environmental science. University of Basilicata. V.le Ateneo Lucano 10, 85100 Potenza, Italy. E: valentina.logiudice@unibas.it; luigi.todaro@unibas.it

<sup>2</sup>Department of Agriculture, University of Naples Federico II, Via Università 100, 80055 Portici, NA, Italy. E: angelo.rita@unina.it

**Keywords:** chemical compounds, electronic nose, heat-treated wood, sensors

### ABSTRACT

As a consequence of the use of thermo-modified wood material, especially for indoor applications, the type of chemical compounds produced by these products plays an important role in indoor environmental issues and human health (Nikoutadbir *et al.* 2023, Kamdem *et al.* 2000). The thermal modification method may influence the chemical properties of wood. Our study aimed to evaluate the effect of thermal modification on the aromatic profile of seven different wood species namely *Cedrus deodara* Roxb, *Pinus laricio* L, *Alnus cordata* L, *Populus nigra* L, *Fagus sylvatica* L (thermo-modified at a temperature of 200 °C), *Quercus Cerris* L, and *Castanea sativa* Mill (thermo-modified at a temperature of 170 °C). An olfactory machine so-called PEN 3 AIRSENSE electronic nose (e-nose) (Fig. 1) consisting of 10 different doped semi-conductive metaloxide gas sensors (MOS) (Tab. 1) was used for this purpose. Wood boards were prepared for thermal modification and then cut into small wood blocks for autoclave sterilization to obtain the final extracts in water solution for olfactory detection. Multivariate analysis, including principal component analysis (PCA) and linear discriminant analysis (LDA) provided an evaluation of the ability of the e-nose sensor array to associate a response pattern with the corresponding class of compounds. The loadings analysis was used to identify the sensors responsible for discrimination in the current pattern file. Results showed that the W5S sensor, with its characteristic of reacting on nitrogen oxides (NO<sub>2</sub>), explained most of the variance attributed to the odor of both thermo-modified and unmodified woods.



Figure 1: Electronic nose (e-nose) AIRSENSE Analytics GmbH, Schwerin, Germany.

**Table 1: Sensors used and their main applications in PEN 3 (Gómez *et al.* 2006)**

Number in array	Sensor name	General description	Reference
1	W1C	Aromatic compounds	Toluene, 10mg/kg
2	W5S	React on nitrogene oxides	NO <sub>2</sub> , 1mg/kg
3	W3C	Ammonia, aromatic compounds	Benzene, 10mg/kg
4	W6S	Mainly Hydrogen	H <sub>2</sub> , 0.1MG/KG
5	W5C	Alkanes, aromatic compounds	Propane, 1mg/kg
6	W1S	Methane	CH <sub>3</sub> , 100 mg/kg
7	W1W	Sulfur compounds, terpenes, limonene, pyrazine	H <sub>2</sub> S, 1mg/kg
8	W2S	Alcohol, partially aromatic compounds	CO, 100mg/kg
9	W2W	Aromatic compounds, sulfur organic compounds	H <sub>2</sub> S, 1mg/kg
10	W3S	React on high concentrations > 100mg/kg, sometimes very selective (methane)	CH <sub>3</sub> , 10 CH <sub>3</sub> , 100mg/kg

## REFERENCES

- Gómez, A.H., Wang, J., Hu, G. and Pereira, A.G. (2006). Electronic nose technique potential monitoring mandarin maturity. *Sensors and Actuators B: Chemical*, **113**(1), 347-353.
- Kamdem, D.P., Pizzi, A. and Triboulot, M.C. (2000). Heat-treated timber: potentially toxic byproducts presence and extent of wood cell wall degradation. *European Journal of Wood and Wood Products*, **58**(4), 253-257.
- Nikoutadbir, A., Tarmian, A., Mohtasebi, S.S. and Abdulkhani, A. (2023). Emission of volatile organic compounds from heat-treated Scots pine wood as affected by wood drying method: Results obtained with olfactory machine and headspace gas chromatography-mass spectrometry. *Drying Technology*, **41**(4), 577-589.

## Oral 6.03 - Wood Modification Methods and Fire Resistance of Façades/Cladding

Joris Van Acker<sup>1</sup>, Marcy Durimel<sup>1</sup>, Liselotte De Ligne<sup>1</sup>, Bogdan Parakhonskiy<sup>1,2</sup>, Andre Skirtach<sup>2</sup>, Jan Van den Bulcke<sup>1</sup>

<sup>1</sup>Ghent University, Laboratory of Wood Technology (UGent-Woodlab), Department of Environment, Faculty of Bioscience Engineering, Coupure links 653, 9000 Ghent, Belgium, Joris.VanAcker@UGent.be, Marcy.Durimel@UGent.be, Liselotte.DeLigne@UGent.be, Jan.VandenBulcke@UGent.be

<sup>2</sup>Ghent University, Research Group Nano-Biotechnology, Department of Biotechnology, Faculty of Bioscience Engineering, Proeftuinstraat 86, 9000 Ghent, Belgium, Bogdan.Parakhonskiy@UGent.be, Andre.Skirtach@UGent.be

**Keywords:** cladding, façades, fire resistance, fire safety, thermal wood modification

### ABSTRACT

The last decade a considerable increase in façades/cladding applications have been linked to modified wood. It is becoming very common to use cladding products based on thermal modification using species like spruce, poplar, ayous, limba. Often these treatments alter the equilibrium moisture content and chemical composition and products are optimized in view of dimensional stability and decay resistance. Since fire safety is of high importance for building with wood not only the construction methods like massive timber using cross laminated timber or light timber-frame structure are relevant to be assessed, also the building envelope needs consideration. It remains crucial to adhere to local building codes and regulations related to fire safety when choosing materials for facades and cladding, especially in areas prone to wildfires or when dealing with multi-storey buildings.

Regarding the impact of (thermal) wood modification on the fire resistance of facades/cladding, it's essential to understand that when certain aspects of wood performance are improved, it is not necessarily without impact on its fire resistance. To improve the fire resistance of wood facades/cladding, additional fire-retardant treatments or coatings can be applied. These treatments are designed to inhibit or delay the ignition and spread of fire. The overall performance of wooden façades/cladding as part of the building envelope could benefit from combining wood protection options to enhance resistance against decay and to improve fire safety. Hence solutions for fire retardant treatments with adequate weather resistance could benefit from wood preservation and wood modification treatment technologies.

A range of performance testing is available to assess fire resistance but this paper focusses on the cone calorimeter method. A variety of commercial modified wood products have been assessed and benchmarked with non-modified wood including some common (tropical) hardwoods used for cladding.

## Oral 6.04 - Comparison of Major Wood Heat Treatment Technologies Paves the Way for a Generalized Mass Loss Kinetic Model

Bertrand Marcon<sup>1</sup> and Giacomo Goli<sup>2</sup>

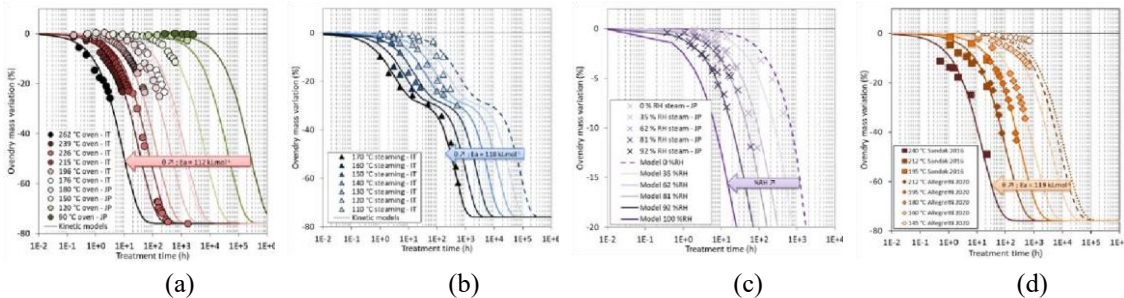
<sup>1</sup>Laboratoire des Matériaux et Procédés (LaBoMaP) – Arts et Métiers Institute of Technology, HESAM, UBFC, Rue porte de Paris, 71250 Cluny, France. E: bertrand.marcon@ensam.eu

<sup>2</sup>Dipartimento di Scienze e Tecnologia Agricola, Alimentari, Ambientali e Forestali (DAGRI), Università di Firenze, Via S. Bonaventura 13, 50145 Firenze (FI), Italy. E: giacomo.goli@unifi.it

**Keywords:** kinetic, mass loss, modelling, poplar, Sitka spruce, thermal modification, time-temperature equivalency

### ABSTRACT

Bibliographic data about dry mass loss coming from different thermal and hygrothermal modification processes versus time were collected in this work and analysed together. The data sets were collected from 4 experimental campaigns involving different modification technologies: A) poplar wood thermally modified in dry air with temperatures ranging from 90 to 260 °C up to a dry mass loss of 76% from (Goli *et al.* 2014; Marcon *et al.* 2018); B) poplar wood hygrothermally modified under saturated steam conditions with temperatures ranging from 110 to 170 °C up to a dry mass loss of 65% from (Marcon *et al.*, 2021); C) spruce wood hygrothermally modified under superheated steam conditions performed at relative humidity ranging from 35 to 92% at temperatures ranging from 110 to 170 °C from (Zeniya *et al.* 2019); and D) poplar wood samples thermally modified with Thermo-vacuum® technology performed at temperatures ranging from 150 to 240 °C from (Sandak *et al.* 2016) and (Allegretti *et al.* 2021). For all those processes, conversation rates master curves at 150 °C were identified on the experimental points as shown in Fig. 1 using the time-temperature and the time-temperature-humidity superposition method.



**Figure 1: Model curves (lines) identified on the experimental dry mass variation (points) measured in: (a) the dry air treatment in an open furnace from (Goli *et al.* 2014) and (Marcon *et al.* 2018), (b), (c) the superheated steam treatment conditions from (Zeniya *et al.*, 2019) with a model plotted up to 20% ML for lack of experimental data for higher ML, and (d) (c) the thermo-vacuum process from (Sandak *et al.*, 2016) and (Allegretti *et al.*, 2020),**

The different master curves were then compared and a generalized kinetic model able to predict the mass losses when modifying wood at different temperatures and relative humidity implemented. A generalized kinetic model is expressed by the equations (Equ. 1) with a master curve at  $\theta_{ref}$  and  $RH_{ref}$  composed of 2 stages:

$$ML_{(t,T,RH)} = \sum_{i=1}^2 ML_{(t \rightarrow \infty)}^i \times \left[ 1 - \exp\left(\frac{t(T)}{\tau_{(RH)}^i}\right)^{p^i} \right] \quad (1)$$



where  $i$  is the number of the considered stage,  $ML^i(t \rightarrow \infty)$  is the maximum mass loss after an infinite treatment time for the considered stage,  $t(T)$  is the time at the considered treatment temperature computed with Equ 2,  $\tau^i(RH)$  is the characteristic time of the considered stage at the considered treatment relative humidity (computation provided in Table 1),  $p^i$  is the slope parameter of the considered stage. Moreover, this general model requires a time shifting with the temperature following the expression (Equ. 2):

$$t(T) = t_{(T_{ref})} \times \frac{A(T)}{A(T_{ref})} \times \exp \left[ \frac{E_a}{R} \left( \frac{1}{T} - \frac{1}{T_{ref}} \right) \right] \quad (2)$$

where  $t_{(T_{ref})}$  is the time considered for the master curve under the reference conditions  $\theta_{ref}$  and  $RH_{ref}$ ,  $A(T)/A(T_{ref})$  is the pre-exponential factor of the Arrhenius law influence by the treatment temperature,  $E_a$  is the energy of activation at the reference temperature, and  $R = 8.314 \text{ J.mol}^{-1}\text{K}^{-1}$  is the universal gas constant. This generalized model involves peculiar setting of 2 kinetic stages, identified on the experimental data sets, as in Table 1.

**Table 1: Generalized model parameters taking into account both the temperature and the relative humidity influences determined for the reference conditions  $\theta_{ref} = 150 \text{ }^\circ\text{C}$  and  $RH_{ref} = 0\%$ .**

1 <sup>st</sup> stage (S1)				2 <sup>nd</sup> stage (S2)			
$ML^1(t \rightarrow \infty)$ [%]	$\tau^1(RH_{ref})$ [h]	$p^1$	Slope [%/log(h)]	$ML^2(t \rightarrow \infty)$ [%]	$\tau^2(RH_{ref})$ [h]	$p^2$	Slope [%/log(h)]
-28	2800	0.8	-18.2	-48	9000	1.2	-46.6
Treatment temperature effect: $E_a = 115 \text{ kJ.mol}^{-1}$ and $A(T)/A(T_{ref}) = 0.0064 \times T$							
Treatment medium relative humidity effect:							
$\tau^1(RH) = \tau^1(RH_{ref}) \times \exp(0.043 \times RH)$				$\tau^2(RH) = -62 \times RH + \tau^2(RH_{ref})$			

To conclude, that model can predict the mass variation occurring during any hygrothermal modification whatever the temperature applied and the environment medium relative humidity.

## REFERENCES

- Allegretti, O., Cuccui, I., Terziev, N., and Sorini, L. (2021). A model to predict the kinetics of mass loss in wood during thermo-vacuum modification. *Holzforschung*, **75**(5), 474-479. <https://doi.org/10.1515/hf-2020-0127>
- Goli, G., Marcon, B., and Fioravanti, M. (2014). Poplar wood heat treatment: effect of air ventilation rate and initial moisture content on reaction kinetics, physical and mechanical properties. *Wood Science and Technology*, **48**, 1303–1316. <https://doi.org/10.1007/s00226-0140677-5>
- Marcon, B, Goli, G., Matsuo-Ueda, M., Denaud, L., Umemura, K., Gril, J., and Kawai, S. (2018). Kinetic analysis of poplar wood properties by thermal modification in conventional oven. *iForest - Biogeosciences and Forestry*, **1**, 131–139. <https://doi.org/10.3832/ifer2422-010>
- Sandak, A., Allegretti, O., Cuccui, I., Sandak, J., Rosso, L., Castro, G., Negro, F., Cremonini, C., and Zanuttini, R. (2016). Thermo-Vacuum modification of poplar veneers and its quality control. *BioResources*, **11**, 10122–10139. <https://doi.org/10.15376/biores.11.4.10122-10139>
- Zeniya, N., Obataya, E., Endo-Ujii, K., and Matsuo-Ueda, M. (2019). Application of time– temperature– humidity superposition to the mass loss of wood through hygrothermally accelerated ageing at 95–140 °C and different relative humidity levels. *Springer Nature Applied Sciences*, **1**(3). <https://doi.org/10.1007/s42452-018-0009-8>

## Oral 6.05 - Natural weathering of Thermally Modified Wood Cladding Treated with Fire Retardants at Different Exposure Levels

Inge Wuijstens<sup>1</sup>, Imke De Windt<sup>1</sup>, Kurt De Proft<sup>2</sup>, Lieven De Boever<sup>1</sup>

<sup>1</sup>Wood.be, Hof ter Vleestdreef 3, 1070 Brussel, Belgium. E: inge@wood.be; imke@wood.be; lieven@wood.be

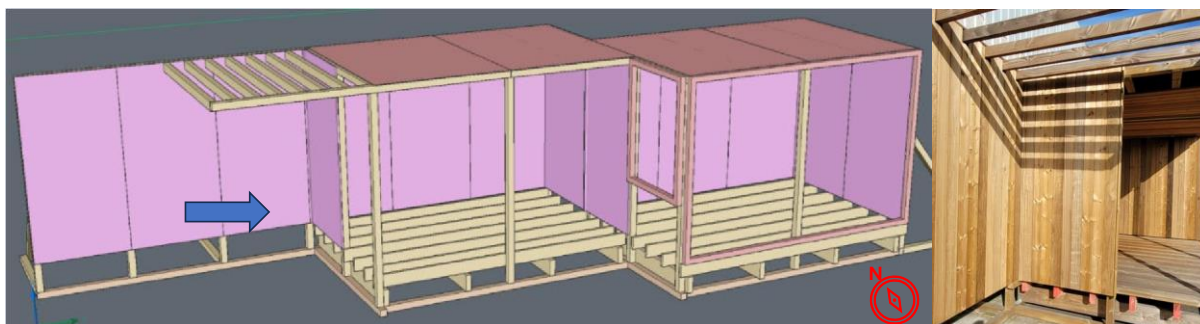
<sup>2</sup>Buildwise, Avenue P. Holoffe 21, 1342 Limelette, Belgium. E: kurt.de.proft@buildwise.be

**Keywords:** cladding, fire retardant treatment, natural weathering

### ABSTRACT

Due to stricter regulations regarding fire safety, fire retardant treatments of wooden cladding become more common for low and medium height buildings. Knowledge is available on how to perform these treatments to obtain a sufficient retention for a specified reaction to fire classification, both on modified and unmodified timber. However, little is known about the impact of added fire retardants to the appearance of these surfaces under natural weathering.

A mock-up (Figure 1) was constructed to encompass different exposure levels.



**Figure 1:** Schematic overview of the mock-up, building height of 2.4 m (left) and photo taken at indicated view point (blue arrow) (right).

Four wood species were tested: *Terminalia superba* (fraké), *Pinus Sylverstris* (pine), *Picea abies* (spruce) and *Triplochiton scleroxylon* (ayous). Based on availability (AVCP system 1 certified), 5 combinations were tested (Table 1).

**Table 1 Examined combinations of wood species, thermal modification process, type of fire retardant treatment**

Test combination	Wood species	Thermal modification process	Fire retardant treatment
1	Heat treated fraké	TMT 1	FR1
2	Heat treated pine	TMT 1	FR1
3	Heat treated fraké	TMT 2	FR2
4	Heat treated spruce	TMT 2	FR2
5	Heat treated ayous	TMT 2	FR2

Simultaneously, a test set-up was monitored for the selected wood species without the fire retardants. For this test, small test specimens (150x75 mm) were mounted vertically for all geographical directions.

The appearance was monitored by means of colour measurements and visual observations, during 1 year of outdoor exposure (Lokeren, Belgium).

The findings reveal a spectrum of discolouration effects, including fading and colour shifts. Moreover, significant differences were observed between softwoods and hardwoods. These differences are linked to a difference in fire retardant, retention and leaching behaviour. Furthermore, the rate and scale of the above described effects are significantly linked to the exposure level.

As such, the study highlights how these discolouration patterns contribute to the overall aesthetic evolution of wooden cladding, providing valuable information for architects, builders, and anyone involved in building projects.

### ACKNOWLEDGEMENTS

This research was funded by VLAIO (Flemish Government) and by FOD Economy (Federal Belgium Government).

### REFERENCES

Ayadi, N., Lejeune, F., Charrier, F. and Merlin, A. (2003). Color stability of heat-treated wood during artificial weathering. *Holz als Roh- und Werkstoff*, **61**, 221-226.

Defoirdt, N., Wuijtens, I., De Boever, L., Coppens, H., Van den Bulcke, J. and Van Acker, J. (2012). A colour assessment methodology for oak wood. *Annals of Forest Science*, **69**, 939-946.

Kržišnik, D., Lesar, B., Thaler, N. and Humar, M. (2018). Influence of Natural and Artificial Weathering on the Colour Change of Different Wood and Wood-Based Materials. *Forests* **9**(8), 488.

Rüther, P. and Jelle, B. P. (2013). Color changes of wood and wood-based materials due to natural and artificial weathering. *Wood Material Science & Engineering*, **8**(1), 13-25.

Oltean, L., Teischinger, A. and Hansmann, C. (2008). Wood surface discolouration due to simulated indoor sunlight exposure. *Holz als Roh- und Werkstoff*, **66**, 51-56.

Östman, B. (2017). Fire performance of wood products and timber structures. *International Wood Products Journal*, **8**(2), 74-79.

## **DAY 2**

### **SESSION SEVEN**

#### **DENSIFICATION AND MINERALISATION**

## Oral 7.01 - Frictional Behaviour of Modified-in-Surface Hardwoods Preliminary Obtained Through Strong Tribological Transformation

Pierre-Henri Cornuault<sup>1</sup>, Stani Carbillet<sup>1</sup> and Luc Carpentier<sup>1</sup>

<sup>1</sup>Université de Franche-Comté, CNRS, Institut FEMTO-ST, F-25000 Besançon, France. E: pierre\_henri.cornuault@univ-fcompte.fr

**Keywords:** friction, hardwood, Tribological Transformation of Surface (TTS), wear

### ABSTRACT

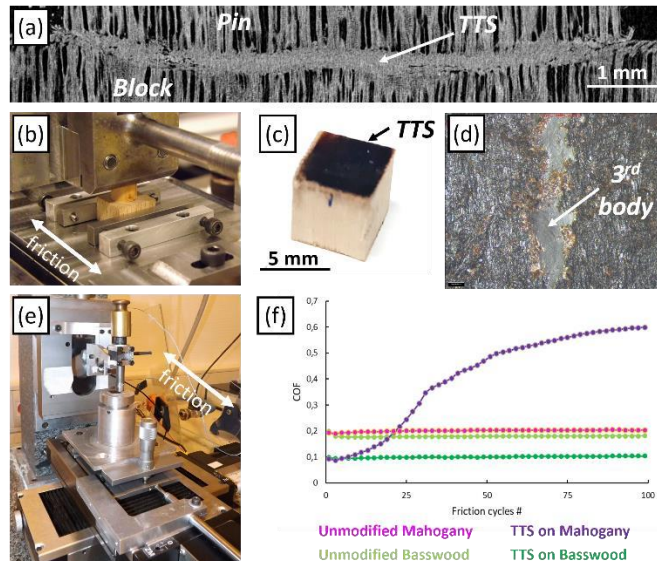
This study addresses the general issue of the potential use of bulk wood as structural industrial parts that are subjected to mechanical contact and friction. Indeed, polymeric material (petro-sourced materials) are very frequently employed as frictional parts such like gears and bearings in many industrial mechanisms. This is mainly due to their favourable tribological behaviour and their low density allowing for lighter parts and structures. However, it is suggested that hardwoods could be considered as challenging materials for such applications since: (i) they offer an eco-responsible alternative to the widespread use of petro-sourced materials, (ii) the density and mechanical properties of thermoplastics and hardwood species are almost similar. While employed in the very first studies of friction conducted by Da Vinci and Coulomb (Pitenis *et al.* 2014), the frictional behaviour of wood was subsequently poorly documented. The authors nevertheless pointed the weak tribological reliability of wood due to wear and severe surface damages during friction. Such observation motivates to functionalize-in-surface or to modify the pristine hardwood material in order to ensure good tribological properties. Meanwhile, recent advances have been achieved in the field of wood modification and wooden surfaces functionalization. Some of them led to the optimization of material properties that are essential for tribological applications such as hardness (Chen *et al.* 2021), thermal conductivity (Tan *et al.* 2022), and surface energy (Jia *et al.* 2016). To the best of our knowledge, only a single paper dealt with the tribological behaviour of modified wood, i.e. delignified wood in this case (Waßmann and Ahmed, 2020).

In this study, we investigate the frictional behaviour of hardwoods that were previously modified-in-surface through a Tribological Transformation of Surface (TTS) achieved by Friction Wood Welding (FWW) process. Briefly, this process is carried out by strongly rubbing two wooden samples together, inducing the interface transformation through frictional heating (Cornuault and Carpentier, 2020). It results in a strongly densified and hardened layer composed of a complex mixture of cellulose, hemicellulose and molten lignin (Fig. 1a). Three wood species were first surface-modified by FWW to further investigate the tribological behaviour of the resulting TTS: beech, mahogany and basswood.

The TTS were achieved at room temperature thanks to a FWW instrumented tribometer (Cameron Plint TE77) operating a linear reciprocating motion with a  $\pm 2.2$  mm relative displacement stroke at 45 Hz, and applying a constant normal force of 150 N between the two samples (Fig. 1b). Afterward, the contact between the samples was separated to reveal the TTS formed on the unmoving sample, which was then investigated (Fig. 1c). The mechanical, physico-chemical and topographical properties of the pristine TTS were characterized through SEM, ATR-FTIR, instrumented indentation testing, variable focus optical microscopy, and X-ray tomography.

Friction tests were then performed at room temperature using a custom tribometer (Fig. 1e) achieving reciprocating linear motion with a  $\pm 2$  mm displacement stroke at 3 Hz. Tests were carried out with the three above-mentioned modified hardwoods using a 5 mm diameter 100Cr6 steel ball as a counterpart. The three pristine and unmodified wood were also tested as references. Various testing conditions were investigated in terms of normal loading (0.5 to 3 N) and sliding distance (5 to 100

m). The friction force was acquired during friction tests thanks to a piezoelectric sensor. After experiments, friction tracks were analysed through SEM and variable focus optical microscopy to characterized the wear and surface damages.



**Figure 1: Samples, tribometers, surface analysis and coefficient of friction (COF) measured.**

Results show that modified hardwoods can result in friction coefficients two times lower than those measured on unmodified wood (Fig. 1f). In this case, surfaces analyses highlight the establishment of a third-body in the contact interface (Fig. 1d) which ensures low wear rates. Nonetheless, under certain experimental conditions (wood specie, contact pressure, sliding distance...etc.), the lubricating effect of this third-body is no longer achieved. The tribo-mechanisms involved in such observations are then discussed based on the surface analyses performed after friction tests.

## REFERENCES

- Chen, B., Leiste, U. H., Fourney, W. L., Liu, Y., Chen, Q., & Li, T. (2021). Hardened wood as a renewable alternative to steel and plastic. *Matter*, 4(12), 3941-3952.
- Cornuault, P. H., & Carpentier, L. (2020). Tribological mechanisms involved in friction wood welding. *Tribology International*, 141, 105963.
- Jia, S., Liu, M., Wu, Y., Luo, S., Qing, Y., & Chen, H. (2016). Facile and scalable preparation of highly wear-resistance superhydrophobic surface on wood substrates using silica nanoparticles modified by VTES. *Applied Surface Science*, 386, 115-124.
- Pitenis, A. A., Dowson, D., & Gregory Sawyer, W. (2014). Leonardo da Vinci's friction experiments: An old story acknowledged and repeated. *Tribology Letters*, 56, 509-515.
- Tan, Y., Wang, K., Dong, Y., Gong, S., Shi, S. Q., & Li, J. (2022). High performance, shape manipulatable transparent wood based on delignified wood framework and exchangeable dynamic covalent vitrimers. *Chemical Engineering Journal*, 448, 137487.
- Waßmann, O., & Ahmed, S. I. U. (2020). Slippery wood: low friction and low wear of modified beech wood. *Tribology Letters*, 68, 1-10.

## Oral 7.02 - Removal of Non-Cellulosic Wood Constituents and Subsequent Densification for Improved Mechanics of Wood

Matthias Jakob<sup>1</sup>, Ulrich Müller<sup>1</sup> and Wolfgang Gindl-Altmutter<sup>1</sup>

<sup>1</sup>Department of Materials Science and Process Engineering, BOKU – University of Natural Resources and Life Science Vienna, Konrad Lorenz Strasse 24, 3430 Tulln, Austria. E: wolfgang.gindl@boku.ac.at

**Keywords:** densification, extraction, mechanics, wood polymers

### ABSTRACT

The delignification and subsequent densification of wood has been repeatedly demonstrated as being a useful and efficient route to improving the mechanics of wood and non-wood materials (Jakob *et al.* 2022). Most of literature applies rather harsh chemical treatment, focusing on almost complete removal of lignin e.g. Frey *et al.* (2018). Own work with hardwoods has shown that delignification is not always required and that significant improvements in strength can also be achieved by focussing on mild extraction of non-cellulosic cell wall polysaccharides.

While the majority of publications on the subject rightly emphasise the enormous improvements in mechanics, particularly when working with low-density species, a number of phenomena are underrepresented in literature. E.g. own work has shown that while delignification and densification almost always improve wood mechanics in fibre direction, the side effects of the process with regard to transverse strength, wettability and adhesive bonding are less studied. Thus, in the present paper, we summarise our experience from five years of work on delignification and densification of wood and nonwood materials, with a particular focus on hardwoods as a raw material.

### REFERENCES

Frey, M., Widner, D., Segmehl, J.S., Casdorff, K., Keplinger, T., and Burgert, I. (2018). Delignified and densified cellulose bulk materials with excellent tensile properties for sustainable engineering. *ACS Applied Materials & Interfaces*, **10**, 5030-5037.

Jakob, M., Mahendran, A.R., Gindl-Altmutter, W., Bliem, P., Konnerth, J., Müller, U., and Veigel, S. (2022). The strength and stiffness of oriented wood and cellulose-fibre materials: A review. *Progress in Materials Science*, **125**, 100916.

## Oral 7.03 - Bending Performance of Thermo-hydro-mechanically Treated Scots pine (*Pinus sylvestris* L.) at Elevated Temperature

Lei Han<sup>1</sup>, Dick Sandberg<sup>2</sup> and Andreja Kutnar<sup>1</sup>

<sup>1</sup>InnoRenew CoE, Livade 6a 6310 Izola, Slovenia, E: lei.han@innorenew.eu; andreja.kutnar@innorenew.eu

<sup>2</sup>Wood Science and Engineering, Luleå University of Technology, Forskargatan 1, 931 87 Skellefteå, Sweden. E: dick.sandberg@ltu.se

**Keywords:** creep compliance, hardness, thermal modification, wood modification, viscoelasticity

### ABSTRACT

Thermo-hydro-mechanical (THM)-densified timber is rarely used in construction, although its mechanical properties are in many cases excellent. The reason is not only due to its set-recovery, which reduces the degree of densification over time so that the mechanical properties deteriorate, but also our knowledge of the long-term creep deformation of densified timber is insufficient. Limited research reports that either of the single densification process or combination with pre-resin impregnation or post-thermal modification could decrease the creep compliance by 50% at stable environment (20 °C and 65% RH), compared with untreated Scots pine (Han *et al.* 2022). However, it is still unclear which modification induced properties changing such as equilibrium moisture content (EMC), crystallinity, microstructure etc. contributes to the creep compliance reduction. Therefore, in this study the influence of EMC on bending creep behaviour of densified Scots pine will be determined. To achieve this objective, both densified wood and reference untreated samples were conditioned to near totally dry state before creep test. All specimens were loaded at  $20 \pm 2$  °C and  $5 \pm 5\%$  RH for 14 days under 3-point bending at 35% of the short-term ultimate load, and the bending deformation were registered. The vertical density profile (VDP) (i.e. in the thickness direction of the specimens) was measured before, after densification by a Siemens medical computertomography (CT) scanner at Luleå University of Technology. Dynamical mechanical analysis (DMA) was used to assess the creep of different layer of densified wood which may correspond with the result of density profile. DMA creep testing was also performed in three-point bending in the radial direction at 35% of their short-term ultimate load in a TA Instruments Q800 DMA. Creep phase at target stress was 1 hour. Before and after the DMA creep test, a dynamic bending segment at 1 Hz, 0.01% strain was performed with a 15-min iso-thermal segment at 20 °C to assess changes in dynamic moduli affected by the creep test. The study is still on going and the result will be present later during the full-paper submission.

### REFERENCES

Han, L., Kutnar, A., Couceiro, J., Sandberg, D. (2022). Creep Properties of Densified Wood in Bending. *Forests* 13, 757. <https://doi.org/10.3390/f13050757>



## Oral 7.04 - Wood Modification via Geopolymer Impregnation: Effects on Decay, Mechanical Properties and Fire Retardancy

Aitor Barbero-López<sup>1</sup>, Paivo Kinnunen<sup>2</sup> and Antti Haapala<sup>3</sup>

<sup>1</sup>Department of Chemistry, University of Eastern Finland, P.O. Box 111, Joensuu 80101, Finland E: aitorb@uef.fi

<sup>2</sup>Fibre and Particle Engineering Research Unit, PO Box 4300, Oulu 90014, Finland E: Paivo.Kinnunen@oulu.fi

<sup>3</sup>Department of Chemistry, University of Eastern Finland, P.O. Box 111, Joensuu 80101, Finland E: antti.haapala@uef.fi

**Keywords:** biodegradation, durability, inorganic preservation, wood protection

### ABSTRACT

A geopolymer is an inorganic and amorphous material formed through the chemical reaction of aluminosilicate precursors, often employed as eco-friendly alternatives to Portland cement. Geopolymers exhibit promising properties for wood modification, an area that has yet to be extensively studied.

In this paper we aim to modify wood impregnating it with 1, 3 and 5% geopolymer dilutions, to check its performance against fungal decay, leachability, its mechanical properties and fire retardancy. The results show the potential of geopolymer impregnation as a promising new wood modification treatment.

Geopolymer was prepared by adding Metakaolin (Metaver® M, Newchem GmbH, Austria) in ice bath to activate the solution of sodium hydroxide, sodium silicate and water stirred in a closed plastic container for 24 hours with a ratio between Si:Al:Na:H<sub>2</sub>O of 4:1:1:23 together with a surfactant (5% w/w) of Sodium dodecyl sulphate (ICN Biomedicals, OH, USA). To be used as a wood impregnant, the resulting geopolymer mixture was diluted to 1%, 3% and 5% using MilliQ water (Merck KGaA, Darmstadt, Germany) and the pH of these dilutions was adjusted to 11 using NaOH 0.1M. The Scots pine wood specimens (40x10x5 mm<sup>3</sup> for the decay and leaching tests; 20x20x340 mm<sup>3</sup> for the mechanical test; 100x100x20 mm<sup>3</sup> for the fire retardancy test) were submerged in the geopolymer dilutions and were kept at 0.2 Bar for 20 min, and afterwards the pressure was increased to 10 bars for 1 hour. The geopolymer-treated wood specimens were then taken out of the geopolymer solution and sealed into Ziplock bags and kept 24 hours at 40°C and 14 days at room temperature for curing. A miniblock test for checking the wood decay was done exposing the geopolymer treated specimens and controls to decay by *Coniophora puteana* in Petri dish for 16 weeks. The dry mass loss was compared to that of the controls. Extra specimens treated with 5 % geopolymer solution and untreated sapwood specimens were exposed to leaching following the EN 84 to analyze the geopolymer leachability. These specimens, together with unleached 5% geopolymer treated wood and unleached controls were exposed to another miniblock test to assess their wood preserving potential. The mechanical testing of the wood specimens was performed using a Zwick Roell 050 materials testing setup and modulus of elasticity ( $E_{mod}$ ), and modulus of rupture (MOR) were calculated by performing a three-point bending test. The fire resistance of the geopolymer-treated wood specimens was analyzed following the ISO 5660-2 Standard Test Method, with a cone calorimeter (ConeTool 1; SGS Govmark Ltd., Farmingdale, IL, USA).

The geopolymer impregnation did not affect significantly the mechanical properties of wood, but significantly reduced the mass loss caused by the decay fungus *C. puteana* at 3 and 5% (table 1).

**Table 1: Elastic modulus ( $E_{mod}$ ), modulus of rupture (MOR) and mass loss caused by *Coniophora puteana* to sapwood specimens with 1, 3 and 5 % geopolymer and controls (N=30).**

Treatment (%)	$E_{mod}$ (Gpa)	MOR (Mpa)	Mass loss (%)
1	12.8 ± 0.5	121.2 ± 6.0	16.5 ± 3.5
3	13.1 ± 0.4	131.0 ± 5.7	2.5 ± 0.9
5	12.4 ± 0.3	117.3 ± 4.8	2.2 ± 0.6
Control	12.0 ± 0.4	113.0 ± 4.7	9.9 ± 2.4

The mass loss caused by *C. puteana* to leached 5% geopolymer-treated wood was as high as the mass loss of controls (Table 2). This indicates a high leachability of the geopolymer indicating lack of effective bonding and integration between the geopolymer and wood. The reason behind remains unclear and needs to be studied, as reasons as an inadequate curing or penetration and the low geopolymer concentration used may cause these effects in the end-result.

**Table 2: Comparison of mass loss caused by *C. puteana* to leached and unleached sapwood specimens treated with 5 % geopolymer and untreated controls (N=8).**

Concentration %	Mass Loss (%)	
	Unleached	Leached
5	3.9 ± 1.0	17.4 ± 2.8
Control	13.7 ± 5.2	15.3 ± 2.6

The geopolymer impregnation increased the time to ignition of the wood specimens when compared to untreated wood, but also higher amount of smoke released by the wood was caused by the treatments. The geopolymer, containing silicon polymers and aluminum compounds, creating a surface layer that may be responsible of increasing the fire resistance of wood.

**Table 3: Fire test results of Scots pine sapwood specimens treated with geopolymer and untreated specimens (N=2).**

Sample	Time to ignition (s)	Mean heat release rate (kW/m <sup>2</sup> )	Peak heat release (kW/m <sup>2</sup> )	mass loss (%)	Total smoke (m <sup>2</sup> /m <sup>2</sup> )
Control	15.5 ± 0.5	117.9 ± 2.6	184.0 ± 8.2	47.0 ± 0.2	184.7 ± 16.2
1 %	16.5 ± 2.5	110.0 ± 11.4	176.9 ± 4.5	66.9 ± 15.2	344.5 ± 139.5
3 %	21.0 ± 1.0	105.3 ± 3.8	213.0 ± 27.9	63.1 ± 16.0	356.0 ± 162.2
5 %	20.5 ± 1.5	115.7 ± 5.1	183.5 ± 9.9	49.4 ± 1.9	254.7 ± 31.6

As a conclusion, geopolymer impregnation of wood shows promising results as a modification method increasing the resistance to fire and decay caused by *C. puteana* and not causing any effects in the mechanical properties of wood. However, geopolymer leaches out from wood easily, what requires further studies to understand how to treat wood with geopolymer. If this issue is addressed, geopolymer can be considered as a promising modification method for wood.

## Oral 7.05 - Wood modification by bio-inspired hydroxyapatite mineralization

Matic Sitar<sup>1,2</sup>, Boštjan Lesar<sup>2</sup>, Andreja Pondelak<sup>1</sup>

<sup>1</sup>Slovenian National Building and Civil Engineering Institute, Dimičeva ulica 12, 1000 Ljubljana, Slovenia. E: matic.sitar@zag.si; andreja.pondelak@zag.si

<sup>2</sup>University of Ljubljana, Biotechnical Faculty, Department of Wood Science and Technology, Jamnikarjeva 101, 1000 Ljubljana, Slovenia. E: bostjan.lesar@bf.uni-lj.si

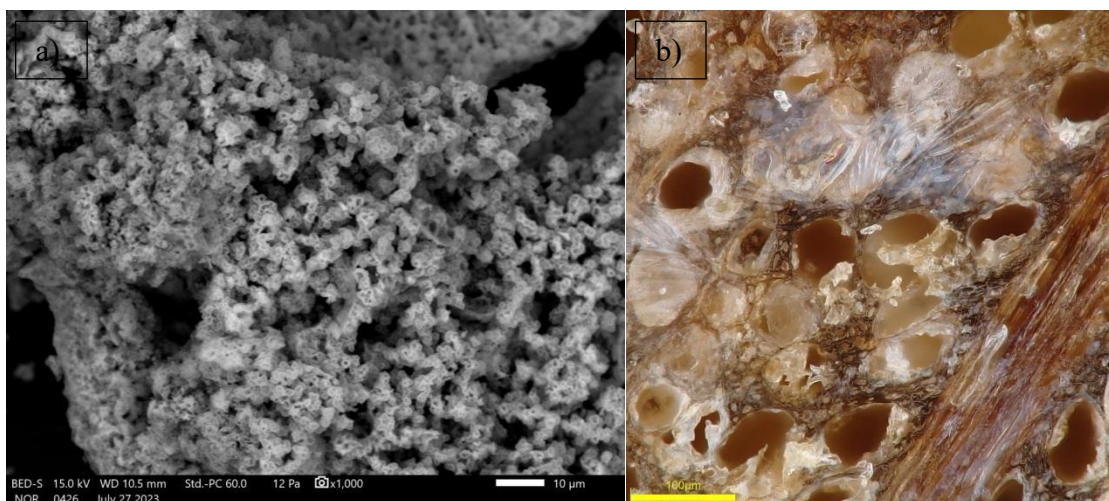
**Keywords:** environmentally friendly, hydroxyapatite, properties of mineralized wood, wood mineralization

### ABSTRACT

In outdoor applications wood is exposed to biotic and abiotic degradation factors. In nature, these processes are desirable, but when we use wood for commercial purposes, the decomposition should be slowed down as much as possible (Humar *et al.* 2020). Unfortunately, most European wood species do not have naturally resistant wood (CEN 2017).

Different methods and procedures have been used to overcome above mentioned drawbacks. However, most wood protection methods involve using harmful chemicals or processes, which raise ecological concerns. In recent years more attentions has been paid to the development of green modified wood (Dong *et al.* 2020). Mineralization of wood by calcium carbonate (CaCO<sub>3</sub>) has already been introduced by our research team (Pondelak *et al.* 2021, Repič *et al.* 2022). We have reported that by in-situ formation of CaCO<sub>3</sub> deep inside the wood structure, improved fire retardancy (Pondelak *et al.* 2021) and fungal durability (Repič *et al.* 2022) can be achieved. However, there are other minerals that can be introduced in the wood structure. One such mineral is hydroxyapatite (short HAp), with the chemical formula Ca<sub>10</sub>(PO<sub>4</sub>)<sub>6</sub>(OH)<sub>2</sub>. HAp is one of the most widely used biomaterials in the field of biomedicine and the main mineral component of human bones and teeth. Due to its unique properties (biocompatibility, low cytotoxicity,...), it is used in various fields of medicine, such as coating material for metal prostheses, in dentistry, as an antimicrobial agent, etc. (Hendi 2017, Sadat-Shojai *et al.* 2013). Until now, the incorporation of HAp into wood has not been published in the literature. The aim of the study is to present a new way of mineralization using HAp as an environmentally friendly wood modification technique.

HAp was incorporated into the wood structure by a two-stage vacuum-pressure impregnation process. First stage includes impregnation with aqueous solutions containing calcium and second stage aqueous solutions containing phosphate. Proper selection of chemicals and the impregnation method is crucial in achieving sufficient levels of compounds in the wood to form hydroxyapatite. The successfulness of HAp mineralization was determined by different methods. By FTIR spectroscopy and X-ray diffraction analysis (XRD) crystallinity of the formed mineral was determined. Scanning electron microscopy (SEM) was used to determine the size and the shape of formed HAp particles. SEM micrograph on the surface of the beech mineralized with HAp shows agglomerates with spherical like shape (figure 1a). By in-situ incorporation HAp into the wood structure, a certain filling of empty spaces in the wood, both lumens and cell walls, was shown using digital microscope (figure 1b). Wood mineralized by HAp has increased density, as well as improved other essential properties (i.e. increased fungal durability, reaction to fire) which will be the subject of ongoing research.



**Figure 1: a) SEM image of HAp formed on the surface of the substrate and b) digital microscope Olypus OLX1000 image of hydroxyapatite mineralized beech wood**

### ACKNOWLEDGEMENTS

The authors acknowledge the financial support of the Slovenian Research Agency (ARIS) within research program P4-0430 (The forest-timber chain and climate change: transition to a circular bioeconomy) and P4-0015 (Wood and lignocellulosic composites).

### REFERENCES

- CEN. (2017). EN 350 - Durability of wood and wood-based products - Testing and classification of the durability to biological agents of wood and wood-based materials. Brussels, European Committee for Standardization.
- Dong, Y., Wang, K., Li, J., Zhang, S., Shi, S. Q. (2020). Environmentally benign wood modifications: A review. *ACS Sustainable Chemistry & Engineering*, **8**(9), 3532-3540.
- Hendi, A. A. (2017). Hydroxyapatite based nanocomposite ceramics. *Journal of Alloys and Compounds*, **712**, pp. 147-151
- Humar, M., Lesar, B., Kržišnik, D. (2020). Technical and aesthetic service life of wood. *Acta Silvae et Ligni*, **121**, pp. 33-48.
- Pondelak, A., Sever Škapin, A., Knez, N., Knez, F., Pazlar, T. (2021). Improving the flame retardancy of wood using an eco-friendly mineralisation process. *Green Chemistry*, **23**(3), 11301135.
- Repič, R., Pondelak, A., Kržišnik, D., Humar, M., Sever Škapin, A. (2022). Combining mineralisation and thermal modification to improve the fungal durability of selected wood species. *Journal of Cleaner Production*, **351**(3), 131530.
- Sadat-Shojai, M., Khorasani, M.T., Dinpanah-Khoshdargi, E., Jamshidi, A. (2013). Synthesis methods for nanosized hydroxyapatite with diverse structures. *Acta Biomaterialia*, **9**(8), 75917621.

## Oral 7.06 - An Innovative Process of Mineralisation with Magnesium Compounds Improves Fire Properties of Wood

Andreja Pondelak<sup>1</sup>, Andrijana Sever Škapin<sup>1</sup>, Nataša Knez<sup>1</sup>

<sup>1</sup>Slovenian National Building and Civil Engineering Institute, Dimičeva ulica 12, 1000 Ljubljana, Slovenia. E: andreja.pondelak@zag.si

**Keywords:** carbonates, environment friendly, fire performance mineralisation

### ABSTRACT

The main disadvantage of wood is its flammability and combustibility and its tendency to biodegrade (Lu *et al.* 2014). The resistance of wood can be improved by adding so-called fire retardants (e.g. halogenated fire retardants) whereas the biological degradation can be improved by the use of biocides. The use of such materials can be environmentally problematic and should therefore be restricted and strictly controlled (Morgan and Gilman 2013). The development of new, more environmentally acceptable systems to improve fire performance and at the same time protection of wood against fungi is therefore crucial. The incorporation of minerals into the wood structure (so-called mineralisation) represents an environmentally friendly alternative to conventional fire retardants (Merk *et al.* 2016). At the Slovenian National Building and Civil Engineering Institute, we have developed a new, environmentally friendly process for mineralisation, based on vacuum-pressure impregnation with soluble organic compounds which are converted into carbonates (e.g. CaCO<sub>3</sub>) in the wood structure (Pondelak *et al.* 2021, Pondelak *et al.* 2023). We have shown that mineralisation with CaCO<sub>3</sub> results in wood with improved fire properties (Pondelak *et al.* 2021, Pondelak *et al.* 2023) and resistance to certain fungi (Pondelak *et al.* 2023, Repič *et al.* 2022). The possibility of incorporating other carbonates (i.e. MgCO<sub>3</sub>) in the wood structure, by means of soluble organic compounds can be also achieved by such a process, which is stated also in our patent (Pondelak *et al.* 2023). It is expected that the latter will have better fire properties than those mineralised with CaCO<sub>3</sub>, mainly due to the following reasons. The temperature of thermal decomposition of MgCO<sub>3</sub> is lower (~400 °C) (Mahon *et al.* 2021) compared to CaCO<sub>3</sub> (~700 °C) (Karunadasa *et al.* 2019). Moreover, MgCO<sub>3</sub> exists in various hydrate forms where each of them has an important effect on improving fire performance.

Mineralised wood was prepared by vacuum-pressure impregnation with a water solution of magnesium compounds and exposed to post-treatment parameters. Full description can be found elsewhere (Pondelak *et al.* 2021, Pondelak *et al.* 2023). Figure 1 shows incorporated MgCO<sub>3</sub> particles in the structure (mainly pores) of the beech wood in the radial and longitudinal direction determined by Scanning electron microscopy (SEM).

Wood mineralised by magnesium carbonate significantly improved fire properties compared to reference beech wood. Moreover, wood mineralised by magnesium carbonate has shown to have better fire properties than those mineralised with calcium carbonate.

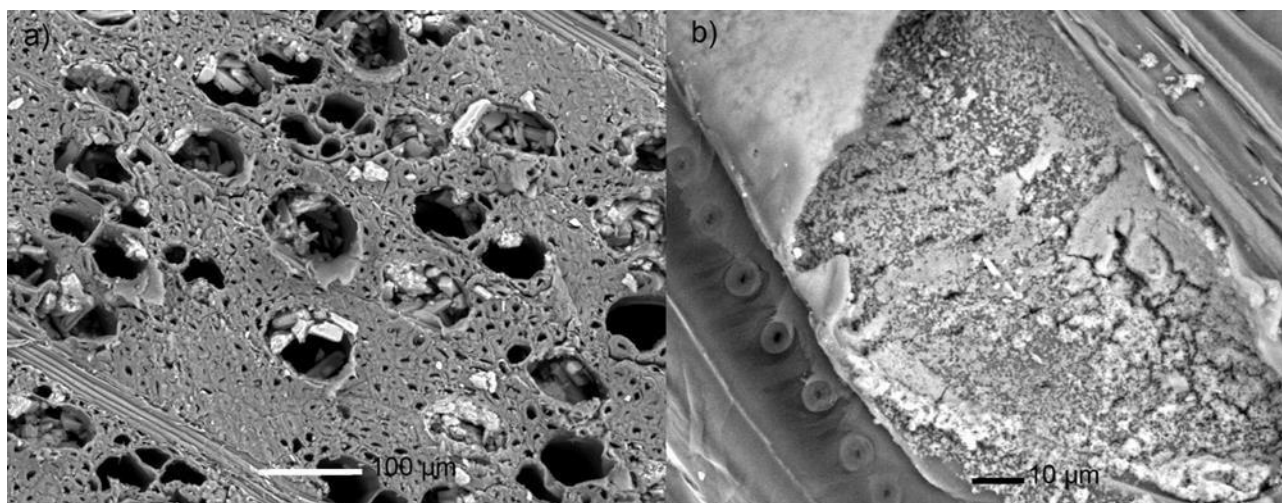


Figure 1: SEM images of MgCO<sub>3</sub> mineralised beech wood in a) radial and b) longitudinal direction.

### ACKNOWLEDGEMENTS

The authors acknowledge the financial support of the Slovenian Research Agency (ARIS) within research programme P4-0430 (Forest-wood value chain and climate change: transition to circular bioeconomy) and P2-0273 (Building structures and materials).

### REFERENCES

- Karunadasa K.S.P., Manoratne C.H., Pitawala H.M.T.G.A., Rajapakse R.M.G. (2019). Thermal decomposition of calcium carbonate (calcite polymorph) as examined by insitu high-temperature X-ray powder diffraction. *Journal of Physics and Chemistry of Solids* 134: 21-28.
- Klaithong S, Opdenbosch D.V., Zollfrank C, Plank J (2017). From the journal *Zeitschrift für Naturforschung B* Preparation of magnesium oxide and magnesium silicate replicas retaining the hierarchical structure of pine wood. *Zeitschrift für Naturforschung B* 72: 341-349.
- Lu Y, Feng M, Zhan H.B. (2014). Preparation of SiO<sub>2</sub>-wood composites by an ultrasonic-assisted sol-gel technique. *Cellulose* 21: 4393-4403.
- Mahon D, Claudio G, Eames P (2021). An Experimental Study of the Decomposition and Carbonation of Magnesium Carbonate for Medium Temperature Thermochemical Energy Storage. *Energies* 14: 1316.
- Merk V, Chanana M, Gaan S, Burgert I (2016). Mineralization of wood by calcium carbonate insertion for improved flame retardancy. *Holzforschung* 70: 867-876.
- Morgan A.B., Gilman J.W (2013). An overview of flame retardancy of polymeric materials: application, technology, and future directions. *Fire and Materials* 37: 259279.
- Pondelak A, Sever Škapin A, Knez N, Knez F, Pazlar T (2012). Improving the flame retardancy of wood using an eco-friendly mineralisation process. *Green chemistry* 23: 1130-1135.
- Pondelak A, Sever Škapin A, Knez N, Repič R, Škrlep L, Pazlar T, Knez F, Legat A (2023). A process of wood mineralization using acetoacetate solutions to improve the essential properties of wood: European patent specification EP 3 934 869 B1, 2023-0712. Munich: European Patent Office.

Repič R, Pondelak A, Kržišnik D, Humar M, Sever Škapin A (2022). Combining mineralisation and thermal modification to improve the fungal durability of selected wood species. *Journal of cleaner production* 351: 1-9.

Wu Y.Q., Yao C. H, Hu Y.C., Zhu X.D., Qing Y, Wu Q.L. (2014). Comparative Performance of Three Magnesium Compounds on Thermal Degradation Behavior of Red Gum Wood. *Materials* 7: 637-652.

## **DAY 2**

### **SESSION EIGHT**

#### **POSTERS 2**



## Poster 8.01 - Modification of Wood by Fast Pyrolysis Bio-Oil – Results from the Screening Test

Anna Sandak<sup>1,2,3</sup>, Jakub Sandak<sup>1,3</sup>, Faksawat Poohphajai<sup>1,4</sup>, Rene Herrera Diaz<sup>1,5</sup>, Ana Gubenšek<sup>1</sup>, Karen Butina Ogorelec<sup>1</sup>, Wojciech Pajerski, Lex Kiezebrink<sup>6</sup>, Klaas Jan Swager<sup>6</sup>, Hans Heeres<sup>7</sup>, Bert van de Beld<sup>7</sup>

<sup>1</sup>InnoRenew CoE, Izola, Slovenia. E: anna.sandak@innorenew.eu; jakub.sandak@innorenew.eu; faksawat.poohphajai@innorenew.eu; rene.herdi@innorenew.eu; ana.gubensek@innorenew.eu; karen.butina@innorenew.eu

<sup>2</sup>Faculty of Mathematics, Natural Sciences and Information Technologies, University of Primorska, Koper, Slovenia. E: anna.sandak@famnit.upr.si

<sup>3</sup>Andrej Marušič Institute, University of Primorska, Koper, Slovenia. E: jakub.sandak@upr.si

<sup>4</sup>Department of Bioproducts and Biosystems, Aalto University School of Chemical Engineering, P.O. Box 16300, 00076, Aalto, Finland. E: faksawat.poohphajai@aalto.fi

<sup>5</sup>Department of Chemical and Environmental Engineering, University of the Basque Country (UPV/EHU), Plaza Europa 1, 20018, Donostia-San Sebastián, Spain. E: renealexander.herrera@ehu.eus

<sup>6</sup>Foreco Dalmsholterweg 5, 7722 KJ Dalfsen, Netherlands. E: l.kiezebrink@foreco.nl; k.swager@foreco.nl

<sup>7</sup>BTG Biomass Technology Group, Enschede, the Netherlands. E: Heeres@btgworld.com; vandebeld@btgworld.com

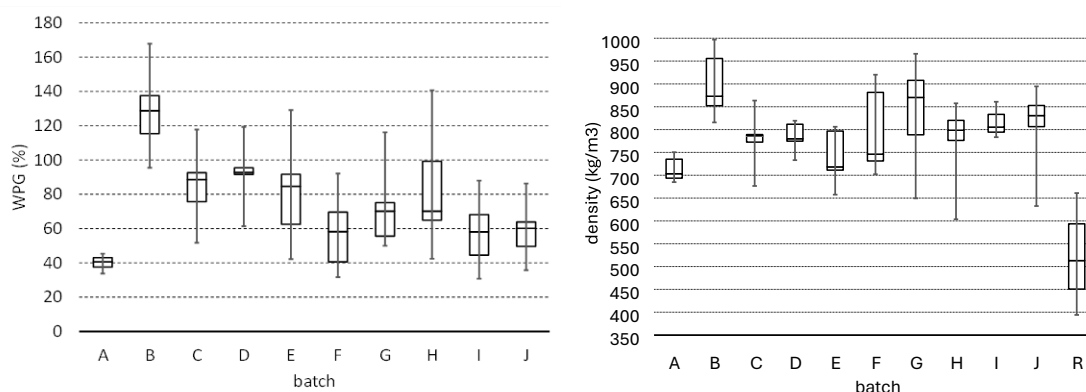
**Keywords:** biobased treatment, durability, Fast Pyrolysis Bio-Oil, screening tests

### ABSTRACT

Modification processes lead to the enhancement of selected wood properties through chemical, biological, or physical agents. Although several treatments, including both active and passive modifications, are recently available development of alternative entirely biobased processes is highly desired. One of the research lines conducted within the NewWave project is focused on the optimization of impregnation based on Fast Pyrolysis Bio-Oil (FPBO) used as a wood modification agent. This research focuses on the modification of wood with FPBO to develop an entirely biobased alternative to currently used toxic and fossil-based preservation agents such as copper salts, organic biocide ingredients, and creosote. Ten formulations based on FPBO were prepared and characterized in terms of pot life, viscosity, and curing behaviour among others. The impregnation process of radiata pine samples was performed in the bench-scale reactor. The uptake of the impregnation liquor was assessed by calculation of weight percent gain (WPG) for each specimen. Penetration depth was assessed with hyperspectral imaging. Characterization methods included moisture uptake, dimensional stability, density, mechanical strength, UV stability, durability tests against fungi and moulds, fixation of components, and VOCs emission. The overview of conducted characterization methods and examples of results are presented in Table 1 and in Figure 1 respectively.

**Table 1: List of conducted characterization methods.**

Activity/characterization method	Procedure
Density	ISO 13061-2
Moisture uptake	CEN 16818:2018
Leaching test	EN 84:2020
Dimensional stability	ISO 13061
Mechanical performance – impact bending strength	ISO 13061-10
UV stability	ISO 16474
Biological durability	EN 113 2021
VOCs emission	ISO 16000-6:2021
Wettability	ISO 19403-2:2020
Appearance (color and gloss)	custom
Hyperspectral imaging	custom
Hygroscopic properties	custom
Thermal properties	ISO 8301:1991



**Figure 1: WPG (left) and density of impregnated wood (right) measured at EMC (20°C, 50% RH) for samples treated in bench-scale reactor.**

The characterization campaign aims to select 3-4 best-performing treatments and prepare a set of experimental samples, that will be evaluated regarding their performance in outdoor applications. After extensive laboratory tests, new construction products will be manufactured at an industrial scale and used at a demonstration site. The performance of the materials, moisture content, and temperature in envelope layers will be monitored in situ, allowing for observing the deterioration of materials and estimating service life regarding functionality and aesthetics. This information will be used for future optimization of the formulation and modification process as well as the scheduling of recommended maintenance actions.

## ACKNOWLEDGMENTS

The authors gratefully acknowledge the funding of the Horizon Europe Program within the project NewWave under grant agreement No 101058369, and support from the consortium partners. AS, JS, FP, RHD, AG and KBO gratefully acknowledge the European Commission for funding the InnoRenew CoE project (Grant Agreement #739574) under the Horizon2020 Widespread-Teaming program and the Republic of Slovenia (Investment funding of the Republic of Slovenia and the European Union of the European Regional Development Fund).

## Poster 8.02 - Anatomical Variations Between Natural and Delignified Wood: a Case of Study of some Italian “Minor” Wood Species

Francesco Bolognesi<sup>1,2</sup>, Alessandra Bianco<sup>2</sup>, Francesca Romana Lamastra<sup>2</sup>, Marco Togni<sup>1</sup>

<sup>1</sup>Dipartimento di Scienze e Tecnologie Agrarie, Alimentari, Ambientali e Forestali (DAGRI)  
Università degli Studi di Firenze, Piazzale delle Cascine 18, Firenze, Italy.

<sup>2</sup>Università degli Studi di Roma “Tor Vergata”, Dipartimento Ingegneria dell’Impresa “Mario Lucertini”, Consortium INSTM RU “Roma Tor Vergata”, Via del Politecnico, 00133 Roma, Italy.

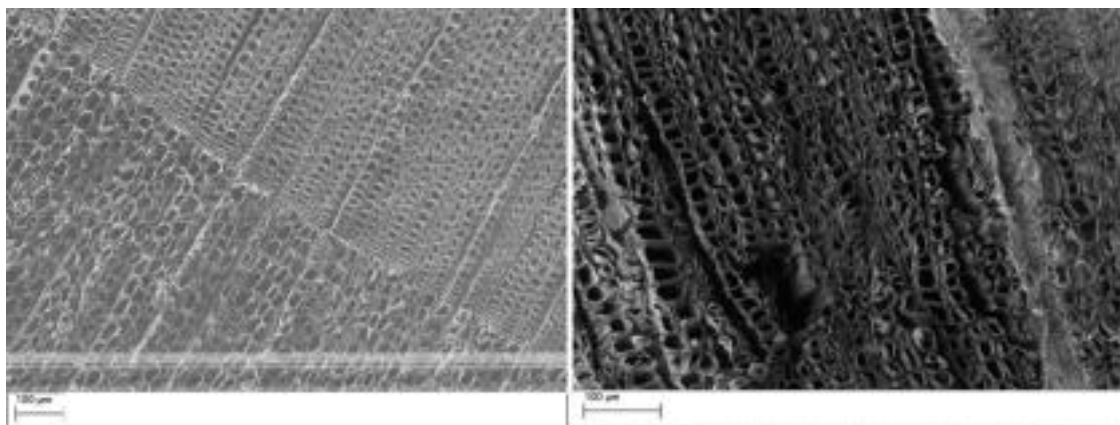
**Keywords:** Corsican pine, delignification, European hop hornbeam, optical microscopy, scanning electron microscopy, Turkey oak, wood anatomy

### ABSTRACT

Delignified wood plays a crucial role in various industrial processes and emerging technologies, making it a key focus within the field of materials science. While beech wood (*Fagus sylvatica* L.) has served as a well-studied model substrate for chemical delignification processes (Montanari *et al.* 2021, Yu *et al.* 2017, Yaddanapudi *et al.* 2017), the anatomical differences of other lesser-known “minor” species remain relatively unexplored. This study aims to address this knowledge gap by examining the anatomical differences between natural and chemically delignified wood samples from “minor” species broadly diffused in Italian forests, such as Corsican pine (*Pinus nigra* Arnold subsp. *laricio* (Poir.) Maire), Turkey oak (*Quercus cerris* L.), European hop hornbeam (*Ostrya carpinifolia* Scop.), thus providing insights into potential advantages and applications of these “minor” species in wood-based industries.

Our investigation utilized a range of cutting-edge microscopic techniques, including optical microscopy and scanning electron microscopy, to capture intricate details of the wood's microstructure in both the anatomical directions: transversal (T) and axial (A). We assessed various anatomical parameters, including cell wall thickness, flaws characteristics and vessel/tracheid morphology, with the objective of discerning the effects of delignification in single species and identifying significant variations between the different wood types.

Our research findings highlight several significant anatomical distinctions between natural wood and delignified wood derived from the previously mentioned species. One of the more interesting is the unalteration in wood mesostructure, but the complete sharp modification in wood cell morphology caused by the delignification process (Figure 1). These disparities in microstructure could have profound implications for the development of innovative biomaterials in various fields, including building sector and electronics (Li *et al.* 2016, Mi *et al.* 2020, Samanta *et al.* 2021).



**Figure 1: SEM micrographs of transversal wood of Corsican pine: natural (left), delignified (right)**

Understanding these anatomical differences offers an exciting opportunity to optimize various delignification processes for specific applications. Moreover, the unique anatomical features of “minor” species may enable more efficient and sustainable utilization of these underutilized resources, reducing environmental impact and enhancing economic viability. In addition, these findings expand our knowledge of wood-based materials and open doors for further exploration of the untapped potential of “minor” species in the ever-evolving landscape of materials science and technology.

In conclusion, this research contributes to the growing body of knowledge surrounding delignified wood and its applications. By shedding light on the anatomical variations between delignified and natural wood from “minor” species, it provides a foundation for the targeted utilization of these “minor” species in wood industries, fostering sustainability and innovation within this critical sector.

## REFERENCES

- Li, T., Zhu, M., Yang, Z., Song, J., Dai, J., Yao, Y., Luo, W., Pastel, G., Yang, B., Hu, L. (2016). Wood Composite as an Energy Efficient Building Material: Guided Sunlight Transmittance and Effective Thermal Insulation. *Advanced Energy Material*, 6: 1601122. doi: 10.1002/aenm.201601122
- Mi, R., Chen, C., Keplinger, T. et al. (2020). Scalable aesthetic transparent wood for energy efficient buildings. *Nature Communications* 11, 3836. <https://doi.org/10.1038/s41467-020-17513-w>
- Montanari, C., Ogawa, Y., Olsén, P., Berglund, LA. (2021) High performance, fully bio-based, and optically transparent wood biocomposites. *Advanced Science* 8:2100559. <https://doi.org/10.1002/advs.202100559>
- Samanta, A., Chen, H., Samanta, P., Popov, S., Sychugov, I. & Berglund, L. (2021). Reversible dual-stimuli responsive chromic transparent wood bio-composites for smart window applications. *ACS Applied Materials and Interfaces*, 13, 3270-3277
- Yaddanapudi, HS., Hickerson, N., Saini, S., Tiwari, A. (2017) Fabrication and characterization of transparent wood for next generation smart building applications. *Vacuum* 146:649–654. <https://doi.org/10.1016/j.vacuum.2017.01.016>
- Yu, Z., Yao, Y., Yao, J., Zhang, L., Chen, Z., Gao, Y., Luo, H. (2017) Transparent wood containing Cs x WO<sub>3</sub> nanoparticles for heat-shielding window applications. *Journal of Material Chemistry A* 5:6019–6024. <https://doi.org/10.1039/c7ta00261k>

## Poster 8.03 - Improving the Energy Storage Properties of Wood by Using Lauric Acid

Ahmet Can<sup>1</sup>

<sup>1</sup>Wood Science and Engineering Department, Bartin University, 74100 Bartin, Turkey. E: acan@bartin.edu.tr

**Keywords:** chemical properties, energy storage, phase change material, wood

### ABSTRACT

While global energy consumption varies based on countries' economic prosperity, a significant portion is attributed to approximately 30-40 buildings (Cao *et al.* 2016). The predominant share of this energy usage is allocated for the purposes of heating and cooling. On a yearly basis, our nation's energy demands are escalating by 8-9 percent. To cope with this mounting energy requirement, there's a steady upsurge in the reliance on fossil fuels. The advancement of energy storage systems holds equivalent importance to the exploration of novel energy sources. Energy storage serves as the mechanism that adeptly captures and transforms energy into the desired usable form (Liu *et al.* 2010). This framework stands as the optimal approach to amplify the integration of renewable energy sources. Phase-change materials (PCMs) are extensively favored for thermal energy storage due to their elevated energy density and uniform temperature behavior during heat exchange (Nazir *et al.* 2019). In this study, three different wood species (*Populus euroamericana* I214, *Pinus sylvestris*, *Fagus orientalis*) were impregnated with lauric acid under vacuum.

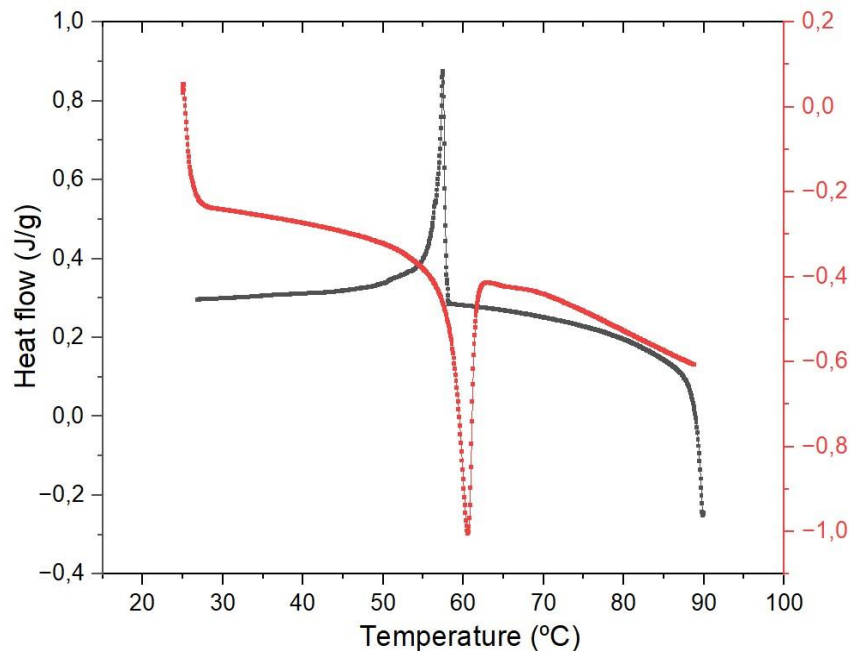


Figure 1: DSC results of lauric acid modified poplar wood

The results show that lauric acid modified wood thermal stability at working temperature. After modification, approximately 14.87%, 12.45 %, and 15.41 % weight gain was obtained in poplar, beech, and pine wood, respectively. Latent heat storage of 50.85 J/g, 48.1 J/g, 52.0 J/g, and phase change temperatures of 48.29 °C, 45.22 °C and 50.46 °C (medium-temperature zone (buildings fields)) was measured for poplar, beech and pine wood, respectively. Ma *et al.* (2018) were conducted involving the creation of a form-stable phase change material (FSPCM) using porous wood flour (WF) and a mixture of lauric acid (LA) and myristic acid (MA). This FSPCM was made using a

vacuum impregnation method. The results from differential scanning calorimetry indicated that the ideal melting temperature for this composite material was 33.1 °C, and it possessed a latent heat of 98.2 kJ kg<sup>-1</sup>.

## REFERENCES

- Cao, X., Dai, X. and Liu, J. (2016). Building energy-consumption status worldwide and the state-of-the-art technologies for zero-energy buildings during the past decade. *Energy and buildings*, 128, 198-213.
- Liu, C., Li, F., Ma, L. P. and Cheng, H.M. (2010). Advanced materials for energy storage. *Advanced materials*, 22(8), E28-E62.
- Ma, L., Guo, C., Ou, R., Sun, L., Wang, Q. and Li, L. (2018). Preparation and characterization of modified porous wood flour/lauric-myristic acid eutectic mixture as a form-stable phase change material. *Energy & Fuels*, 32(4), 5453-5461.
- Nazir, H., Batool, M., Osorio, F. J. B., Isaza-Ruiz, M., Xu, X., Vignarooban, K. Phelan, P., Inamuddin, and Kannan, A.M. (2019). Recent developments in phase change materials for energy storage applications: A review. *International Journal of Heat and Mass Transfer*, 129, 491-523.

## Poster 8.04 - Improving Evaluation of Treatments for Preventing Resin Exudation through Coatings

Dennis Jones<sup>1</sup>, Aubin Vieillescazes<sup>2</sup>, Micael Öhman<sup>1</sup>, Olov Karlsson<sup>1</sup>, Rostand Moutou Pitti<sup>3</sup> and Dick Sandberg<sup>1</sup>

<sup>1</sup>Wood Science and Engineering Department, Luleå University of Technology, Forskargatan 1, 931 87 Skellefteå, Sweden. E: dennis.jones@ltu.se; micael.ohman@ltu.se; olov.karlsson@ltu.se, dick.sandberg@ltu.se

<sup>2</sup>Civil engineering department, Polytech Clermont Ferrand, 2, Av. Blaise Pascal – TSA 60206 – CS 60026 - F 63178 Aubière Cedex, France. E: dept.gc@polytech.univ-bpclermont.fr

<sup>3</sup>Université Clermont Auvergne, CNRS, Clermont Auvergne INP, Institut Pascal, F-63000 Clermont-Ferrand, France. E: rostand.moutou\_pitti@uca.fr

**Keywords:** Wood, resin exudation, phenol-formaldehyde resin, coatings

### ABSTRACT

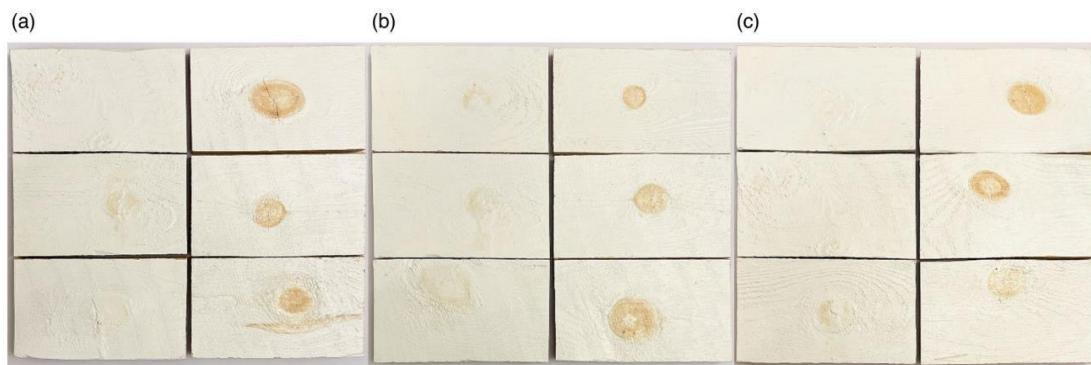
A common problem in surface-coated wood products is that extractives migrate from inside the wood, especially through the knots, and resurface under or penetrate through the paint layer, and thereby change the adhesion and appearance of the painted surface. The discoloration present on the paint is due to the processes of photo-oxidation of lignins, as well as a result of extractives present within the knots. Among the more common extractive groups associated with discolouration are terpenes, stilbenes, lignans and resin acids (Hundhausen *et al.* 2020). This problem has increased with the introduction of water-borne paints on the market (Ekstedt 2000). Among methods to prevent such discolouration of paint by the extractives include:

- creating a dense barrier between the knot and the paint to prevent contact between the extractives and the paint or to limit contact between the knot and external agents, and
- including compounds in the primer to create a chemical reaction with the extractives.

Among methods considered herein have been:

1. Water glass: This product has been used for a long time in the protection of wood against bad weather. It creates a solid waterproof layer on the surface of the wood.
2. Epoxy resin: Applied as a bicomponent resin which, when exposed to a minimum temperature of 10 to 15 °C, becomes solid.
3. Ferrous sulphate + hydrogen peroxide: This mixture is supposed to interact with the extractable compounds present in the knot. The ferrous sulphate must first be mixed with water according to the ratio 1/10. Then the hydrogen peroxide is applied on the knot and a few drops from the previous mixture are also applied to the knot.
4. Cactus juice: this product is a stabilising resin used to harden and stabilise wood. This product is then to be heated to approximately 100 °C.
5. Heat treatment + water glass: The wood is placed in an oven at around 100 °C for 2 hours before the treatment is applied.
6. Heat treatment + epoxy resin: The wood is placed in an oven at around 100 °C for 2 hours before the treatment is applied.
7. Epoxy resin (painted): The epoxy resin will be painted in this treatment to create a thinner treatment layer.
8. Epoxy resin + acetone (painted): The epoxy resin will be painted in this treatment to create a thinner treatment layer and mixed with acetone to make the treatment more liquid. The share of acetone in the mixture is 15%.

9. Pre-heating the wood to a temperature of 130 °C or 160 °C and applying PF resin. Very promising results were obtained with the epoxy resin treatment, with the complete avoidance of yellowing of the paint. Water glass was also effective when compatibility between the paint and the treatment was possible (was noted with excessive cracking of the coating). The creation of a relevant discoloration calculation method made it possible to accurately compare the different samples. The primary goal of the subject was to find a treatment to delay the discoloration, in the end, a treatment was found to reverse the discoloration. The phenol formaldehyde treatment using the studied variation of the hot-and-cold bath process was tested as a means of preventing resin exudation from knots (Figure 1). The studied treatment greatly reduced the migration of resin and extractives through knots in coated wood. This method can be considered as a pre-treatment prior to painting in order to reduce migration of extractives from knots through coatings on wood panels. Other treatments had little or no effect.



**Figure 1: Treated (left) and untreated reference (right) specimens after accelerated aging: (a) Preheating at 130°C and soaking in pure PF, (b) Preheating at 130°C and soaking in PF diluted with methanol 1:1, and (c) Preheating at 160°C and soaking in pure PF.**

It would also be interesting to look for:

- Products similar to epoxy resin that are more environmentally friendly and possibly improve indoor air quality.
- Additional research at high temperatures to extract the extractable compounds in the knots.
- Additional research using more conventional heat treatment conditions to extract the extractable compounds.

## REFERENCES

- Ekstedt, J. (2000). Determination of Discolouration of Paints on Wood Due to Resin Exudation from Knots. Report P0006009 (Stockholm: Träteknik – Swedish Institute for Wood Technology Research).
- Hundhausen, U., Peitzmeier, J. and Johnsen, I. (2020). The influence of pinosylvin, pinoresinol, abietic acid and alpha-pinene on knot yellowing of coated scots pine (*Pinus Sylvestris* L.). 16th Annual Meeting of the Northern European Network for Wood Science and Engineering—WSE 2020, Helsinki, Finland.



## Poster 8.05 - Preliminary Evaluation of Wood Impregnated with Oak Bark-derived Residuals

Rene Herrera<sup>1,2</sup>, Mariem Zouari<sup>1,3</sup>, Faksawat Poohphajai<sup>1,2,4</sup>, Jakub Sandak<sup>1,2</sup>, Anna Sandak<sup>1,2,3</sup>

<sup>1</sup>InnoRenew CoE, Izola, Slovenia. E: rene.herdiaz@innorenew.eu, mariem.zouari@innorenew.eu, faksawat.poohphajai@innorenew.eu, jakub.sandak@innorenew.eu, anna.sandak@innorenew.eu

<sup>2</sup>Andrej Marušič Institute, University of Primorska, Koper, Slovenia

<sup>3</sup>Faculty of Mathematics, Natural Sciences and Information Technologies, University of Primorska, Koper, Slovenia

<sup>4</sup>Department of Bioproducts and Biosystems, Aalto University School of Chemical Engineering, Aalto, Finland

**Keywords:** bio-based treatments, oak bark extractives, ultrasound and alkaline extraction, UVprotection, decay resistance, wettability

### ABSTRACT

Modified wood and engineered wood products have shown enhancing performance during the service life. However, sustainable strategies that not only enhance key wood properties but also align with circular and green principles for further industrial upscaling are still highly desired (Herrera *et al.* 2023). Forest industries generate different valuable side-streams, such as bark from the logging process. Bark is traditionally utilized for energy generation, animal bedding or soil mulching, but it presents an intriguing potential as a feedstock for the preparation of bio-based wood treatments (Liu *et al.* 2020). In this preliminary study, residual wet outer oak bark (*Quercus robur*) supplied by DHT (Koszkowo, Poland) was dried and milled (1-4mm) and modified by three distinct procedures. They included: (1) extraction employing ultrasonic cavitation using both water and ethanol-water (Picot-Allain *et al.* 2021); (2) alkaline aqueous extraction with NaOH carried out in a pressure reactor (Borrega *et al.* 2022); (3) thermochemical conversion to biochar collecting the water-condensed extractives fractions (Marrot *et al.* 2022). The obtained solutions were chemically characterized, and subsequently waterbased formulations with the incorporation of additives were prepared for wood impregnation. A set of samples (*Pinus sylvestris*) was vacuum-pressure impregnated at room temperature with all three solutions, and then underwent curing process via plasma or heat treatment (103 °C). Impregnated samples were physically characterized, and tested regarding the leachability, dimensional stability, wettability, UV-stability, and decay resistance against white and brown rot. The result of this study reveals the improvements and limitations of each configuration along with effectiveness of bio-based treatments derived from residual bark. This research contributes to the development of sustainable wood treatment strategies with potential applications in the wood and construction industry.

### REFERENCES

- Borrega, M., Kalliola, A., Määttänen, M., Borisova, A. S., Mikkelsen, A., & Tamminen, T. (2022). Alkaline extraction of polyphenols for valorization of industrial spruce bark. *Bioresource Technology Reports*, **19**, 101129.
- Herrera, R., Poohphajai, F., Sandak, A., & Gordobil, O. (2023). Simultaneous Improvement of Surface Wettability and UV Resistance of Wood with Lignin-Based Treatments. *Polymers*, **15**(16), 3409.

Liu, L. Y., Patankar, S. C., Chandra, R. P., Sathitsuksanoh, N., Saddler, J. N., & Renneckar, S. (2020). Valorization of bark using ethanol–water organosolv treatment: Isolation and characterization of crude lignin. *ACS Sustainable Chemistry & Engineering*, **8(12)**, 4745-4754.

Marrot, L., Meile, K., Zouari, M., DeVallance, D., Sandak, A., & Herrera, R. (2022). Characterization of the Compounds Released in the Gaseous Waste Stream during the Slow Pyrolysis of Hemp (*Cannabis sativa* L.). *Molecules*, **27(9)**, 2794.

Picot-Allain, C., Mahomoodally, M. F., Ak, G., & Zengin, G. (2021). Conventional versus green extraction techniques—A comparative perspective. *Current Opinion in Food Science*, **40**, 144156.

## Poster 8.06 - Optical Properties of Spectrally Irradiated Wood

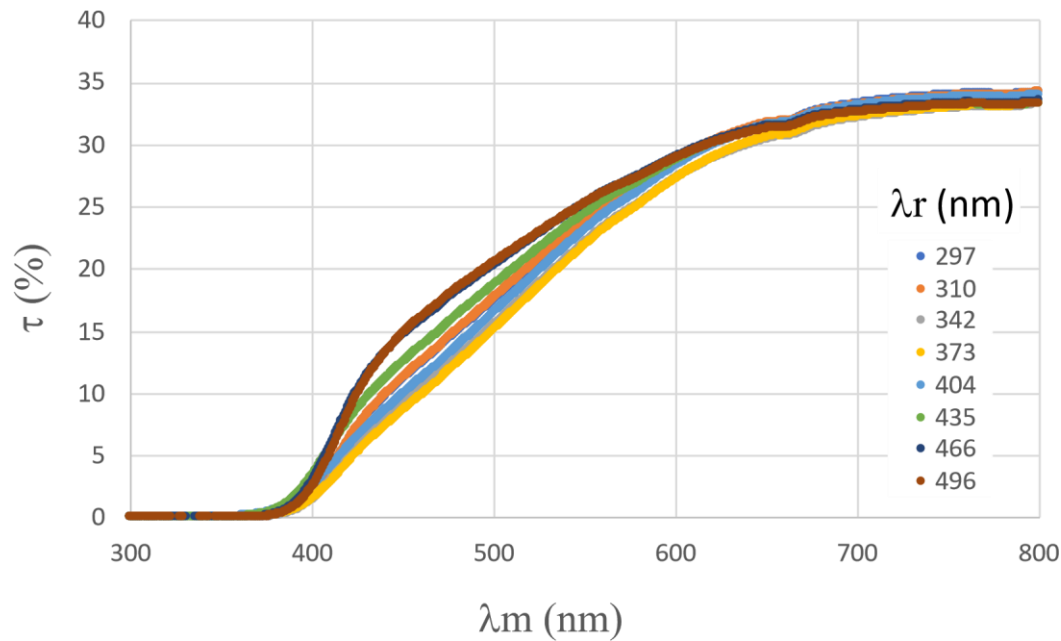
Hiroyuki Sugimoto<sup>1</sup>, Kai Maruyama<sup>1</sup> and Masatoshi Sugimori<sup>1</sup>

<sup>1</sup>Department of Forest Resources, Faculty of Agriculture, Ehime University, 3-5-7, Tarumi Matsuyama Ehime, Japan. E: sugimoto.hiroyuki.rw@ehime-u.ac.jp

**Keywords:** appearance change, degradation, optical properties, reflectance, transmittance

### ABSTRACT

Wood is a familiar material used for a variety of purposes in daily life, including wooden furniture, flooring, and decorative objects. On the other hand, a problem in the use of wood is that light reduces its value of appearance. This degradation is known to have different effects depending on the wavelength of light (Refs. 1-3). In recent years, there have been various light sources for indoor lighting (fluorescent lamps, LEDs, UV-cut window glass, etc.), each of which has different wavelength characteristics. Since lightresistant design of wood is required for the several types of indoor light, it is necessary to clarify the appearance changes in wood according to the irradiation wavelength. Some studies of wood colour change by the irradiation of various wavelengths light have shown that sapwoods of larch (*Larix decidua*), hinoki (*Chamaecyparis obtusa*) and sugi (*Cryptomeria japonica*) become darker when exposed to light in the 350-390 nm<sup>1)</sup>, 300390 nm<sup>2)</sup> or 246-403 nm<sup>3)</sup> wavelength ranges, and lighter in the 390-580 nm<sup>1)</sup>, 440-560 nm<sup>2)</sup> or 434-496 nm<sup>3)</sup> wavelength ranges, respectively. The reason for these various variations in optical properties have been attributed to the differences in their constituent molecules, such as lignin or extractives<sup>4)</sup>. However, these previous studies did not consider the effects of light, heat and leakage of extracted components during sample preparation, and it is questionable whether the effects of light irradiation alone have been examined. On the other hand, the authors have shown that the transmission of incident light into the material is strongly influenced by its cell structure<sup>5)</sup>. Therefore, in this study, for the purpose of evaluating only the colour change caused by light irradiation, samples were prepared in such a way that they were as free as possible from the effects of light, heat, leakage of extractives component and difference of cell structure from cutting and drying, and the resulting samples were treated with spectral light irradiation and the changes in optical properties, such as reflectance and transmittance, were measured and considered. Fig.1 show the wavelength( $l_m$ ) dependence of the transmittance ( $t$ ) of sugi irradiated with various wavelengths( $l_r$ ). Depending on  $l_r$ , the change in transmittance at  $l_m$  of 400-500 nm was distinctly different. These details, including reflectance, will be presented at the conference.



**Figure 1: The wavelength( $\lambda_m$ ) dependence of the transmittance ( $\tau$ ) of sugi irradiated with various wavelengths( $\lambda_r$ ).**

## REFERENCES

- Minemura, S., Umehara, M. (1979). *Hokkaido Shikenjo Hokoku*, **68**, 99-107.
- Mitsui, K. (2004). Changes in the properties of light-irradiated wood with heat treatment. Part 2. Effect of light-irradiation time and wavelength. *Holz Roh-Werkst.* **62** (1), 23-30.
- Kataoka, Y., Kiguchi, M., Williams, R.S., Evans, P.D. (2007). Violet light causes photodegradation of wood beyond the zone affected by ultraviolet radiation. *Holzforschung*, **61** (1), 23-27.
- Nishihara, Y., Sugimoto, H., Sugimori, M. (2023). Changes in visible light spectral properties of wood due to extraction treatments. In: Proceedings of the 73rd Annual Meeting of Japan Wood Research Society. Fukuoka, Japan, C000548.
- Sugimoto, H., Kawabuchi, S., Sugimori, M., Gril, J. (2018) Reflection and transmission of visible light by sugi wood: effects of cellular structure and densification. *Journal of Wood Science*, **64**(6), 738-744.

## Poster 8.07 - Exploring the Potential of Carbon Nanodots as an UV Protection Reagent for Wood

Sarah Jué<sup>1,2</sup>, Chia-feng Lin<sup>1</sup>, Alexander Scharf<sup>1</sup>, Dennis Jones<sup>1</sup>, Rostand Moutou Pitti<sup>2,3</sup> and Dick Sandberg<sup>1</sup>

<sup>1</sup>Wood Science and Engineering, Luleå University of Technology, Forskargatan 1, 931 87 Skellefteå, Sweden. E: sarjuc-3@student.ltu.se, chia-feng.lin@ltu.se, alexander.scharf@ltu.se, dennis.jones@ltu.se, dick.dsandberg@ltu.se

<sup>2</sup>Université Clermont Auvergne, Clermont Auvergne INP, CNRS, Institut Pascal, Clermont Ferrand, France. E: rostand.moutou\_pitti@uca.fr

<sup>3</sup>CENAREST, IRT, BP 14070, Libreville, Gabon. E: rostand.moutou\_pitti@uca.fr

**Keywords:** anti-discoloration, carbon nanospheres, quantum dots, wood aesthetic, wood protection

### ABSTRACT

The greying of wood exposed to weathering is mainly due to the removal of photodegraded lignin, leaving a layer of remaining cellulose-rich polymers on the wood surface. The synergistic effect of photostabilizing UV absorbers (UVA) and hindered amine light stabilizers (HALS) has been well-known for slowing down the greying process. Inorganic nanoparticles, such as TiO<sub>2</sub> or ZnO have attracted recent attention for slowing down wood greying (Jirouš-Rajković and Miklečić 2021). Carbon nanoparticles can be produced from biomass or synthetic polymers under relatively mild hydrothermal carbonisation (Adolfsson *et al.* 2018, 2020). In addition, carbon nanodots prepared from the precursors citric acid and urea exhibit fluorescent properties, absorbing UV light upon excitation with visible light (Strauss *et al.* 2020). This UV-absorption property, along with both precursors previously being used as wood modification reagents, has attracted our interest in exploring the potential of carbon nanodots as a new wood-modification reagent. Urea has been reported as a reagent for wood carbamylation because the thermal decomposition of urea produces highly reactive isocyanic acid, which can further react with hydroxyl groups of wood polymers to form-stable covalent bonds (Lin *et al.* 2023). Citric acid has also been used as a wood-modification reagent, capable of undergoing esterification to enhance the dimensional stability (Lee *et al.* 2020). In this study, the potential of using citric acid and urea for wood protection was explored. The preliminary study involved the deposition of water-soluble fluorescent materials synthesised from citric acid and urea onto the wood surface, revealing photoluminescent properties (Fig. 1). Further evaluation will explore the potential for surface modification (e.g. combining it with crosslinker or polymers) or using citric acid/urea directly for bulk wood modification with a focus on slowing down the greying process. Additionally, the physicochemical properties of the modified wood will also be evaluated.

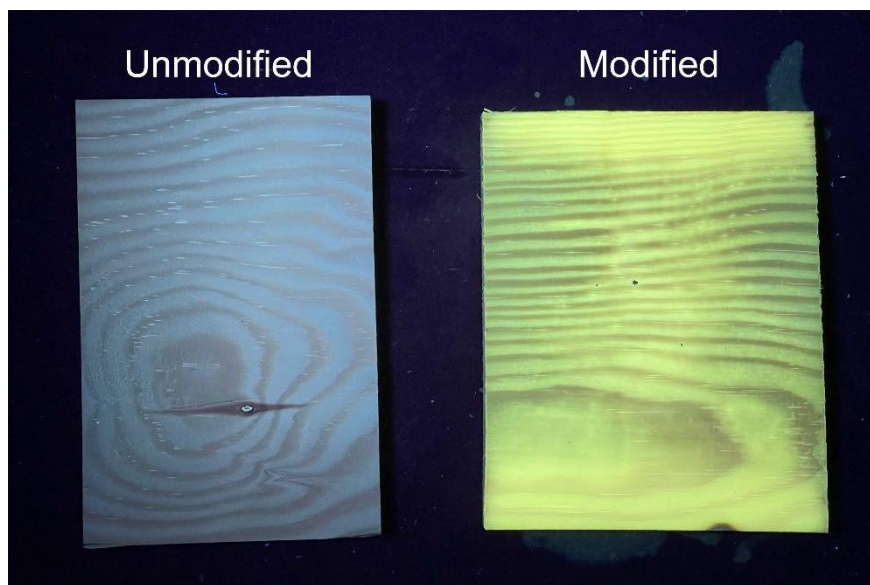


Figure 1. The unmodified and modified wood under UV-A light lamp.

## REFERENCES

- Adolfsson, K.H., Lin, C.F. and Hakkarainen, M. (2018). Microwave assisted hydrothermal carbonization and solid state postmodification of carbonized polypropylene. *ACS Sustainable Chemistry and Engineering*, **6**(8), 11105–11114.
- Adolfsson, K.H., Yadav, N. and Hakkarainen, M. (2020). Cellulose-derived hydrothermally carbonized materials and their emerging applications. *Current Opinion in Green and Sustainable Chemistry*, **23**, 18–24.
- Jirouš-Rajković, V. and Miklečić, J. (2021). Enhancing weathering resistance of wood - A review. *Polymers*, **13**(12), Article ID: 1980.
- Lee, S.H., Tahir, P.M., Lum, W.C., Tan, L.P., Bawon, P., Park, B.D., Al Edrus, S.S.A.O. and Abdullah, U.H. (2020). A review on citric acid as green modifying agent and binder for wood. *Polymers*, **12**(8), Article ID: 1692.
- Lin, C-f., Myronycheva, O., Karlsson, O., Mantanis, G.I., Jones, D. and Sandberg, D. (2023). A new wood-modification process based on in situ grafting of urethane groups: biological resistance and dimensional stability of carbamylated Scots pine wood. *Wood Material Science & Engineering*, **18**(3), 1160-1162.
- Strauss, V., Wang, H., Delacroix, S., Ledendecker, M. and Wessig, P. (2020). Carbon nanodots revised: The thermal citric acid/urea reaction. *Chemical Science*, **11**(31), 8256– 8266.

## Poster 8.08 - Identifying Influential Factors Affecting Wettability Patterns on Wood Surfaces through Multilevel Analysis

Valentina Lo Giudice<sup>1</sup>, Petar Antov<sup>2</sup>, Lubos Kristak<sup>3</sup>, Nicola Moretti<sup>1</sup>, Angelo Rita<sup>4</sup> and Luigi Todaro<sup>1</sup>

<sup>1</sup>School of agricultural, forestry, food and environmental science. University of Basilicata. V.le Ateneo Lucano 10, 85100 Potenza, Italy. E: valentina.logiudice@unibas.it, nicola.moretti@unibas.it, luigi.todaro@unibas.it

<sup>2</sup>Faculty of Forest Industry, University of Forestry, 1797 Sofia, Bulgaria. E: p.antov@ltu.bg

<sup>3</sup>Faculty of Wood Sciences and Technology, Technical University in Zvolen, Zvolen, Slovakia. E: kristak@tuzvo.sk

<sup>4</sup>Department of Agriculture, University of Naples Federico II, Via Università 100, 80055 Portici, NA, Italy. E: angelo.rita@unina.it

**Keywords:** modelling, statistical test, wettability, wood surface

### ABSTRACT

The reduction of hydrophilic characteristics and the migration of extractives induce important changes in terms of wood surface wettability. This research involves the application of two different strategies of wood modification, namely thermo-vacuum cylinder (TVP), and press-vacuum plant (PVP) (Ferrari *et al.* 2013, Cetera *et al.* 2019). The details of these treatments are provided in Table 1. The control group (NT-NS) did not undergo any treatment. To test whether variation in contact angle could be attributed to hydrothermal modification, we performed multilevel modeling (MLM). Multilevel modelling has gained popularity, particularly for hierarchically structured data, and surpasses classical regression in predictive accuracy (Barton 2016). This approach gains advantages when dealing with data that involves repeated. One intriguing feature of multilevel models is their capacity to independently estimate the predictive effects of a predictor and its group-level mean, which are sometimes interpreted as “direct” and “contextual” effects of the predictor. Wettability was modeled to assess the timedependent influence of two independent variables: steaming/thermal modification and heartwood/sapwood, on the dependent variable, wettability. Both marginal which quantifies the proportion of variance explained by the fixed factor(s) alone and conditional, which quantifies the proportion of variance explained by both the fixed and random factors, were computed. Nested replication/modification/wood-type random effects were also included in the models to account for the variability among samples. Intraclass Correlation (ICC) was also determined: it is a measure of reliability or dependence among individuals (Aguinis *et al.* 2013) and can help to assess whether random effects are present in the data. For all analyses, standardized regression coefficients, confidence interval (CI), and significance levels are reported (Tab. 2). Overall, the model emphasizes the significant contribution of wood surface modification to wettability patterns, with minimal influence related to intrinsic characteristics of the wood surface.

**Table 1: Treatment schedules.**

Method code	Steaming]	heating
NT-NS	-	-
PVP-S	x	x
PVP-NS	-	x
TVP-S	x	x
TVP-NS	-	x
NT-S	x	-

NS=unsteamed; NT= unmodified; S=steamed; PVP= press-vacuum plant; TVP= thermo-vacuum plant

**Table 2: Linear mixed-effect models. Contact angle (Ca) as a function of time and treatment.** *std Beta*: standardized coefficients of predictor variables; *CI* = confidence interval.; *ICC* = Intra-class Correlation; *Obs*: number of observations; *R<sup>2</sup><sub>m</sub>* = marginal R-square (proportion of variance explained by fixed factors); *R<sup>2</sup><sub>c</sub>* = conditional R-square (proportion of variance explained by fixed and random factors); Akaike Information Criterion. \*, \*\* and \*\*\* denote statistical significance at  $P < 0.05$ ,  $P < 0.01$  and  $P < 0.001$ , respectively.

	angle		angle	
	<i>std. Beta</i>	<i>CI</i>	<i>std. Beta</i>	<i>CI</i>
<b>Fixed Parts</b>				
(Intercept)				
time	-0.527 ***	-0.57 – -0.49	-0.527 ***	-0.56 – -0.49
(NT-S)			0.243 ***	0.20 – 0.29
(PVP-NS)			0.103 ***	0.06 – 0.15
(PVP-S)			0.371 ***	0.32 – 0.42
(TVP-NS)			0.311 ***	0.26 – 0.36
(TVP-S)			0.147 ***	0.10 – 0.19
<b>Random Parts</b>				
$\sigma^2$	287.683		238.100	
N <sub>replication:(direction:woodtype)</sub>	40		40	
N <sub>direction:woodtype</sub>	4		4	
N <sub>woodtype</sub>	2		2	
ICC <sub>replication:(direction:woodtype)</sub>	0.043		0.056	
ICC <sub>direction:woodtype</sub>	0.004		0.005	
ICC <sub>woodtype</sub>	0.006		0.007	
Observations	1680		1680	
R <sub>c</sub> <sup>2</sup>	0.327		0.443	
AIC	14337.623		14039.452	

*NS=unsteamed; NT= unmodified; S=steamed; PVP= press-vacuum plant; TVP= thermo-vacuum plant*

## REFERENCES

- Aguinis, H., Gottfredson, R.K. and Culpepper, S.A. (2013). Best-practice recommendations for estimating cross-level interaction effects using multilevel modeling. *Journal of Management*, **39**(6), 1490-1528.
- Barton, K. (2016). MuMIn: Multi-Model Inference. R package version 1.15.6. <https://CRAN.R-project.org/package=MuMIn>.



Cetera, P., Todaro, L., Lovaglio, T., Moretti, N. and Rita, A. (2016). Steaming treatment decreases MOE and compression strength of Turkey oak wood. *Wood Research*, **61**(2), 255-264.

Ferrari, S., Allegretti, O., Cuccui, I., Moretti, N., Marra, M. and Todaro, L. (2013). A reevaluation of turkey oak wood (*Quercus cerris* L.) through combined steaming and thermo- vacuum treatments. *BioResources*, **8**(4), 5051-5066.

## Poster 8.09 - Dimensional Stability and Sorption Properties of Acetylated and Non-Acetylated Birch Plywood as a Function of the Face Veneer Grain Angle

Jure Žigon<sup>1</sup>, Yue Wang<sup>2</sup>, Tianxiang Wang<sup>2</sup>, Aleš Straže<sup>1</sup> and Magnu Wålinder<sup>2</sup>

<sup>1</sup>Department of Wood Science and Technology, Biotechnical Faculty, University of Ljubljana, Jamnikarjeva ulica 101, 1000 Ljubljana, Slovenia. E: jure.zigon@bf.uni-lj.si, ales.straze@bf.uni-lj.si

<sup>2</sup>Building Materials Division, Department of Civil and Architectural Engineering, KTH Royal Institute of Technology, Brinellvägen 23, 100-44 Stockholm, Sweden. E: yue4@kth.se, tiawan@kth.se, magnus.walinder@byv.kth.se

**Keywords:** Acetylation, birch, dimension stability, plywood, sorption

### ABSTRACT

The dimensional stability of solid wood is problematic in most cases of its use in construction and represents one of the main disadvantages of solid wood in general. To overcome this problem, plywood as a wood engineering product represents a promising and widely used example. Plywood consists of several layers of veneer glued together crosswise in up to 50 mm thick boards. In addition to less pronounced in-plane anisotropy and resulting higher dimensional stability compared to solid wood, plywood can also show superior mechanical properties. Various adhesives and veneers are used in the production of plywood, of which phenol-resorcinol-formaldehyde resin and birch veneer are commonly used.

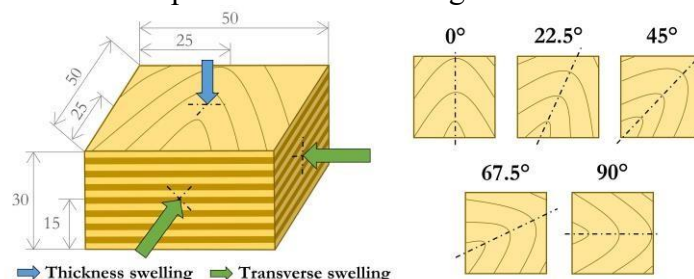
In addition, especially in applications, where improved resistance against elevated humidity and associated biological attack is required, the plywood consisting of acetylated wood veneers comes to expression. Acetylation of wood is generally a modification process in which the wood material is impregnated with liquid acetic anhydride at a temperature of 130 °C to 150 °C for up to 24 hours. In this way, the wood surface becomes more hydrophobic and resistant to mold and blue stain fungi (Yin *et al.* 2023). As shown in our previous study (Wang *et al.* 2022a), acetylated plywood has a slightly higher density but up to 50% lower moisture content than comparable nonacetylated plywood (Table 1). This also contributes to improved mechanical properties of acetylated plywood conditioned under various climatic conditions, such as bending strength and stiffness, compressive and shear strength. Due to the nature of plywood, the mechanical properties are highly dependent on the load-to-face veneer grain angle. For example, when the wood grain angle of the face veneer is shifted from 0° to 22.5°, 45°, 67.5°, and finally 90°, the edgewise bending strength is the highest at an angle of 0° and 90°, and lowest at an angle of 45° (Wang *et al.* 2022b).

**Table 1: Differences of moisture content of non-acetylated and acetylated birch plywood.**

Relative humidity [%] at 20 °C	Birch plywood moisture content		
	Non-acetylated	Acetylated	Difference [%]
35	8.4	4.2	-50
65	10.8	5.9	-45
100	21.6	10.8	-50

Despite numerous studies dealing with the mechanical properties of various types of plywood, not much has been reported on the dimensional stability and sorption properties of acetylated plywood, including its dependence on the grain angle of the face veneer. For the present study, panels of non-

acetylated and acetylated birch plywood with a nominal thickness of 30 mm were manufactured and supplied by Koskisen (Järvelä, Finland). From these panels, the samples of 50 mm × 50 mm (SIST EN 317) were prepared with five different angles of face veneer grain, as shown in Figure 1. The sorption properties and dimensional changes of both types of plywood were studied by conditioning the samples at a temperature of 23 °C and a relative humidity increasing from 10% to 98%. The relative humidity was increased by 10% every 7 days. The equilibrium moisture contents of non-acetylated and acetylated plywood obtained under each condition were calculated by drying the specimens at 103 °C for 48 hours after completion of conditioning.



**Figure 1: Schematic representation of the plywood sample with marked points for monitoring swelling (left; dimensions in mm) and the explanation of the face veneer grain angles (right).**

The results showed lower equilibrium moisture content and sorption hysteresis of acetylated birch plywood compared to non-acetylated birch plywood. In addition, the acetylated birch plywood exhibited lower dimensional changes in all three recorded directions than non-acetylated birch plywood. Both results confirm the contribution of acetylation to the higher hydrophobic character of the modified wood. In the case of thickness swelling, the positive contribution of acetylation was confirmed by the calculated values of the anti-swelling efficiency (ASE). However, for both types of plywood, the transverse swelling of the specimens was found to correlate with the grain angle of the face veneers. The results of this study are certainly important for the use of acetylated plywood in construction and for other purposes.

## REFERENCES

SIST EN 318 (2004) Wood based panels - Determination of dimensional changes associated with changes in relative humidity. *Slovenian Institute for Standardization*, 8 p.

Yin, H., Sedighi Moghaddam, M., Tuominen, M., Dédinaité, A., Wålinder, M., Swerin, A. (2022) Wettability performance and physicochemical properties of UV exposed superhydrophobized birch wood. *Applied Surface Science*, **584**, 152528.

Wang, Y., Wang, T., Crocetti, R., Wålinder, M. (2022a) Effect of moisture content on the angledependent edgewise flexural properties of unmodified and acetylated birch plywood. In: Proceedings of the 18<sup>th</sup> Annual Meeting of the Northern European Network for Wood Science and Engineering. Universität Göttingen, Göttingen, Germany, pp. 165-167.

Wang, T., Wang, Y., Crocetti, R., Wålinder, M. (2022b) Influence of face grain angle, size, and moisture content on the edgewise bending strength and stiffness of birch plywood. *Materials & Design*, **223**, 111227.

## Poster 8.10 - Upgrading Sawdust from Wood Bark to Produce New Thermoplastic Materials

Firmin Obounou Akong<sup>1</sup>, Célia Pinto<sup>1</sup>, Ania Belarbi<sup>1</sup>, Prabu Sejati Satria<sup>1,2</sup> and Philippe Gérardin<sup>1</sup>

<sup>1</sup>LERMAB, INRAE, Université de Lorraine, 54000 Nancy, France. E: Firmin.obounou-akong@univ-lorraine.fr

<sup>2</sup>Research Center for Biomass and Bioproducts, National Research and Innovation Agency (BRIN), 16911 Bogor, Indonesia

**Keywords:** bark, fatty acid, esterification, solvent free, thermoplastic

### ABSTRACT

Over the past few decades, there has been growing interest in developing new materials with thermoplastic properties obtained from biodegradable and renewable materials. Wood is one of the world's most abundant renewable natural materials. However, there are certain disadvantages, such as water sensitivity, insolubility in common organic solvents and high softening temperature, associated with the material wood. These drawbacks make it unsuitable for certain industrial uses currently served by synthetic polymers derived from petrochemicals. Against this backdrop, it is becoming increasingly urgent to develop new bio-based materials and innovative technologies to minimize widespread dependence on fossil resources. Bark is an important by-product of wood first transformation industries, which is currently valorized for low cost applications as energy or horticulture. A study on the conversion of wood bark sawdust into thermoplastic materials was undertaken to develop new ways of efficient valorization of wood waste with added value. Sawdust from wood bark was thermoplasticized by esterification. The hydroxyl groups of this sawdust were functionalized with various fatty acids in the presence of trifluoroacetic anhydride (TFAA) as an impelling agent. To optimize the esterification conditions and better understand reactivity of bark, series of experiments were carried out to determine the weight percent gain and ester content of acylated bark sawdust as a function of the length of the fatty chain used. Various reaction parameters, such as the presence or absence of solvent, the amount of TFAA used relative to the wood, temperature and reaction time, were investigated to produce esterified sawdust from wood bark with different ester contents. FTIR, <sup>13</sup>C solid state NMR, DSC, TMA and SEM were used to characterize the chemical, structural and thermoplastic properties of sawdust derived from esterified and unesterified wood bark. Experimental data showed that pre-swelling, the amount of TFAA and the length of the fatty chain grafted to the sawdust from wood bark had significant effects on the esterification reaction. Different wood barks from different species showed some variation in the ester content obtained. Thermoplasticized wood bark sawdust exhibited good softening properties and was easily moulded into materials in the form of translucent, flexible films. A wide range of softening temperatures from 110 to 180°C for esterified sawdust from wood bark was obtained, and these were largely dependent on mass gain. Films derived from esterified wood bark sawdust also showed surface hydrophobicity by contact angle measurement.

## Poster 8.11 - Micromorphological and chemical changes of Densified Ash Wood (*Fraxinus americana*)

Alexandra Miguel Guevara Castillo<sup>1</sup>, Raúl Rodríguez Anda<sup>1</sup>, Francisco Javier Fuentes Talavera<sup>1</sup> and José Antonio Silva Guzmán<sup>1</sup>

<sup>1</sup>Departamento de Madera Celulosa y Papel, Universidad de Guadalajara, Km. 15.5 Carretera Guadalajara Nogales, Predio Las Agujas, Mpio. Zapopan, Jalisco, 45020, México. E: alexandra.guevara@alumnos.udg.mx; jantonio.silva@academicos.udg.mx

**Keywords:** ash wood, chemical changes, micromorphology, microstructure, thermocompression, wood densification.

### ABSTRACT

In order to improve some wood properties for a better eco-friendly performance different modification methods have been developed. One of the most physical methods commonly used is thermocompression densification, which increases wood density and hardness (Yu *et al.* 2017, Yu *et al.* 2022), and consequently improving dimensional stability, permeability, mechanical strength, natural durability and surface quality (Chen *et al.* 2020, Li *et al.* 2016). The objective of this study was to determine the effect of the thermomechanical process into the cell wall structure and chemical composition of ash wood (*Fraxinus Americana*) previously densified (Revilla, 2017) by combining moisture content (12 and 30%), temperature (120, 140 and 160°C) and compression rates (20,30 and 40%). A softening effect of the cell walls occurred in early wood causing its viscous compaction (Fig. 1) and some fractures were observed specifically in treatments carried out at 12% MC (Fig. 2). FTIR analysis (Fig. 3) and chemical component extractions studies showed significant chemical changes especially in the maximum compression and temperature treatments where the extractives and carbohydrates were degraded due to the exposure times at elevated temperatures. There was an increase in the lignin contents due to the cross-linking of the hydrolyzed holocelluloses with lignin chains. Consequently, some alfa cellulose contents were reduced resulting in an increase in amorphous cellulose (beta cellulose) and inverse.

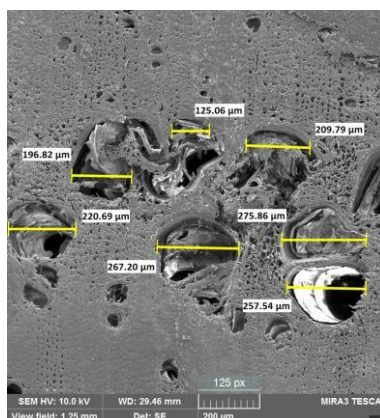


Figure 1: Densified ash wood. Wood plastification, no ruptures observed

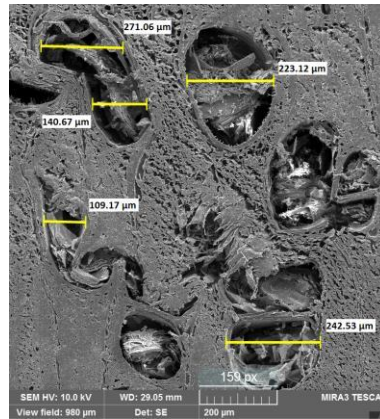


Figure 2: Densified ash wood at 12% MC. Microfractures present

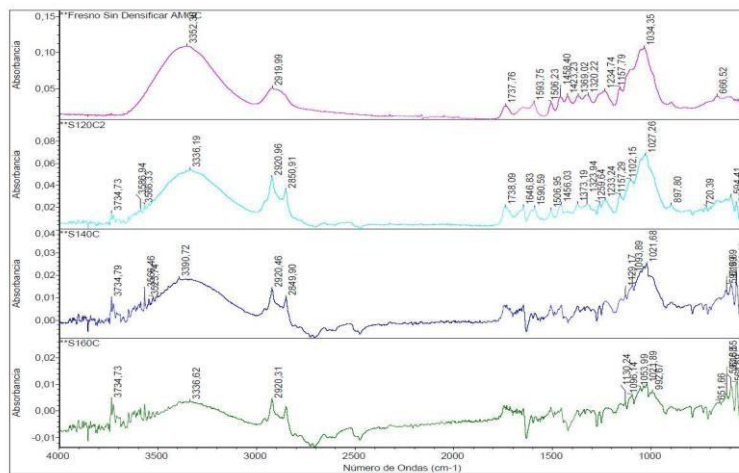


Figure 3: FTIR analysis of dry specimens at different temperatures and 40% C.R

## REFERENCES

- Chen, C. J.; Kuang, Y. D.; Zhu, S. Z.; Burgert, I.; Keplinger, T.; Gong, A.; Li, T.; Berglund, L.; Eichhorn, S. J.; Hu, L. B. (2020). Structure-Property-Function Relationships of Natural and Engineered Wood. *Nat. Rev. Mater.* 2020, 5(9), 642–666.
- Li L., Wang X., and Wu F. (2016). Chemical Analysis of Densification, Drying, and Heat Treatment of Scots pine (*Pinus sylvestris* L.) through a Hot-Pressin. *BioResources*, 11(2), 3856-3874.
- Revilla E. (2017). Ash (*Fraxinus* sp.) wood densification and the effect on its physicochemical properties. Master's degree thesis. Universidad de Guadalajara, México.
- Yu Y., Zhang F., Zhu S., and Li H. "Effects of High-Pressure Treatment on Poplar Wood: Density Profile, Mechanical Properties, Strength Potential Index, and Microstructure". (2017). *BioResources*, (12)6283-6297.
- Yu L., Chang-Hua, F., Yi-Fan M., and Ben-Hua Fei (2022). Wood mechanical densification: a review on processing, *Materials and Manufacturing Processes*, 37:4, 359-37.

## Poster 8.12 - Development of Innovative Methods for Assembling Lignocellulosic Materials for the Manufacture of Glasses

Adrien Magne<sup>1</sup>, Juliette De Nas De Tourris<sup>1</sup>, Jennifer Afrim<sup>1</sup>, Teldja Benzid<sup>1</sup>, Prabu Satria Sejati<sup>1</sup>, Firmin Obounou Akong<sup>1</sup>, Robin Féron<sup>2</sup> and Philippe Gérardin<sup>1</sup>

<sup>1</sup>LERMAB, INRAE, Université de Lorraine, 54000 Nancy, France. E: Firmin.obounou-akong@univ-lorraine.fr

<sup>2</sup>IN'Bô Z.A Les Bouleaux – 88240 les Voivres. E: adrien.magne@univ-lorraine.fr

**Keywords:** mapple wood, fatty acid, esterification, solvent free, thermoplastic

### ABSTRACT

The combination of lignocellulosic fiber fabrics bonded with resin is widely used to prepare multi-layer materials with improved mechanical strength and dimensional stability. In'Bô uses natural materials to manufacture glasses frames. More specifically, flax fibers are shaped and given their final strength by the use of a binder (cellulose acetate). As part of its product improvement approach, In'bô is keen to develop thermoformable materials for adjusting frames to customer morphology, and to design eyewear made solely from flax fibers (without using cellulose acetate as a matrix). In collaboration with the Laboratoire d'études et de recherche sur le matériau bois (Lemab), this research was carried out to provide a new alternative to flexible, thermoplastic materials based, initially developed with wood, on natural fibers through the esterification of flax fiber using myristic acid, propionic acid and trifluoroacetic anhydride (TFAA) as impelling agent without any solvent. A series of experiments was carried out to graft the aforementioned fatty acids onto ground or unground flax fibre, using trifluoroacetic anhydride (TFAA) as impelling agent without any solvent. The reaction was carried out rapidly, leading to a high ester content. Most of the hydroxyl groups in the flax structure reacted with the target fatty acids, as demonstrated by FTIR and CPMAS 13C NMR spectroscopy. Flax esterified with myristic acid showed higher thermal stability than wood esterified with propionic acid by TGA and DSC, and produced several softening temperatures observed by TMA. Thermoplastic and translucent films were obtained after high-temperature pressing. Scanning electron micrographs revealed that esterified flax fiber pressed at high temperature showed complete disappearance of the fibrous structure to make way for a smooth, homogeneous surface, indicating that thermal fluidity was achieved during pressing. Films obtained from ground or unesterified flax fiber also showed surface hydrophobicity by contact angle measurement. Finally, the mechanical properties of the materials studied were determined. Another alternative developed during this study concerns the modification of solid wood samples using the preceding esterification procedure followed by hot pressing of the obtained modified wood to yield a new thermoplastic material, which can be involved in the fabrication of glasses frames.

## Poster 8.13 - Exploring the Solid Wood Modification with Preserved Hierarchical Structure via Non-Cellulosic Substance Removal

Chin Yi Hien<sup>1,2,3</sup>, Pascal Biwolé<sup>1,4</sup>, Gril Joseph<sup>1,5</sup>, Vial Christophe<sup>1</sup>, Moutou Pitti Rostand<sup>1,6</sup>, Ouldboukhitine Salaheddine<sup>1</sup>, Labonne Nicolas<sup>2</sup>, Horikawa Yoshiki<sup>3</sup>

<sup>1</sup>Université Clermont Auvergne, CNRS, SIGMA Clermont, Institut Pascal, France.

<sup>2</sup>Dagard Company, 23600 Boussac, France.

<sup>3</sup>Institute of Agriculture, Tokyo University of Agriculture and Technology, Fuchu, Tokyo 183-8509, Japan.

<sup>4</sup>MINES Paris, PSL Research University, PERSEE - Center for Processes, Renewable Energies and Energy Systems, France.

<sup>5</sup>Université Clermont Auvergne, INRAE, PIAF, 63000 Clermont Ferrand, France.

<sup>6</sup>CENAREST, IRT, BP 14070, Libreville, Gabon. E: yi\_hien.chin@uca.fr

**Keywords:** bio-based material, delignification, drying technologies, nanotechnology, poplar wood, wood modification

### ABSTRACT

Wood is a bio-based material that has always been used for various purposes due to its excellent mechanical, thermal and acoustic properties. Innovations have been made in wood modification technology to create new functionalities while improving its current advantages. Wood is composed of three main chemical elements: cellulose, hemicellulose and lignin. Cellulose contributes to the mechanical strength of wood fibers through its polymerization and crystallinity. Hemicellulose, which is hygroscopic and sensitive to heat, creates a bond between cellulose and lignin to consolidate mechanical properties. Lignin is a natural glue between the wood cells and the middle lamella. This study focuses on the modification of wood by the removal of non-cellulosic substance to create a cellulose skeleton and to increase porosity in wood. The creation of pores contributes to lower density and therefore lower thermal conductivity. Besides, a new composite can be invented by impregnation of other materials between the cellulose fibers. Top-down approach combining chemical and mechanical treatments increases the production scalability for further application. This work starts by reviewing the protocol of various wood delignification methods that preserve the natural hierarchical structure of solid wood, as summarized in Table 1. Then, it focuses on experimental trials on French poplar wood using kraft, alkali and organosolv methods, followed by the drying comparison.

**Table 1: Summary of delignification methods for different wood samples with thickness > 5 mm.**

Wood species & dimension [mm]	Chemical reagents	Cooking duration [hour]	Purpose	Authors
Basswood 120 x 30 x 12	Step 1: NaOH + Na <sub>2</sub> SO <sub>3</sub> Step 2: H <sub>2</sub> O <sub>2</sub>	Step 1: 3 Step 2: 3	Thermal insulator	T. Li <i>et al.</i> , 2018
Beech 40 x 50 x 5	Step 1: NaOH + Na <sub>2</sub> SO <sub>3</sub> Step 2: H <sub>2</sub> O Step 3: H <sub>2</sub> O <sub>2</sub>	Step 1: 48 Step 2: 12 Step 3: Until white	Transparent wood by nanoparticles infiltration	Yu <i>et al.</i> , 2017
Spruce 100 x 20 x 10	H <sub>2</sub> O <sub>2</sub> + Acetic acid	6	Densification by compression	Frey <i>et al.</i> , 2018



Basswood 100 x 50 x 40	Steam of H <sub>2</sub> O <sub>2</sub> + Acetic acid	20	Transparent wood by resin infiltration	H. Li <i>et al.</i> , 2019
Balsa 20 x 5 x 5	Peracetic acid	6	Flame retardancy by clay impregnation	Fu <i>et al.</i> , 2017
Balsa 10 x 15 x 15	NaClO <sub>2</sub>	18	Oil/water separation by epoxy impregnation	Fu <i>et al.</i> , 2018
Cryptomeria japonica 10 x 10 x 10	Step 1: Ethylene glycol + H <sub>2</sub> O + 97% H <sub>2</sub> SO <sub>4</sub> Step 2: NaClO <sub>2</sub>	Step 1: 1 Step 2: 56	Lignin-free wood for understanding of anatomical structure	Horikawa <i>et al.</i> , 2020
Paulownia 600 x 45 x 12.5	NaOH	1	Thermal insulator	Zhao <i>et al.</i> , 2023
Poplar 40 x 20 x 20	Deep eutectic solvent	12	Water transportation in solar steam generation devices	Chen <i>et al.</i> , 2019

## REFERENCES

- Chen, Z., Dang, B., Luo, X., *et al.* (2019). Deep Eutectic Solvent-Assisted In Situ Wood Delignification: A Promising Strategy To Enhance the Efficiency of Wood-Based Solar Steam Generation Devices. *ACS Applied Materials & Interfaces*, 11(29), 26032–26037.
- Frey, M., Widner, D., Segmehl, *et al.* (2018). Delignified and Densified Cellulose Bulk Materials with Excellent Tensile Properties for Sustainable Engineering. *ACS Applied Materials & Interfaces*, 10(5), 5030–5037.
- Fu, Q., Ansari, F., Zhou, Q., & Berglund, L. A. (2018). Wood Nanotechnology for Strong, Mesoporous, and Hydrophobic Biocomposites for Selective Separation of Oil/Water Mixtures. *ACS Nano*, 12(3), 2222–2230.
- Fu, Q., Medina, L., Li, Y., *et al.* (2017). Nanostructured Wood Hybrids for Fire-Retardancy Prepared by Clay Impregnation into the Cell Wall. *ACS Applied Materials & Interfaces*, 9(41), 36154–36163.
- Horikawa, Y., Tsushima, R., Noguchi, K., *et al.* (2020). Development of colorless wood via twostep delignification involving alcoholysis and bleaching with maintaining natural hierarchical structure. *Journal of Wood Science*, 66(1), 37.
- Li, H., Guo, X., He, Y., & Zheng, R. (2019). A green steam-modified delignification method to prepare low-lignin delignified wood for thick, large highly transparent wood composites. *Journal of Materials Research*, 34(6), 932–940.
- Li, T., Song, J., Zhao, X., *et al.* (2018). Anisotropic, lightweight, strong, and super thermally insulating nanowood with naturally aligned nanocellulose. *Science Advances*, 4(3), eaar3724.
- Yu, Z., Yao, Y., Yao, J., *et al.* (2017). Transparent wood containing Cs x WO<sub>3</sub> nanoparticles for heat-shielding window applications. *Journal of Materials Chemistry A*, 5(13), 6019–6024.
- Zhao, X., Liu, Y., Zhao, L., *et al.* (2023). A scalable high-porosity wood for sound absorption and thermal insulation. *Nature Sustainability*.

## Poster 8.14 - Malic acid/glycerol Polyester treated beech boards: Curing Kinetics and Density Distribution

Emmanuel Fredon<sup>1</sup>, Romain Rémond<sup>1</sup>, Adele Chabert<sup>1,2</sup>

<sup>1</sup>Laboratoire d'Etude et de Recherche sur le Matériau Bois, Université de Lorraine, 27 rue Philippe Seguin, 88051 Epinal, France. E: emmanuel.fredon@univ-lorraine.fr, romain.remond@univ-lorraine.fr

<sup>2</sup>Wood Biology and Wood Products, Georg-August-Universität Göttingen, Büsgenweg 4, 37077 Göttingen, Germany. E: adele.chabert@uni-goettingen.de

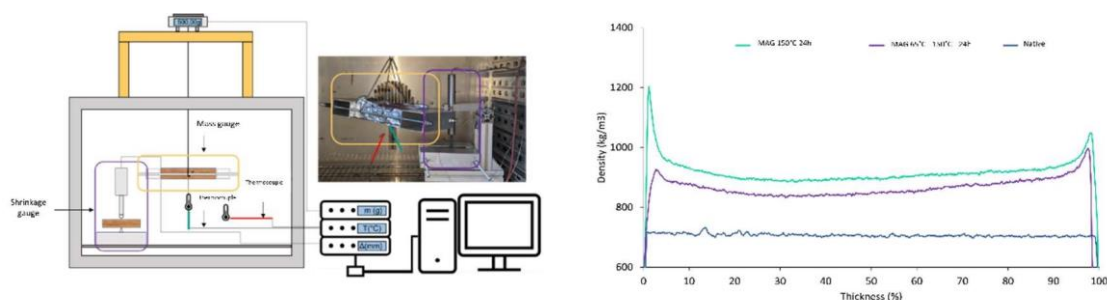
**Keywords:** beech, density profile, glycerol, malic acid, polyester

### ABSTRACT

Over the last decade, the study of polyester treatment has emerged and greatly increased. This wood modification method lies in the impregnation of wood with polyacids and polyol monomers followed by a curing step that ensures the polymerization and fixation of the esterified oligomers, setting the wood cells in a swollen state. This treatment limits water induced adsorption and desorption and confers durability and hardness improvements. State of art shows that different combinations of monomers have been successfully investigated in this way, such as lactic acid (Grosse 2019), citric acid and glycerol (L'Hostis 2018), succinic anhydride and glycerol (L'Hostis 2020), citric acid and sorbitol (Larnoy 2018). These approaches, as reviewed by Kurkowiak (2022), are alternatives to now well-known industrial scale polyfurfurylic (Kebony trademark) and anhydride acetic (Accoya trademark) chemical modification treatments (Rowell 2009, Lande 2004). Regarding our recent work on malic acid-glycerol (MaG) treatment on small samples (Chabert 2022), our team became interested in the up-scaling of this treatment. The following questions then arose. What are the best curing conditions? Could the different phases of the treatment be observed in the kinetics? Is the treatment homogeneous in the final product?

For this purpose, the kinetics were measured during the curing of a beech wood board (100x27x150mm<sup>3</sup>) impregnated with MAG (figure 1a). A heat resistant shell was coated on the board's extremities, then it was hanged and linked to a scale. Thermal and mass measures were gathered using a data capture station. The final distribution of the density in the modified wood was measured using a X-ray densitometer, DAX 5000 (GreCon GmbH, Alfeld, Germany). Additionally, the wood cell deformations were observed using SEM.

The evolution of the board's mass and temperature enabled us to identify the different phases characteristics of a single-deck drying and curing process at 150 °C for 24 hours. Despite the severity of the dry curing conditions, the polyester treatment does not appear to cause cracks or cell collapses or deformations, whereas it has been observed on the water-impregnated board, as shown by SEM analyses. After treatment, a significant increase in density is shown and specifically at the surface of the board. This phenomenon has previously been described by Kurkowiak (2022) and Klüppel and Mai (2013). Several strategies (reducing thickness, pre-drying, waterless hot impregnation) were then studied to evaluate their effects on the kinetics and on the reduction of density gradient but provided limited improvements. Nevertheless, the denser 'shell' at the periphery of the board can be an interesting feature depending on the desired use.



**Figure 1: a) Tests set-up for mass loss and shrinkage measurement, b) density profile along the dimensionless thickness of the modified wood**

## REFERENCES

- Chabert A. J, Fredon, E, Rémond R. (2022). Improving the stability of beech wood with polyester treatment based on malic acid, *Holzforschung*, 76(3), 268–275.
- Grosse, C., Grigsby, W. J., Noel, M., Treu, A., Thevenon, M.F. and Gérardin, P. (2019). Optimizing chemical Wood Modification with Oligomeric Lactic acid by screening of processing condition, *Journal of wood Chemical Technology*, 39, 385–398.
- Klüppel, A and Mai, C (2013). The influence of curing conditions on the chemical distribution in wood modified with thermosetting resins, *Wood science and technology*, 47(3), 643–658.
- Kurkowiak K, Emmerich L. and Militz H (2022). Wood chemical modification based on biobased polycarboxylic acid and polyols - status quo and future perspectives, *Wood Material Science and Engineering*, 17(6) 1040-1054.
- Kurkowiak K, Mayer A. K, Emmerich L and Militz H. (2022). Investigations of the Chemical Distribution in Sorbitol and Citric Acid (SorCA) Treated Wood—Development of a Quality Control Method on the basis of Electromagnetic Radiation, *Forests*, 13, 151-165.
- Lande, S., Westin, M., Schneider, M. (2004). Properties of furfurylated wood, *Scandinavian Journal of Forest Research*, 19, 22–30.
- Larnøy, E., Karaca, A., Gobakken, L. R. and Hill, C. A. S. (2018). Polyesterification of Wood Using Sorbitol and Citric Acid under Aqueous Conditions. *International Wood Products Journal*, 9(2), 66–73.
- L’Hostis, C., Fredon, E., Thévenon, M. F., Gerardin, P. (2018) Improvement of beech wood properties by in situ formation of polyesters of citric acid and tartaric acid in combination with glycerol, *Holzforschung*, 72(4), 291-299.
- L’Hostis, C., Fredon, E., Thévenon, M. F., Santiago-Medina, F. J. and Gerardin, P. (2020). Beech Wood Treated with Polyglycerol Succinate a New Effective Method for Its Protection and Stabilization, *Holzforschung*, 74(4), 351–61.
- Rowell, R. M. Ibach R. E, McSweeney, J, Nilsson, T. (2009). Understanding decay resistance, dimensional stability and strength changes in heat-treated and acetylated wood, *Wood Material Science Engineering*, 4, 14–22.

## Poster 8.15 - Implementing Fire Retardants into a Biobased Adhesive System for Wood-Based Composites

Luka Kopač<sup>1,2</sup>, Alexander Scharf<sup>1</sup>, Dennis Jones<sup>1</sup>, Dick Sandberg<sup>1</sup> and Sergej Medved<sup>2</sup>

<sup>1</sup>Wood Science and Engineering, Luleå University of Technology, Forskargatan 1, 931 87 Skellefteå, Sweden. E: lukkop-3@student.ltu.se, alexander.scharf@ltu.se, dennis.jones@ltu.se, dick.sandberg@ltu.se

<sup>2</sup>Department of wood science and technology, Biotechnical Faculty - University of Ljubljana, Rožna dolina, Cesta VIII/34, Slovenia. E: sergej.medved@bf.uni-lj.si

**Keywords:** density profile, surface densification, test method, wear, wood protection

### ABSTRACT

In recent years, there has been a push to create composite materials using nonformaldehyde resins, preferably from bio-based sources. Traditional interior particleboards typically use urea-formaldehyde (UF) resins, while exterior-grade boards may use melamine-urea-formaldehyde, epoxy, or polyurethane resins. Polyesterification reactions involving polyacids and polyols produce thermoset resins (Tham *et al.* 2015). Recent research has expanded these reactions to solid wood, employing bio-based compounds like citric acid, glycerol, glucose, and sorbitol. The cost-effective use of citric acid and sorbitol has led to commercial wood modification treatments, such as CIOL® in Norway and corresponding SorCA treatment in Germany. This approach of using citric acid/sorbitol was adapted by Lin *et al.* (2022) in the manufacture of particleboard. As the popularity of these products continues to rise, concerns about their fire resistance become increasingly significant. Consequently, implementing fire-retardant treatments for panel products is necessary. This study investigated the feasibility of implementing either a proprietary fire-retardant ionic liquid (IL) supplied by Palonot Oy, Finland, or ammonium dihydrogen phosphate (ADP) into the previously established citric acid and sorbitol adhesive system.

Table 1 shows the chemical used in combination with wood processing residual sawdust containing Scots pine (*Pinus sylvestris* L.) and Norway spruce (*Picea abies* (L.) Karst.) particles.

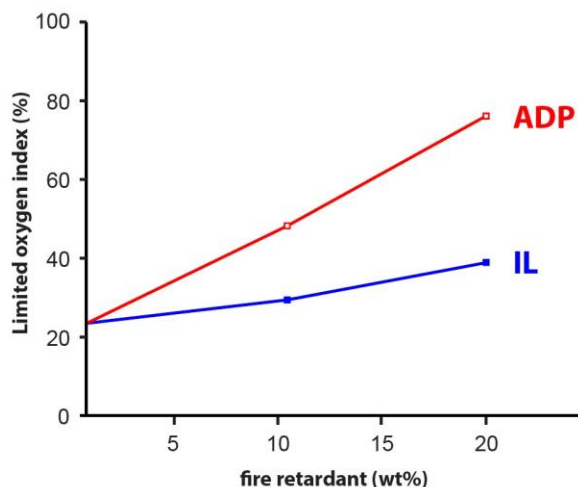
**Table 1: Overview of chemical combinations of the modification treatment of sawdust.**

Group	Citric acid/sorbitol <sup>a</sup> [wt%]	IL <sup>b</sup> [wt%]	ADP <sup>c</sup> [wt%]	Solid content solution [wt%]
Control	10	0	0	10
IL_10	10	10	0	20
IL_20	10	20	0	30
ADP_10	10	0	10	20
ADP_20	10	0	20	30

<sup>a</sup>citric acid and sorbitol in a 3:1 molar ratio, <sup>b</sup>ionic liquid system, <sup>c</sup>ammonium dihydrogen phosphate

The chemicals were dissolved in deionized water to create a solution containing 10 to 30 wt% solids, as shown in Table 1. For each group, dried wood particles were combined with the respective solution in a rotating vessel through spraying to achieve the desired wt% chemical content relative to the particle mass. These particle-chemical mixtures were then dried in an oven at 70 °C for 24 hours to remove most of the water. Following the drying process, the resulting mat was subjected to open-system pressing for 10 minutes at temperatures of 180 °C, 200 °C, or 220 °C, to a target thickness of 7.5 mm and a target density of 700 kg/m<sup>3</sup>. After pressing and cooling, the boards were trimmed and were conditioned at 20 °C and 65% RH for one week. Limited Oxygen Index (LOI) was tested to

determine fire retardancy. Thickness swelling, internal bonding strength, bending performance were tested according to the EN 317, EN 319 and EN 310 standards, respectively (CEN 1993a, b, c).



**Figure 1: Measurements of Limited oxygen index for different fire retardants and loadings.**

Initial results of LOI showed a linear trend of fire-retardant loading to LOI for both chemicals (Fig. 1), with ADP exhibiting significantly higher values than IL. Ongoing work will study the effect of the fire-retardant presence on the hygroscopic and mechanical properties of the boards, particularly whether the fire retardants exhibit any detrimental effects for the polymerisation of citric acid/sorbitol.

## REFERENCES

CEN (1993a). EN 317. Particleboards and fibreboards–Determination of swelling in thickness after immersion in water. European Committee for Standardisation, Brussels. Belgium.

CEN (1993b). EN 319. Particleboards and fibreboards–determination of tensile strength perpendicular to the plane of the board. European Committee for Standardisation, Brussels. Belgium.

CEN (1993c). EN 310. Wood-based panels–Determination of modulus of elasticity in bending and of bending strength. European Committee for Standardisation, Brussels. Belgium.

Lin, C.-F., Karlsson, O., Jones, D. and Sandberg, D. (2022). Bio-based adhesive derived from citric acid and sorbitol for wood-composite manufacture. *Wood Material Science & Engineering* **17**(5), 397-399.

Tham, W.H., Wahit, M.U., Kadir, M.R.A., Wong, T.W. and Hassan, O. (2016). Polyol-based biodegradable polyesters: a short review. *Reviews in Chemical Engineering* **32**(2), 201-221.

**Poster 8.16 - Laser incising – a Philosophical Shift: From timber Treatment to Wood Modification**

Morwenna Spear<sup>1</sup>, Paul Mason<sup>2</sup>, Geraint Williams<sup>2</sup> and Graham Ormondroyd<sup>1</sup>

<sup>1</sup>The BioComposites Centre, Bangor University, Bangor, LL57 2UW, U.K. E: m.j.spear@bangor.ac.uk

<sup>2</sup>Millennium Lasers, Llandarcy, Neath, U.K.

**Keywords:** laser incision, permeability, refractory species, wood anatomy, wood modification

**ABSTRACT**

Timber incising has been known and investigated for many years, yet remains a very diverse field. Often simplified into three main categories: mechanical incision, laser incision and bioincising, this group of technologies is commonly seen as the domain of timber preservation. To date only mechanical incising has been fully commercialised. Yet laser incision offers essential differences which mark it out as suitable for wood modification and high value products, with great aesthetic appeal and high market value. Wood modification requires near-perfect distribution of the treatment agent. This is achieved in laser incision through careful matching of incision patterns and strategies with the timber species. Building on successful collaborative research, we have demonstrated the technology's application in a full scale (plank length 3m) resin modification process for both softwoods and hardwoods. In this poster we present the concept of laser incision and the reasons it is so well suited to revolutionise all fluid-based wood modification systems (e.g. chemical modifications, resin and polymer modifications) as well as other protective treatments.

## Poster 8.17 - X-ray CT Scanning as a Method for Quantifying Mineralization in Spruce and Beech Woodblock

Marcy Durimel<sup>1,2</sup>, Liselotte De Ligne<sup>1,2</sup>, Bogdan Parakhonskiy<sup>3</sup>, Jan Van den Bulcke<sup>1,2</sup>, Andre Skirtach<sup>2</sup> and Joris Van Acker<sup>1,2</sup>

<sup>1</sup>UGent-Woodlab, Department of Environment, Faculty of Bioscience Engineering, Ghent University, 9000 Gent, Belgium. E: marcy.durimel@ugent.be

<sup>2</sup>UGCT, UGent Centre for X-ray Tomography, Proeftuinstraat 86, 9000 Gent, Belgium

<sup>3</sup>Nano-Biotechnology Laboratory, Department of Biotechnology, Faculty of Bioscience Engineering, Ghent University, 9000 Ghent, Belgium

**Keywords:** Earlywood, latewood, wood based-building product, wood mineralization, X-ray CT scanning

### ABSTRACT

Wood and wood-based building materials are considered key elements of our future socio-economic environment. They are a relevant nature-based solutions to climate change, as they are critical for addressing various aspects of the sustainable development goals highlighted by international, European, and national political structures. Wood is mainly used as a structural material, in building structure envelopes, and as insulation. Its biodegradability is an asset at the end of its service life, but also an important parameter to control to maintain a good product lifespan (Pomponi et al. 2020).

Wood mineralization has attracted a great deal of interest in recent years in the field of wood modification. Compared to traditional wood modification processes, mineralization is a simple, environmentally-friendly process that improves wood properties. By incorporating minerals into the biological matrix of wood, increased fire resistance can be obtained (Huang et al. 2018; Hernandez et al. 2022). Weight gain of the material is often used to characterize the effectiveness of the treatment (Merk et al. 2015; 2016). The main techniques used to show the distribution of mineralization inside wood are visualization techniques such as SEM (scanning electron microscopy) and Raman spectroscopy. However, these techniques only visualize the distribution in 2D and do not allow to quantify it. In this paper, we propose the use of X-ray CT scanning, a nondestructive technique to visualize and quantify mineralization in 3D to quantify the difference in mineralization in early- and latewood in spruce and beech wood blocks, the average penetration depth of mineralization, and the mineralization in different regions of the woodblocks.

The normalized grey value ( $(grey\ value_{post} - grey\ value_{pre}) / grey\ value_{pre}$ ) is associated with the mineralization inside the wood blocks. Spruce samples have the highest weight gain and the highest normalized grey value for the full blocks. Beech samples have a lower weight gain, which is represented by a lower normalized grey value in X-ray CT scanning. Spruce samples show a higher grey value difference in the earlywood region than in the latewood region, which means that mineralization occurs to a greater extent in the earlywood region than in the latewood region. The grey value difference for the beech samples in the earlywood and latewood regions is similar, meaning that mineralization does not occur to a significantly larger extent in either the earlywood or latewood regions.

### REFERENCES

Hernandez, Vicente, Romina Romero, Sebastián Arias, and David Contreras. 2022. 'A Novel Method for Calcium Carbonate Deposition in Wood That Increases Carbon Dioxide Concentration and Fire Resistance'. *Coatings* 12 (1): 72.

Huang, Lili, Xiaolin Yao, Yongtong Huang, and Qingsong Wang. 2018. 'The Preparation of CaCO<sub>3</sub>/Wood Composites Using a Chemical Precipitation Method and Its Flame-Retardant and Mechanically Beneficial Properties'. *BioResources* 13 (3): 6694–6706.

Merk, Vivian, Munish Chanana, Sabyasachi Gaan, and Ingo Burgert. 2016. 'Mineralization of Wood by Calcium Carbonate Insertion for Improved Flame Retardancy'. *Holzforschung* 70 (9): 867–76.

Merk, Vivian, Munish Chanana, Tobias Keplinger, Sabyasachi Gaan, and Ingo Burgert. 2015. 'Hybrid Wood Materials with Improved Fire Retardance by Bio-Inspired Mineralisation on the Nano- and Submicron Level'. *Green Chemistry* 17 (3): 1423–28.

Pomponi, Francesco, Jim Hart, Jay H. Arehart, and Bernardino D'Amico. 2020. 'Buildings as a Global Carbon Sink? A Reality Check on Feasibility Limits'. *One Earth* 3 (2): 157–61.



## Poster 8.18 - Wood Surface Modification Using Metal and Ceramics to Make Wood Fire and Termite Resistant

Laurence Podgorski<sup>1</sup> and Alain Denoirjean<sup>2</sup>

<sup>1</sup>Technological Institute FCBA, Allée de Boutaut, BP227, Bordeaux, France. E: Laurence.podgorski@fcba.fr

<sup>2</sup>IRCER, Université de Limoges, 12 rue Atlantis, 87000 Limoges, France. E: alain.denoirjean@unilim.fr

**Keywords:** ceramic, fire, metal, modification, termites, thermal spraying, wood

### ABSTRACT

Thermal spraying can be used to coat wood surfaces with metal (Nejad *et al.* 2017) or ceramics (Podgorski *et al.* 2020). The characteristics of the sprayed materials can modify the wood surface and bring new properties such as termite resistance and fire reaction improvement. In this project, Scots pine samples and spruce plywood were used. All the samples have been coated with a first layer of metal, which is essential for the adhesion of a ceramic layer. Pre-tests with copper as an undercoat had previously demonstrated the feasibility of ceramic deposits on wood (Podgorski *et al.* 2020). In this project, brass has been chosen as the metallic undercoat because it is cheaper than copper. Three oxide ceramics were deposited on the brass-coated wood: i) Al<sub>2</sub>O<sub>3</sub>, ii) Al<sub>2</sub>O<sub>3</sub>/ZrO<sub>2</sub>, iii) Al<sub>2</sub>O<sub>3</sub>/TiO<sub>2</sub>. Test specimens were also produced with brass coating only. The treatments were carried out at the IRCER premises using thermal spraying. The mean thickness (brass + ceramic) was 380 µm including 228 µm for brass. The surface modification provided an original look to the wood surfaces and brought different colors (Fig. 1).

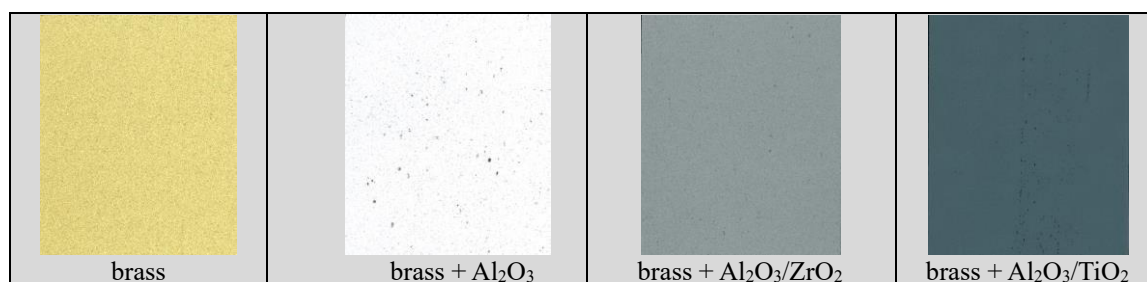


Figure 1: Aspect of the Scots pine samples modified using brass and ceramics.

The metal/ceramic deposits led to almost systematic warping of the specimens. This deformation was estimated by measuring the maximum deflection at the centre of the samples. The greater the quantity of metal/ceramic deposited on the wood, the greater the deflection.

Liquid water permeability tests (EN 927-5) have shown that the brass and ceramic coatings on wood had high water uptake (mean 2 825 g/m<sup>2</sup>) compared with organic coatings (water uptake < 175 g/m<sup>2</sup>) as shown in Fig.2.

The resistance of coatings to termites (*Reticulitermes grassei*) was assessed by adapting method EN 118 (6 replicates/coating, 150 termites/replicate). No termites were able to penetrate the modified wood surfaces and the insects died during the 8 weeks of exposure. The effectiveness of the treatments was attributed to the high hardness of the coatings. Reaction to fire tests (SBI) on spruce plywood coated with brass and with brass+Al<sub>2</sub>O<sub>3</sub>/ZrO<sub>2</sub> have shown an improvement of the fire growth rate index (FIGRA) and total heat release (THR600s) compared to unmodified spruce plywood (Fig.3). Both coatings lead to a reaction to fire class very close to C compared to class D for the control (uncoated spruce plywood). The brass+Al<sub>2</sub>O<sub>3</sub>/ZrO<sub>2</sub> coating slightly improved the results compared with the brass coating. In terms of smoke performance, the classification for brass was s1 as for uncoated wood and s2 for the brass+Al<sub>2</sub>O<sub>3</sub>/ZrO<sub>2</sub> coating.

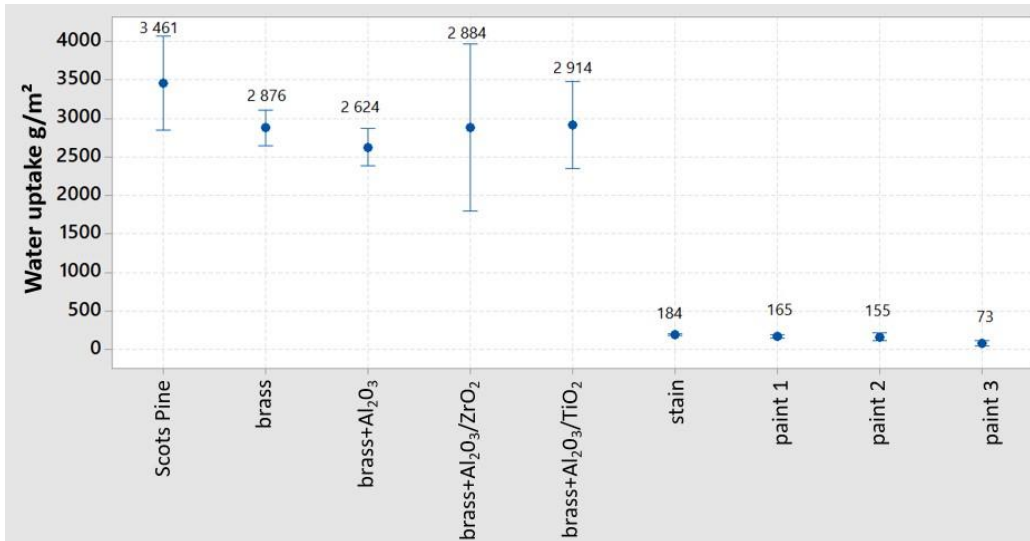


Figure 2: Water uptake (mean and confidence interval 95%) of the brass and ceramics coatings compared to organic coatings.

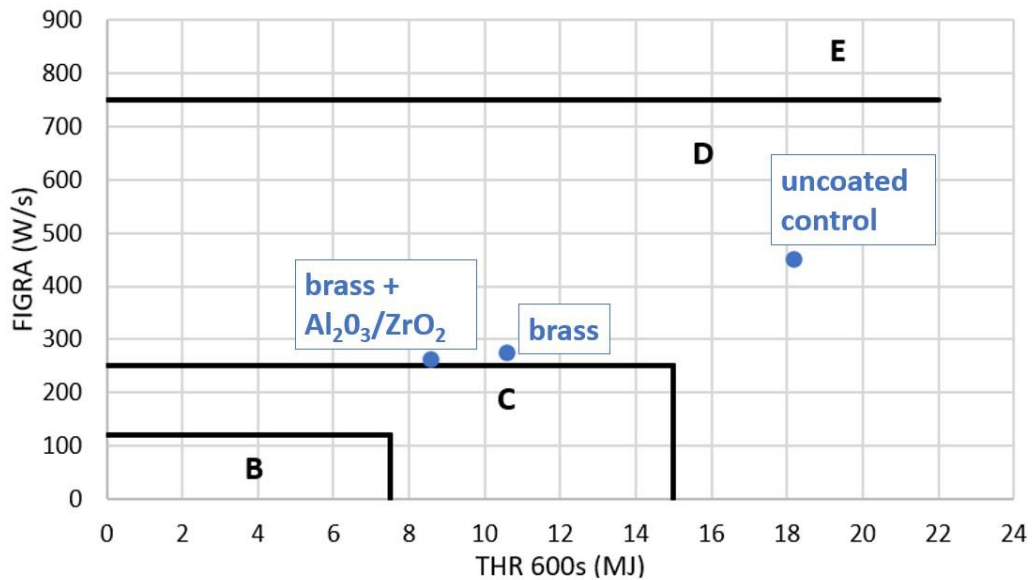


Figure 3: SBI test results

### REFERENCES

Nejad M., Shafaghi R., Pershin L., Mostaghimi J., Cooper P. (2017). Thermal Spray coating: a new way of protecting wood. *BioResources* 12 (1), 143-156.

Podgorski L., Myalska H., Denoirjean A., Kutnik M (2020). Thermal spray to protect wood from termites. International Research Group on Wood Protection, Document IRG/WP 20-40905.

## Poster 8.19 - Production and Application of Chemically Modified Cellulose Nanofibrils

Primož Oven<sup>1</sup>, Ida Poljanšek<sup>1</sup>, Vesna Žepič<sup>2</sup>, Jaka Levanič<sup>1</sup>, Urša Osolnik<sup>1</sup>, Viljem Vek<sup>1</sup>

<sup>1</sup>Department of Wood Science and Technology, Biotechnical Faculty, University of Ljubljana, Jamnikarjeva 101, 1000 Ljubljana. E: primoz.oven@bf.uni-lj.si

<sup>2</sup>TECOS, Slovenian Tool and Die Development Centre, Kidričeva ulica 25, SI-3000 Celje, Slovenija. E: vesna.zepic.bogataj@tecos.si

**Keywords:** nanocellulose, TEMPO mediated oxidation, acetylation, properties, bionanocomposites

### ABSTRACT

Nanocellulose is nanostructured natural and sustainable material produced mainly from wood or other lignocellulosic feedstock. Nanocellulose has exceptionally good properties which makes this component suitable for development of new materials with the aim to replace those that are nowadays produced from building blocks of fossil origin or as addition to improve properties of materials with less favourable performance. Production of cellulose nanofibrils, which is one type of nanocellulose, and its material application usually demands some chemical modification, which is done with the goal to improve defibrillation or its compatibility with polymer matrices, respectively.

The aim of this contribution is first to review research on chemical modification of woody biomass and pulp done at the Department of wood science and technology at University of Ljubljana with the aim of nanocellulose production and its application. Lignocellulose nanofibrils were produced directly from wood mill by treating it with maleic anhydride and high pressure homogenization, whereas TEMPO ((2,2,6,6-tetramethylpiperidin-1-yl)oxyl) mediated oxidation of pulp following by post oxidation with potassium peroxydisulfate and high pressure homogenization were employed for production of cellulose nanofibrils with increased carboxyl content and improved colour stability for application in nanocomposites. Another type of chemically modified nanocellulose were acetylated cellulose nanofibrils obtained in a heterogeneous system with acetic anhydride, pyridine and dimethylformamide. The effect of acetylation of variously dried cellulose will be shown as well. Morphological, structural, physical and thermal properties of chemically modified nanocellulose and bionanocomposites produced from it will be discussed.

### REFERENCES

Žepič, V. Poljanšek, I., Oven, P., and Čop, M. (2016). COST-FP1105: Properties of PLA films reinforced with unmodified and acetylated freeze dried nanofibrillated cellulose. *Holzforschung*, **70**(12): 1125–1134.

Levanič, J., Poljanšek, I., Oven, P. (2018) Chlorine-Free Method for the Oxidation of Residual Aldehydes on TEMPO-Oxidized Cellulose. *BioResources* **13**(4), 7969-7982.

## Poster 8.20 - Effects of Microwave Treatment on the Retention of a Preservative Product in two Portuguese Wood Species

Fernando Júnior Resende Mascarenhas<sup>1,2</sup>, Alfredo Manuel Pereira Geraldes Dias<sup>1,2</sup>, André Luis Christoforo<sup>3</sup>, Rogério Manuel dos Santos Simões<sup>4</sup>

<sup>1</sup>University of Coimbra, ISISE, ARISE, Department of Civil Engineering, Rua Luís Reis Santos—Pólo II, University of Coimbra (UC), Coimbra, 3030-788, Portugal. E: fer.jr.resende@hotmail.com

<sup>2</sup>SerQ – Innovation and Competence Forest Centre, Rua J, N° 9, Zona Industrial da Sertã, Sertã, 6100711, Portugal

<sup>3</sup>Department of Civil Engineering, Federal University of São Carlos (UFSCar), Rodovia Washington Luís (SP-11 310), Km 235, São Carlos, 13565-905, Brazil

<sup>4</sup>University Beira Interior (UBI), Department of Chemistry, Unit of Fiber Materials and Environmental Technologies (FibEnTech-UBI), Covilhã, Portugal

**Keywords:** eucalyptus, maritime pine, microwave treatment, preservative product, treatability

### ABSTRACT

Wood is an important material for construction, with structural or non-structural applications. Depending on the environmental conditions where the wood elements are used, they require protection against biological agents through impregnation with a preservative product. In Portugal, two wood species stand out due to their predominance in forests, different uses, and economic importance: eucalyptus (*Eucalyptus globulus* Labill.) and maritime pine (*Pinus pinaster* Ait.) (Lopes *et al.* 2021). Although eucalyptus wood has satisfactory mechanical properties, its heartwood is not durable against fungi and termites, and it is difficult to impregnate with preservative products (CEN 2016). It happens because eucalyptus has low permeability (treatability) (Torgovnikov and Vinden 2010). In this sense, researchers and engineers have been carrying out investigations in the diverse fields of wood modification, looking for techniques to address the inherent limitations of wood. Microwave (MW) treatment of wood has gained prominence in the field of wood modification to improve wood permeability. Hence, assuming that MW treatment can make wood species more permeable to preservative products, this research work aimed to improve the impregnability of Portuguese maritime pine and eucalyptus after being submitted to MW treatment (drying). Small clear wood specimens of both wood species, containing only heartwood, measuring 20 mm x 20 mm x 320 mm, were used. The wood samples were divided into MW-treated and control groups (with a conventional drying method). Each wood group had 16 wood specimens. MW drying was performed using a conventional MW device with 2.45 GHz under a power of 400 W. The green wood samples were submitted to MW drying until a final moisture content of 12 % was reached. Four wood samples were placed inside the MW oven, and successive MW cycles of 25 min were applied, with cooling intervals of 30 s to weigh the samples (Fig. 1).

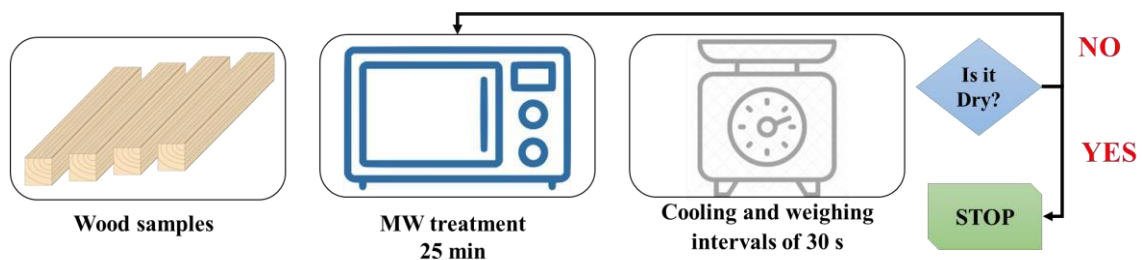


Figure 1: MW treatment routine.

The control samples (conventional drying) from maritime pine and eucalyptus were named PP\_Control and EG\_Control, respectively. The MW-treated samples were named PP\_400W\_25min and EG\_400W\_25min, respectively, for maritime pine and eucalyptus. After that, the wood samples (control and MW-treated) were impregnated with a 5 % water-soluble solution of Tanalith E 8001 under industrial conditions (vacuum-pressure process), and the Tanalith E 8001 retention was calculated. The results of MW-treated and control samples were assessed by classical analysis of variance (ANOVA) at a 5% significance level with the Tukey Test.

The results demonstrated that the PP\_Control retained 10.47 kg/m<sup>3</sup> of preservative product, whereas the corresponding samples treated with the MW (PP\_400W\_25min) retained 18.19 kg/m<sup>3</sup>. Based on the recommendations of usages for Tanalith E 8001, the MW-treated maritime pine samples could be applied in circumstances of Use Classes (UC) 1, 2, 3, and 4. The results for the MW-treated eucalyptus samples were remarkable. The EG\_Control samples only retained 4.94 kg of Tanalith E 8001 per cubic meter of wood, while the corresponding samples MW-treated (EG\_400W\_25min) retained 11.18 kg/m<sup>3</sup> (statistically different), showing that after MW treatment, the wood samples were satisfactorily impregnated with the preservative product. Based on the guidelines for applying Tanalith E 8001, the MW-treated eucalyptus wood samples could be placed in conditions of UC 1, 2, and 3, while the EG\_Control did not retain the minimum required quantity for any UC. These results are in agreement with the well-known refractory nature of the eucalyptus wood regarding impregnation (Torgovnikov and Vinden 2010), and the present results have shown that the use of MW energy improved its treatability. It is important to recall that eucalyptus wood species lack natural durability and cannot be treated by regular impregnation processes (CEN 2016). The MW treatment generates heat almost uniformly in the sample, producing water vapor at appreciable pressure that moves from inside to outside, opening the wood structure, namely the tracheids, pit membranes, and parenchyma cells, increasing wood permeability (Weng *et al.* 2020). The conjugated effect of MW treatment and Tanalith E 8001 impregnation had a marginal effect on wood density. The density of PP\_400W\_25min ranged from 583±9 kg/m<sup>3</sup> (before the MW drying) to 576±6 kg/m<sup>3</sup> (after the MW treatment and impregnation), a decrease of 1.2 %. EG\_400W\_25min had a density of 852±30 kg/m<sup>3</sup> before the MW drying and 836±42 kg/m<sup>3</sup> after the MW drying and impregnation, a decrease of 1.9 %. The present results are in good agreement with Torgovnikov and Vinden's (2010) results, which indicate that it is possible to obtain an increase in permeability by 1.5 times with no statistically significant decreases in densities and mechanical properties.

In conclusion, MW treatment showed to be an effective technology in the field of wood modification to improve the impregnability of maritime pine (an increase of 74 %) and overcome the low permeability of eucalyptus, a refractory wood species, with an increase of 126 % in the retention of the preservative product. Finally, the notable upgrade in impregnability due to the MW drying can be very beneficial for enhancing the uses of both wood species as construction materials.

## REFERENCES

- CEN. (2016). EN 350. Durability of Wood and Wood-Based Products - Testing and Classification of the Durability to Biological Agents of Wood and Wood-Based Materials, Brussels.
- ISO. (2014). ISO 13061-2. Physical and mechanical properties of wood — Test methods for small clear wood specimens - Part 2: Determination of density for physical and mechanical tests, Switzerland.
- Lopes, A. F., Batista, E., Hilário, S., Santos, L., and Alves, A. (2021). Occurrence of Diaporthe species in *Eucalyptus globulus*, *Pinus pinaster* and *Quercus suber* in Portugal. *Forest Pathology*, **51**(2), 1–13.
- Torgovnikov, G., and Vinden, P. (2010). Microwave wood modification technology and its applications. *Forest Products Journal*, **60**(2), 173–182.

Weng, X., Zhou, Y., Fu, Z., Gao, X., Zhou, F., and Fu, F. (2020). Effects of microwave treatment on microstructure of Chinese fir. *Forests*, **11**(7), 1-9.

## Poster 8.21 - Wood Modification as an Opportunity for Local Wood Species in Musical Instrument Making

Mario Zauer<sup>1</sup>, Tobias Dietrich<sup>1</sup>, Herwig Hackenberg<sup>1</sup> and André Wagenführ<sup>1</sup>

<sup>1</sup>TU Dresden University of Technology, 01062 Dresden, Germany. E: mario.zauer@tudresden.de

**Keywords:** ammonia treatment, densification, musical instruments, thermal treatment

### ABSTRACT

The use of wood in high-quality musical instruments requires high-quality material. This essentially includes particularly select, partly rare, European and non-European as well as tropical types of wood. Depending on the component or function in the instrument, the wood must have high dimensional stabilities, sound radiation coefficients, stiffness's and hardness's. An essential evaluation criterion for customer acceptance is, first and foremost, the colour and grain of the wood. A wood which has wrong look, colour or feel may be rejected purely for these reasons, even if their performance is similar or even superior to other materials (Wegst 2006). In addition to a comparatively low dimensional stability, most regional types of wood also only have a slight, correspondingly fascinating optical quality. Furthermore, the hardness and vibration properties are sometimes significantly weaker compared to the tonewood ranges currently used. To solve this problem, modified regional wood species were examined using three different modification processes in three case studies or groups of instruments:

- (1) Thermal modification for the use in selected components of classical guitars,
- (2) Combination of mechanical densification and subsequent thermal treatment for the use as fingerboard material of electrical bass guitars,
- (3) Combination of ammonia treatment and subsequent mechanical densification for the use as fingerboard material of electrical guitars.

In all cases the material, modified regional wood, were developed in laboratory scale. Component-specific preferred variants were then manufactured on a pilot scale. Ultimately, entire test instruments were made using the modified wood and were checked comparatively with identical reference guitars by means of objective (automatic plucking method) and subjective (professional musicians) testing methods.

Thermal modification alone (1) improved especially the dimensional stability, acoustical properties and the optical aspects, in the sense darker colour nuances, of the regional wood species. In particular, black locust (*Robinia pseudoacacia* L.) as well as the wood from service tree (*Sorbus torminalis* (L.) Crantz), plum tree (*Prunus domestica* L.) and cherry tree (*Prunus avium* L.) in a special modified state could be used as a substitute for Indian rosewood (*Dalbergia latifolia* Roxb.), cedro or Spanish cedar (*Cedrela odorata* L.) and ebony (*Diospyros crassiflora* Hiern.) in classical guitars.

Mechanical densification in combination of thermal treatment (2) led to higher density and thus hardness of the regional wood species. The latter process step resulted in a darker colour and reduction or even compensation of the set recovery effect of the densified wood. Due to thermal treatment of the densified wood dimensional stability is improved depending on the treatment intensity. However, compared to the non-densified wood the swelling and shrinking of the combined modified wood in the direction of densification (in this case radial direction) was clearly higher. Nevertheless, special combined treatment of both densification and thermal modification could enable the use of European beech (*Fagus sylvatica* L.) and black walnut (*Juglans nigra* L.) to substitute Indian rosewood as fingerboard material in electric bass guitars.

Gaseous ammonia treatment effected a plasticisation of the wood structure for its lowforce and low destructive mechanical densification (3). Additionally, the wood received a darker colour and the set

recovery effect of the densified wood was reduced or compensated after the treatment. The whole treatment procedure led to higher density and thus hardness. Here too, it should be noted that swelling and shrinking in the direction of densification (in this case radial direction) was higher compared to non-densified wood. Nevertheless, special combined treatment of both ammonia treatment and densification could enable the use of European beech, black locust and European oak (*Quercus* spp.) to substitute Indian rosewood as fingerboard material in electric guitars (Figure 1).



**Figure 1: Fingerboards in electrical guitars: modified beech (left), untreated rosewood (middle), modified oak (right).**

Although the modified woods appear to be excellently suited to the tested musical instruments in terms of acoustic and mechanical requirements, they were always rated slightly or significantly worse in terms of optical quality compared to the types of wood to be substituted. In this context, a rethink would have to take place in the musical instrument sector, which is sometimes very traditional. Apparently only 0,5% of tropical wood deforestation goes into making musical instruments worldwide. However, there is obviously a problem with the availability of appropriate high-quality tonewoods. In the course of numerous discussions with tonewood dealers, musical instrument makers and musicians, it became clear that the availability of the special woods is obviously generally given and perhaps slightly reduced in some cases. Though, the available qualities are no longer available to the extent that they are predominantly required in the musical instrument sector. We think that gives opportunities for wood modification for both nontonewood with good quality and tonewood already used but with relative weak properties.

## REFERENCES

- Wegst, U.G.K. (2006). Wood for Sound. In: *American Journal of Botany*, **93**(10), 1439-1448.
- Pfriem, A. (2018). Tropenholzproblematik im Musikinstrumentenbau, Chancen für alternative Materialien? In: Proceedings of the 18. Holztechnologische Kolloquium, Dresden, Germany. **23**, 79-88.



## Poster 8.22 - Maximum Compressibility Along the Grain of Different Wood Species

Mátyás Báder<sup>1</sup>, Miklós Szauer<sup>1</sup> and Róbert Németh<sup>1</sup>

<sup>1</sup>Institute of Wood Technology and Technical Sciences, Faculty of Wood Engineering and Creative Industries, University of Sopron, Bajcsy-Zsilinszky 4, 9400 Sopron, Hungary. E: bader.matyas@uni-sopron.hu

**Keywords:** anatomical properties, compression ratio, different regions, diffuse porous beech, pleating, pliability, ring-porous sessile oak

### ABSTRACT

This study deals with beech and oak wood species in order to determine the maximum compression ratio along the grain of these species. It may provide useful information for filling scientific gaps and for the wood industry dealing with pliable wood. Hungarian logs were used from different locations: beech (*Fagus Sylvatica* L.) and sessile oak (*Quercus petraea* (Matt.) Liebl.) from the region of Sopron, Hungary and sessile oak from region Zala, Hungary. After the sample preparation, they were stored frozen to preserve their moisture content. In each case, the specimens were steamed for 45 minutes and then compressed along the grain (aka. pleating) at a rate of 25 %/min in a heated compression equipment, followed by a fixation period for one minute (Báder and Németh 2023). The ratio of pleating was carried out up to the preset value or until something unusual was observed on the graph plotted by the software of the material testing machine. All pleated samples were visually inspected and graded for any damage that may have occurred. The level of pleating was considered adequate if at least 90% of the samples could withstand compression without damage. Figure 1 shows a typical damage. Bending tests were carried out after conditioning in a normal climate (20 °C / 65% relative humidity) as described by Báder and Németh (2018).



Figure 1: Deep splits along the rays due to pleating in a beech specimen.

As a result, we concluded that the highest compression ratio is 30% for beech, 27-28% for oak sapwood and 20-21% for oak heartwood. Thus, diffuse porous hardwood species have a significantly higher compressibility than ring-porous hardwood species, and that oak sapwood is easier to compress due to its different structural-chemical structure compared to oak heartwood. Logs of the same species but with different anatomical characteristics can bear pleating differently. The density of the wood and the width of the latewood are inversely proportional to the compressibility. The higher density, i.e. the wider latewood results in greater spring-back of the specimens after pleating. Comparing specimens of the same wood species, those with a smaller fibre length can be compressed to a greater ratio. The cell wall thickness of the samples does not show any difference and do not

affect the highest compaction ratio. Pleated wood has significantly better bending properties compared to the untreated one and the higher the compression ratio, the more pliable it became. Therefore, it is important to know the highest compressibility ratio because this makes it easier to adapt the modification ratio to a specific bending requirement.

## REFERENCES

Báder, M. and Németh, R. (2018). Further treatment option after longitudinal wood compression. In: Proceedings of the 9th European Conference on Wood Modification. Practicum, Arnhem, The Netherlands, 7 p.

Báder, M. and Németh, R. (2023). A review of wood compression along the grain—After the 100th anniversary of pleating. *Forests*, **14**(4), 763. <https://doi.org/10.3390/f14040763>

## Poster 8.23 - Studies on the Durability of the Reaction to Fire Performance of Melamine Formaldehyde Resin and Phosphorus Polyol Treated Wood

Muting Wu<sup>1</sup>, Lukas Emmerich<sup>2</sup> and Holger Militz<sup>1</sup>

<sup>1</sup>Wood Biology and Wood Products, Georg-August-Universität Göttingen, Büsgenweg 4, 37077 Göttingen, Germany. E: muting.wu@uni-goettingen.de, hmilitz@gwdg.de

<sup>2</sup>Landesbetrieb Wald und Holz NRW, Zentrum Holz, Team Holzwirtschaft, Carlsauestraße 91A, 59939 Olsberg, Germany. E: lukas.emmerich@wald-und-holz.nrw.de

**Keywords:** chemical modification, EN84, flame retardant, heat release, thermal setting resin

### ABSTRACT

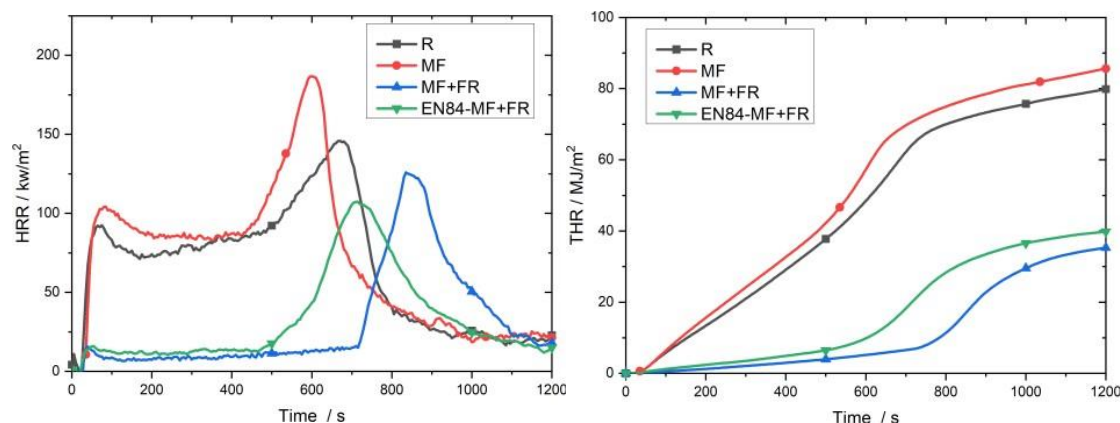
Wood modification using melamine formaldehyde (MF) resin has been recognized for its efficacy in enhancing the resistance against wood-destroying organisms and reducing moisture-induced swelling and shrinking processes (Behr 2019, Zelinka *et al.*, 2022). However, melamine treatments show low impact on the flammability and combustion behaviour of wood (Xie *et al.*, 2016). To address the aspect of a fire protection, one approach could be a combination of MF resins with compatible flame retardant agents (Lin *et al.*, 2021). In this study, Scots pine sapwood (*Pinus sylvestris* L.) has been treated with an aqueous MF resin formulation, to which a phosphorus polyol was added. Both, fixation of the flame retardant (FR) inside the wooden structure and the fire performance were studied on treated wood specimens. The fixation of the FR was assessed on the basis of mass changes which treated specimens experienced during an accelerated ageing process according to EN 84 (2020). The fire performance was evaluated by heat release measurements using a mass loss calorimeter according to ISO 13927 (2015).

A positive weight percent gain (WPG) indicated, that both MF and FR were deposited inside the wooden structure. A permanent cell wall swelling (Cell wall bulking (CWB) effect), which was measured after the treatments, confirmed that both MF resin and the phosphorus polyol were able to penetrate into the wood cell wall (Tab. 1). After an accelerated ageing process (EN 84, 2020), noticeable reductions in WPG were measured on FR and MF + FR treated specimens. In case of purely FR treated wood specimens, a WPG around 0 % indicated, that almost the entire FR compound had been removed during a cold-water leaching (EN 84, 2020), thus had not been fixed inside the wooden structure. Even though WPG reductions were measured on MF + FR treated wood specimens, the extent of the latter indicated that the FR compounds experienced a certain fixation by the incorporation in a polymeric network formed by cured MF resin. However, macroscopic WPG measurements before and after a cold-water leaching did not allow to differentiate between the removal of MF and FR compounds. Thus, it remains questionable to which extent WPG reductions in MF + FR treated specimens were based on non-fixed MF or FR compounds (Tab. 1).

**Table 1: Weight percent gain (WPG) and cell wall bulking (CWB) of MF, FR and MF+FR treated Scots pine sapwood specimens before and after an accelerated ageing according to EN 84 (2020). The prefix “EN84” in the sample ID indicates that the collective was leached (EN 84).**

Sample	WPG [%]	CWB [%]	Sample	WPG [%]	CWB [%]
MF	33.5 (±1.5)	4.1 (±0.4)	EN84-MF	31.6 (±1.4)	2.9 (±0.3)
FR	10.2 (±1.4)	2.1 (±0.7)	EN84-FR	-1.5 (±0.4)	-2.2 (±0.7)
MF+FR	52.2 (±2.2)	4.2 (±0.4)	EN84-MF+FR	37.7 (±2.0)	0.9 (±0.3)

With the phosphorus and nitrogen (P-N) synergistic effect of phosphorus polyol and MF resin, the combined treatment of MF+FR significantly reduce the heat release. The THR<sub>600s</sub> values for MF+FR (5.2MJ/m<sup>2</sup>) showed an 89.3% and 91% reduction compared to untreated wood (48.5MJ/m<sup>2</sup>) and pure



MF treated wood (57.3MJ/m<sup>2</sup>), respectively. Notably, this enhancement persisted even after the leaching process (9.9 MJ/m<sup>2</sup>), indicate MF can fix the flame retardant inside wood.

**Figure 1: Heat release rate (left) and total heat release (right) curve of untreated and treated Scots pine sapwood specimens with dimensions of 100 (ax.) x 100 x 18 mm<sup>3</sup>.**

## REFERENCES

- EN ISO 13927: 2015. Plastics. Simple heat release test using a conical radiant heater and a thermopile detector. European Committee for Standardization, Brussels, Belgium.
- EN 84: 2020. Wood preservatives – Accelerated ageing of treated wood prior to biological testing - Leaching procedure. European Committee for Standardization, Brussels, Belgium.
- Georg, B. (2019) The influence of melamine treatment in combination with thermal modification on the properties and performance of native hardwoods. Doctoral Thesis (Göttingen, Germany: Georg-August-University Göttingen).
- Lin, C., Karlsson, O., Martinka, J., Rantuch, P., Garskaite, E., Mantanis, G. I., Jones, D., & Sandberg, D. (2021). Approaching highly leaching-resistant fire-retardant wood by in situ polymerization with melamine formaldehyde resin. *ACS Omega*, 6(19), 12733–12745.
- Xie, Y., Xu, J., Militz, H., Wang, F., Wang, Q., Mai, C., Xiao, Z., (2016). Thermo-oxidative decomposition and combustion behavior of Scots pine (*Pinus sylvestris* L.) sapwood modified with phenol- and melamine-formaldehyde resins. *Wood Science Technology* 50, 1125–1143.
- Zelinka, S.L., Altgen, M., Emmerich, L., Guigo, N., Keplinger, T., Kymäläinen, M., Thybring, E.E., Thygesen, L.G., (2022). Review of Wood Modification and Wood Functionalization Technologies. *Forests* 13, 1004.

## Poster 8.24 - Effect of Aspen face Veneer Thickness on the Fire performance of Post-manufacture Fire-retardant Treated Birch Plywood

Percy Alao<sup>1</sup>, Anti Rohumaa<sup>1,2</sup>, Karl Harold Dembovski<sup>1</sup>, Jussi Ruponen<sup>3</sup> and Jaan Kers<sup>1</sup>

<sup>1</sup>Department of Material and Environmental Technology, Tallinn University of Technology, Ehitajate tee 5, 19086 Tallinn, Estonia. E: percy.alao@taltech.ee, kademb@taltech.ee; jaan.kers@taltech.ee

<sup>2</sup>Fiber Laboratory, South Eastern Finland University of Applied Sciences, Vipusenkatu 10, FI- 57200 Savonlinna, Finland. E: anti.rohumaa@xamk.fi

<sup>3</sup>Department of Bioproducts and Biosystems, Aalto university, Vuorimiehentie 1, FI-00076 AALTO, Espoo, Finland. E: jussi.ruponen@aalto.fi

**Keywords:** aspen, fire retardant, fire test, heat release, plywood, veneer

### ABSTRACT

The demand for plywood is increasing significantly as renewable resources become more important. Plywood is a versatile material with many uses, such as subfloors, roof sheathing, and wall panels in construction (Kawalerczyk *et al.* 2019). However, health and safety standards regarding fire risk in construction and transportation sectors limit the use of plywood in some applications. Additionally, birch (*Betula pendula* Roth), one of the most popular wood species used to make plywood in Northern Europe (Akkurt *et al.* 2022), has a high heat combustion rate (Rudzīte and Bukšāns 2021), making it difficult to meet fire safety standards. Furthermore, using underutilized wood species increases the possibility to effectively valorize wood as a renewable material. Consequently, this study designs a post-manufacturing fire-retardant (FR) treatment approach to study the fire performance of birch plywood laminated with aspen (*Populus tremula* L.) veneers of varying nominal thicknesses (see Table 1). Aspen is chosen because it is a common wood species in many parts of Europe that is underutilized. Besides, aspen is lighter, has a lower heat combustion rate, is cheaper, more durable to decay, is more pliable, and grows faster than birch. The FR used in the study is a novel protic-ionic liquid based aqueous solution of bisphosphonate acid and an alkanol amine. The presented reaction to fire performance is based on ISO 5660-1 using a cone calorimeter, since the larger scale single burning item test is not suitable for analytical investigation. Additionally, the transportation industry follows the indicators deriving from ISO 5660-1. Table 1 shows that the density of the plywood generally decreases as the face veneer thickness (FVT) increases.

**Table 1: The properties of the panels and corresponding reaction to fire performance**

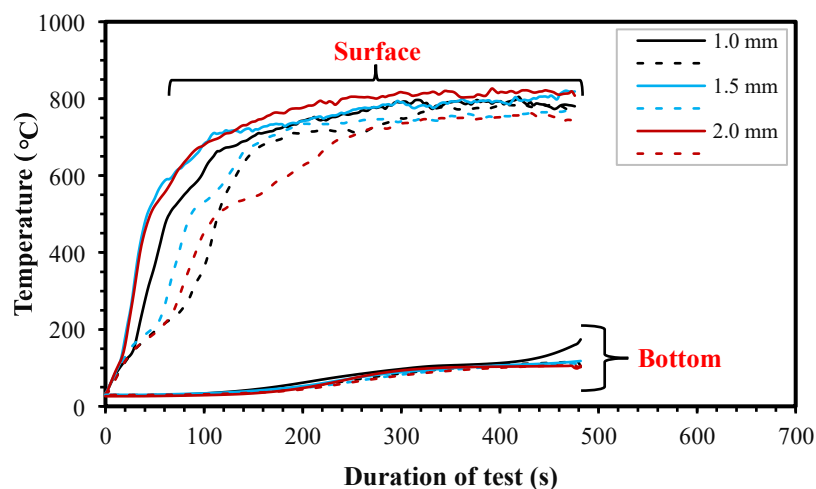
Nominal thickness [mm]	Panel	FVT	Density		pHRR		*MAHRE		Mass loss	
			[kg/m <sup>3</sup> ]		[kWm <sup>-2</sup> ]		[kWm <sup>-2</sup> ]		[%]	
			**C	FR	C	FR	C	FR	C	FR
1.0	15.1		695.9	705.2	250.8	226.5	160.3	141	71.0	60.7
1.5	17.2		658.3	678.5	273.9	177.4	161.1	123.3	65.6	53.6
2.0	19.2		656.1	645.6	267.7	169.6	154.3	122.6	56.9	48.3

\* Maximum average heat rate of emission; \*\* Control

The FR-treated plywood with 2 mm FVT is lighter than the corresponding control batch because different panels were used for the control and FR treatments. The fire performance of the control panel is not affected by the FVT, as measured by the peak heat release rate (pHRR) and MAHRE. However, the FVT does affect the mass loss, with thicker face veneers causing lower mass loss,

indicating less thermal decomposition. The fire performance of FR-treated plywood is more conclusive, with pHRR, MAHRE, and mass loss all correlating to FVT, suggesting that the FVT affects the fire-retardant treatment. 2 mm FVT plywood shows the best fire performance.

The self-ignition temperature-time curve presented in Fig. 1 shows a longer ignition delay and slower heating rate for the FR-treated aspen-laminated plywood samples compared to untreated variants at the beginning of exposure (up to 260 seconds), which is likely because the FR treatment inhibits the chemical reactions that lead to ignition. The linearization of temperature rise, which begins at about 110 seconds and over 650 °C for the untreated samples, is significantly delayed for the FR-treated variants, especially with the batch having FR-treated 2 mm veneers, which suggests that the FR treatment also affects the post-ignition combustion behavior of the plywood. No meaningful changes in temperature transfer were observed during the test duration (480 seconds), mainly due to the test span. The results demonstrate the potential application of underutilized wood species as a functional material for plywood.



**Figure 1: The temperature-time curve showing the surface and bottom temperature rise of birch plywood laminated with untreated (-) and FR-treated (- -) aspen veneers.**

## REFERENCES

- Akkurt, T., Kallakas, H., Rohumaa, A., Hunt, C. G., and Kers, J. (2022). Impact of Aspen and Black Alder Substitution in Birch Plywood, *Forests*, **13**(2), pp. 142.
- Kawalerczyk, J., Dziurka, D., Mirski, R., and Grześkowiak, W. (2019). The effect of veneer impregnation with a mixture of potassium carbonate and urea on the properties of manufactured plywood. *Drewno*, **62**(203), pp. 107-116.
- Rudzīte S., and Bukšāns E. (2021). Impact of High-pressure Impregnation and Fire Protective Coatings on the Reaction to Fire Performance of Birch Plywood. *Rural Sustainability Research*, **45**(340), pp. 65-75.

## **DAY 2**

### **SESSION NINE**

#### **NEW TRENDS**

## Oral 9.01 - Ultrafast Self-Propelling Directionally Water Transporting Wood via Constructing Multi-Hierarchical Structures on Cell Wall

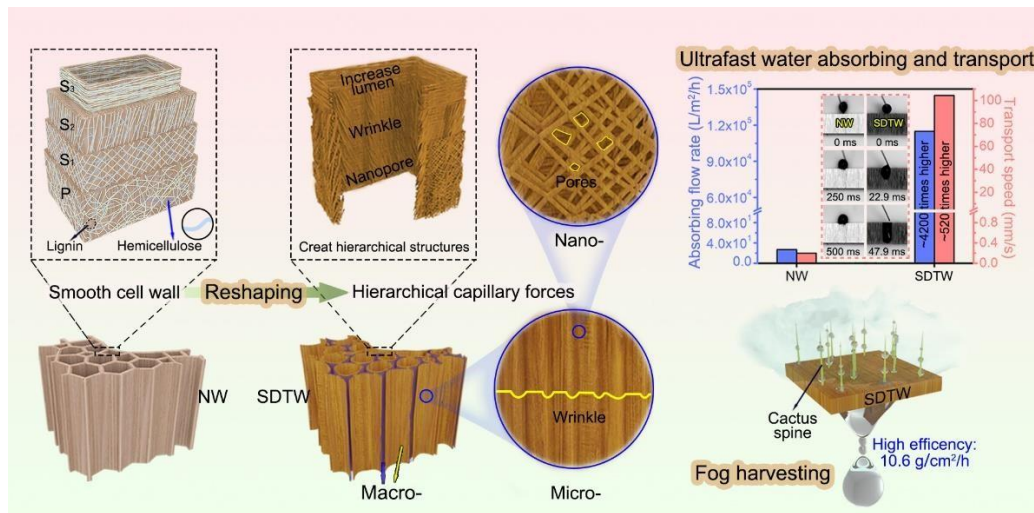
Yanjun Xie<sup>1</sup>

<sup>1</sup>Material Science and Engineering College, Key Laboratory of Bio-based Material Science and Technology (Ministry of Education), Northeast Forestry University, 150040 Harbin, China. E: yxie@nefu.edu.cn

**Keywords:** cell wall reconstruction, directional water transport, hierarchical structures, wood functionalization

### ABSTRACT

The inherent hydrophilic longitudinal channel structure of wood has inspired the design of materials for water transport (Luo *et al.* 2020, Che *et al.* 2019, Jia *et al.* 2017, Wu *et al.* 2020). However, it was limited by the low transport speed due to the lack of propulsion. Inspired by the micro-grooves structure of *Nepenthes alata* and shrinkage property of wood cell wall (Chen *et al.* 2016, Rafsanjani *et al.* 2014), a self-propelling directionally water transporting wood (SDTW) was constructed by redesigning the cell wall to generate robust capillary forces due to aligned longitudinal hierarchical wood cell structures. The hierarchical structures consisted of directional parallel macro- and micro-sized ridgegroove structures and creating nano-voids. They are further perfectly fixed by modification with maleic anhydride to improve the dimensional stability and environmental durability without altering surface hydrophilicity. Prepared SDTW shows an ultrafast water transport speed of 200.4 mm/s (~260 % of *Nepenthes alata*, which shows a fast speed of 78 mm/s as a native structure) and water absorbing rate of  $1.15 \times 10^5$  L/m<sup>2</sup>/h (over 4200-fold higher than that of natural wood). Moreover, and SDTW based fog harvesting system showed a high efficiency of 10.6 g/cm<sup>2</sup>/h. The excellent water transport properties combined with its facility, durability, scalability, and sustainability, demonstrates potential for practical water manipulation applications.



**Figure 1: Preparation of self-propelling directionally water transporting wood. It showed high water transport speed and potential as a fog harvesting material.**

### REFERENCES

Luo, Y., Song, F., Xu, C., Wang, X. and Wang, Y. (2020). Bioinspired fabrication of asymmetric wood materials for directional liquid manipulation and transport. *Chemical Engineering Journal* **383**, 123168.



Che, W., Xiao, Z., Wang, Z., Li, J., Wang, H., Wang, Y. and Xie, Y. (2019). Wood-based mesoporous filter decorated with silver nanoparticles for water purification. *ACS Sustainable Chemistry & Engineering* **7** (5), 5134-5141.

Jia, C., Li, Y., Yang, Z., Chen, G., Yao, Y., Jiang, F., Kuang, Y., Pastel, G., Xie, H., Yang, B., Das, S. and Hu, L. (2017). Rich mesostructures derived from natural woods for solar steam generation, *Joule* **1** (3), 588-599.

Wu, Q., Wang, C., Wang, R., Chen, C., Gao, J., Dai, J., Liu, D., Lin, Z. and Hu, L. (2020). Salinity gradient power generation with ionized wood membranes. *Advanced Energy Materials*, **10**, 1902590.

Chen, H., Zhang, P., Zhang, L., Liu, H., Jiang, Y., Zhang, D., Han, Z. and Jiang, L. (2016). Continuous directional water transport on the peristome surface of *Nepenthes alata*. *Nature* **532** (7597), 85-89.

Rafsanjani, A., Stiefel, M., Jefimovs, K., Mokso, R., Derome, D. and Carmeliet, J. (2014). Hygroscopic swelling and shrinkage of latewood cell wall micropillars reveal ultrastructural anisotropy. *Journal of The Royal Society Interface*, **11**(95),1-10.

## Oral 9.02 - Delignified Wood as Substrate for Nanostructured Composites with Extended Range of Functionalities

Lars Berglund<sup>1</sup>

<sup>1</sup>KTH Royal Institute of Technology, Dept of Fiber and Polymer Technology, Wallenberg Wood Science Center, SE-10044 Stockholm, Sweden, E: blund@kth.se

**Keywords:** ecoindicators, nanoporosity, wood cell wall

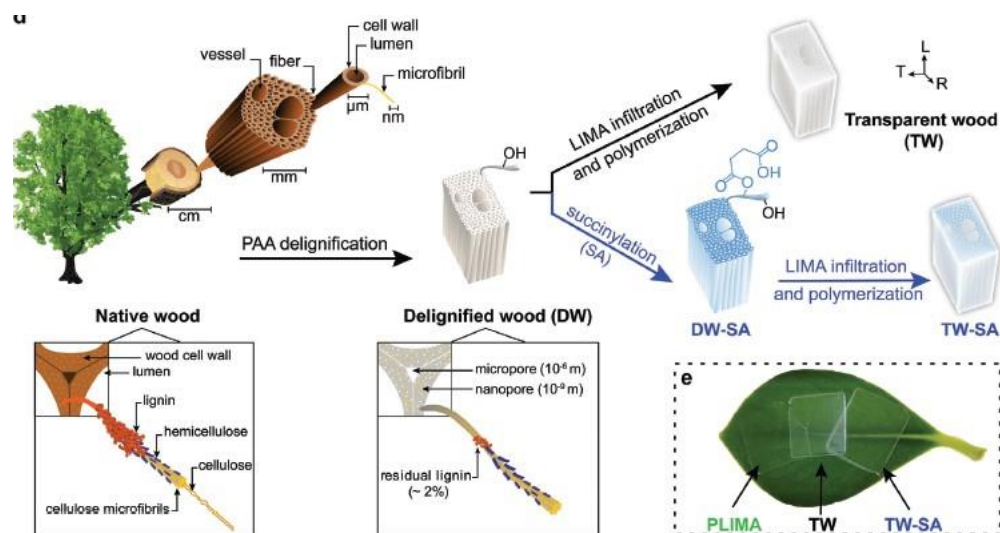
### ABSTRACT

Wood can be delignified by chemical means to remove most of the lignin and increase the small-scale porosity of the wood cell wall. The methods, eg peracetic acid treatment, need to be tuned so that the delignified wood substrate can be physically handled, without disintegrating into pulp fibers. Here, focus is on considerations for chemical and physical tailoring of the delignified wood substrate.

In our lab, we initially used polymer matrices to create transparent wood composites of high optical transmittance and a large variety of optical functionalities. For example, we have functionalized wood by photochromic and thermochromic additives and we have designed electrochromic devices and solar cells. We have dispersed quantum dots, earth metal and metal nanoparticle additives for optical effects and phase change materials for heat storage (Montanari *et al.* 2023). Devices for electrochemical energy storage, transistors (Tran *et al.* 2023) and piezo-electric sensing as well as piezoelectric energy harvesting (Ram *et al.* 2022) have been realized. Exceptional thermal insulation properties (Garemark *et al.* 2022) and shape-memory effects have been demonstrated, all relying on nano-structural control of the wood substrate during the preparation phase. Nanoscale impregnation and interaction mechanisms were investigated to generate fire retardant composites with inorganics inside the cell wall (Samanta *et al.* 2022).

By focusing on the desirable wood nanostructure and mechanisms for cell wall modification, new approaches have been developed including TEMPO-oxidation (Li *et al.* 2020), enzymatic oxidation (Koskela *et al.* 2023), cell wall crosslinking for physical stability and specific drying procedures (Wang *et al.* 2023). For sustainable development purposes, biobased monomers and green chemistry modification methods are preferable; see Figure 1, and suitable eco-indicators must be analyzed such as cumulative energy demand and carbon dioxide emissions (Montanari *et al.* 2021).

Most investigations on cellulosic nanocomposites are based on nanocelluloses disintegrated from chemical wood pulp fibers. The nanocellulose fibrils are used as a colloidal dispersion, and polymer composites, inorganic hybrids and related materials are prepared by a bottom-up approach. This allows the use of colloid chemistry for functionalization and nanostructural control. The approach has a disadvantage in that controlled cellulose fibril orientation and dispersion (separation from neighboring fibrils) is difficult and energy demanding. The present top-down approach utilizes the cellulose fibril orientation in the wood cell wall and the good dispersion of fibrils in the native cell wall. In addition, it is much easier to preserve the native state of the cellulose fibrils, without significant chemical or mechanical degradation.



**Figure 1: Preparation of fully biobased transparent wood composites. *Betula pendula* is subjected to peracetic acid (PAA) delignification, then succinylation followed by impregnation by acrylic monomer from limonene oxide and polymerization (Montanari *et al.* 2021).**

## REFERENCES

- Garemark, J.; Perea-Buceta, J. E.; Rico Del Cerro, D.; Hall, S.; Berke, B.; Kilpeläinen, I.; Berglund, L. A.; Li, Y. Nanostructurally Controllable Strong Wood Aerogel toward Efficient Thermal Insulation. *ACS Applied Materials and Interfaces* **2022**, *14*, 24697-24707.
- Koskela, S.; Wang, S. N.; Li, L. W.; Zha, L.; Berglund, L. A.; Zhou, Q. An Oxidative Enzyme Boosting Mechanical and Optical Performance of Densified Wood Films. *Small* **2023**, *19*, 2205056.
- Li, K.; Wang, S. N.; Chen, H.; Yang, X.; Berglund, L. A.; Zhou, Q. Self-Densification of Highly Mesoporous Wood Structure into a Strong and Transparent Film. *Advanced Materials* **2020**, *32*, 2003653.
- Montanari, C.; Chen, H.; Lidfeldt, M.; Gunnarsson, J.; Olsen, P.; Berglund, L. A. Sustainable Thermal Energy Batteries from Fully Bio-Based Transparent Wood. *Small* **2023**, 2301262.
- Montanari, C.; Ogawa, Y.; Olsen, P.; Berglund, L. A. High Performance, Fully Bio-Based, and Optically Transparent Wood Biocomposites. *Advanced Science* **2021**, *8*, 2100559.
- Ram, F.; Garemark, J.; Li, Y. Y.; Berglund, L. Scalable, efficient piezoelectric wood nanogenerators enabled by wood/ ZnO nanocomposites. *Composites Part a-Applied Science and Manufacturing* **2022**, *160*, 107057.
- Samanta, A.; Høglund, M.; Samanta, P.; Popov, S.; Sychugov, I.; Maddalena, L.; Carosio, F.; Berglund, L. A. Charge Regulated Diffusion of Silica Nanoparticles into Wood for Flame Retardant Transparent Wood. *Advanced Sustainable Systems* **2022**, *6*, 2100354.
- Tran, V.; Mastantuoni, G. G.; Zabhipour, M.; Li, L. W.; Berglund, L.; Berggren, M.; Zhou, Q.; Engquist, I. Electrical current modulation in wood electrochemical transistor. *Proceedings of the National Academy of Sciences of the United States of America* **2023**, *120*, 2218380120.
- Wang, S.; Li, L.; Zha, L.; Koskela, S.; Berglund, L. A.; Zhou, Q. Wood xerogel for fabrication of high-performance transparent wood. *Nature communications* **2023**, *14*, 2827.

## Oral 9.03 - Optical Wood with Switchable Solar Transmittance for Allround Thermal Management

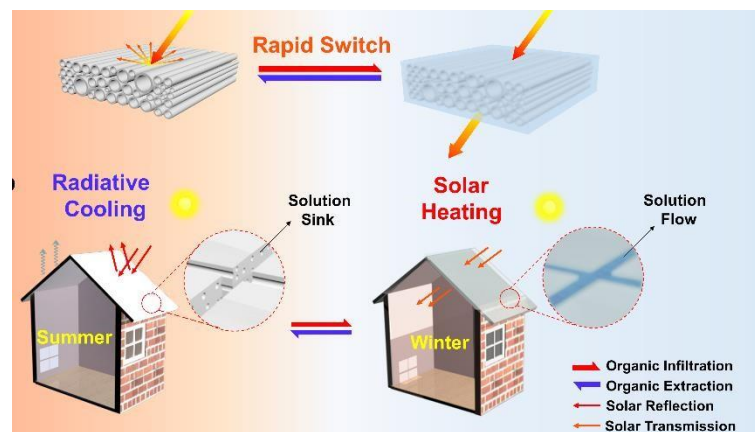
Daxin Liang<sup>1</sup> and Yanjun Xie<sup>1</sup>

<sup>1</sup>College of Materials Science and Engineering, Northeast Forestry University, Harbin 150040, China. E: daxin.liang@nefu.edu.cn

**Keywords:** daylight harvesting, radiative cooling, switchable, thermal management, wood

### ABSTRACT

Technologies enabling passive daytime radiative cooling and daylight harvesting are highly relevant for energy-efficient buildings. Despite recent progress demonstrated with passively cooling polymer coatings, however, it remains challenging to combine also a passive heat gain mechanism into a single substrate for all-round thermal management. Herein, we developed an optical wood (OW) with switchable transmittance of solar irradiation enabled by the hierarchically porous structure, ultralow absorption in solar spectrum and high infrared absorption of cellulose nanofibers. After delignification, the OW shows a high solar reflectance (94.9%) in the visible and high broadband emissivity (0.93) in the infrared region (2.5-25  $\mu\text{m}$ ). Owing to the exceptional mass transport of its aligned cellulose nanofibers, OW can quickly switch to a new highly transparent state following phenylethanol impregnation. The solar transmittance of optical wood (OW-II state) can reach 68.4% from 250 to 2500 nm. The switchable OW exhibits efficient radiative cooling to 4.5  $^{\circ}\text{C}$  below ambient temperature in summer (81.4  $\text{W m}^{-2}$  cooling power), and daylight heating to 5.6  $^{\circ}\text{C}$  above the surrounding temperature in winter (heating power 229.5  $\text{W m}^{-2}$ ), suggesting its promising role as a low-cost and sustainable solution to all-season thermal management applications.



**Figure 1:** Schematic diagram. (a) OW-I (left) and OW-II (right). (b) The working principle of the energy-efficient building made with an OW roof, based on the controlled impregnation and rinsing of the wood panels with either phenylethanol (OW-I to OW-II) or ethanol (OW-II to OW-I).

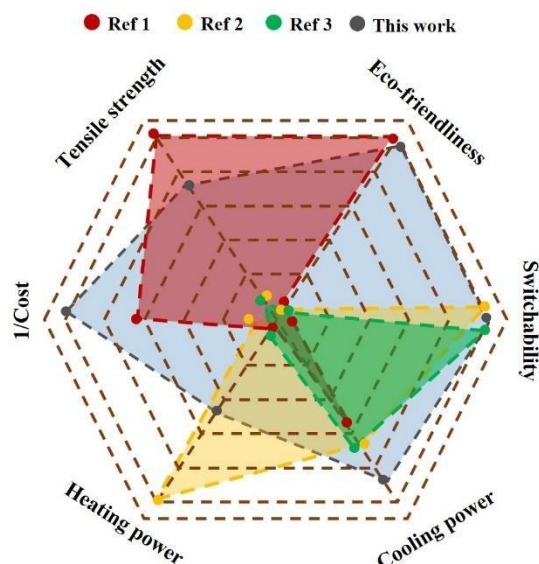


Figure 2: The radar map composed of optical wood and current methods reported in the literature.

Table 1: Comparison of six character of optical wood and current methods reported in the literature.

	Ref.1	Ref.2	Ref.3	Ref.4	Ref.5	Ref.6	This work
1/Cost	1/11.27	1/444.10	1/9.44	1/2.46	1/6.24	1/ 1.73	1/1.21
Tensile strength	404.3	0	0	5.4	0	0	22.3
Cooling power	63	71.6	~71	43.4	85.1	47.0	81.4
Natural	100	0	0	0	0	0	100
Heating power	0	643.4	Not stated	744	Not stated	197	229.5
Switchable	0	100	100	100	100	100	100

## REFERENCES

- Fei, J., Han, D., Ge, J., Wang, X., Koh, S. W., Gao, S., Sun, Z., Wan, M. P., Ng, B. F., Cai, L., Li, H. (2022). Switchable surface coating for bifunctional passive radiative cooling and solar heating. *Advanced Functional Materials*, **32**(27), 2203582.
- Li, T., Zhai, Y., He, S., Gan, W., Wei, Z., Heidarinejad, M., Dalgo, D., Mi, R., Zhao, X., Song, J., Dai, J., Chen, C., Aili, A., Vellore, A., Martini, A., Yang, R., Srebric, J., Yin, X., Hu, L. (2019). A radiative cooling structural material. *Science*, **364**, 760-763.
- Li, X., Sun, B., Sui, C., Nandi, A., Fang, H., Peng, Y., Tan, G., Hsu, P. C. (2020). Integration of daytime radiative cooling and solar heating for year-round energy saving in buildings. *Nature Communication*, **11**, 1-9.
- Mandal, J., Jia, M., Overvig, A., Fu, Y., Che, E., Yu, N., Yang, Y. (2019). Porous polymers with switchable optical transmittance for optical and thermal regulation. *Joule*, **3**, 3088-3099.
- Mei, X., Wang, T., Chen, M., Wu, L. (2022). A self-adaptive film for passive radiative cooling and solar heating regulation. *Journal of Materials Chemistry A*, **10**, 11092-11100.

Zhang, C., Yang, J., Li, Y., Song, J., Guo, J., Fang, Y., Yang, X., Yang, Q., Wang, D., Deng, X. (2022). Vapor–liquid transition-based broadband light modulation for self-adaptive thermal management. *Advanced Functional Materials*, **32**(48), 2208144.

## Oral 9.04 - Functional Transparent Wood Through Incorporation of Modified Antimony-Doped Tin Oxide Nanoparticles

Zhe Qiu<sup>1</sup>

<sup>1</sup>Material Science and Engineering College, Key Laboratory of Bio-based Material Science and Technology (Ministry of Education), Northeast Forestry University, 150040 Harbin, China. E: zxqiuzhe@163.cn

**Keywords:** functionalization, infrared heat shielding, nanoparticles, transparent wood, ultraviolet resistance

### ABSTRACT

Transparent wood (TW), prepared by infiltrating suitable resin into delignified wood, has the advantages of high light transmission, good mechanical properties and low thermal conductivity, and is considered as an emerging candidate to replace traditional glass for applications in energy efficient building (Li *et al.* 2016, Zhu *et al.* 2016). However, the actual application faces many challenges, such as the inherently poor properties of ultraviolet resistance and infrared heat shielding. To address the above problems, the antimony-doped tin oxide (ATO) nanoparticles were introduced into TW. The ATO was first modified by silane coupling agent to improve its dispersibility. Then, it was directly added into pre-polymerized methyl methacrylate, after which the mixed functional resin impregnated the delignified wood to prepare functional TW. The obtained ATO/TW exhibited high transparency, excellent near infrared heat shielding performance (transmittance: < 10% at 0.3% addition, 1700 nm), and ultraviolet shielding (transmittance: < 20% at 0.7% addition, 360 nm) properties according to the ultraviolet–visible spectrophotometer measurement, the infrared heat shielding simulation test, and UV shielding test. Moreover, the ATO/TW has good mechanical strength and low thermal conductivity, the results indicate that the multifunctional and durable ATO/TW has potential to be used as energy-saving building material.

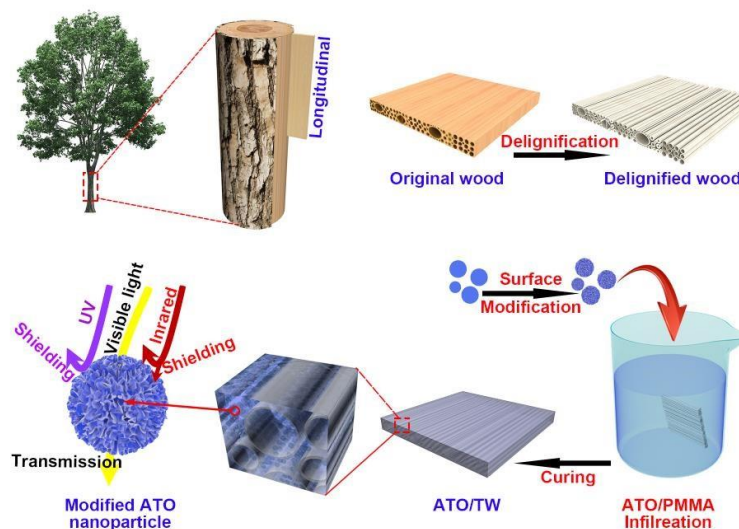


Figure 1: Fabrication and function of ATO/TW

## REFERENCES

Li, Y., Fu, Q., Yu, S., Yan, M. and Berglund, L. (2016). Optically transparent wood from a nanoporous cellulosic template: combining functional and structural performance. *Biomacromolecules*, **17**(4), 1358-1364.

Zhu, M., Song, J., Li, T., Gong, A., Wang, Y., Dai, J., Yao, Y., Luo, W., Henderson, D. and Hu, L. (2016). Highly anisotropic, highly transparent wood composites. *Advanced Materials*, **28**, 5181-5187.



## Oral 9.05 - Enhancing Building Energy Efficiency: Impregnation of Wood with Phase Change Materials

Jakub Grzybek<sup>1,2</sup> and Thomas Schnabel<sup>1,3</sup>

<sup>1</sup>Department of Green Engineering and Circular Design, Salzburg University of Applied Sciences, 5431 Kuchl, Austria E: jakub.grzybek@fh-salzburg.ac.at; thomas.schnabel@fh-salzburg.ac.at

<sup>2</sup>Faculty of Forestry and Wood Technology, Department of Wood Science and Technology, Mendel University in Brno, 613 00 Brno, Czech, Republic

<sup>3</sup>Faculty for Design of Furniture and Wood Engineering, Transilvania University of Brasov, Romania

**Keywords:** green engineering, latent heat storage, thermal energy storage, wood impregnation, wood modification

### ABSTRACT

In the European Union, buildings account for 40% of energy consumption and 36% of greenhouse gas emissions. This underlines the crucial role that improving the energy efficiency of buildings plays in achieving the carbon neutrality target set by the European Green Deal for 2050. One potential solution to the growing energy demand for heating and cooling is the use of phase change materials (PCMs) in building structures. PCMs could enhance thermal inertia, thereby improving overall energy performance. The incorporation of PCMs into wood provides an opportunity for its application in building construction to reduce energy consumption for indoor heating and cooling.

In this study, the process of wood impregnation with PCMs was carried out and the thermal properties were characterised. Special attention was given to preventing the PCM from leaking out of the wood in the liquid state. The results showed that the specimens achieved a high level of impregnation, resulting in a significant weight percentage gain of the phase change material. The treated wood also exhibited a longer time delay during heating and cooling, which can be attributed to the temperature shift during the phase transition. In addition, by implementing a bio-based approach, leakage was successfully minimised. These results suggest that the incorporation of fatty acids as phase change materials into wood matrix has great potential to increase the heat storage capacity of wood and reduce energy consumption for heating and cooling indoor spaces.

## Oral 9.06 - Optical Smart Transparent Wood via Based on Phase-Change Copolymer

Yonggui Wang<sup>1</sup>

<sup>1</sup>Material Science and Engineering College, Key Laboratory of Bio-based Material Science and Technology (Ministry of Education), 150040 Harbin, China. E: wangyg@nefu.edu.cn

**Keywords:** Energy saving, flexible wood, optical responsiveness, thermal phase change

### ABSTRACT

Thermal phase change materials possess the property of optical change when undergoing a phase transition, which has great potential in smart homes, energy-saving buildings, and surveillance of temperature (Oro *et al.* 2012, Prajapati and Kandasubramanian 2019, Li *et al.* 2020). However, they are either easy to leak, have low mechanical strength, or high transition temperature (Mohseni *et al.* 2020, Cui *et al.* 2017, Golestaneh *et al.* 2016). In this study, a copolymer consisting styrene and butyl acrylate with a low content of 1-octadecene was infiltrated into flexible wood to obtain transparent wood with flexibility and thermo-reversible optical properties (SBO/TW). The 1-octadecene endowed the SBO/TW with obvious revisable optical performances, and benefited from the low content, the SBO/TW could repeatedly rapidly turn from opaque (~23.7% of transmittance, ~98.3% of haze) to transparent (~74.9% of transmittance, ~36% of haze) at common condition of room temperature to 50 °C. Moreover, the SBO/TW showed excellent flexibility with a good tensile strength of 15.3 MPa. In addition, it had excellent thermal insulation property with a thermal conductivity lower than 0.2 W/(mK). These properties make this thermal optical responsive wood attractive for intelligent lighting control systems.

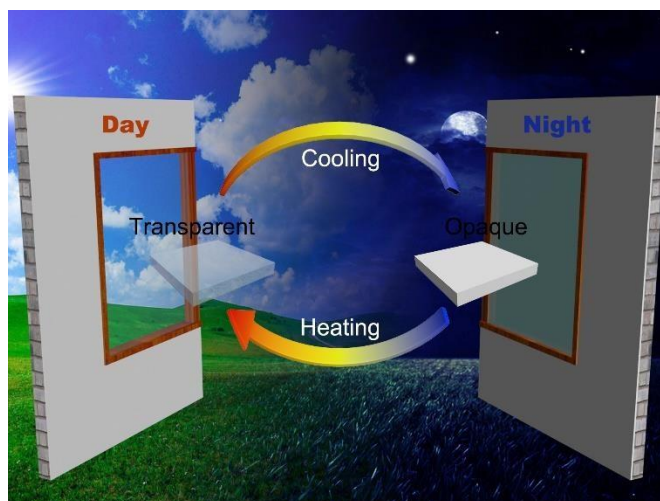


Figure 1: SBO/TWs were applied as smart window element: transparent in the day was in favour of lighting and opaque in the night was good for privacy protection.

## REFERENCES

- Oro, E., Gracia, A. de, Castell, A., Farid, M.M., Cabeza, L.F. (2012) Review on phase change materials (PCMs) for cold thermal energy storage applications. *Applied Energy* **99**, 513-533.
- Prajapati, D.G. and Kandasubramanian B. (2019). Biodegradable polymeric solid frameworkbased organic phase-change materials for thermal energy storage. *Industrial & Engineering Chemistry Research* **58** (25), 10652-10677.
- Li, S., Zeng, W., Wang, B., Xu, H. and Peng Y. (2020). Obtaining three cleaner products under an integrated municipal sludge resources scheme: struvite, short-chain fatty acids and biological activated carbon. *Chemical Engineering Journal*, **380** (15), 122567.
- Mohseni, E., Tang, W., Khayat, K.H. and Cui, H. (2020). Thermal performance and corrosion resistance of structural-functional concrete made with inorganic PCM. *Construction and Building Materials*, **249**, 118768.
- Cui, H., Tang, W., Qin, Q., Xing, F., Liao, W., Wen, H. (2017). Development of structuralfunctional integrated energy storage concrete with innovative macro-encapsulated PCM by hollow steel ball. *Applied Energy*, 185, 107-118.
- Golestaneh, S.I., Mosallanejad, A., Karimi, G., Khorram, M. and Khashi M. (2016). Fabrication and characterization of phase change material composite fibers with wide phase-transition temperature range by co-electrospinning method. *Applied Energy*, 182, 409-417.

## Oral 9.07 - Thermoplastic from Wood: Dream or Reality?

Prabu Satria Sejati<sup>1,2</sup>, Firmin Obounou Akong<sup>1</sup>, Frédéric Fradet<sup>3</sup>, Philippe Gérardin<sup>1</sup>

<sup>1</sup>LERMAB, INRAE, Université de Lorraine, 54000 Nancy, France. E: philippe.gerardin@univ-lorraine.fr

<sup>2</sup>Research Center for Biomass and Bioproducts, National Research and Innovation Agency (BRIN), 16911 Bogor, Indonesia

<sup>3</sup>PLASTINNOV, IUT de Moselle-Est, Université de Lorraine, 57500 Saint-Avold, France

**Keywords:** esterification, fatty acid, thermoplastic, wood

### ABSTRACT

Thermoplastics are ubiquitous in the materials sector due to their facility of processing using different polymerization processes and numerous starting monomers. However, despite the many advantages of these materials, their petrochemical origins and low biodegradability are major drawbacks to their use. According to the French Environment and Energy Management Agency, bioplastics made from renewable raw materials are set to develop rapidly over the coming years, helping to limit greenhouse gas emissions and the impact of these materials on the environment. Cellulose acetate was one of the first bioplastics invented in 1865. More recently, a number of biobased polymers with structures similar to those of fossil-based polymers (PE and PET from sugar cane) or with new structures (PLA from starch) have been developed. However, the non-biodegradable nature of the former and competition with food resources for the latter are disadvantages for the development of these biobased polymers.

Using renewable resources to produce plastics helps to reduce the need for petrochemicals, making the sector less dependent from fossil resources. In this context, the use of wood could be an interesting raw material for the production of bioplastics. The development of wood/polymer composites (WPC) using mixtures of different thermoplastics and wood powder has already been widely studied in the past decades. However, despite early work on cellulose acetate and WPC, very little work has been carried out directly on wood to confer it thermoplastic properties. A few studies have reported on different attempts to make wood thermoplastic involving either reduction of cellulose crystallinity, either destruction of hydrogen bonds responsible for cellulose crystallinity. Some other attempts describe modification of wood structure using chemical modification reactions like benzylation, octanoylation or cyanoethylation.

The aims of this presentation are twice: first we will propose a review of the state of the art of previous studies described in the literature to confer to wood thermoplastic properties and secondly, we will focus on a new method developed in our laboratory to confer to wood intrinsic thermoplastic properties without addition of any petrosourced polymer or plasticizer. For this purpose, wood sawdusts have been modified with mixed anhydride obtained from reaction of fatty acid with trifluoroacetic acid anhydride used as impelling agent. Reactivity of wood cell wall polymers as well as properties of esterified sawdust using different spectroscopic and thermal analysis will be presented.



