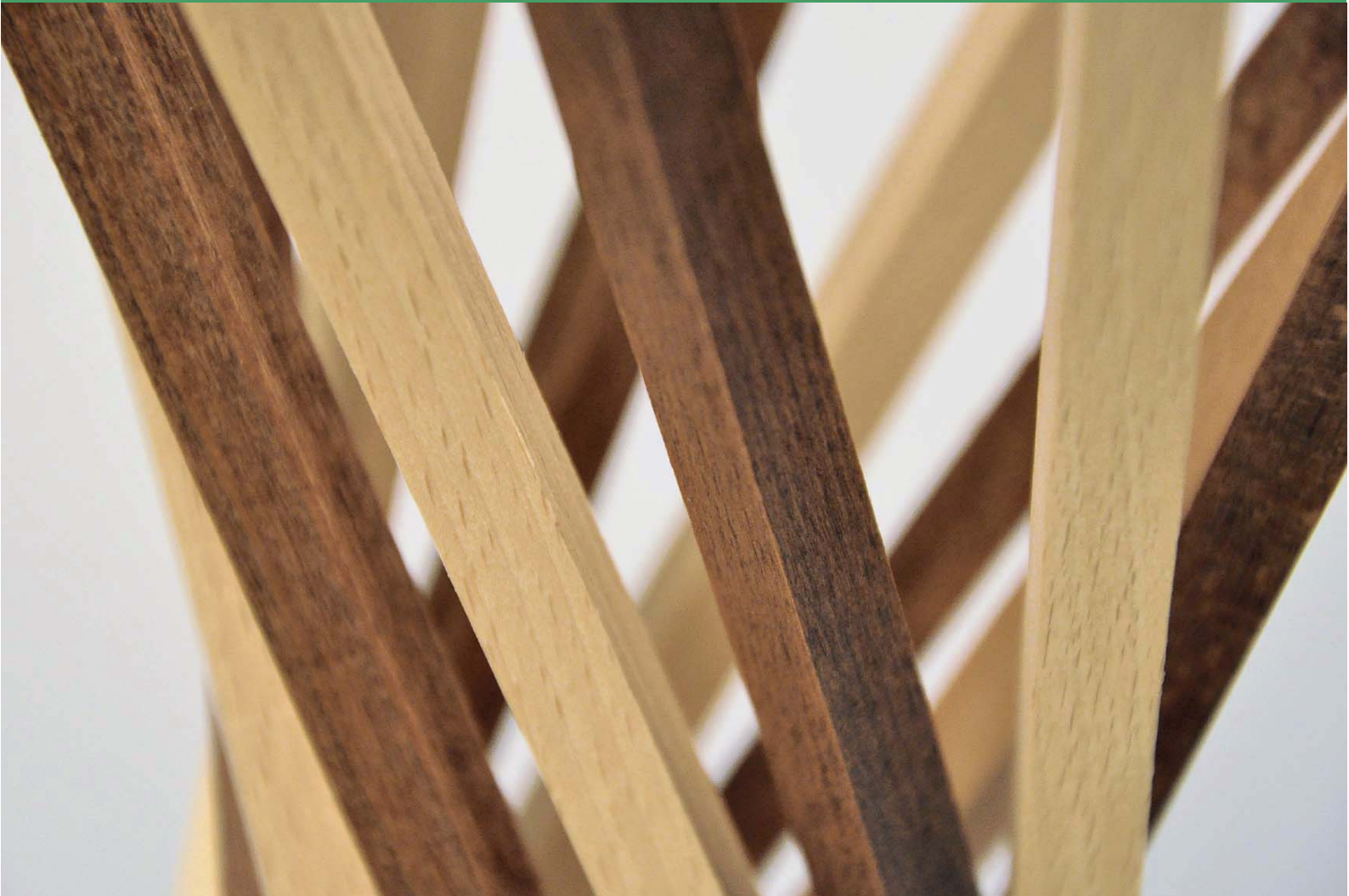


Book of Abstracts



COST Action FP1407 - 3rd Conference „Wood modification research & applications“

Kuchl, September 14-15, 2017

**Salzburg University of Applied Sciences
Forest Products Technology & Timber Constructions**

in collaboration with
the Society of Wood Science and Technology &
the European Conference on Wood Modification



FH Salzburg



ModWoodLife

COST Action FP1407

Understanding wood modification through an integrated scientific and environmental impact approach (ModWoodLife)

Wood modification research & applications

Third COST Action FP1407 International Conference

Kuchl, Austria

14-15 September 2017

Editors: Gianluca Tondi, Marko Posavčević, Andreja Kutnar and Rupert Wimmer

Salzburg University of Applied Sciences

Kuchl, 2017

Print ■ Druckerei Schönleitner

Print-run ■ 150 copies

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COST Action FP1407

3rdInternational Conference

Kuchl, Austria

14-15 September 2017

“Wood modification research & applications”

Organizer ■ Salzburg University of Applied Sciences; Forest products technology & Timber constructions

Co-organizers ■ Society of Wood Science and Technology (SWST) & European Conference on Wood Modification (ECWM).

Editors ■ Gianluca Tondi, Marko Posavčević, Andreja Kutnar, Rupert Wimmer

ISBN 978-3-200-05255-0

Proceedings of the 3rd COST Action FP1407 International Conference - Wood modification research & applications

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All papers have been reviewed.

Cover pictures ■ Gianluca Tondi, Hermann Huber, Alexander Petutschnigg;
Salzburg University of Applied Sciences

Cover design ■ Gianluca Tondi, Ingrid Seidl; Salzburg University of Applied Sciences

Published by ■ Salzburg University of Applied Sciences Press, Marktstraße 136a, A-5431 Kuchl, 2017

ISBN 978-3-200-05265-9 (digital edition)

ISBN 978-3-200-05255-0 (printed edition; not for sale)

The organizers would like to acknowledge the scientific and the editorial committees of the third COST Action FP1407 International Conference “Wood modification research & applications” and the coorganizers:

- Gianluca Tondi - Local Organizer
- Andreja Kutnar - COST FP1407 Action chair
- Rupert Wimmer - Society of Wood Science and Technology – Co-organizer
- Marko Posavčević - Editing
- Holger Militz - European Conference on Wood Modification – Co-organizer
- Michael Burnard - Editing
- Ingrid Seidl - Local Organizer

Book of Abstracts

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Local organizer preface

Isn't a great occasion to combine the newest developments in wood modification with the wonderful late-summer landscape of Austria? So, welcome to the department of Forest products technology & Timber construction of the Salzburg University of Applied Sciences!

This International Conference is the fourth event in eight years after the first meeting of 2010 and the workshop of 2012 of the COST action FP1006 and the PTF BPI "Processing Technologies for the Forest & Bio-based Products Industries" of 2014. This highlights our position in the International scientific position in wood science research.

The present conference was organized in collaboration with the Society of Wood Science and Technology (SWST) and with the European Conference on Wood Modification (ECWM). Many thanks to both organizations for contributing to give this meeting such a high impact.

You might have great expectation now and therefore I personally feel very challenged because this conference is the first that I have organized actively. Hence, thank you all for being here and for your support during the preparation of this event. I wish we will be able to render this conference at least as much pleasant as the previous ones were.

The considerable amount of submitted papers has involved a great effort for the scientific and editorial committees and therefore the resulting conference program has become very intense. I wish this format will be easy to follow and I am looking forward to listen to all of these "pills of science" and I wish you too!

Finally, I take this occasion to thank all the researchers who submitted their contributions and in particular the Andreja Kutnar, Rupert Wimmer, Holger Militz, Marko Posavčević and Mike Burnard as well as the essential support of Ingrid Seidl.

Thank you again and... Enjoy the conference!

Gianluca Tondi

Local organizer, Salzburg University of Applied Sciences.

Action chair preface

Welcome to the third international conference of COST Action FP1407 “Understanding wood modification through an integrated scientific and environmental impact approach” (ModWoodLife). The conference, “Wood modification research and applications”, held in Kuchl, Austria September 14th and 15th, 2017 brings together researchers from across Europe and beyond. The Society of Wood Science and Technology co-organised this conference, giving our Action an opportunity to extend beyond Europe. It is important to be open to the world, especially for our Action, because only global collaboration in research and development will lead to the most impactful breakthroughs in wood modification. This conference will focus on presenting innovative materials and process developments for various wood modification technologies, ecological solutions, and other challenges related to improving the properties of timber to guarantee more sustainable wood use. Special emphasis will be given to innovative, bio-friendly, wood protection techniques and preservatives.

During the first 24 months of the Action we have made steady progress towards COST FP1407’s main objective thanks to your contributions. That objective, to characterise the relationship between wood modification processing, product properties, and the associated environmental impacts in order to maximise sustainability and minimize environmental impacts, has great value for the forest sector, for researchers, and society at large. The Action delivered the state of the art of generic Life Cycle Assessments and Environmental Products Declarations of wood products in different Member States. Our other achievements so far include: a systematic comparison of modification processes including their technical characteristics and environmental performance; delivered the state of the art of life cycle analyses of different commercial modification processes; used “cradle to gate” and “cradle to grave” to examine scenarios for the service life and end of life of wood products; recruited industrial stakeholders to become active in the Action; characterized selected modified wood materials and products; reviewed carbon sequestration calculation methodologies; delivered the state of the art on end of life management of wood products; and examined the state of policy positions/actions related to wood/biobased material use.

The Action has delivered two scientific international conferences, both in COST inclusiveness countries (Slovenia and Czech Republic), and each produced proceedings that were published in print and on-line. In the first two grant periods, two training schools were organized (Finland and UK) and 24 STSMs were performed. Within the Action’s network 8 joint SCI publications were published and 5 proposals submitted. The Action has released 3 newsletters, is active on social media, and has contributed professional journal articles that were published in Greece, Hungary, Portugal, Slovenia, Serbia, and Ukraine in those countries national languages.

Thank you so much and please keep up the great work!

Wishing you a successful and memorable conference in Kuchl full of fruitful discussions.

Andreja Kutnar
Chair, COST FP1407

Conference Program

COST Action FP1407 3rd Conference:
“Wood modification research and applications”
*co-organized with Society of Wood Science and Technology &
the European Conference of Wood modification*

Wednesday, September 13th, 2017

18:00-21.00: Welcome evening. “Drinks and Blues”, with live music featuring Doug Gardner and Rupert Wimmer

Thursday, September 14th, 2017

8:30: **Registration**

8:50-9:05: Welcome to Kuchl: **Alexander Petutschnigg & Gianluca Tondi**

9:05-9:10: Welcome from COST action chair: **Andreja Kutnar**

9:10-9:15: Welcome from the ECWM chair: **Holger Militz**

Keynote session		Chairs: A. Kutnar, R. Wimmer
9:15	New strategies for modifying wood with biopolymers	W. Grigsby
9:45	Advanced analysis tools and programs to accelerate the adoption of more natural structures	C. G. Hunt, J. Jakes, C. Frihart
10:15	Presentation of SWST and its presence in Europe	V. L. Herian
10:30	Presentation of "ForestValue"	M. Greimel

10:45-11:15: **Coffee break**

ECWM Session: Novel wood modification technologies		Chairs: H. Militz, I. Burawska
11:15	Effects of methyl methacrylate impregnation on physical properties of timber	S. Curling , M. Spear, R. Gibson, G. Ormondroyd
11:28	Catalytically induced <i>in situ</i> polymerisation of ethylene in the hierarchical porous wood structure	J. Gurr , G.A. Luinstra, A. Krause
11:40	Modified wood with lactic acid oligomers: Assessment of performance	C. Grosse , M. Noël, MF. Thévenon, P. Gérardin
11:53	Improvement of beech wood properties through chemical modification with bio-sourced polyesters	C. L’Hostis, E. Fredon , MF. Thévenon, P. Gérardin

12:05	Laminated veneer lumber (LVL) made of beech wood veneers modified with lignin-phenol formaldehyde solutions	M. Fleckenstein , V. Biziks, C. Mai, H. Militz
12:18	Ionic liquid pre-treatment to reduce the elastic spring-back and set-recovery of surface densified Scots pine	B. Neyses , D. Sandberg
12:30	Humidity optimization considerations for thermal wood modification	W. Willems

12:45-14:15: **Lunch break**

Session 2 : Characterisation of modified wood		Chairs: J. Sandak, A. Rozanska
14:15	Exploration of hypothesis that limitation of fungal oxidant diffusion underlies decay resistance in acetylated wood	C. Hunt , S. Lacher, K. Hirth, L. Lorenz, K.Hammel, C.Houtman, E. Engelund Thybring, S. Zelinka
14:23	Dimensional stable and durable laminated veneer lumber (lvl) from european beech (<i>fagus sylvatica</i>) by impregnation with low molecular weight phenolic resin	S. Bicke , V. Biziks, H. Militz
14:40	Fixing tannin in wood: Characterization of the treated wood	L. Sommerauer, D. Bartosch, R. Waschak, A. Oberle, MF. Thevenon, G. Tondi
14:53	Assessment of chemical fingerprint of modified wood	J. Sandak , A. Sandak, O. Allegretti, I. Cuccui
15:05	Wood modification method efficiency evaluation by cyclic hydrothermal treatment	A. Morozovs , A. ee, L. Fišere, U. Spulle
15:18	Machinability of thermally modified wood assessed with a new objective method	G. Goli , J. Sandak, A. Sandak, P. Cetera, L. Todaro
15:30	Microscopic investigation of acetylated hornbeam and densified beech	R. Rousek , F. Fodor

15:45-16:15: **Coffee break**

Session 3 : Innovative use of wood modification processes		Chairs: L. Ross Gobakken, W. Grigsby
16:15	Modification of the particleboard core layer by non-wood lignocellulosic raw materials	R. R�h
16:28	The use of novel modified wood fiber for manufacturing structural wood plastic composite timber for an innovative marine application	D. J. Gardner , Y. Han, D. Mayes, J. Pynnonen, S. Ruell
16:40	Sawdust-based activated carbon for wastewater treatment from textile industry	B. Bestani , N. Benderdouche
16:53	Sustainable use of <i>Eucalyptus globulus</i> residues for polyurethane foam production	A.P. Fernandes, J. Ferreira, I. Domingos, J. Labidi, L. Cruz-Lopes, B. Esteves

17:05	Mechanochemo modification of cellulose powders	M. Wolcott , M. Azadfar, L.Huang, J. Wang
17:18	Using pre-stressed or non-pre-stressed curved members of concentrically composited laminated timber in structures of non-controllable environment	T. Teppand

17:30: **Close of the first day**

17:30-19:00: **Core group meeting**

19:00: **Bus transfer to dinner from Campus Kuchl to Sternbräu (Griesgasse 23), Salzburgcentre**

19:30: **Conference dinner**

Friday, September 15th, 2017

Short term scientific mission session		Chair: M. Schwarzkopf
9:00	Micro-distribution of epoxidized oils in wood	G. Kose Demirel, A.Temiz , E. D. Gezer
9:07	Enhancement of coatings for wooden claddings via plasma pre-treatment and environmental impact	J. Žigon , M. Petrič, S. Dahle
9:15	Understanding of the effect of natural saltwater treatment on durability, fibers densification and chemical modification of palm wood	M. T. Elaieb, A.Namsi, K. Candelier
9:22	The effect of wood drying and heat modification on some physical and mechanical properties of radiata pine (results from STSM 35419)	R. Herrera , J. Labidi, R. Llano-Ponte, R. Ananias
9:30	Approach for cascading – Analysis of the application potential of different tree materials with antimicrobial properties	K. Wagner , C. Roth, G. Oosting, M. Musso, A. Petutschnigg, T. Schnabel
9:37	Analysis of fracture toughness in Mode II on modified wood	M. Redon , V. Sebera, M. Brabec, D.Decky, P. Čermák, J. Milch, J.Tippner
9:45	Environmental profiles of alternative tannin extraction scenarios	T. Ding, S. Bianchi, C. Ganne-Chédeville, P. Kilpeläinen, A. Haapala, T. Rätty
9:52	Review of biogenic carbon in carbon footprint of modified wood	L.G.F. Tellnes , C. Ganne-Chedeville, A. Dias, F. Dolezal, C. Hill, E. Zea Escamilla
10:00	Advanced understanding of the structural influences of bio-based lignocellulosic materials - Future industrial applications in high added value wood modification products	G. Schmidt , J. Gurr, O. Mertens, M. Nopens

Poster Session		Chairs: D. Sandberg, L.G.F. Tellnes
10:10	Thermo-mechanical treatment of flooring elements	I. Burawska , P. Boruszewski
10:12	Improvement of wood heat treatment via an acoustic field	E. A. Silveira , A. Pétrissans, A. Caldeira - Pires, M. V. Girão, B. Colin, P. Rousset, M. Pétrissans
10:14	Surface modification of solid wood by charring	M. Kymäläinen , S. Hautamäki, K. Lillqvist, K. Segerholm, L. Rautkari
10:16	Development of UV – colour modification of wood surface	P. Daniel , V. Kotradyová, R. Tiňo, V. Dvonka
10:18	Wood modification by alkali-activated composition coatings for fire protection	D. Vaičiukynienė , R. Bistrickaitė, A. Kielė
10:20	Wood modification with n-methylol compounds – effects of modification agent and process conditions	L. Emmerich , S. Bollmus, H. Militz
10:22	Prediction of mass loss dynamics during wood thermal modification under industrial conditions	B-J. Lin , E. Silveira, B. Colin, A. Pétrissans, W-H. Chen, P. Rousset, M. Pétrissans
10:24	Mechanical properties of densified and thermally modified timber	J. Wehsener , C. Brischke, L.Meyer-Veltrup, P. Haller
10:26	The ability to layered wood composites pressing time modification	A. Gumowska , G. Kowaluk, E. Robles
10:28	Investigation of periodical effect on modified woods outdoor colour change	M. Bak , R. Németh
10:30	Nanoscale mechanical properties of wood: effects of heat treatment	S. Saleh , M. Wentzel, H. Militz, C. Volkert
10:32	Color and wettability changes of heat-treated wood finished with UV-rad cured coating after artificial weathering	E. Robles , R. Herrera, J. Sandak, J. Labidi
10:34	Surface behaviour of poplar and spruce wood after immersion in extractives solution achieved from thermally treated Hungarian oak	P. Cetera , L. Todaro, T. Meints, W. Gindl-Altmatter
10:36	Studies of the gluability of the pine wood veneers after TM modification with the use of PVAC adhesives	A. Bernaczyk, T. Krystofiak , B. Lis
10:38	Impact of selected modification systems on elasto-mechanical properties of wood	S. Bollmus, C. Leitch, H. Militz
10:40	Investigation of tropical wood modification under hydro-mechanical loadings with digital image correlation and X-ray microtomography	R. Moutou Pitti , S-E. Hamdi, B. Odounga, M. C. Teguedi
10:42	Acoustic emission technique to monitor real-time wood fracture properties in room temperature	M. Diakhate, R. Moutou Pitti , N. Angellier, E. Bastidas-Arteaga, S.-E. Hamdi
10:44	Performance of 3-layer composites with densified surface layers of Nothofagus species of Southern Patagonian forests	M.Schwarzkopf , M. Burnard, G.Martínez Pastur, L. Monelos, A. Kutnar
10:46	The activity of moulds on wood surfaces modified with laser	L. Reinprecht , Z. Vidholdová
10:48	Do extractive compounds of thermally modified woods play an important role in the decay and termites resistances of these modified materials? A preliminary study.	K. Candelier , MF. Thévenon, R. Collet, P. Gérardin, S. Dumarçay

10:50	In-situ SEM / TEM fracture tests on (modified) tracheids of pine latewood	M.-C. Maaß , M. Wentzel, H. Militz, C. Volkert ¹
10:52	Traditional wood finishing substances and their influence on surface roughness	A. Rozanska , P. Kieblesz
10:54	Biobased phenolic resins for wood protection against fire	P.L. de Hoyos Martínez , F. Charrier El-Bouhtoury, J. Labidi
10:56	The activity of bacteria on surfaces of wooden composites painted with acrylate coating with addition of silver nanoparticles	J. Iždinský , L. Reinprecht, E. Nosál, Z. Vidholdová, J. Krokošová
10:58	Hydrophobicity of ϵ -caprolactone-modified wood materials	Z. Candan , M. Yildirim, A. Satir, M. A. Ermeýdan, O. Gonultas
11:00	Natural adhesives from liquefied wood based resins and their applications	M. H. Alma , T. Salan
11:02	Bio-based foams from renewable and sustainable polyols obtained via liquefaction of wood and other lignocellulosics	T. Salan , M. H. Alma
11:04	Analysis on the quality of vegetable charcoal derived from Sapucaia's endocarp (<i>lecythis pisonis</i>)	R.S. de Araújo , W.S. L. da Costa, T.dos Santos Farias, A. R. Souza Reis, S.H.F. da Silva, P. S. B. dos Santos
11:06	Spectroscopic characterisation of bio-based filaments for the fused deposition modeling proceeding	S. Kain , M. Musso, A. Petutschnigg

11:10-11:50: **Coffee break**

Session 4 : Modified wood in sustainable built environment		Chairs: C. Hill, F. Dolezal
11:50	Common themes in wood modification and environmental impact assessment of wood	M. Burnard , M. Posavčević, E. Kegel ³
12:03	Carbon sequestration in the built environment – the role of harvested wood products	C. Hill
12:15	Comparative assessment of carbon uptake and release of wooden and concrete building materials	F. Dolezal , P. Boogman
12:27	Performance of thermally modified radiata pine facade, gallery and decking in a passive house in Spain after six years exposure. Research and applications in a real case.	D. Lorenzo , A. Lozano, J. Benito, M. Touza, J. Fernández-Golfín ⁵
12:40	Architects' perception of modified wood: a parallel study in selected countries in Europe and selected regions in USA	M. Kitek Kuzman , E. Haviarov, D. Sandberg

12:55-14:00: **Lunch**

14:00- 15:00: **Working group meeting (WG1, WG2, WG3, WG4)**

15:00- 15:15: **Reports of WG leaders and conference conclusions**

15:15- 15:45: **Coffee break**

15:45- 17:00: **Management committee meeting**

Thursday 14th September,
COST Action FP1407: 3rd meeting
“Wood modification research
& applications”

DAY 1

Keynote session

From 9:15 to 10:45

Chairs: A. Kutnar, R. Wimmer

New strategies for modifying wood with biopolymers

Warren Grigsby¹

¹Scion, Te Papa Tipu Innovation Park, 49 Sala Street, 3010 Rotorua, New Zealand;

warren.grigsby@scionresearch.com

Keywords: wood drying, dewatering, SCF processing, furfuryl alcohol

Introduction

Wood is susceptible to reversible and irreversible changes induced by varying responses to moisture which include degradation on drying, difficulty in removing water on drying, and dimensional instability on undergoing sorption processes with liquid or atmospheric water. This paper will follow a recent review on successful processing technologies and profile strategies which target overcoming moisture-induced responses of wood (Graichen 2017). These approaches also consider the green credentials of the wood products and build on sustainability, energy and employing renewables to be cost-effective approaches to manufacturing decorative, high value timbers for exterior use.

Dewatered wood a new substrate produced by supercritical processing

While used globally, kiln drying timber can be lengthy, energy intensive and induce stresses within the wood. As alternatives, the use of compressed gases, such as air or carbon dioxide have been evaluated to accelerate water removal from timber (Jones and Walker 1999, Stahl and Bentz 2004). In using compressed gases, the wood is subjected to pressure cycles to induce water migration from the wood. Scion has focused activity on employing supercritical carbon dioxide (SCF) processing to remove water from green timbers including fundamental understandings of this process (Dawson *et al.* 2015, Franich *et al.* 2014). This process coined “dewatering” similarly employs successive pressure cycles to reduce green timber to its fibre saturation point (*ca.* 35-40% mc). This process can be very rapid (<1 h), energy efficient and avoid introducing drying stresses or colour found with kiln-dried timber. This technology has been evaluated at pilot- and commercial-scale for drying softwood radiata pine and also extended to other wood species. Fundamental studies including magnetic resonance imaging have provided understandings of mechanisms of water movement within lignocellulosics under SCF conditions employed in this process (Newman *et al.* 2016).

While a paradigm shift in wood-drying technology, this SCF dewatering technology does require initial capital investment in more expensive processing equipment compared to kiln drying facilities. Higher-value applications than just sawn timber are required for SCF processed timbers in order to recover this additional cost. Being at fibre saturation point, dewatered timber can be considered as a new substrate for wood modification given its relatively high moisture content (mc) compared to modifying conventional kiln-dried material (<20% mc). As such, there is a need to design and

understand new treatment technologies for this material. Treatment options for dewatered wood may include a second, sequential treatment or post-treatment and include either addition of preservatives or other additives (Kang *et al.* 2005, Kjellow and Henriksen 2009) or acetylation (Matsunaga *et al.* 2016). As a new substrate for industry, dewatered wood offers unique properties.

Processing and modification of dewatered wood using renewables

In approaches to treating and modifying wood it is of interest to ensure any introduced functionality is both sustainable and environmentally benign. Developing *in situ* polymerisation of bio-derived monomers within dewatered wood to modify timber performance can achieve these expectations and include glycolic acid (Noël *et al.* 2015), furfuryl alcohol (Lande *et al.* 2004) and acetic acid. With wood furfurylation a well understood process, the feasibility of modifying dewatered wood with furfuryl alcohol has been initially demonstrated. Furfurylation of dewatered wood was found to be influenced by both the initial moisture content of the dewatered wood and the furfuryl alcohol content of the treatment solution. Uptake of aqueous solutions with high furfuryl alcohol content provide moderate weight gains using dewatered timber that has undergone SCF processing immediately prior to the furfurylation treatment. These observed lower uptakes with dewatered timber at fibre saturation may be due to penetration of the furfuryl alcohol solution into the cell wall being inhibited. However, treatment uptakes may also be aided by the increased porosity within dewatered wood compared to kiln dried material (Grigsby *et al.* 2013). Nonetheless, furfurylation of both dewatered wood and kiln-dried wood to a similar furfuryl alcohol weight uptake result in the modified wood having comparable dimension stability and hardness.

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Advanced analysis tools and programs to accelerate the adoption of more natural structures

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Keywords: Wood properties, structure-property relationships, durability, adhesives

While wood is a highly desirable building material from an ecological and sustainability perspective, we do not understand its fundamental properties nearly as well as we understand competing materials such as steel and concrete. We can avoid toxic preservatives by acetylating wood, but we don't fundamentally understand why acetylation works. Hydroxymethylated resorcinol (HMR) can dramatically improve the water resistance of wood bonds, but no one has been able to explain the mechanism. This lack of understanding slows progress by forcing wood technology industries to rely on inefficient empirical strategies to develop and validate new products and technologies.

A more efficient route to developing new wood-based materials is to develop a detailed fundamental understanding of the properties of wood and wood-derived materials. A detailed understanding allows selection of the proper design values and provides a roadmap for achieving those values. This talk will provide an overview of new tools and approaches being taken by USFS Forest Products Laboratory scientists to understand the fundamental mechanisms behind decay resistance of modified wood and moisture durability of wood adhesives. Advances in these areas will accelerate the development of improved wood modifications, a wide range of bio-based (lignin, tannin, protein) adhesives, traditional adhesives, and other biomass utilization strategies. The improved performance of these technologies will generate new market opportunities for wood and bio-based materials.

Understanding what makes wood decay resistant

The leading hypothesis explaining the decay resistance mechanism of modified wood is that nanoscale water-swollen regions of the wood allow transport of fungal "digestive juices" to move into the cell wall and allow partially digested sugars to diffuse out of the cell wall to feed the fungus. What is lacking is a test of this hypothesis: at the nanometer scale, where is the water? What are the properties of these water swollen regions? What are the diffusion rates in these regions before and after acetylation? What chemicals are fungi using to digest the wood cell wall? Some approaches include:

- Diffusion measurements –Testing hypotheses behind the effectiveness of bulking and crosslinking treatments in enhancing decay resistance (Zelinka *et. al.*, 2014)
- Decay mechanisms – Understanding the chemical systems fungi depend on to degrade wood

Durable adhesive performance

Many years of empirical experience have provided general rules about which adhesives will work and which will not under a given set of conditions, but little in the way of fundamental, quantifiable principles that can be used to design durable bonds, especially with novel substrates or adhesive systems. Until recently, there was little understanding of such questions as why phenol formaldehyde works so well in moisture cycling and epoxy does not, why melamine formaldehyde does not work well for acetylated wood, how isocyanate adhesives stick to the wood, or how HMR improves bonding. The following techniques and programs are attempting to provide the detailed understanding needed to answer these questions, and design adhesive properties to match the substrate and environmental conditions. Some approaches include:

- Theoretical models of the critical components of adhesive durability (Frihart 2009)
- Improved measurements of mechanical properties of wood cell walls – nanoindentation advances, applied to effects of adhesive infiltration, moisture response, viscoelastic properties
- Nanoscale swelling: Small Angle Neutron Scatter (SANS) to measure nanoscale swelling inside the wood cell wall; native and modified wood, with and without adhesive. (Plaza *et. al.*, 2016)
- Measuring swelling forces and moisture sorption of the interphase wood cells
- Detailed understanding of chemical structures of wood – NMR techniques that allow a detailed look at the chemistry inside a wood cell wall

Summary

A solid theoretical understanding of materials properties has given us rust-resistant cars and amazingly powerful computers. Unfortunately, we do not have the deep fundamental understanding of wood to fully utilize the potential of wood and wood-derived products. This talk provides an overview of some of the efforts underway to generate the fundamental understanding required to get the best, most economical performance from wood-based materials.

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Thursday 14th September,
COST Action FP1407: 3rd meeting
“Wood modification research and
applications”

ECWM session:

Novel wood modification technologies

From 11:15 to 12:45

Chairs: H. Militz, I. Burawska

Effects of methyl methacrylate impregnation on physical properties of timber

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Keywords: methacrylate, modification, impregnation, mechanical properties, hardness, stability

The methacrylation of wood was investigated as a method of modifying timber in order to improve its mechanical properties. Samples of pine and spruce wood were impregnated under vacuum with methyl methacrylate monomer using azodiisobutyronitrile as an initiator. Samples cured at moderately elevated temperature (70°C) were conditioned and tested for a number of physical characteristics. Weight percent gains (WPG) of over 80% were observed with pine and over 70% with spruce. In spruce wood in particular, the WPG achieved was dependent on the presence of juvenile or mature wood in the samples. In Three-point bending tests methacrylation significantly increased the modulus of rupture (Fig 1) and modulus of elasticity (Fig 2) values compared to untreated control samples. Compression strength and Janka hardness values were also significantly increased by methacrylate impregnation in both spruce and pine, although the effect was more pronounced in the pine samples.

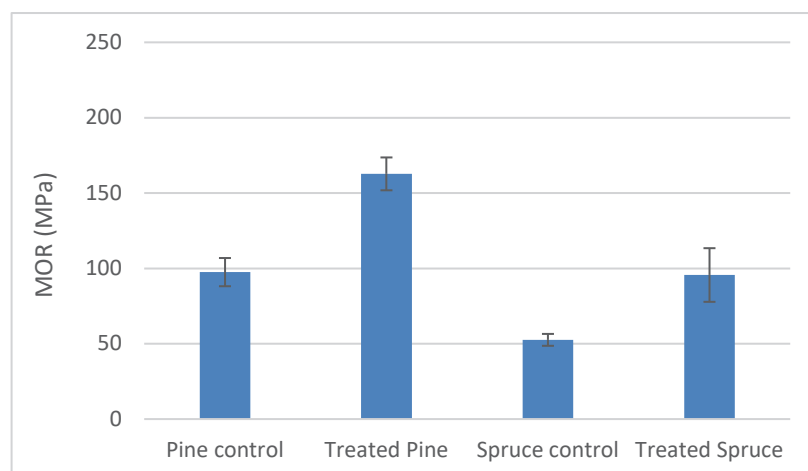


Figure 1: Effect of methacrylation on modulus of rupture (error bars = standard deviation of the mean).

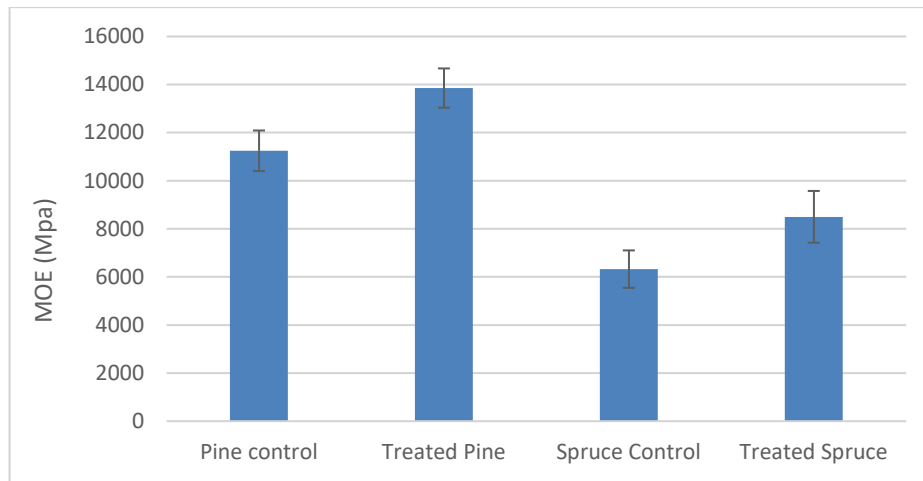


Figure 2: Effect of methacrylation on modulus of rupture (error bars = standard deviation of the mean).

The results of this investigation demonstrate an improvement in timber properties after methacrylation.

Catalytically induced *in situ* polymerisation of ethylene in the hierarchical porous wood structure

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Keywords: wood modification, metallocene catalyst, trimethylaluminium co-catalyst, polyethylene, *in situ* polymerisation

Introducing catalytically induced polyolefin polymerization methods into the field of wood science may open up a vast number of novel wood modification possibilities, i.e. tuning the hydrophobic properties and with that creating the option of new functionalization to enhance the property profile further. The scope of our ongoing research is focused on polymerizing ethylene within the wood structure by *in situ* polymerization techniques. This is achieved by a highly specialized catalytic system, consisting of a metallocene catalyst and an aluminiumalkyl co-catalyst. This system exhibits promising features in the fields of polyolefin nanocomposite production, and it has attracted interest in the macromolecular science community as well as in the industry. The approach followed in this study comprises three steps. In the first step, small solid wood samples of pine sapwood are pre-treated with the co-catalyst trimethylaluminium, which is adsorbed by the wood surface and adsorbed within the pores. In the second step the metallocene catalyst is introduced, which is binding to the immobilized co-catalyst. Hence, catalytically active sites are foremost formed on the wood surface and within the pores. In the third step, ethylene is introduced under low pressure. Upon initiation of the ethylene polymerization, polyethylene is formed on the wood surface and inner pores (Kaminsky 2013).

Field emission scanning electron microscopy (FESEM) and energy-dispersive X-ray spectroscopy (EDX) were utilized to analyse cross sections of the treated wood samples. The FESEM image (Fig. 1) displays filled, partially filled as well as empty cell lumen. The EDX analysis of that same cross section (Fig. 2) displays the distribution of the elements aluminium (blue) and oxygen (green). The distribution of the aluminium is attributed to the co-catalyst. High shares of oxygen are distributed

along the cell walls, whereas negligible shares are distributed in the empty and filled cell lumen. This finding makes us assume that the cell lumen contains or even is filled with polyethylene, as wood cell wall tissue chemically incorporates oxygen whereas polyethylene does not.

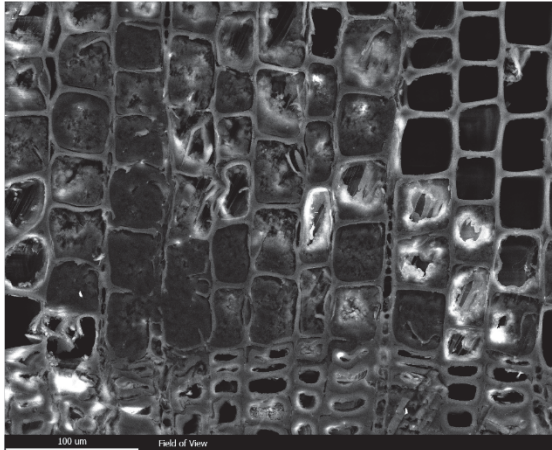


Figure 1: FESEM image of solid pine wood cross section with partially polymer filled cell lumen.

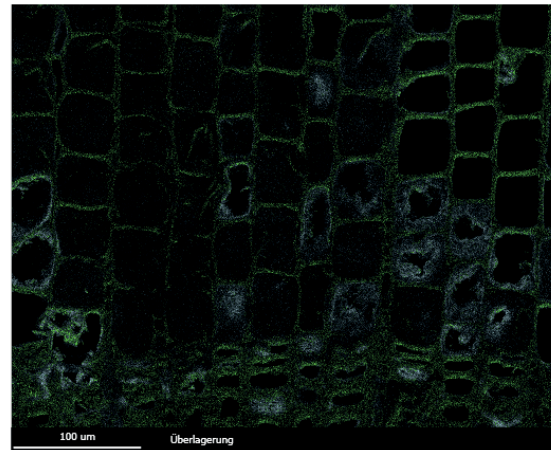


Figure 2: EDX analysis of same cross section. Colour code: green for oxygen, blue for aluminium.

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Acknowledgments:

The authors would like to thank Dr. Frank Friedrich and his workgroup, specifically Ms. Renate Walter, of the Zoology of the University Hamburg for providing expertise and equipment for FESEM and EDX analysis.

Modified wood with lactic acid oligomers: assessment of performance

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Keywords: wood modification, lactic acid, dimensional stability

Treatment based on lactic acid has been developed and optimized for wood modification by Noël *et al.* (2009, 2015) and Grosse *et al.* (2016). This treatment aims to improve wood dimensional stability and biological resistance for its usage outdoor. The process consists in oven-dried beech wood (*Fagus sylvatica L.*) impregnation with lactic acid oligomers (OLA), followed by curing in dry conditions.

Treatment efficiency depends on curing conditions and increases with temperature (Grosse *et al.*, 2016). At 160°C, treatment conferred very promising properties to wood, in particular regarding anti-swelling efficiency (ASE), moisture exclusion efficiency (MEE) and biological resistance and it was persistent in wood (respectively ASE*, MEE, WL_{CV} and WL_{CP}, and LR in Table 1). Mechanical performance evaluation through 3 points bending test, showed the modified wood was more brittle but could resist higher loads. OLA polycondensation was assessed by Fourier transform infrared (FTIR) spectroscopy (Fig. 1).

In order to evaluate possible up-scaling of this treatment, larger samples were impregnated. Density profile was studied on 100x100x550 mm³ samples and showed a homogeneous repartition of product in wood. Homogeneity of resistance against fungal decay was assessed with EN113 on samples of 15x25x500 mm³ (Fig. 2).

Table 1: Results of test.

Treatment	ASE* [%]	MEE [%]	WL _{CV} [%]	WL _{CP} [%]	LR [%]
OLA	61.1 ±3.2	39.4 ±0.7	2.0 ±0.2	2.7 ±0.2	2.3 ±0.2

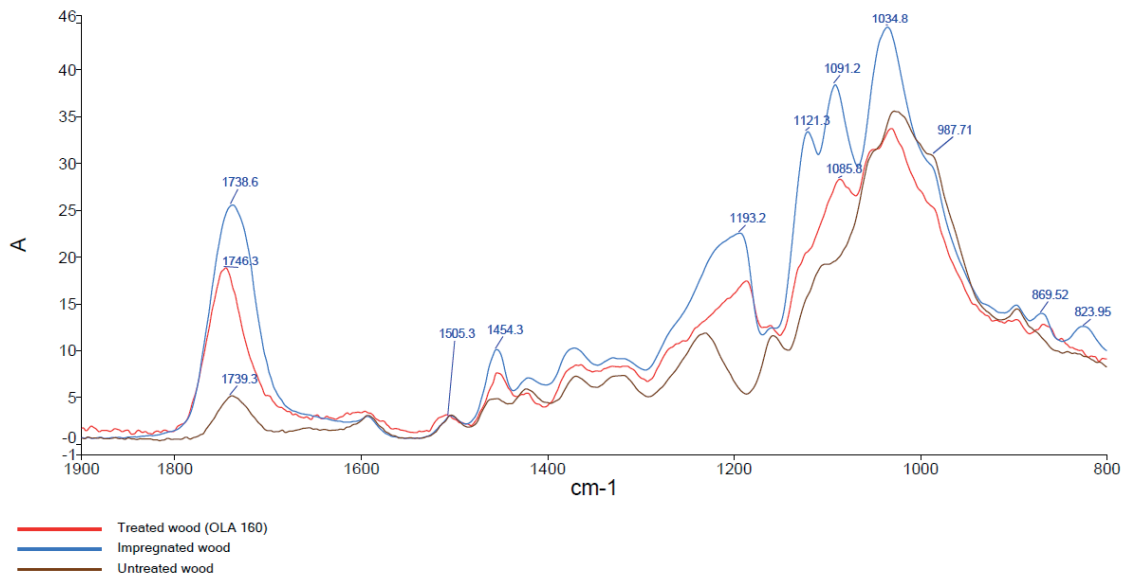


Figure 1: FTIR spectra of untreated wood, impregnated wood and treated wood.



Figure 2: Samples exposed to *Coriolus versicolor* following the EN 113 standard procedure

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Acknowledgments

The research leading to these results was supported by funding from the WoodWisdom-Net Research Program, which is a transnational R&D programme jointly funded by national funding organisations within the framework of the ERA-NET+ Action WoodWisdom-Net+.

Improvement of beech wood properties through chemical modification with bio-sourced polyesters

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Keywords: wood chemical modification, polyesters, ATR-MIR spectroscopy.

The present work deals with the improvement of wood durability and physical properties through innovative and sustainable bio-based chemical treatment. It constitutes a part of the "BIOCOPOL" project, a research program conducted in the frame of WoodWisdom net. In order to avoid the use of biocidal formulations facing environmental concerns, wood chemical modification appears as one of the most promising alternative ways. Among them, acetylation (Rowell 2014) and furfurylation (Lande et al. 2004) are now commercial treatments which allow indigenous wood species to be used in quite severe conditions, as far as significant improvements of decay resistance and dimensional stability can be obtained. Lactic acid based polymers had also been used for wood modification (Noël et al. 2009).

Objectives and principles of the study

The originality of this study is based on the selection of bio-based, available and harmless monomers to be driven in the wood cell walls, and in-situ polymerized to fix them irreversibly. Beech has been chosen for many reasons: outdoor uses of this abundant wood species are strongly limited as it suffers from a low durability and high dimensional instability. Moreover, its high impregnability is an advantage for submit it to chemical impregnation process. Different treatments consisting in grafting or polymerization of bio-based molecules have been investigated: mixtures made of glycerol and citric acid (GCA), glycerol and tartaric acid (GTA), glycerol and succinic anhydride (GSA), once dissolved in aqueous solutions, have been impregnated in beech wood (*Fagus sylvatica*), and oven cured from 103°C to 160°C. Similar processes were conducted using citric acid (CA) and tartaric acid (TA) aqueous solutions as control treatments in order to bring to light some benefits of polymerization: it is expected to offset the weakness induced in terms of mechanical performances usually occurring with thermal degradation.

Results

Evaluated according to the ENV 1250-2 standard, resistance against chemical water leaching was first minimized by tuning the COOH equivalent/OH ratio at 0.5, and this ratio was kept constant during the study. It has been also shown the influence of curing temperature: a correct fixation, keeping leaching under 1% can be achieved using temperature treatment starting at 140°C. Regarding dimensional stability and mechanical resistance, CA and TA treatments present similar behaviour. An increase of dimensional stability and a decrease of three points bending resistance with the increase of treatment temperature are observed for those two treatments, and can be explained by acidic catalysis of wood thermal degradation. GCA and GTA treatments which allow polymerization due to presence of glycerol in solution showed the same tendency concerning dimensional stability, but present totally different results concerning the mechanical aspect: GCA shows a weakening of the material compared to untreated wood, especially when treated at the highest temperature (160°C), while GTA induced an increase when treated at 140°C. GSA treated wood does not present any weakening compared to control samples. Regarding decay resistance against *basidiomycetes* fungi, rapid screening tests (fig 1) and EN 113 standardized tests have been conducted and also show major differences among the treatments. An increase in curing temperature makes treated wooden substrates more resistant to fungi degradation. Concerning formulations, the presence of tartaric acid give unsatisfying results compared to the two others. The differences in chemical structures between the three polycarboxylic acids and the characterizations by means of ATR-MIR spectroscopy help us to explain the significant discrepancies obtained, highlighting the polymerization reactions and the ability of acid end groups to graft on the wood cell walls. Moreover, wood thermal degradation is also observable, explaining the high dimensional stability obtained when the impregnated beech samples were cured at the highest temperatures (160°C). Finally, GSA treatment, presenting the best compromise regarding the different properties, has been studied in terms of economic viability, and appears particularly promising from a commercial point of view.



Figure 1: Screening test samples after 6 weeks decay by *Coriolus Versicolor*

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Acknowledgments:

The research was supported by funding from the WoodWisdom-Net Research Program.

Laminated veneer lumber (LVL) made of beech wood veneers modified with lignin-phenol formaldehyde solutions

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Keywords: lignin, phenol substitution, laminated veneer lumber

The treatment of wood with water-soluble PF resins is a well-known and effective method to improve the dimensional stability, biological durability, and outside weathering properties of wood (Stamm and Seborg 1936; Rowell and Banks 1985; Bicke and Militz 2015). However, there are several disadvantages of PF resins primarily related to economic, environmental, and health issues. For decades scientists have been exploring the possibility of replacing crude-oil based phenol with a renewable alternative such as lignin.

Lignin is an amorphous, three-dimensional bio-polymer (Fengel and Wegener 1989), one of the most important structural elements in higher plants (Suparno et al. 2015) and the most important bio-polymer on earth containing aromatic structures (Tejado et al. 2007). Today, most of the produced lignins are burned to generate energy (Kleinert and Barth 2008). Because of the high availability, current low price and aromatic structure, technical lignins are considered to be a suitable renewable source which could be used as a substitute for crude oil based phenol in PF resins (Wang et al. 2013; Kleinert and Barth 2008; Panday and Kim 2011).

The objectives of our study were to investigate the influence of different technical lignins and lignin cleavage products (LCP) on the curing behavior of the synthesized lignin-phenol-formaldehyde solutions (LPF solutions) and on the dimensional stability, moduli of rupture (MOR) and elasticity (MOE), accelerated weathering and fungal resistance of LVL made from treated beech wood veneers.

Four different LPF solutions in dimethyl sulfoxide (DMSO) were individually produced using three technical lignins (Indulin AT, BioChoice and Organosolv lignin) and LCP at a ratio of 60 wt. %: 40 wt. % (PF resin: technical lignin/LCP). Rotary cut beech (*Fagus sylvatica* L.) veneers were treated with the synthesized resins. Four of these modified veneers were bonded with a commercial PF adhesive to produce four-layer laminated veneer lumber (LVL). Curing characteristics using differential scanning calorimetry (DSC) showed a higher curing temperature for the LPF resins compared to the commercial PF resin. The mechanical and water-related properties of the LPF-modified LVL were quite similar or slightly better than those of PF-modified LVL. LVL specimens exposed to a white-rot fungus (*Trametes versicolor*) and a brown-rot fungus (*Coniophora puteana*) exhibited a very low mass loss and did not show significant differences between the LPF-modified and PF-modified samples except in one case: LVL made from veneers treated with Indulin AT exposed to the white-rot fungus suffered higher mass loss. Artificial weathering results showed a low resistance of the LVL specimens made from veneers treated with technical lignins; however, specimens treated with LCP and the reference PF resin displayed a higher resistance to artificial weathering. To conclude, our study shows that technical lignins and LCP can, to a certain extent, be used as a substitute for crude-oil based phenol in PF resins.

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Ionic liquid pre-treatment to reduce the elastic spring-back and set-recovery of surface densified Scots pine

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Keywords: wood compression, densification, wood modification, shape-memory effect

Although it is not a new research area in the field of wood science, in recent years, surface densification of wood became a subject of increasing popularity. It was shown that creating a layer of densified wood cells of a few millimetre thickness just beneath the surface can lead to an increase in hardness by approximately 100% (Gong *et al.* 2010, Laine *et al.* 2013).

One of the major obstacles regarding the wide-spread commercialisation of surface-densified wood products is the reduction of the elastic spring-back and set-recovery of the deformed wood cells in the context of an industrial process. The elastic spring-back can be greatly reduced by introducing a cooling stage before releasing the pressure at the end of the densification process (Neyses 2016). The set-recovery can be nearly eliminated by applying different methods, such as chemical modification or by thermal-hydro-mechanical treatments of the densified wood cells (Kutnar *et al.* 2015). A theory describing the underlying mechanisms of the elastic spring-back and the set-recovery was presented by Navi and Sandberg (2012) and further described by Navi and Pizzi (2014). It is suggested that the set-recovery occurs due to elastic deformation of cellulose, which is 'frozen' inside the plastically deformed matrix of lignin and hemicellulose. Plasticisation of the wood after it has been densified, e.g. through moisture, leads to a recovery of the elastic deformation. Hence, the set-recovery can be greatly reduced if plastic deformation of the cellulose can be achieved. This is, however, difficult to accomplish with the established methods of plasticising wood, such as the combination of moisture and heat, or treatment with gaseous ammonia.

In the past decade, several studies explored the possibility of plasticising cellulose by treatment with so-called ionic liquids (Swatloski *et al.* 2002, Kilpeläinen *et al.* 2007). Apart from being considered 'green solvents' of cellulose, ionic liquids are reported to have a positive effect on antifungal and antimicrobial activity, and on fire and UV degradation resistance. Ou *et al.* (2014) reported that ionic liquid treatment transforms wood into a thermoplastic material, without or only

little elastic deformation when put under load. This behaviour suggests that the elastic spring-back and the set-recovery can be largely reduced.

The objective of the present study was to evaluate the effect of ionic liquid pre-treatment on the elastic spring-back and set-recovery of surface-densified wood.

The study was carried out on Scots pine (*Pinus sylvestris* L.) specimens treated with a 20% weight solution of 1-Ethyl-3-methylimidazolium chloride, solved in ethanol. The specimens were soaked in the solution prior to being surface-densified on the sapwood side in a hot press at up to 250°C for 2 to 7 minutes.

As no cooling stage was applied before releasing the pressure, the elastic spring-back of the untreated control group was 50-60% (based on the compressed thickness). The ionic liquid treatment reduced the spring-back to less than 20%, similar to values obtained if a cooling stage would have been applied. Depending on the pressing time, the set-recovery could be reduced from about 90-100 % (control group) to 20-30%, where it appears that part of the remaining set-recovery can be attributed to inadvertent densification of earlywood regions in the untreated core of the specimens. The high pressing temperature led to thermal degradation of the densified wood cells, which could be an additional contribution to the reduction in set-recovery. Visual inspection and Brinell hardness tests suggest that the specimens treated with ionic liquids have a more pronounced density peak close to the surface in comparison to the untreated specimens.

The results of the present study indicate the potential of ionic liquids as a strong plasticiser of wood, reducing the elastic component of deformation of wood under load. In this way, the elastic spring-back and set-recovery are to a certain extent prevented from occurring in the first place. Further studies are needed to optimize the treatment and to gain a better understanding of the treatment mechanism.

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Humidity optimization considerations for thermal wood modification

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Keywords: pressurised heat treatment, wood moisture, thermal mass loss kinetics, uniformity, mechanical stress

Thermal wood modification (TWM) can be performed at any gas pressure in a heating compartment. Few studies have been attributed to the influence of a large variation of total pressure and the partial water vapour pressure on the thermal modification rate and resultant product material properties (Seborg *et al.* 1953; Ladner and Halmschlager 2002; Borrega and Kärenlampi 2008; Altgen *et al.* 2016), since most industrial processes are currently conducted at (near) atmospheric pressure. Previous work of the author gave evidence that the chemistry of thermal wood modification is not significantly affected by the choice of process, *i.e.* only the rate of modification is affected (Willems *et al.* 2013).

The current work is devoted to pressurised processes that have the unique ability to maintain finite wood moisture content (MC) levels during the thermal modification process. The local temperature T and the partial water vapour pressure p_v in wood determine the relative humidity (RH), controlling the local MC. In a process with a trapezium temperature profile with constant heating/cooling rates and an intermediate constant temperature stage, p_v can be (T -dependent) chosen to keep the RH constant (Willems 2009), preferably set at the product service RH. Taking a raw material MC in equilibrium with this RH, moisture movements and dimensional changes during the thermal modification process are then reduced to a minimum extent, practically eliminating product degrade by checks, deformation and internal stresses.

In saturated water vapour, in-process product dimensional changes can be optimally avoided, but the resultant material is significantly weakened by moisture softening and a lack of crosslinking between the hydrolysed wood polymers (Obataya and Higashihara 2017). Subsequent drying to service RH may then result in wood anatomical damage. In the opposite case of dry heating, the material will maximally shrink and subsequently become extensively crosslinked. Subsequent moistening to service RH may then result in problematic tensile stresses in the core of the boards. Moreover, the crosslinked wood polymers in the fully shrunken state render the material excessively brittle. In the intermediate water vapour pressure range between saturation and absolute dryness, wood can be crosslinked in a partially swollen state, leading to a less brittle material with preserved stiffness.

An increased total pressure at constant temperature above the thermal modification threshold has been shown to accelerate the rate of thermal modification (Seborg *et al.* 1953; Ladner *et al.* 2002; Borrega and Kärenlampi 2008). Altgen *et al.* (2016) and Willems *et al.* (2015) found that at elevated pressure, the modification rate becomes explicitly determined by pressure, independent of temperature. A practically important consequence of the explicit pressure dependence of the modification rate is an improved product uniformity, since pressure gradients will equalise faster than temperature gradients. The addition of a non-condensable gas to raise the total pressure in the reactor will aid the rate and uniformity of the heat treatment, although it then becomes more difficult to keep control over the partial water vapour pressure (hence MC), which is important for the mechanical properties of the product.

It is deduced that the general mechanical properties of modified wood by thermal treatment may be best preserved in a pressurised pure superheated steam environment.

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Thursday 14th September,
COST Action FP1407: 3rd meeting
“Wood modification research
& applications”

Session 2:

Characterization of modified wood

From 14:15 to 15:45

Chairs: J. Sandak, A. Rozanska

Exploring the hypothesis that limiting diffusion of fungal oxidants underlies decay resistance in acetylated wood

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Keywords: decay resistance, diffusion, free volume, acetylated wood

The mechanisms by which chemical modifications, specifically acetylation, improve the decay resistance of wood are a topic of active research. In the early stages of decay, fungi secrete low-molecular-weight oxidants or oxidant precursors. These oxidants diffuse through the wet wood cell wall and oxidize cell wall polymers, which enable the decay process to proceed. One hypothesis states that acetylation stops decay by dramatically inhibiting the diffusion of these oxidants through the cell wall (Zelinka *et al.* 2016). Data showing a drastic difference in Fe uptake from solution between control and acetylated wood appear to support this (Hoseinipour and Mai 2016).

It has been proposed that carbohydrate-rich domains in the cell wall (hemicellulose and non-crystalline cellulose) go through a glassy-rubbery transition upon water swelling and become the medium for ion transport (Jakes *et al.* 2013, Zelinka *et al.* 2016). Bulking treatments that reduce the free volume are hypothesized to prevent the carbohydrates from entering the rubbery phase, thus dramatically limiting diffusion inside the cell wall.

Figure 1 shows ICP measured Fe(III) uptake with ICP in pre-wetted loblolly pine wood acetylated to 0, 10, and 20 WPG after exposure to a large excess of solution mimicking the conditions of fungal brown rot: 5 mM oxalate, 0.1 mM Fe(III), and pH 3.9, with Fe(III) ·(oxalate)₃³⁻ as the dominant iron species. Fe uptake was measured between 1 and 79 hours of exposure. 10 samples, 2 mm longitudinal and weighing ~2 g, were removed at each time point, weighed wet, weighed dry, and then analysed for Fe content. Our original hypothesis predicted that acetylated wood would take up Fe(III) at a far lower rate and saturate at a lower concentration than control wood.

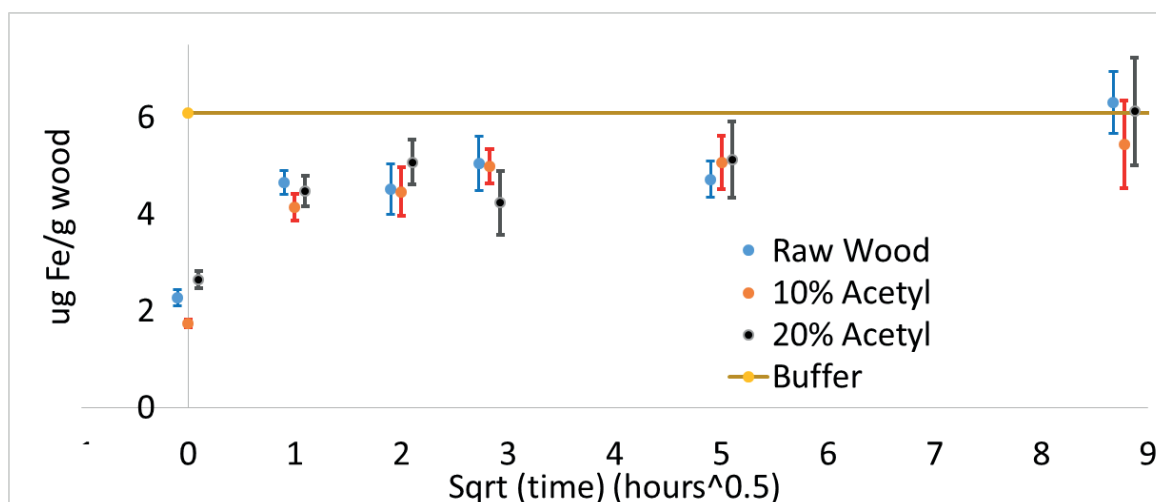


Figure 1: Fe content of pine wood by ICP after various times soaking in 6 $\mu\text{g Fe/g}$ oxalate buffer. Error bars represent 95% confidence interval. 79 hour data is statistically different ($p < 0.005$) from 1 hour data for all specimen types.

Using 5mM oxalate buffer, we observed that both control and acetylated wood contained the same amount of iron after 79 hours of soaking: 1.4x the solution concentration. The fact that pore volume was halved by acetylation to 20%, but carboxylate $[\text{COO}^-]$ in wood is unchanged suggests that the amount of Fe observed in the wood was determined by the ability of wood COO^- to compete with the very strongly chelating oxalate buffer for Fe. We conclude that the similar experiments of Hosseinipour and Mai (2016), where the wood adsorbed and retained 3180x as much Fe per ml as the surrounding solution, reflect the ability of wood COO^- and hydroxyl groups to compete effectively for Fe against their acetate buffer. Since acetylation interfered with Fe uptake from acetate buffer, it appears likely that OH groups have a role in Fe chelation when oxalate is absent.

In our experiments, diffusion was largely complete by the first data point, 1 hour. A full diffusion model must be run to establish the lowest diffusion rate of Fe(III) in acetylated wood consistent with the data.

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Dimensional stable and durable laminated veneer lumber (LVL) from European beech (*fagus sylvatica*) by impregnation with low molecular weight phenolic resin

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Keywords: Beech, durability, *Fagus sylvatica*, laminated veneer lumber, phenol formaldehyde

This work deals with the evaluation of Laminated Veneer Lumber (LVL) made from rotary cut beech (*Fagus sylvatica* L.) veneers, which were treated with low molecular weight alkaline phenolic resins with the aim of cell wall modification. Beech-LVL, and derived products from it, are already used and accepted for structural purposes, e.g. fabrication halls and multi-storey buildings, though they benefit from its higher mechanical properties compared to softwoods. On the other hand, susceptibility to biological decay and possibility of dimensional changes limit the applicability to dry climate conditions. Therefore, it was the aim of the BMBF funded research project DauerBuche, belonging to the BioEconomy Cluster e.V., to achieve durability against fungal decay, increase of weathering performance and dimensional stability. As modification of the cell wall is the best way to provide dimensional stability and durability to protect wood and wood products against these corrupting influences, a modification on basis of phenol formaldehyde was chosen. This seemed to be more attractive than other chemical treatments or thermal treatment, because earlier work showed a sufficient preservation of the mechanical properties, which is crucial for building applications. The dimensional stability was evaluated in a 4h-boiling test with a subsequent drying and evaluation for delamination. Volumetric swelling was found to be reduced by 57% at moderate Weight-Percent-Gain (WPG) for undensified boards. The durability of the LVL against white rot fungus *Trametes versicolor* has been tested for 16 weeks according to the standard ENV 12038. This test offered that the mass loss was decreased to less than 1% by the PF-treatment. The mechanical

properties modulus of rupture (MOR), modulus of elasticity (MOE) and impact bending strength have been assessed. It came out that for the undensified products the MOR could be preserved and MOE was significantly increased. The modification also enabled densification which resulted in further increase of MOR and MOE. Due to the loss of flexibility the absorbed energy at the impact bending test was considerably lower. Over all it was concluded that the WPG could be much lower than it was found in former studies for this type of modification. Especially for products that are wished to have high strength values and a resistance against fungal decay, but do not have to have the highest dimensional stability, 15% WPG was sufficient. The possibility of reducing the product volume for a building project because of its elevated mechanical strength qualifies the additional costs for the modification.

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Fixing tannin in wood: Characterization of the treated wood

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Keywords: wood preservatives, flavonoid, aldehyde, anhydride, weathering

The tree protects its wood physically through the bark, but also chemically by synthesizing substances which prevent degradation against biological (e.g. animal, insects, fungi) and radiative attacks (UV –rays). The idea we would like to pursue is simple increase the concentration of substances that the nature already synthesizes trying to fix them properly in wood without affecting their efficacy. It appears easy but it is not. Sensibility against leaching, fungal decay and in particular against weathering were observed (Tondi *et al.* 2012). During outdoor exposures, indeed, relatively fast discoloration and increased crack formation were observed. Two phenomena were identified to explain these weaknesses: i) the stiffness of the tannin polymers which do not resist the continuous swelling and shrinking cycles and ii) the polymerized tannin which do not maintain the typical radical-scavenging properties of the native tannin and therefore it is strongly degraded by the sun rays (Tondi *et al.* 2013). Recently, several studies were done by adding molecules which enhanced the elasticity of the formulation, but only contained improvements were registered against weathering (Tondi *et al.* 2017).

In this contribution new flavonoid co-polymers of formaldehyde, glyoxal, maleic anhydride, furfural and furfuryl alcohol are presented for their leaching resistance and their biocidal activity were investigated. In Fig.1 the effect of the hardening time and temperature on the situ-polymerization of tannin are presented.

It was observed that selecting tailored hardening parameters it is possible to achieve very satisfactory fixation of the polymer in wood. However, these formulations presented only limited biological properties against fungi and therefore contained amount of boric acid and copper sulphate were added. The results of the biological screening tests against *Coriolus versicolor* are presented in Fig.2 and they show high efficacy for all the formulations added of 1% boric acid and satisfactory results also for the tannin-hexamine and tannin-furfural formulations added of 3% copper sulphate.

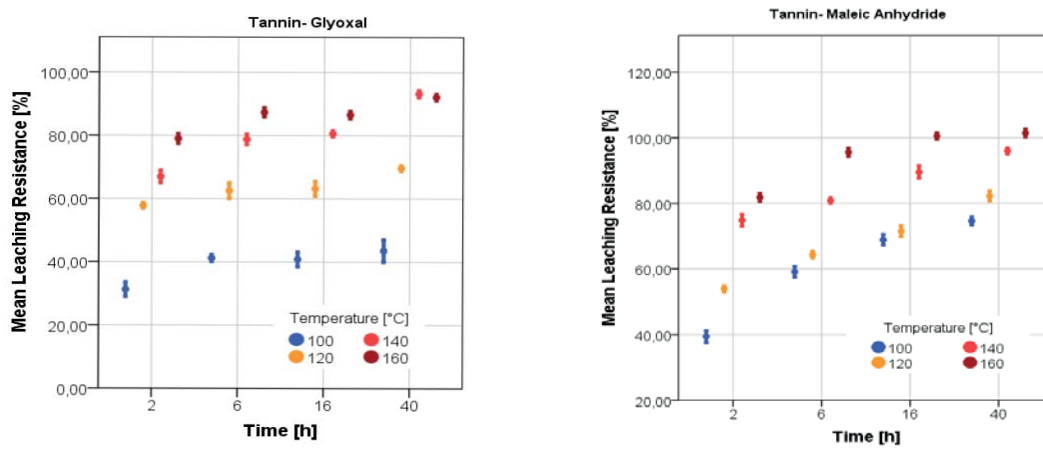


Figure 1: Time and temperature effect in the leaching resistance of tannin-based polymers.

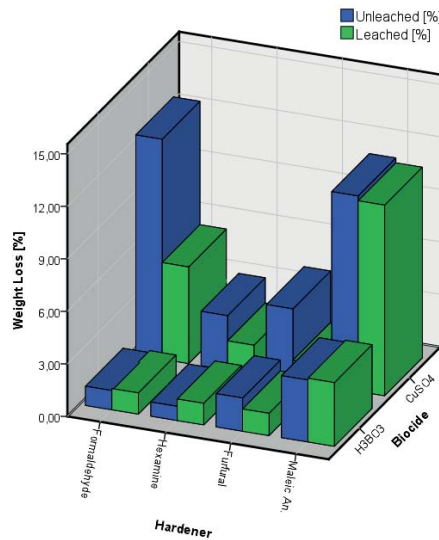


Figure 2: Weight loss of tannin-copolymers specimens added of different amount of biocide.

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Acknowledgments: The authors acknowledge the support of the Austrian ministry of Agriculture, Forestry, Environment and Water Management for the ERA-NET Wood-wisdom project “BioCoPol”.

Assessment of chemical fingerprint of modified wood

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Keywords: wood thermal treatment, spectroscopy, chemical fingerprint, xylograms

Thermal treatments include several alternative processes that differ in terms of intensity (temperature and duration), treatment atmosphere (vapour/nitrogen/vacuum), use of catalyst, and system configuration (open/close, wet/dry) (Hill 2006). Thermal modification of wood leads to several chemical reactions: dehydration, depolymerisation, degradation, thermo-oxidation. Those reactions take place with different rates depending on the modification process, wood species, process duration and treatment temperature. As a result, chemical composition and in consequence physical properties of wood treated in different processes vary significant.

Exact assessment of changes to particular chemical components is rather difficult, since a range of complex chemical reactions occur simultaneously while exposing wood to elevated temperatures. This manuscript proposes an original approach for visualization of chemical fingerprint of thermally modified wood (Sandak *et al.* 2016). Fourier transform near infrared spectroscopy (FT-NIR) was selected here as technique for fast and non-destructive screening of chemical composition. Selected NIR bands assigned to functional groups of woody polymers were extracted from the spectra and were used as “information hubs”, inspired by work of Tsenkova (2013) and her aquagrams concept. Eight European wood species (both softwood and hardwood) were thermally treated under vacuum (250 mbar) for 3 hours in a Thermovacuum processor. Additional details regarding the experimental plant, treatment conditions and applied schedule can be found in Sandak *et al.* (2015).

The new method for spectra visualization, called “xylograms” is capable of highlighting peculiarities in chemical changes to woody polymers due to the thermal treatment. Comparison of xylograms allows observation of kinetic and permits evaluation of thermal stability of investigated species and/or comparison of process parameters influence.

Deep understanding of chemical changes might be helpful for further optimization of thermal treatment procedures at industrial scale. Moreover, xylograms as a simple and illustrative method

might be suitable for visualization of other modification/degradation processes of wood as well as other materials.

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Acknowledgments:

Part of the work was conducted within the BIO4ever (RBSI14Y7Y4) project which is funded within a call SIR (Scientific Independence of young Researchers) by MIUR and the project TV4NEWOOD, Eco/12/333079 co-funded by Eco-Innovation Initiative of the European Union.

Wood modification method efficiency evaluation by cyclic hydrothermal treatment

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Keywords: acetic anhydride, acetylation, aspen wood, cyclic hydrothermal testing, rapeseed oil

The alterations in wood biopolymers network with modification decreases its swelling probability (Thybring, 2013). It is assumed that water sorption decreases due to wood hydroxyl group bulking by acetylation (Hill, 2006). The bulking of hydroxyl groups on cell wall surface theoretically might be performed with bulky molecules like fat's triglycerides, Impregnation with vegetable oils of soft-wood have been used to reduce the wood's ability to absorb moisture (Temiz *et al.*, 2013). According to Hyvönen *et al.* (2006), the effectiveness of a water repellent treatment is probably dependent on both the amount of deposit and its precise location within the treated wood.

Objective of presented research was to evaluate properties of acetylated, acetylated and impregnated with rapeseed oil, and jointly acetylated and impregnated woods in comparison with un-modified-reference by cyclic hydrothermal treatment (CHT). Each CHT cycle consists of wood swelling in hot water at 75 °C with following freezing at -20 °C) and drying at 103 °C. Duration of each cycle's stage was 3 days. Wood samples were weighed and measured after hot water treatment and drying. Dynamics of wood mass loss, water absorption, volume swelling, and shrinking during CHT were calculated on the base of weight and dimensions measurement data set.

The four different modification schedules were used for aspen (*Populus tremula* L.) wood modified samples:

- A) vacuuming 6.5 h, impregnation with acetic anhydride (AA) and acetylation at $(110\pm 10)^{\circ}\text{C}$, 24,5 h;
- B) vacuuming 2,5 h, impregnation with AA 14,5 h, acetylation at $(110\pm 10)^{\circ}\text{C}$, 8.3 h, then impregnation with rapeseed oil (RSO) at 120°C , 5,5h with simultaneous acetic acid distil-off;
- C) impregnation off wood with AA at 20°C , 6 h and impregnation with RSO at $(120\pm 10)^{\circ}\text{C}$, 16 h;
- D) vacuuming 5.5 h, and impregnation with RSO at $(100\pm 10)^{\circ}\text{C}$, 1.3 h.

Rapeseed oil, impregnated in wood, was cured at 103°C till hardening.

Wood substances (cellulose, hemicelluloses and partly lignin) hydroxyl groups are assumed to determine water sorption in to the wood cell wall mostly (Skaar, 1988). Water has lesser molecule volume than acetic anhydride, and it may penetrate into wood substance and interact with not acetylated hydroxyl groups that result as wood swelling and dimensional changes and finally lead to wood collapse by cracking.

Modification efficiency on wood durability during CHT in comparison with natural wood were: 315%, 163%, 122%, and 244% for A, B, C and D schedules accordingly based on multi-parameter evaluation of wood mass loss, water absorption, volume swelling, and shrinking dynamics.

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Acknowledgments:

The authors express their thanks to National Research Programme Forest and Earth Entrails Resources: Research and Sustainable Utilization – New Products and Technologies (ResProd) 2014.-2017 for financial support.



Machinability of thermally modified wood assessed with a new objective method

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Keywords: thermally modified, wood, machining, surface, quality

The surface machined by peripheral milling can be evaluated by different means, where visual assessment or surface roughness measurement are most frequently applied. A new method, alternative to the state of the art methodologies (Goli and Sandak 2016), is proposed here to provide objective evaluation of thermally modified and unmodified wood by means of an automatic system. The formation of defects is stimulated by a purposely-designed cylindrical sample. The surface characterization is done on a prototype multi-sensor platform. The platform is composed of a laser triangulation system that reconstructs the 3D surface topography and a video camera recording the image of the same surface while the sample is rotated around its central axis. The surface is automatically reconstructed by a specially designed software that allows quality assessment and the detection of specific defects. The method is relatively rapid and allows an easy comparison of different cutting conditions useful for determination of optimal configurations. The scanner was successfully used to assess the machinability of thermally modified wood as well as of

untreated control samples. The effect of sharp and dull tool on thermally modified and unmodified wood was also assessed (Sandak *et al.* 2017).

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Acknowledgments:

Authors would like to acknowledge COST Actions FP0904, FP1303 and FP1407 for creating research networks and providing inspirations to plan these studies. Part of the work was conducted within the BIO4ever (RBSI14Y7Y4) project which is funded within a call SIR (Scientific Independence of young Researchers) by MIUR. Special thanks to ALAC srl (Porto Recanati, Italy) for assistance in the preparation of experimental samples.

Microscopic investigation of acetylated hornbeam and densified beech

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Keywords: acetylation, densification, microscopy, wood modification

Wood properties can be improved by various methods of wood modification. This research focuses on two well-known methods – acetylation and densification. The main reason for wood acetylation is significant improvement of dimensional stability and durability (Hill 2006); depending on wood species and process settings, acetylation can increase density, strength and hardness too (Fodor *et al.* 2017). This process is suitable for hornbeam, which has excellent service life indoors but is non-durable outdoors. Wood densification, which improves mainly strength and hardness (Navi and Sandberg 2012), is suitable for beech wood. Although its density is not low, it is easy to densify. Beech wood is the most common hardwood species in Central Europe and the process can enhance utilisation of this renewable material. Investigation of these materials with altered properties requires new approach. Several methods of sample preparation for microscopy are evaluated in this research.

Hornbeam (*Carpinus betulus* L.) used for this research was acetylated at Accsys Technologies under industrial conditions using standard process parameters (Fodor *et al.* 2017). Acetylated specimens after durability test and reference samples were included too. Sections were cut with sledge microtome Leica SM2000R after softening with water or acetone in the case of acetylated samples. The sections were stained with safranin and astra blue or potassium permanganate (1% solution) for 2 minutes and embedded in Canada balsam.

Beech (*Fagus sylvatica* L.) wood was plasticized in steam, compressed and fixed between stainless steel plates. Hydrothermal stabilization of selected specimens was carried out in saturated steam at a temperature of 90 °C for 10 days based on previous results (Rousek and Horáček 2016). Finally, the specimens were dried while kept between the plates. Sections were cut by the sledge microtome after wetting the cross section with water. Microtomed surface was prepared by the same method but compression set recovery was restrained by a special stainless-steel clamp.

Results showed a different behaviour of modified wood compared to the reference. Acetylated wood cannot be stained with safranin and astra blue (Fig. 1a) due to chemical changes of all wood components. New deposits were found in the cell lumens. Although acetylated hornbeam had no

sign of fungal decay and it had less than 1% of mass loss, some hyphae can be observed in the microscopic sections (Fig. 1a). This was experienced by other researchers too (Mohebbi and Militz 2010). Potassium permanganate stained brown both densified beech and acetylated hornbeam (Fig. 1b). Middle lamella got darker in both cases. Densified wood recovered structure during cutting sections depending on the post-treatment time. Stabilized wood showed closed lumens of fibres and partially open vessels and parenchyma cells (Fig. 1b). Reflected light microscopy revealed cracks in radially compressed specimens (Fig. 1c). The method is less suitable for single cells observation, because the contrast is lower due to light scattering in cell walls. Scanning electron microscopy should be used instead.

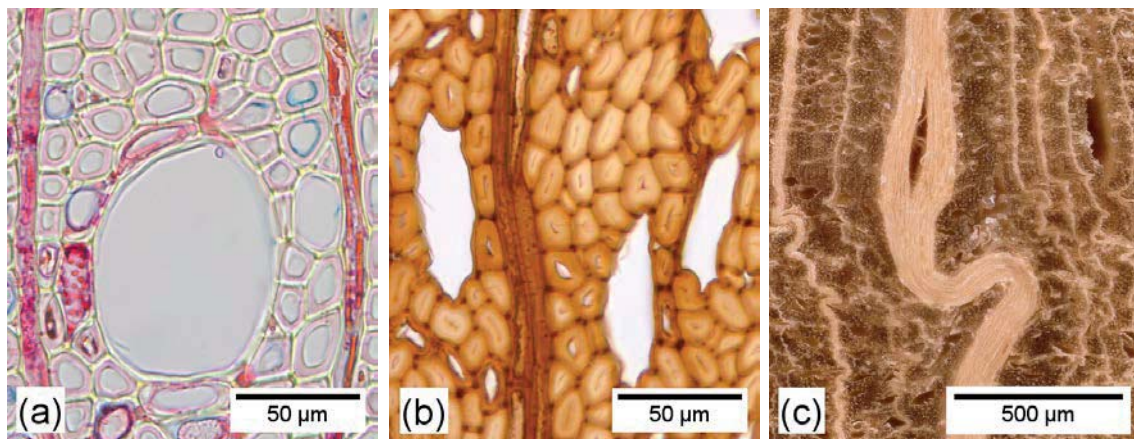


Figure 1: Bright field microscopy of acetylated hornbeam (a) (cross section). Brown deposits in cell lumens are stained red with safranin, hyphae of brown rot *Coniophora puteana* are stained with astra blue. Bright field microscopy (b) and reflected light microscopy (c) of beech wood densified in tangential direction (b) and radial direction (c) (cross section). The sample (b) is stained brown with potassium permanganate.

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Acknowledgments:

This work was supported by the ÚNKP-16-3-1 New National Excellence Program of the Ministry of Human Capacities (Hungary) and the Specific University Research Fund of the FFWT Mendel University in Brno (LDF_VP_2017040).

Thursday 14th September,
COST Action FP1407: 3rd meeting
“Wood modification research and
applications”

Session 3:

**Innovative use of wood modification
processes**

From 16:15 to 17:30

Chairs: L. Ross Gobakken, W. Grigsby

Modification of the Particleboard Core Layer by Non-wood Lignocellulosic Raw Materials

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Keywords: particleboard, hemp shives, cereal straw, mechanical properties

The aim of the research was to utilize non-wood lignocellulosic raw materials as potential substituents of wooden particle shares in particleboard core layer. The idea of higher utilization of e.g. hemp shives and cereal straw is based on the reduced availability of real wood raw material, on its continuous prices increase and on considerable amounts of lignocellulosic residues suitable for composites production worldwide. Particleboard core layer modification was made from the reason to incorporate "cheaper" material into the particleboard structure. Surface particles were made from wood with the aim not to change the particleboard surface properties or appearance.

For the laboratory production of modified particleboard core layer the similar technological procedures were used, as are utilized for standard particleboard production in production lines. Core particles were produced as the combination of wood and non-wood lignocellulosic materials (hemp shives or cereal straw) in the following ratios: wood from 100% decreased to 70% by the step of 5%, non-wood lignocellulosic materials from 0 increased to 30% by the step of 5% (between 10 – 20% of lignocellulosic materials was the step 1%). Mat forming was done manually. The average density of modified particleboard was $625 \pm 10 \text{ kg.m}^{-3}$. Moisture content of all materials produced during the time of testing was $10 \pm 2\%$.

Results of bending strength, modulus of elasticity and tensile strength perpendicular to plane of the board for modified particleboard by hemp shives or cereal straw as well as for unmodified particleboard samples are shown in Table 1.

Experimental research demonstrated that:

1. The reasonable values of bending strength are met for almost all ratios of hemp shives and cereal straw except too high concentration of cereal straw in particleboard. Bending strength slightly decreases with the ratio of non-wood lignocellulosic materials up to 20% which may be due to the fact that non-wood lignocellulosic particles are more flexible than wood particles.
2. The average values for modulus of elasticity in bending are 1 600 - 2 600 MPa depending on particleboard type. This value is met for all ratios of shives and cereal straw. Based on regression analysis it was confirmed the statistically significant degree of correlation

between modulus of elasticity in bending and shives or cereal straw ratio addition in particleboard on the significance level $\alpha = 0.05\%$.

Table 1: Selected mechanical properties of the modified particleboard by hemp shives and cereal straw

Ratio non-wood lignocellulosics to wood particles (%)	Bending strength		Modulus of elasticity in bending		Tensile strength perpendicular to plane	
	hemp shives (MPa)	cereal straw (MPa)	hemp shives (MPa)	cereal straw (MPa)	hemp shives (MPa)	cereal straw (MPa)
0 – 100	14.4		3 600		0.64	
5 – 95	17.8	16.6	3 700	2 800	0.65	0.41
10 – 90	16.4	17.8	3 400	2 750	0.62	0.38
15 – 85	16.6	17.6	3 300	2 700	0.61	0.32
18 - 82	17.2	17.1	3 500	2 800	0.61	0.26
20 – 80	18.0	17.4	3 600	2 850	0.60	0.24
25 – 75	16.8	13.0	3 450	2 800	0.58	0.23
30 – 70	14.8	8.8	3 100	2 200	0.52	0.18

- Typical value of tensile strength perpendicular to plane of the board for particleboard with thickness of 16 mm according to the standard EN 319:1995 is specified at the level of 0.35 - 0.45 MPa depending on particleboard type. This minimum value was complied with all shives ratios. Decrease of tensile strength with the increasing share of shives is probably due to the different shives geometry compared with wood chips. For cereal straw, the minimum value complied with the ratio of 5 % only which excludes the use of particleboard with higher ratio of cereal straw in some applications.

There is a statistical dependence between the modifications of particleboard core layer by hemp shives and cereal straw and its physical and mechanical properties. Non-wood lignocellulosic raw materials may not be used for energy purposes only, but they may have their real use in the composites production. It is possible to produce particleboard with the substitution of wood particles in the core layer with maximum 20 – 30% of hemp shives and with maximum 10 – 15% of cereal straw without the negative change of particleboard properties or without change of the technical and technological production parameters.

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Acknowledgments

The research described in the paper presented was supported by the Slovak Research and Development Agency under the contract No. APVV-14-0506 and APVV-0200-12. The author would like to thank the agency for the support of this research.

The use of novel modified wood fiber for manufacturing structural wood plastic composite timber for an innovative marine application

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Keywords: wood plastic composites, styrene maleic anhydride copolymer, modified wood fiber, mechanical properties, durability

Wood plastic composites (WPCs) have found use in a myriad of building material applications including decking and railings, siding, doors and windows, roof shingles, etc. Outdoor durability of WPCs is a major focus of research and development, and application of WPCs in a marine environment provides a particular challenge because of possible mechanical property loss and durability issues. Marine aquaculture structures operate in one of the most demanding of environments in which most materials do not survive for long. University of Maine researchers have developed a patented WPC technology using an engineering thermoplastic (styrene maleic anhydride (SMA) copolymer) that is stronger, stiffer and less prone to creep than current WPC materials. Along with commercial research partners (Stora Enso and Innovasea), we are evaluating the use of this SMA-WPC technology with a novel modified wood fiber developed by Stora Enso in Innovasea's Aquapod fish cages. The novel modified wood fiber provides a WPC feedstock with improved dimensional stability, lower water absorption, and improved durability which are attractive for marine (fish cage) applications. The benefit of using the SMA-WPC material technology includes: improved structural performance, lower susceptibility to heat damage, and ability to easily apply marine coatings. We will discuss the progress on the production and testing of novel modified wood WPCs in fish cage construction and report on the mechanical property and durability findings.

Sawdust-based activated carbon for wastewater treatment from textile industry

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Keywords: sawdust, chemical treatment, adsorption, isotherms

A simultaneous chemical and heat treatments was used to Sawdust as a lignocellulosic solid waste for surface modification in order to prepare activated carbons. These later were used as an adsorbent for the removal of basic green (textile dye) from aqueous solution. Impregnation in 20% KOH (SD-AcK), and $(\text{NH}_4)_2\text{S}_2\text{O}_8$ (SD-AcP) solutions then pyrolysis at 600°C for 3 hours were found to enhance the adsorption capacity of the pollutant significantly relative to its inactivated state. The batch adsorption experiments resulted in a maximum adsorption capacity determined from Langmuir models of up to 434.78 mg/g, 238.10 and 200.0 for SD-AcK, SD-AcP and Merck respectively as shown in Table.1. Parameters influencing adsorption capacity such as contact time, adsorbent dosage, pH and temperature were studied. FT-IR analyses and iodine number determination were also performed to characterize the prepared adsorbents. Adsorption kinetics was found to comply with the pseudo second order with a good correlation factor ($R^2 = 0.99$) with intraparticle diffusion as the rate determining steps. Thermodynamic analysis and temperature effects of the process confirm that the adsorption reaction was spontaneous ($\Delta G^\circ < 0$) and endothermic ($\Delta H^\circ > 0$). This study showed that sawdust as a waste could prove to be a very useful in removing toxic substances from the environment.

Table 1: Isotherm constants and iodine number for basic green adsorption on *Sawdust* based carbons and Merck AC.

Isotherm type		Adsorbents		
		SD-AcK	SD-AcP	Merck AC
Langmuir	b [mg g ⁻¹]	434,78	238,09	200.00
	K [L g ⁻¹]	0,33	0,012	0.250
	R^2	0,998	0.976	0.996
Freundlich	k_f [L g ⁻¹]	190,57	40.24	118.27
	n	4.78	3.745	12.95
	R^2	0.855	0.957	0.827
Iodine number [mg/g]		491	437	826



Figure 1: Images of (a) Sawdust; (b) Powdered sawdust; (c) Sawdust-based AC.

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Sustainable use of *eucalyptus globulus* residues for polyurethane foam production

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Keywords: bark, branches, *Eucalyptus globulus*, liquefaction, polyurethane foams

Bark and branches produced along *Eucalyptus globulus* life cycle are not used in the paper and pulp industry and are therefore considered as residues. Traditionally, these forest residues are used in the production of energy, directly in furnaces or for pellets production. However, selling these products has revealed an increasing difficulty, so it is urgent to create new options. These forest residues are renewable resources that converted to liquid mixtures can later be used in the production of high value-added products replacing petroleum products for various applications. The liquefied compounds can be separated and used in the production of various chemical compounds or be re-condensed through conventional polymerization techniques to produce adhesives, plastics or polymers. This work focuses on the production and characterization of polyurethane foams (PUs), obtained from *Eucalyptus globulus* forest residues. This work was done in the frame of project VALRESF (PROJ/CI&DETS/CGD/0014- Valorisation of forest residues through liquefaction). This project aims to study low-temperature liquefaction processes that are applied to forest residues to explore its potential for liquid mixtures with specific and functional chemical characteristics, that will lead to the development and testing of various value-added products.

Liquefaction was held on a double shirt reactor heated by hot oil with automatic stirrer at ± 70 rpm using polyalcohols as solvents (glycerol, ethyleneglycol, PEG 400 or a mixture of them) and sulfuric acid or potassium hydroxide as catalysts. In order to achieve the best liquefaction yield, basic and acid catalysts, temperatures from 140°C to 200°C and times ranging from 30-120 min were tested. The best liquefaction yields were used to produce polyurethane foams. The chosen polyol was

obtained from *Eucalyptus globulus* branches, liquefied during 120 minutes at 180° C, using sulfuric acid as catalyst and a mixture of glycerol: ethyleneglycol (1:9) as solvents. Acid number, OH and viscosity index were determined for the polyol used to produce PU foams. The polyol was mixed with a catalyst (Polycat 34), a blowing agent (water) and a surfactant (Tegostab B 8404). This mixture was stirred for about 30 s at 750 rpm, in IKA Ost Basic mixer and then polymeric isocyanate MDI (MDI M229 Voranate) was added. Afterwards the mixture was stirred again for a few seconds at 750 rpm until the chemical reaction started. Physical and mechanical properties (density, compressive modulus and compressive strength) for the PUFs produced were tested.

Results showed that the best liquefaction yields were achieved with acid medium and that temperatures between 160°C-180°C and time between 60-120 min gave the best results for both bark and branches. The hydroxyl number of the liquefied material was 960 mg KOH g-1, acid number 22.6 mg KOH g-1 with a viscosity of around 2 Pa.s (Table 1).

Table 1: Hydroxyl and acid numbers and the viscosity of the polyol produced.

Hydroxyl number [mg KOH g-1]	Acid number [mg KOH g-1]	Viscosity [Pa.s]
960 ± 200	22.6 ± 0.1	2.03

Overall higher percentage of Isocyanate leads to better mechanical properties although only to a certain percentage. Higher percentage of catalyser or blowing agent results in a decrease of density and of compressive strength. There was no clear relation between changes in surfactant percentage and density and compressive strength of the produced polyurethane foams.

The density of the foams ranged between 25.5-67.8 g/dm³ which is similar to most commercial foams, however mechanical properties are still far from those of common rigid polyurethane foams. Compressive modulus and strength ranged between 0.03-0.30 MPa and 1.7-16.0 kPa much lower than common rigid polyurethane foams. The best mechanical properties were achieved with 3 mg of polyol, 14.2 mg of isocyanate, 0.3 mg of catalyst, 0.135 mg surfactant and 0.035 mg of blowing agent (Table 2).

Table 2: Density, compressive modulus, compressive strength and R NCO/OH index of the polyol.

Sample	Density [g/dm ³]	Compressive modulus [MPa]	Compressive strength [kPa]	RNCO/OH
E21	64.7	0.30	16.0	2.2

Overall this study has shown that liquefaction is an efficient process to liquefy both bark and branches and that polyurethane foams can be obtained with the resulting polyol. New studies are underway in order to improve foam properties to match commercial rigid or elastic PUs foams

Acknowledgments:

This work is financed by national funds through FCT - Fundação para a Ciência e Tecnologia, I.P., under the project UID/Multi/04016/2016. Furthermore we would like to thank the Instituto Politécnico de Viseu and CI&DETS for their support.

MechanoChemo modification of cellulose powders

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Keywords: modified cellulose, mechanochemo reaction, composite, PLA

Cellulose fiber and nanocellulose have the potential for a variety of new applications. However, difficulties in achieving a proper compatibility between cellulosic fibers and polymer matrices, dispersion during processing, and processing permissible temperature is needed to realize this potential. In the report presented here, we hypothesize that wood pulps, composed primarily of cellulose, can be subjected to mechanochemical activation with selected chemicals to prepare surface activated and/or functionalized cellulose powders for reinforcements. Mechanochemical activation and modification in a completely solvent-free environment was carried out in a planetary ball mill. Largely amorphous cellulose particles, surface modified cellulose derivatives, and their reinforced polymer composites were prepared and investigated.

The ball milling time alters the particle size, crystallinity, and specific surface area of the milled cellulose particles (Fig. 1). The particle size was measured by a laser scattering particle size analyzer. The crystal structure was investigated by X-ray diffraction(XRD). Morphology characterization was conducted by scanning electron microscopy(SEM).

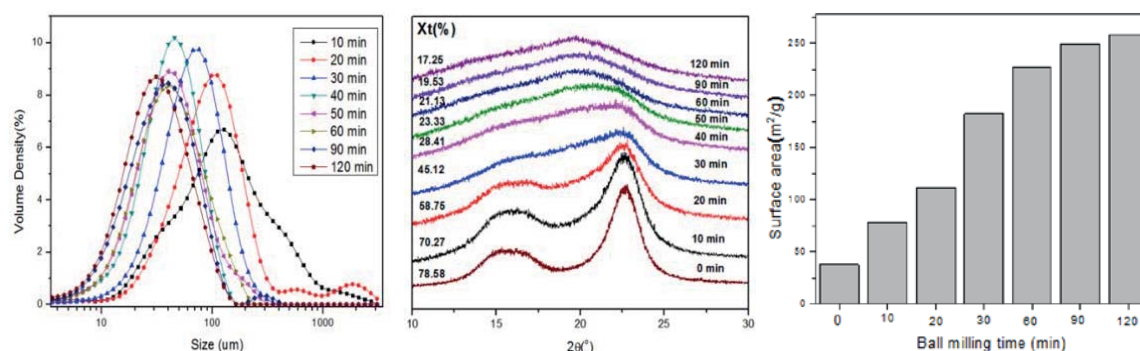


Figure 1: Effect of different ball milling time on the particle size distribution, crystallinity and surface area

Long chain fatty acid can greatly improve water repellency and their amphiphilic nature can facilitate mixture with non-polar polymers. However, the steric hindrance of the fatty chain is a disadvantage for reaction in the solid-state and mixed anhydrides are asymmetric and unstable. Therefore, mixed anhydride of acetic anhydride and oleic acid were combined to obtain two

different PK values moieties of the asymmetric molecule and therefore improve reactivity. The mixed anhydrides were ball milling with the activated cellulose powder for 90 min. The resulting modified powders demonstrated a contact angle with water of 72-deg compared to the untreated powders of 41-deg when measured with a sisal drop method. When the powders were extruded with polypropylene at a mass percent of 10%, the resulting blend demonstrated a lower viscosity during compounding. The resulting composite was used to produce injection molded tension samples. The modified cellulose powders demonstrated comparable reinforcing ability compared to the unmodified powders while resulting in significant improvements in ductility (Fig 2).

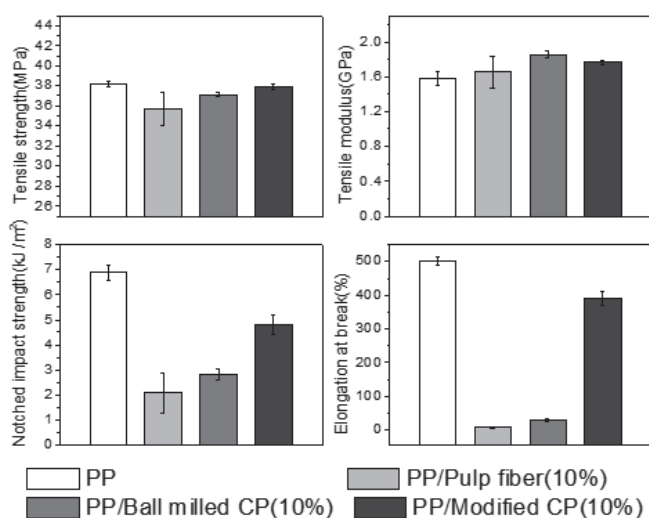


Figure 2: The mechanical properties of the modified CP/PP composite

Finally, co-milling with sodium boride was used for reducing the aldehyde groups produced when cleaving cellulose chains. After washing with DI water, this modification proved extremely effective at increasing thermal stability as shown in Fig 3.

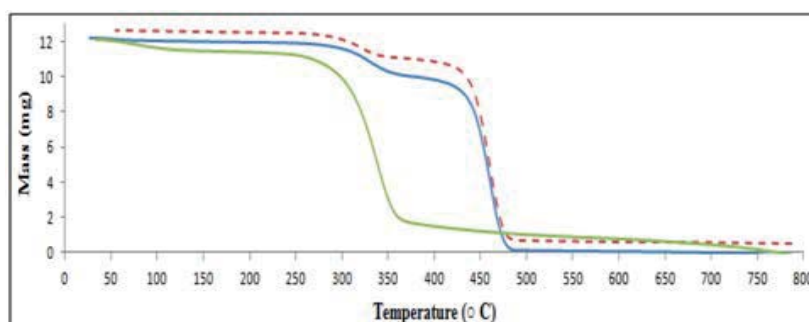


Figure 3: TGA curves for cellulose powders (CP) (bottom), modified CP (middle), modified CP/PP blend (top).

Acknowledgments:

This material is based upon work supported by National Science Foundation- Center for Bioplastics and Biocomposites (NSF-CB2 IUCRC) under Grant No. 1439732. The authors would like to acknowledge the guidance of Alper Kizilas of Ford Motor Corp, Yucheng Peng of Berry Plastics, and Kevin Lewandowski of 3M.

Using pre-stressed or non-pre-stressed curved members of concentrically composited laminated timber in structures of non-controllable environment

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Keywords: concentrically composited laminated timber, carpentry joint

The concept of the presentation belongs to the topic of integration, dissemination and exploitation.

Main features

The biggest problem in civil engineering of timber as anisotropic material and very suggestible to moisture is expansion or shrinkage of timber members because of steadily changing MC in it generating extra inside forces what are very difficult to take into consideration if the structure itself is not very simple. It especially applies to the structures as long span timber bridges, towers, tall timber buildings etc. standing in open space. To avoid exceeding MC in timber structures and its non-controllable expansion it can be covered or treated with other materials. However, it can't guarantee the exact MC never as it has been projected and calculated at the first so it need additionally some other solutions.

Concentrically composited laminated timber (CCLT)

One of them could be introducing of new type of concentrically composited laminated timber (CCLT). As the cross-sections of lamellae are very small the odds of wood industry of different species of timber with various physical properties could use for producing lamellae (Fig. 1).



Figure 1: CCLT lath (ash, aspen, birch, ash; *from inside to outside*), specimen S 01 KK2.

By gradually displacing the square-tubes of lamellae upwards starting from the inside layer, the tenon and mortise would be generated during the gluing process to connect the laths longitudinally on the building site without additional gluing as a new type of carpentry joint. Such type of

technology makes the product very strong but at the same time very flexible. Some of the test results of different experiments (bending, compressing etc.) shown in Table 1 (Nurme *et al.* 2017).

Table 1: Mean results of bending tests.

Specimen	Description [from inside to outside]	Bending Stress [MPa]	Elastic Modulus [MPa]
V 02 KK1 ^a	Solid spruce	45,92	9777
<i>Composited, not shifted</i>			
V 03 KK1	Oak, aspen, birch, oak	82,35	10847
V 03 KK2	Oak, alder, oak, ash	117,30	14699
V 03 KK3	Ash, aspen, birch, ash	123,65	15538
V 03 KK5	Birch, aspen, alder, birch	78,27	13193
<i>Composited, shifted</i>			
S 01 KK1	Oak, aspen, ash, oak	96,52	14082
S 01 KK2	Ash, aspen, birch, ash	128,47 ^b	16565
S 01 KK3	Ash, alder, birch, ash	113,50	15667
S 01 KK4	Birch, aspen, spruce, birch	86,63	13396

^aTo compare as ordinary construction timber, ^bMax bending stress 137,27 MPa

Results and Conclusions

In some cases such as the long span timber bridge (Fig. 2) where we can't guarantee the designed shape or it is danger to have plastic deformations it can used curved, two curved or twisted CCLT beams or posts what act as leaf springs. Some members can be mounted as pre-stressed to have preventive affect to keep the shape under the carrying loads. A special type of pre-stressed two-curved bridge deck used of massive timber balks what take part in stiffing all structure both as vertically and horizontally.

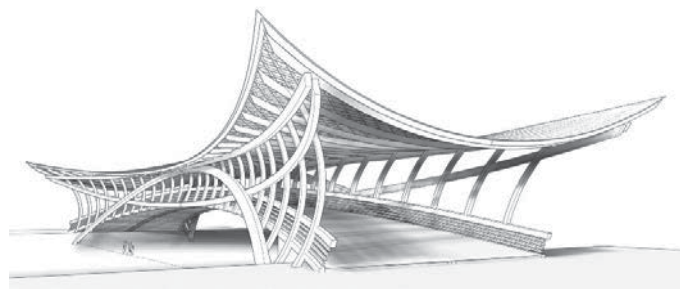


Figure 2: Expansion of Leonardo's Bridge (span 67,14m; Author's design).

This difficult structure and its details can be modelled and analysed using CAD/CAM/FEM-software and produced with CNC-technologies which was impossible only a few decades earlier.

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Acknowledgments: patent application P20170011 from 28th of February 2017 submitted for CCLT.

Friday 15th September,
COST Action FP1407: 3rd meeting
“Wood modification research
& applications”

DAY 2

STSM session

From 9:00 to 10:10

Chair: M. Schwarzkopf

Micro-distribution of Epoxidized Oils in Wood

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Keywords: epoxidized oils, wood modification, FTIR, SEM

Vegetable oils do not chemically bond with wood structure, it only fills in the cavities in wood structure and stands as a barrier to prevent water absorption. High oil absorption level requires for good protection in ground use of oil treated wood. High retention of oils (400-600 kg/m³) causes the process impractical and non-economic (Temiz *et al.* 2008). However, this problem can be solved using modified oils, epoxidation of double bonds at the fatty acid part of triglyceride and converted into oxirane rings for low oil absorption (Chen *et al.* 2002).

In this study, soybean (SO) and linseed oils (LO) having very high amount of unsaturated fatty acid were epoxidized to improve the bonding ability between oil and wood components and reduce oil retention (80-270 kg/m³). The epoxidized linseed oil (ELO), epoxidized soybean oil (ESO) and unepoxidized oils were subjected to FTIR analyses to reveal differences in chemical structures and then the distribution of these oils in wood structure has been examined.

Microscopy Observation

SEM was used determined to distribution of oils in wood. Samples were cut using a sliding microtome (ca. 15 µm) and dried overnight at 30°C. Then mounted on stubs with double-sided tape and coated with gold. SEM micrographs (in transverse section) of treated wood were shown in Figure 1.

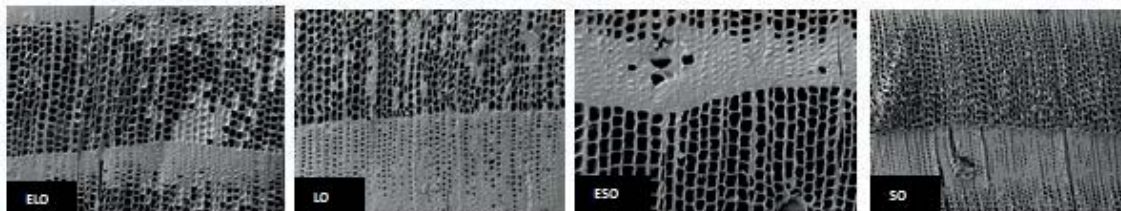


Figure 1: SEM micrographs of treated wood (in transverse section)

According to SEM observation, oils are mainly shown in the cell lumens especially late wood and rarely early wood tracheids.

FTIR Spectrum

The presence of epoxy group and double bonds in oils (linseed, soybean, epoxidized linseed, epoxidized soybean oils) were determined by using Bruker Tensor 37 Spectrophotometer in the range of 4000–400 cm^{-1} , with a 4 cm^{-1} resolution over 32 scans, using Diamond ATR. Figure 2 shows FTIR spectrum for oils.

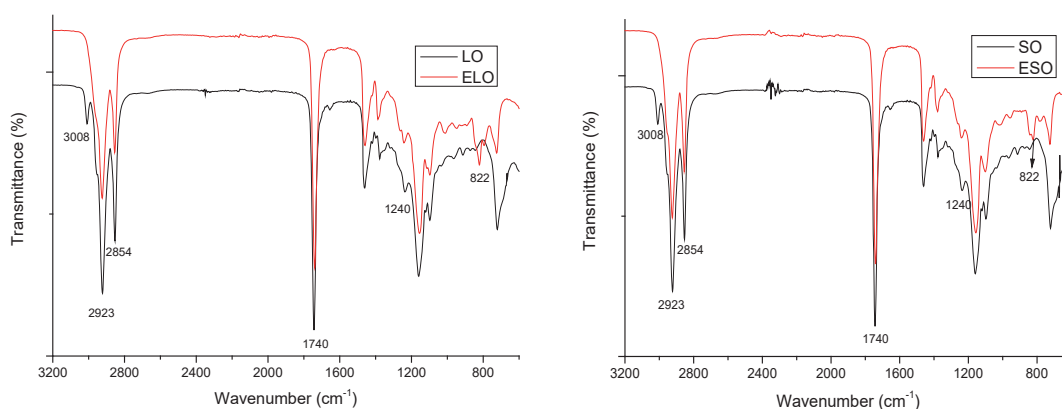


Figure 2: FTIR spectrum of oils

When the obtained FTIR results are analysed, it is seen that 822 cm^{-1} peak appeared in both ELO and ESO. These peaks show us presence of epoxy rings and this is important to show that the epoxidation process is successful (Jebrane *et al.* 2015). 1740 cm^{-1} pikes show carbonyl groups and there is no difference in carbonyl groups after epoxidation process. Peaks between 2855 and 2928 cm^{-1} indicate bonds between C-H and no change is observed in these peaks.

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Acknowledgments:

The authors would like to acknowledge the Scientific and Technological Research Council of Turkey (TUBITAK), for financially supported by TUBITAK 2214/A and TUBITAK 1001 project (Project Code: 114O265).

Enhancement of coatings for wooden claddings via plasma pre-treatment and environmental impact

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Keywords: Dielectric barrier discharge plasma, primer, Norway spruce wood, top coat, wood surface finishing

Plasma treatments have been indicated as environmentally friendly process for modification of intrinsic properties and functionalities of wood and wood based composites (Demirkir *et al.* 2014). In many investigations it was shown that plasma treatment increase surface free energy of wood and therefore increase its wettability (Rehn *et al.* 2003, Rehn and Viöl 2003). On such modified substrates, additional coatings can be applied.

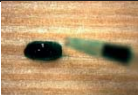






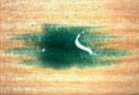


In this research, we quantified the impact of the plasma treatments on contact angle of a liquid coating and on some properties of a cured finish. For our research, Norway spruce wood was chosen. Plasma pre-treatments were carried out prior to the application of the surface coating system. As a coating, we applied a commercial water-borne two-step finishing system. It was selected as an example for applications on claddings in exterior exposure. Three different systems were tested.

Norway spruce (*Picea abies* (L.) Karst.) lamellae, with radial orientation and of the dimensions of (300 × 100 × 3) mm³ were prepared. Before subsequent experiments, they were stored and conditioned for 7 days in a standard climate with relative humidity of 65 % and at 20 °C. The coatings used were the commercial primer and the top coat (Helios d.o.o.). Wood surface plasma treatments were performed using dielectric barrier discharge (DBD) setup, in air at atmospheric pressure. Plasma treatments were carried out after sanding of sample surfaces or prior to the application of the coatings, respectively. Electric power consumption by a high-voltage generator and for plasma generator movement was directly measured on the electric network output. Sessile drop contact angle measurements were performed on untreated and treated surfaces, using distilled water, primer and top coat. After application of a droplet with a volume of 5 µL, photos of drops were taken after 1, 2, 5, 10, 20, 30 and 45 seconds. The spreading rate of droplets on wood surfaces was observed by application of 5 µL red- and green-coloured water. To assess chemical changes after the plasma treatment, in the plasma-generated layer, FT-IR spectroscopy was performed.

Reconstruction of a three-dimensional sample structure was obtained from sets of images (optical sectioning) at different depths within a sample surface by laser scanning confocal microscope. The adhesion of the coating systems on both treated and untreated specimens was evaluated by pull-off and cross cut tests on coating films covering the substrate.

The total electrical power, needed for generation of plasma on the sample surface and for the movement of plasma generator along the specimen, was in average 198.11 W. When the coloured water drop was deposited on a wood surface, a linear expansion along the grain occurred. The treated samples have larger areas covered with water drops compared to the untreated samples (Table 1).

Table 1: Time-dependent evolutions of green coloured water droplet areas on untreated and plasma treated wood surfaces

Type of samples	Time after dyed liquid deposition				
	0 s ^a	1 s ^a	2 s ^a	3 s ^a	24 h
Untreated					
Treated 3 mm/s					

^aPhotographs taken by microscope under 8-fold magnification

In general, the wettability of wood surfaces, on the radial cut, was improved by plasma treatment. Distilled water, primer and top coat droplets formed much lower contact angles on treated surface than on untreated wood surface. Compared to the untreated surface, after plasma-treatment the surface became smoother (Fig. 1). Finally, pull-off strength and cross cut tests showed that plasma treatment of a substrate increased adhesion of the primer coating, while the difference between the adhesion of top coat films on untreated and on treated samples was not so obvious.

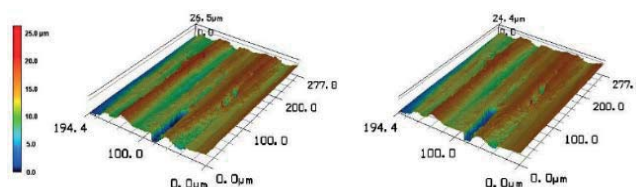


Figure 1: 3D profile of the sample surface segment before (left) and after (right) plasma treatment.

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Acknowledgments: The financial support of Jure Žigon’s short term scientific visit (STSM) to Clausthal University of Technology, by the COST FP1407 Action is greatly acknowledged. Financial support of the Slovenian Research Agency through the research programme P4-0015 “Wood and lignocellulose composites” is also acknowledged.

Understanding of the effect of natural saltwater treatment on durability, fibers densification and chemical modification of palm wood

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Keywords: palm wood, chemical modification, Brining treatment, decay and termite resistance, mechanical properties.

Nowadays, the Tunisian primary wood-processing industry consumes more than 872,000 m³ of wood raw materials, of which only 119,000 m³ are supplied by local forest resources. In addition, several natural lignocellulosic fibers, as those from palm woods, are available around the country but they remain poorly exploited. The palm-tree sector plays a very important role on both the socioeconomic and ecological levels, mainly in southern Tunisia. There are three million trees in Tunisian palm plantations, ensuring a potential significant wood production. This type of wood is used in various specific industries, mainly in the craft and furniture industries. In the past, Palm wood was also used as structural material. Its low natural durability and its low mechanical properties were improved by an ancestral preservative method consisting in the immersion of the trunk of the palm tree trunk freshly slaughtered for a period ranging from 1 to 2 years (depending on the species) in the salt waters of the Lake of *Chot Djerid*. This ancestral practice was disappeared, and it is always difficult to find more information on the different parameters involved in this kind of process. The objective of this STMS work was to assess the main technological qualities of palm wood preserved by salting while trying to retrace the steps of this natural and eco-friendly preservation process. Samples (boards) from two defect-free of common date palm cultivars (*Kentichi* and *Deglet Nour*) with ages ranging from 40- to 50-years and two sample (boards) preserved by salting in the *Chot Djérid* (*Kentichi* and *Deglet Nour*) were used for the experiments. Each wood samples were collected at the Regional Center of Research on Oasis Agriculture - Degache - Southern Tunisia. Densities (air-dried, water saturated, basic), mechanical properties, decay and termites resistances tests were performed on native and water salt treated palm woods.

The first results showed a significant increase of the air-dried density of palm wood samples which increases from 216 to 408 kg/m³ after the wood salt water immersion. Basic and water saturated densities of Palm wood are also increased by salt water treatment but way less important than for the air-dried density. According bending test, salt water treatment allow to improve greatly the palm wood MOR in bending (from 15.8 MPa to 61.1 MPa) and MOR parallel to the fibers (from 11.9 MPa to 22.3 MPa). These results could be explain by the palm fibers densification occurred by the salt water impregnation into the wood.

Figure 1 (a) shows that treated palm wood has better decay resistance for each tested fungus (*Coniophora puteana* [CP]; *Poria placenta* [PP]; *Gloeophyllum trabeum* [GT] and *Corioliolus versicolor* [CV]). Termite’s resistance tests highlighted that native and treated palm wood had a similar degradation level after termite exposure, but the termite mortality rate was higher for the treated wood than that of native palm wood.

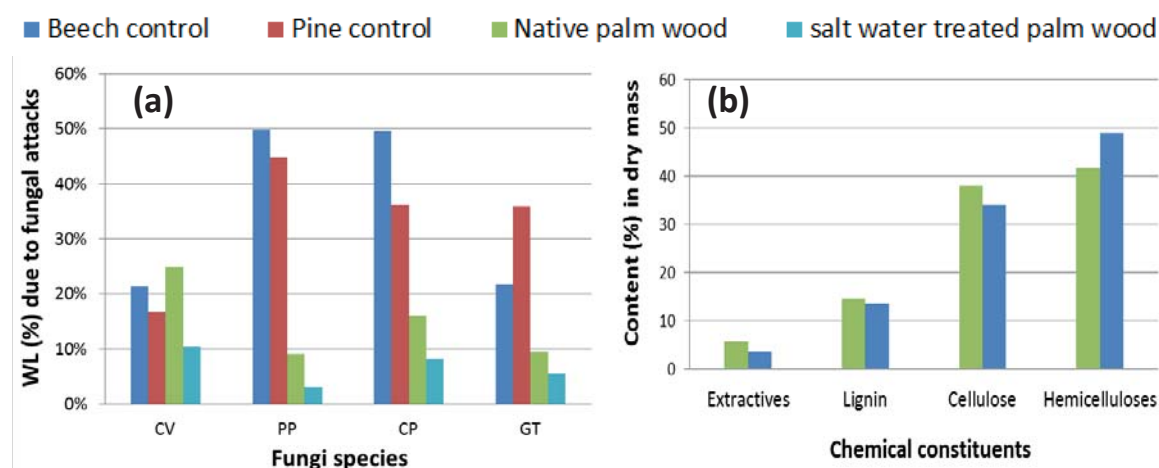


Figure 1: (a) Weight losses due to fungal attacks on native and salt water treated palm wood and (b) Percentage of different chemical constituents of native and salt water immersed palm woods.

According to Figure 1 (b), extractives, lignin and cellulose contents are slightly more abundant in the control samples except for the hemicelluloses which are more abundant in the treated palm wood sample. Mineral compounds analyses performed with MP-AES 4100 Agilent device are in progress, in order to evaluate the presence of several mineral compounds in palm wood, after salt water immersion that could explain the improvement of decay and termite resistance of the treated material.

Acknowledgments:

The authors gratefully acknowledge the Cost Action FP1407 “ModWoodLife” for the STSM financial support granted to Dr. Mohamed Tahar Elaieb allowing him to carry out this work at the CIRAD, with the BioWooEB Unit Research team.

The effect of wood drying and heat modification on some physical and mechanical properties of radiata pine (results from STSM 35419)

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Keywords: heat-treatment, kiln drying, thermal modification validation, wood drying.

The wood industry is particularly interested in the development of drying technologies that provide better energy efficiency, enhancing the drying quality and rate while reducing the air emissions of conventional drying system. Heat modification is an interesting alternative that improving intrinsic properties, reducing energy consumption as well as prevents from biological damage. In this work, the changes that occur in the physical-mechanical properties from drying to heat treatment were follow in order to know the factors that are significantly influenced by the drying process and which alter the wood dimensions or its strength values. The fast growing species *Pinus radiata* was used for modelling the physical-mechanical changes after drying process and heat modification (at 190 °C) by measuring the moisture content, density, shrinkage, MOR and MOE from the same load. The results revealed interesting correlations between intrinsic factors and properties (Table 1); the values of density were highly dispersed after drying or thermal treatment and not categorically correlated with other parameters. In contrast, wood with low density was generally more stable, since the minimum density values were kept constant after heat-treatment. Moreover, weight loss and MC were decreasing proportionally to the treatment intensity due to the release of bound water, and after heat treatment it was due to the degradation of the hemicelluloses fraction and amorphous cellulose (Fig. 1). Regarding the weight loss, it was associated with changes in microporosity or in cell wall thickness. Both weight loss and MC were reasonably correlated with the dimensional stability, improving the dimensional stability after drying treatments but keeping the same order of anisotropy; thus, the anatomical aspects that influenced the wood shrinkage were not significantly affected. Regarding the mechanical properties, the MOE was unaffected by the drying temperature, whereas the values were similar when drying at 100 °C or when thermally modified at 190 °C. However, the MOR was dropped during the drying process, obtaining three differentiated groups with a decrease of around 59% after thermal modification.

Table 1: Results of some physical- mechanical properties

Sample	Density [kg/m ³]		MC [%]	WL [%]	VS [%]	Ψ [T/R]	MOE [MPa]	MOR [MPa]
	ρ ₀	ρ _b						
Air-dried	432.52 ±45.19 ***	388.66 ±38.66 ***	22.61 ±10.95 ***	51.83 ±5.80 ***	10,17 ±0.63 ***	1.55 ±0.36	7036.79 ±1650.96 ***	101.29 ±4.80 ***
Kiln-dried	0(0-b) 454.98 ±45.85 ***	0(0-b) 399.38 ±45.34 ***	1 9.16 ±0.50 ***	1 8.35 ±0.42 ***	1 4,41 ±0.42 ***	1.47 ±0.33	1 9241.89 ±1976.19 ***	1 82.98 ±5.55 ***
Heat-treated	0(0-R) 437.88 ±42.18 ***	1 395.88 ±40.18 ***	1 6.25 ±0.60 ***	0 5.88 ±0.54 ***	1 1,37 ±0.33 ***	1.37 ±0.27	0 9653.65 ±1456.63 ***	1 48.97 ±11.07 ***
	0(0-b-R)	0(0-b)	1	0	1		0	1

ρ₀= Anhydrous density ρ_b= Basic density; MC= Moisture Content; WL= Weight Loss; VS= Volumetric Shrinkage; Ψ= anisotropy coefficient; MOE= Modulus of elasticity; MOR= Modulus of rupture; Significance of each set of values means (one-way ANOVA): (***) significantly different, P<0.001; Bonferroni correction test: (0) difference of the means is not significant at ***, (1) difference of the means is significant at ***

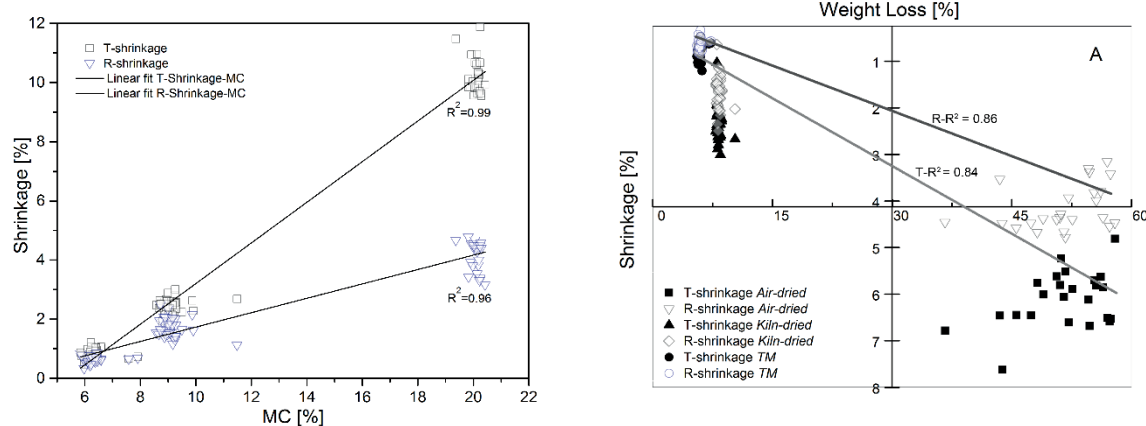


Figure 1: Performance of shrinkage versus moisture content and weight loss from air drying to heat modification

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Acknowledgments:

The authors appreciate the financial support of the Basque Government (scholarship of young researchers training) and the support of COST Action FP1407 through STSM-35419.

Approach for Cascading – Analysis of the Application potential of different Tree Materials with antimicrobial Properties

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Keywords: antimicrobial effects, FT-IR spectroscopy, material extractives

In every culture, plant extractives play a fundamental role in the drug therapy as well as for food supplements and the cosmetics industry (Bart 2011; Bart *et al.* 2012). Natural extractives have a good reputation in society; however, through the growing demand and the industrialization they are pushed to a corner and are replaced with products produced from fossil resources. Nevertheless, there are also a huge potential to find new products with additional benefit to increase the utilization of products from natural resources and the cascading for by-products.

Anti-microbial effects could be divided into passive and active effects, while these terms have not been commonly differentiated in many publications (Laireiter *et al.* 2014). Positive anti-microbial effects of wood are ascribed to passive effects, such as the hygroscopicity and associated dehydration of bacteria. Moreover, the active anti-microbial effect is that wood might contain anti-microbial substances, which directly inhibiting the growth of micro-organisms.

The aim of the research is to characterize the chemical compounds, which are in the liquid extractives of the wood material.



Figure 1: Overview of the various wood materials a) beech, b) pine, c) black locust, d) larch.

A screening approach was used to detect the potential of antimicrobial effects on the extractives. In this research, the focus is on the bark from the following trees species larch (*Larix decidua* Mill.), pine (*Pinus sylvestris* L.), beech (*Fagus sylvatica* L.) and black locust (*Robinia pseudoacacia* L.). To produce the extractive, the following screening method was applied for the extraction with different solvents (e.g. methanol and water). The extraction with solvents was done in the following way on 1g of wood dust (see Figure 1) got pipetted with 10ml of a solvent and extracted for 24 hours. After 24 hours, the solutions were filter and analyzed.

The results show that the various wood materials demonstrate different antimicrobial effects. Also the extractives from the bark show antimicrobial properties.

This screening process evaluated the potential of the antimicrobial properties of the extractives from various wood materials. These findings are useful for the potential estimation of the recovery of valuable products from various wood processing processes (e.g. wood drying and thermal modification). These properties could create a surplus value in the field of natural active compounds and flavors and utilization natural resources such as wood extractives, which would have been in other ways waste. However, further research is needed to obtain more knowledge about antimicrobial effects of wood materials.

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Acknowledgement

The authors gratefully acknowledge for the support of the Austrian Research Promotion Agency (FFG) under grant no 853478.

Analysis of fracture toughness of thermally modified wood in Mode II

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Keywords: thermal modification, fracture toughness, Mode II, DIC.

Thermally modified timber (TMT) has been long recognized as an efficient and eco-friendly alternative to tropical species and wood treated by other techniques. Nevertheless, the range of feasible applications for TMT is limited by undesired side effects, such as reduction of mechanical properties. Therefore, this paper aims to evaluate and understand the problematics of fracture mechanics of modified wood. The measurement of fracture toughness on mode II will combine the three-point bending test performance together with the strain field data obtained from the optical technique applying the principles of the digital image correlation (DIC).

The fracture toughness of thermally modified wood has been tested against reference non-treated samples of the European beech (*Fagus sylvatica* L.) wood as clear orthotropic blocks with dimensions of 20x20x500 mm (radial x tangential x longitudinal length). Twenty control samples were cut from untreated and the same number was prepared at differently thermally modified (180 °C and 200 °C) which took approximately 50 hours of chamber treatment (Figure 1b). Before the sampling, all source material was conditioned in a climate chamber at 20 °C and 65% relative humidity until equilibrium moisture content (EMC) was reached. The test performed was based on the 3-point bending end-notched setup in the mode II. An artificial crack length approximately 162 mm was introduced in longitudinal direction. The artificial crack was in LR plane and during testing a Teflon paper was placed inside it to reduce friction. A stochastic pattern to enable DIC calculation was also applied on all the samples prior to the mechanical test. Afterwards, the samples were measured on a universal testing machine, Zwick Z050/TH 3A equipped with a 50 kN load cell. The deformation induced in the samples was determined by the full-field optical stereovision system

consisting of two CCD cameras. The images were captured every 0.5 seconds (2 Hz) and synchronized with the applied force. The strain fields at the area of interest from the partial derivatives of the displacement using Lagrange notation were calculated in Vic-3D (Correlated Solutions Inc.).

Results

The analytical calculation of toughness follows the equivalent crack length method for the determination of the resistance curve (R-curve). In this sense, the strain energy release rate (G_{II}) for all samples is explicitly determined from the experimental load-displacement (P-u) curve by means of the compliance-based beam method (CBBM). This method allows the determination of the R-curve without requiring the measurement of crack length during test. As depicted in Fig. 1a, the resistance curve of the non-treated wood samples is greater than the thermally modified specimens.

Additionally, the image data from the 3-D digital image correlation (DIC) provides additional data such as displacement and strains helping in the analysis of the crack development (Fig. 1b). So, an independent evaluation of the crack tip opening displacement (CTOD) is successfully accomplished by post-processing DIC measurements. Based on the existing results, the following can be concluded; the strain energy release rate (G_{II}) of thermally modified wood is lower than the non-treated wood which confirms that thermal modification influences fracture toughness properties of wood.

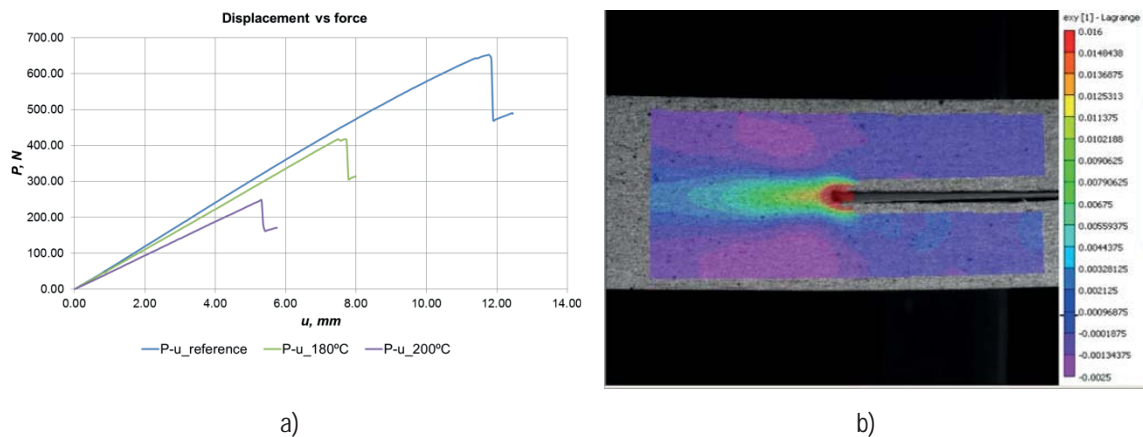


Figure 1: a) Load-displacement curves b) DIC - Crack tip opening displacement

Acknowledgments:

The authors would like to thank COST Action FP1407 for enabling Prof. Miguel Redón-Santafé to participate in an STSM which was completed with Dr. Václav Sebera, Mendel University in Brno, Faculty of Forestry and Wood Technology, Department of Wood Science.

Environmental Profiles of Alternative Tannin Extraction Scenarios

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Keywords: LCA, tannin, spruce bark, hot water extraction, post extraction treatment

Tannins have shown antifungal effects and have been considered a potential natural compound for wood preservation (Anttila *et al.* 2013). Extracts produced from softwood bark through hot water extraction contain both tannins and non-tannin compounds (Bianchi *et al.* 2015). The non-tannin compounds may reduce the effectiveness of tannin used as a wood (Anttila *et al.* 2013). Bianchi *et al.* (2016) have proposed a way to increase the tannin concentration in softwood bark extracts relative to other compounds by inserting a cold-water extraction step before the hot water extractions. The purpose of this research is to study environmental impacts of hot water extraction; identify the hot spots within the tannin extraction and post-extraction treatments as cradle-to-gate life cycle and give suggestions to optimize environmental profile of different tannin production scenarios of spruce bark.

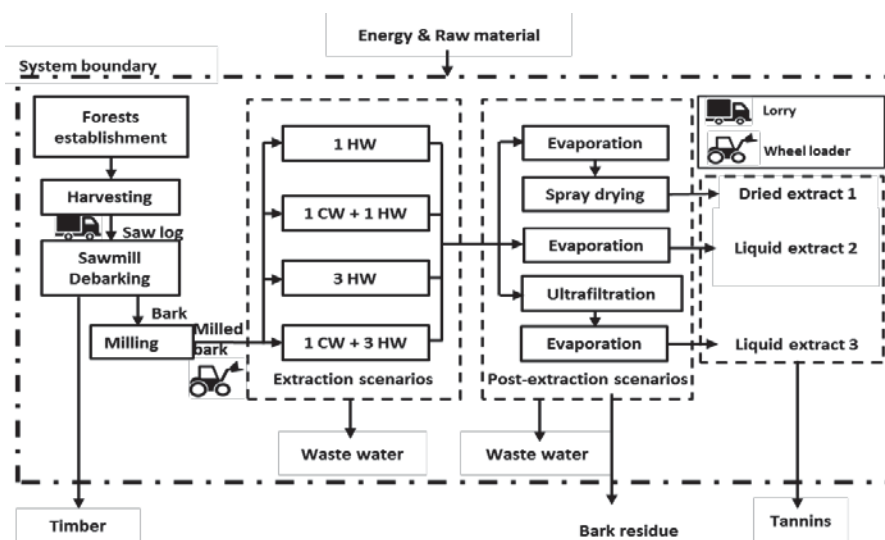


Figure 1: System boundary for the tannin production. Detailed flow chart of the simulated tannin production unit operations from cradle to gate

The system under study includes steps from forests establishment to tannin product (fig.1). Scenarios are explained in Table 1. The functional unit (FU) is 1 kg tannin yield after post extraction treatment.

Table 1: Tannin extraction (E) and post extraction (P) scenarios (30% is weight percentage (w/w) of all compound in extracts solution; 5% is weight percentage (w/w) of tannin in extracts solutions).

Extraction scenarios	
E1(1HW)	One step of hot-water extraction
E2 (1CW+1HW)	One step of cold-water extraction and one step of hot water extraction
E3 (1CW+1HW)	Three steps of hot water extraction
E4 (1CW+3HW)	One step of cold-water extraction and three steps of hot water extraction
Post extraction scenarios	
P1	Extractives → evaporation to 30% → spray drying → dried extract 1
P2	Extractives → evaporation to 5% → liquid extract 2
P3	Extractives → ultrafiltration → evaporation to 5 % → liquid extract 3

Experiments were designed to study the tannin yield under different extraction scenarios; the post-extraction scenario analysis was based on literature review. The results show that the use of ultrafiltration after extraction (P3) can halve the environmental burdens given that loss of tannins is negligible. Although tannin yields are higher, preliminary cold water extraction followed by hot water extraction(s) (1CW +1HW & 1CW+3HW) have a higher environmental impact for the FU than a single hot water extraction. Preliminary cold water extraction and ultrafiltration (1CW +1HW, 1CW+3HW & P3) might be beneficial processes in terms of obtaining less non-tannin compounds. Noticeably, evaporation process is the largest contributor to all environmental impacts and resource use categories, which should be solved in the future to improve the environmental performance of tannin production. One possible solution is to use regular heat recovery or more advanced technologies (e.g. Liu *et al.* 2013) to reduce the energy demand.

The utilization of spruce bark tannins as an antifungal agent is still in development. Some other issues than tannin purity are also arising, e.g. the solubility of tannin in water, and to what extent tannin-based preservatives can increase the service life of wood products. This paper offers a novel platform for future studies on tannin extraction methodologies and applications of tannins such that more solid product level LCA results can be provided. This would allow an accurate study of the entire value-chain of tannin-impregnated wood.

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Review of biogenic carbon in carbon footprint of modified wood

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Keywords: climate change, carbon footprint, modified wood, environmental product declaration (EPD), carbon storage

There is an increasing use of carbon footprinting and Environmental Product Declaration (EPD) for communicating the environmental performance of construction products (Minkov *et al.* 2015). This can be related to increasing concerns regarding greenhouse gas (GHG) emissions from human activities and associated climate change (Stechemesser and Guenther 2012). Product carbon footprint accounts the total amount of GHG emitted during the life cycle of goods and services, based on Life Cycle Assessment (LCA). Thus, this is based on a different approach than the GHG assessments at the level of projects, corporations, nations and individuals which mostly account for direct GHG emissions, not addressing indirect emissions from upstream and downstream activities. Addressing the accounting of biogenic carbon flows and their relation to the global warming impacts associated with a product is specially challenging for forest products (Sandin *et al.* 2016). Therefore, a review was performed on recent research and technical standards in this field to promote a common understanding. The main findings show the need for reporting the contribution of biogenic carbon to the total GHG emissions and removals over the product's lifecycle. In order to facilitate the implementation of more advanced methods from current research, the EPD should also include more detailed information of the used wood, in particular species and origin. The current available EPD for modified wood are used as examples for how this could be included in practice.

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Acknowledgments:

The authors acknowledge COST Action FP1407 in supporting networking activities that resulted in the cooperation behind this publication. Also, specifically for supporting a Short Scientific Mission (STSM) with reference code COST-STSM-ECOST-STSM-FP1407-020117-081905 where a large part of this work was performed. Ana Dias acknowledges the project Sustainfor (PTDC/AGR-FOR/1510/2014) funded under the project 3599-PPCDT by FCT (Science and Technology Foundation - Portugal) and by FEDER (European Regional Development Fund). Edwin Zea acknowledges the Chair of Sustainable Construction ETHZ for their support. Callum Hill acknowledges the European Commission Horizon 2020 project ISOBIO', project number 636835 for financial support.

“Think outside of the wooden box! PhD Workshop Hamburg – COST Action FP1407”

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Keywords: PhD workshop, innovation, wooden box, FP1407 training school

Wood as a porous, three dimensional, hygroscopic, viscoelastic and anisotropic bio-polymer composite is not yet understood completely. The gap in fundamental understanding of the highly complex anatomical, chemical and physical structure of lignocellulosic materials constrains the development of innovative wood-based products.

On the other hand, the European wood-based panel and composite sector is composed of strongly cost-driven industries. Maximizing process yields and minimizing the costs of raw material are important in times of increasing consumer demands (Mantau 2012). During the transformation process from a petro-based industry to a wood-based bio-economy, novel, innovative lignocellulosic products can play a key role (Hagemann *et al* 2016). But why do we still lack high-performance wood-based products that are required by an emerging bio-economy?

Young researchers in independent research institutions are urged to generate, deliver and interlink knowledge. This then may lead towards innovation in research and subsequently industry. Especially in regions in which the wood-based industry is still below its potentials, this innovation drive is even more important. Alfranca *et al* (2014) analysed the effects of innovation on the European wood industry market structure. They confirmed that R&D spending and R&D personnel are key factors in explaining market concentrations, but are no explanation for innovation. However, the influence of these variables may be affected by the initial degree of market concentration in the industry. One must ask the question, which are the specific obstacles to innovation in the sector?

Environmental friendly building materials must necessarily be durable. Hence, they require elevated resistance to physical and chemical factors as well as biodegrading agents. Especially wood modification and bio-composite design aim at enhancing product performance. How do these products enable the European market to strengthen cascade utilization, hence broadening the resource base, storing carbon dioxide and saving process energy in the long-term?

Together with the European COST Action FP1407, the University Hamburg MIN Graduate School International and the Thünen Institute for Wood Research we seek to answer these questions. Our team organises a “PhD workshop” which aims at connecting ideas of young researchers, active in adjacent topics such as process engineering, building physics, wood and material science. The workshop follows a holistic hands-on strategy and involves specialists from the wood biology, chemistry and physics and combines them with “non-wood” experts, e.g. from the automotive sector. The workshop sessions are listed below:

0. Basic session: The essentials of wood physics, anatomy, chemistry and processing technologies
1. Session: Wood-water relations - Is water a main component of lignocellulosic materials?
2. Session: Performance of wood products - What are the theoretical potentials and how do they translate into practical values?
3. Session: How do wood based products impact the environment?
4. Session: Advanced processing of lignocellulosic materials: Where to find higher added value?
5. Session: The 10 big questions of wood science - Which high-value products are currently being researched or produced?

This shall yield an advanced knowledge on how wood structure influences renewable high-performance biomaterials. The results of the workshop shall lead to ideas outside of the “wooden box”, benefiting each partner institute, participant as well as the sector as a whole.

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Acknowledgments: Our thanks go to Andreja Kutnar, Gus Verhaeghe and Johannes Welling. They helped us to get any obstacles out of our way so far. Special thanks go to our excellent speakers. Their willingness to support our workshop on a pragmatic and direct way by contributing to our sessions make it a worth experience for all participants.

Friday 15th September,
COST Action FP1407: 3rd meeting
“Wood modification research
& applications”

Poster session

From 10:10 to 11:10

Chair: D. Sandberg, L. Tellnes

Thermo-mechanical treatment of flooring elements

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Keywords: wood modification, improvement, quality, floorboard

T-M modification is one of the processes that could be used to improve the material properties of wood. Although wood modification was the subject of a lot of consideration at an academic level for over 50 years, there is still a need and a space for development. As a result of T-M treatment, wood changes occurring (depending on the modification conditions) such as: improvement in dimensional stability, reduced hygroscopicity, improved resistance to biodegradation, unfortunately often followed by reduced MOE, MOR, abrasion resistance and tendency for cracks and splits (Hill 2006, Mitsui et. al 2003, Tjeerdsma et. al 1998).

The research was carried out in aim to verify the possibility of enhancement of floorboards properties in terms of selected mechanical and physical parameters. Research material was prepared by industrial partner. Typical flooring material was composed of seven layers of wood. The thickness of the use layer made of oak wood was 3.8 mm. Material was subjected to T-M modification by hot rolling densification. During modification, temperature of hot rolls and a rolling pressure were a variable factor, influencing a thickness of surface deformation.

Floorboards were produced in six variants, to allow the assessment of modification impact (Table 1).

Table 1: Samples used during project

Group	Densification [mm]	Temperature of modification [°C]
K	-	-
1/100	1	100
2/100	2	100
4/100	4	100
1/180	1	180
2/180	2	180

The impact of T-M treatment on the flooring surface was evaluated among others by the density distribution, which allowed to define the thickness of upper layer of modified surface, as well as its adjustment along the element. Then hardness of modified surface (according to EN 1534) was determined. The surface was characterized also by scratch and abrasion resistance. The next stage

of the study included surface wettability and surface free energy determination. Due to potential use of flooring in underfloor heating conditions, thermal properties: thermal conductivity, temperature conductivity (thermal diffusivity) and volume heat capacity were characterized for each group of samples.

It was found that compression of wood during hot rolling increase the average density of the composite. Stimulating effect on density increase was noted in the case of increase in the value of modifying factors. Modification of the top layer of the floorboard increases the hardness of the surface, but the effect of each modification parameter is disturbed due to the specificity of the anatomical structure as well as the anisotropy of the top layer of the composite. For the modification parameters used, no statistically significant differences were found for the thermal properties (thermal conductivity, temperature conductivity - thermal diffusivity and volume heat capacity). The composite structure of the tested materials, composed of several layers (regardless of the surface modification), cause a reduction in thermal conductivity, which demonstrates greater insulation of these materials.

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Improvement of wood heat treatment via an acoustic field

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Keywords: thermal degradation, acoustics, wood heat treatment, thermal modification

Torrefaction is a wood thermal modification process which improves wood properties as moisture content, grindability and material homogeneity. This mild form of pyrolysis is carried out in inert atmosphere under relatively low temperatures (from 200 °C to 300 °C). Resulting thermodegradation reactions of wood polymers are essentially endothermic. Various technologies were developed and implemented in the industry (Acharya et al. 2012). The present work is devoted to develop an innovative technology aiming to improve the wood heat treatment coupling an acoustic field delivered by a sound speaker and temperature. The lab-scale reactor is illustrated on Fig. 1. Device development, characterization and first experimental results will be presented. A acoustic behaviour characterization and mapping within the reactor's cavity was executed.

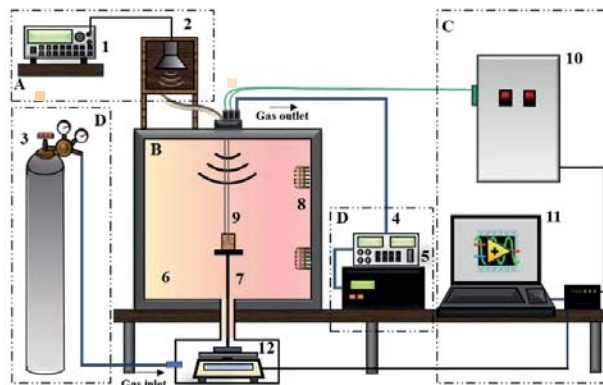


Figure 1: Experimental torrefaction system diagram: 1) Wave generator; 2) Speaker 3) N₂ cylinder; 4) Gas pump; 5) O₂ control; 6) Reactor chamber; 7) Sample support; 8) Electric resistances for heating; 9) Thermocouples; 10) System control; 11) Computer; 12) Weight balance.

This characterization included the flow rate and acoustic intensity measurement at the exact sample's location in the reactor. The analyses enabled the identification of optimal acoustic frequency and intensity to produce a maximum acoustic flux around the wood sample. It has been assumed that the acoustics field within the torrefaction reactor could have the capability to modify the pressure distribution and heating medium velocity field around the wood sample modifying such way the wood surface thermal boundary layer and improving convection heat transfer. Experiments were carried out at 250 °C for 120 min under influence of two acoustic frequencies 1810 and 2696 Hz (103 and 107 dB intensities respectively). Experimentally recorded profiles of wood centre's temperature and mass yield are graphically illustrated on Fig. 2. A maximum temperature gradient of 2 °C was observed between treatments with and without acoustic. These results indicate that the acoustic fields affect the heat transfer under similar experimental conditions and consequently wood thermodegradation. Next step will be to analyse different parameters as temperature, biomass species and frequencies variations. This will be subject to new investigations and research publications.

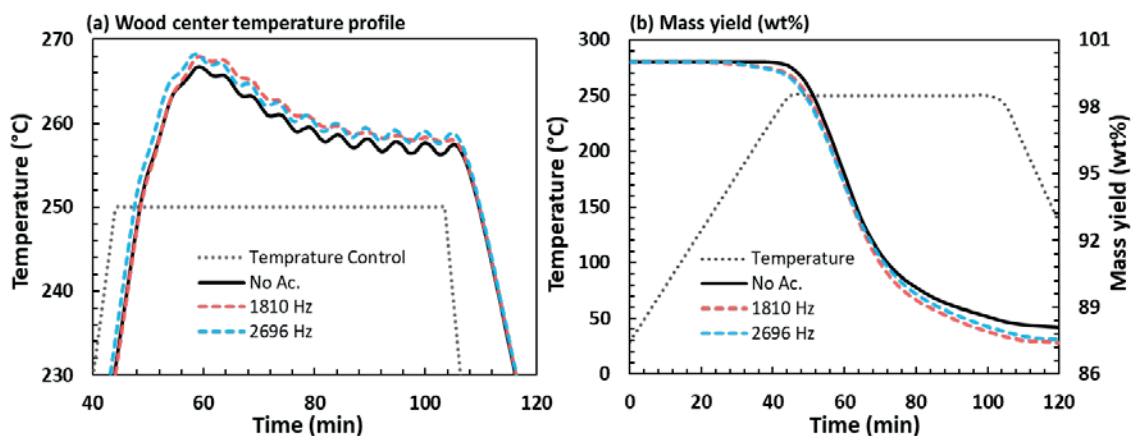


Figure 2: Thermal degradation at 250 °C under acoustic influence.

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Acknowledgments:

The authors gratefully acknowledge the program ANR-11-LABEX-0002-01 (Lab of Excellence ARBRE) and their financial support.

Surface modification of solid wood by charring

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Keywords: cladding; corption; thermal modification; wood; wood char

Modification processes, such as thermal modification, aim to increase the durability of wood in demanding conditions. Still, different modifications do not entirely prevent fungal growth if there is sufficient moisture in the environment. Cupping, erosion and checking may take place (Jämsä *et al.* 2000) and fading of colour is also not always a desired feature. Relatively fast surface charring of wood could be an alternative to increase the service life of wooden claddings. Charring is a traditional Japanese technique used to increase the durability of cladding boards (*shou sugi ban*), where wood is burned from the surface and the appearance is said to last for decades (Miller 2015). To assess some weathering related effects on surface charred wood, spruce (*Picea abies* L.) and pine (*Pinus sylvestris* (Karst.) L.) sapwood were subjected to long (30, 60, 120 min at 250 °C) and short charring processes (30 s at 400 °C) using a hot plate. The wettability was measured by floating and by contact angle measurements. The micromorphological changes were studied with scanning electron microscopy (SEM) on ultra violet (UV) laser ablated samples (Seltman 1995).

Charring had a positive effect on the wood contact angles. Highest contact angles of spruce were obtained with samples modified at 250 °C for 30–60 minutes, and those for pine with samples modified at 250 °C for 30 minutes. The absorption measured by floating reduced from reference to the sample modified at 250 °C for 60 min, but increased slightly with longer treatment time as well as more severe temperature (Fig. 1). The SEM revealed damage on the inner surface of the lumen surface of the lumen of spruce charred at 400 °C close to the charred surface, i.e., approximately

5–15 cell layers from the surface (Fig. 2). In the case of spruce charred at 250 °C, 120 min, the samples were influenced less, but deeper. The damaged surface could be related to fast expansion of extractives, but the effect of laser ablation on charred wood is also unknown.

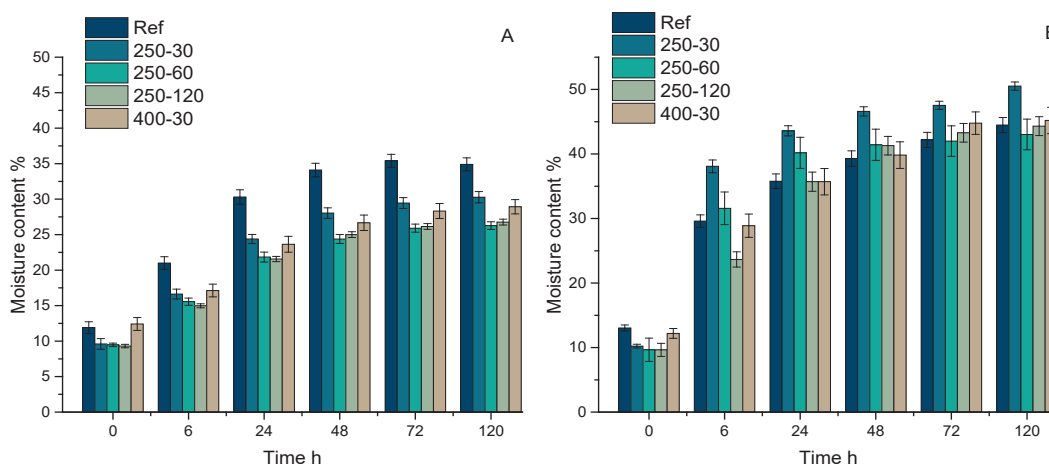


Figure 1: Water uptake of spruce (A) and pine (B)

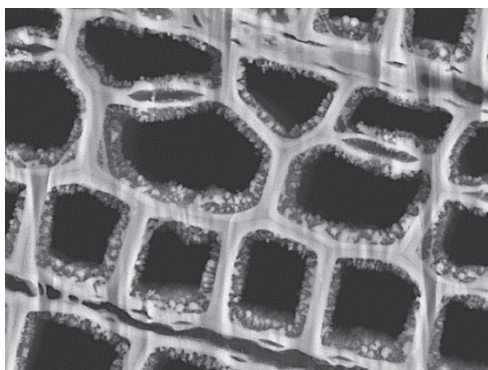


Figure 2: SEM image of spruce charred at 400 °C (30 s)

Fast one-sided charring of wood could provide a durable alternative to contemporary modification methods when less permeable wood species, such as spruce, or pine heartwood are used. With lower temperature and longer time, the wood is affected deeper, creating a wider transition zone from charred surface to inner virgin wood. The effect of this thermally modified layer needs to be investigated in terms of its protective potential when wood cracks during weathering.

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Development of UV – colour modification of wood surface

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Keywords: wood colour, texture, UV-irradiation, individualisation, modification

The colour modification as a secondary effect of thermal or chemical modification of wood does not cover or repress the natural texture, even when the new colouring can be less authentic for the particular wood species. This paper deals with possibilities of intentional visual modification of wood colour that ensures aesthetic innovation and individualisation of surface, by maintaining surface authenticity. The UV-degradation connected with the colour changes – yellowing or browning can serve in a more intensified form for the intentional colour modification and aesthetic purposes, with maintaining an authenticity.

The main challenge is the application of this technique on less „fashionable“ and interesting wood species with less pronounced texture. For the experiment, 4 kinds of hardwood species in form of veneered plywood were selected: poplar, birch, beech and alder, formatted to A4 sheets. These were exposed in the illumination chamber with the high-power UV- lamp (400W). Exposition time and designs of the patterns used for the aesthetic improvement were tested. First pilot tests have shown that the sufficient contrasting colour change is reached within 8 hours of exposure. Different mesh patterns were used to achieve interesting structures. The further development is the illumination with inclination that allows interesting visual patterns (Fig. 1).

Later colour changes due to UV- radiation can be eliminated through UV-protection paint or additives or can be left without some intervention like a „part of the game“. In literature the effect of UV light on wood is usually connected with degradation and deterioration. However, photo-oxidation or photo-chemical degradation affects only a thin surface layer of wood under such conditions, starting immediately after exposure to UV light. Wood is a good absorber of infrared (IR), visible and ultraviolet (UV) light. UV light is and can be fully absorbed by a 75 µm-thick layer of wood (Hon and Ifju 1978). Understanding the chemistry of UV degradation of wood requires knowledge of the chemical nature of wood components, the UV spectrum, and the interactions of UV radiation with various chemical structures in wood. Chemical changes on the UV light treated wood surface are now being observed with FTIR spectroscopy. UV- light portion of sunlight is responsible for the primary photo-oxidative degradation of wood. The first law of photochemistry, the Grotthus-Draper principle, states that for a photochemical reaction to occur, some component

of the system must first absorb light. The second law of photochemistry, the Stark-Einstein principle, states that a molecule can only absorb one quantum of radiation at time (Rabek 1995). The absorbed energy can thus cause the dissociation of bonds in molecules of the wood constituents. This homolytic process produces free radicals as the primary photochemical products. This event, with or without oxygen and water, can lead to depolymerization and to the formation of chromophoric groups such as carbonyls, carboxyls, quinones, peroxides, hydroperoxides, and conjugated double bonds (Feist and Hon 1984). Changes in wood colour are therefore attributed to the modification of chromophores, which are capable of absorbing UV light in the range of 300 to 400nm.



Figure 1: Samples of UV-modification on poplar and beach wood by using different mesh masks and samples with the special transition pattern thanks to inclination of the mask.

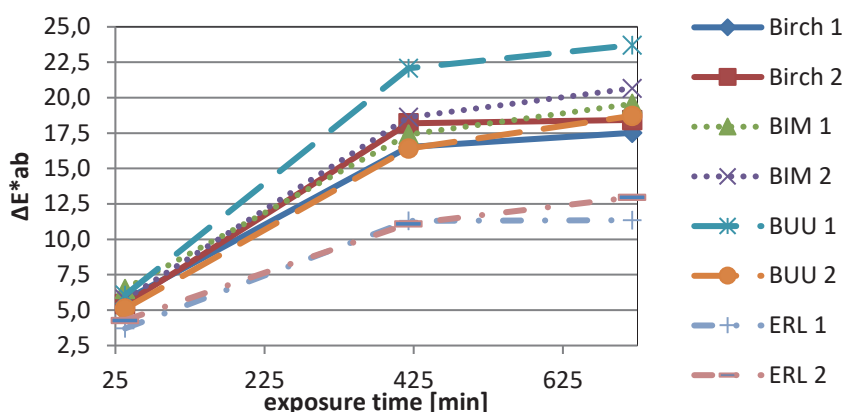


Figure 2: Dependence of ΔE^*_{ab} changes on time of exposure to UV light for various wood surfaces.

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Acknowledgments:

This study was supported by project APVV 0594-12 Interaction of Man and Wood and APVV 16-0567 IDENTITY.SK – common platform of design, architecture and social sciences

Wood Modification by Alkali-Activated Composition Coatings for fire protection

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Keywords: alkali activation materials, coating, fire protection

Wood is natural product, which is widely used in many technical areas as construction material. All commonly used wooden building materials lose strength when exposed to high enough temperatures. It is a great demand in durable fire protection coatings with good adhesion with wood. Recently alkali activated materials have attracted strong interests all over the world due to their advantages of low energy cost, sustainable solution by recycle the hazardous residue material and it undergone green chemistry technique treatment. These materials are known as superior thermal stable and fire resistance. (Krivenko et al. 2013) present data on the protection of timber from combustion and burning using the protective coatings based on alkali aluminosilicates. In combustibility and burning behaviour, the formulated coatings can be classified as hardly combustible and non- burnable materials. In the paper (Guzii et al. 2015), the application of a heat-reflecting geocement coating, containing perlite, to reduce timber combustibility was studied. The study (Giancaspro et al. 2004) was related on determining the quality of fireproof wood treatment with geocement coating using the rapid method.

This research related with alkali activated slag with phosphogypsum addition and providing ways to use waste by-products, help curb carbon dioxide emissions. The main firing properties of these coatings were investigated. The wood (with and without coatings) properties before and after burning tests was evaluated.

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Wood modification with N-methylol compounds – Effects of modification agent and process conditions

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Keywords: curing process, DMDHEU, DMedHEU, impact bending strength, mDMDHEU

Cyclic N-methylol compounds such as DMDHEU (1,3-Dimethylol-4,5-dihydroxyethyleneurea) are water-soluble resins, originally used for textile applications. Since the early 1990s there has been a high amount of research activities to evaluate DMDHEU for its potential for wood modification (Militz 1993). Finally, this modification method was developed up to pilot scale (Schaffert 2006).

The main enhancements achieved by modification with DMDHEU are a high resistance against wood-destroying fungi and a good to high improvement of dimensional stability. Furthermore, a significantly increased surface hardness has been proven. Issues so far unresolved are an increased brittleness and crack sensitivity of uncoated and modified wood during weathering (Xie *et al.* 2014).

The objective of this work was to study the determinants for an increased brittleness and crack sensitivity of wood, modified with crosslinking agents such as DMDHEU. In particular, the first approach was a screening of alternative molecules that might be suitable for modifying wood and lead to the same improvements as DMDHEU, by simultaneously reducing brittleness and crack sensitivity of the modified material. The main focus was on resins based on modified dimethyloldihydroxyethyleneurea (mDMDHEU) and dimethyldihydroxyethyleneurea (DMedHEU). One of the greatest benefit of these molecules is a decreased formaldehyde content (mDMDHEU), whereby in case of DMedHEU there is no formaldehyde.

The Weight Gain [%] was found to be quite similar for the tested resins (Fig. 1). However, increased concentrations of mDMDHEU and DMedHEU were required to reach the same WPG [%] level as for DMDHEU. The impact bending strength [kJ m^{-2}] was significantly reduced due to the modifications (between 50 - 60 %) and found to be determined by the modification agent. Further on the curing process itself (heat treatment at 120 °C) indicated an effect on the brittleness of the wood (Fig. 1).

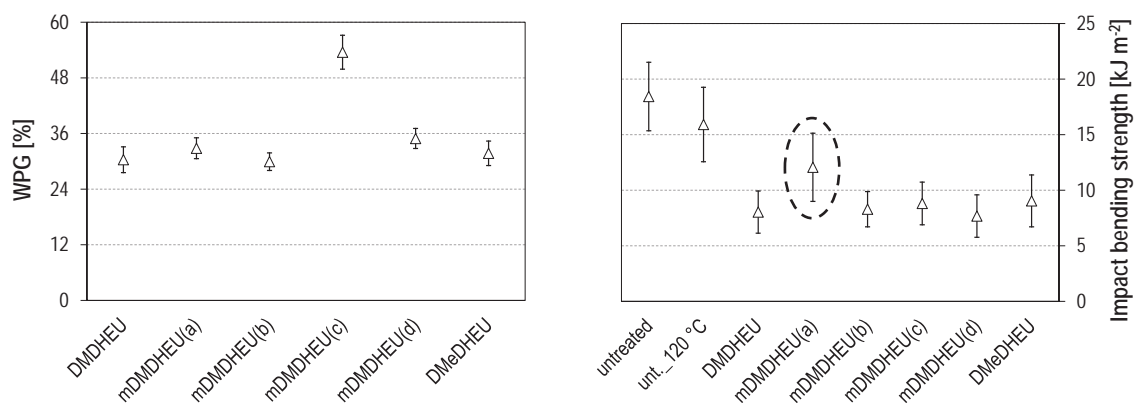


Figure 1: Weight Percent Gain [%] and Impact bending strength [kJ m⁻²] of wood treated with DMDHEU, mDMDHEU and DMedHEU under dry conditions.

As a second approach, the effects of process conditions on later properties of the modified wood were studied. In particular, two significantly different curing processes were investigated, by varying air humidity and temperature profile during the curing of the chemicals. Whilst samples modified under dry conditions tend to a higher Weight Gain [%], a modification under superheated steam atmosphere leads to a significantly lower embrittlement and crack sensitivity (Fig. 2).

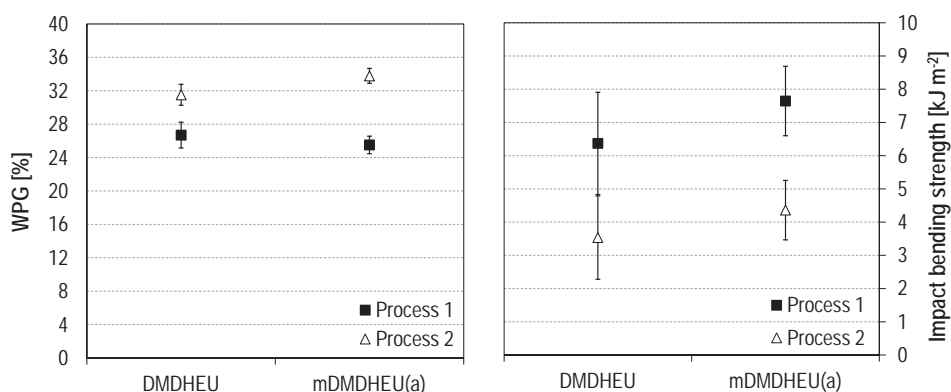


Figure 2: Weight Percent Gain [%] and Impact bending strength [kJ m⁻²] of wood impregnated with DMDHEU and mDMDHEU(a) and modified under superheated steam atmosphere (filled symbols) and dry conditions (open symbols)

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Acknowledgments:

The author thanks the Archroma Management GmbH (Reinach, Switzerland) for the provision of test chemicals and fruitful discussions.

Prediction of mass loss dynamics during wood thermal modification under industrial conditions

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Keywords: hardwood and softwood, kinetics model, thermal degradation, wood heat treatment

Thermal modification is an efficient way to improve some wood properties, like equilibrium moisture content (EMC), dimensional stability and durability (Esteves and Pereira 2009), to produce a wood modified material. According to the literature (Candelier et al. 2016), it has been observed that the thermal degradation of wood has a high dependence on the initial wood characteristics (wood specie, density) and process parameters, such as drying stage, heating medium, and treatment intensity (heating rate, temperature and duration). The objective of this study is to predict the treatment duration in order to reach a particular level of wood modification under industrial conditions. For that, the mass loss dynamics during the treatment are recorded and modelled. The flow chart of research methodology is shown in Fig. 1.

Thermal modification of poplar and fir

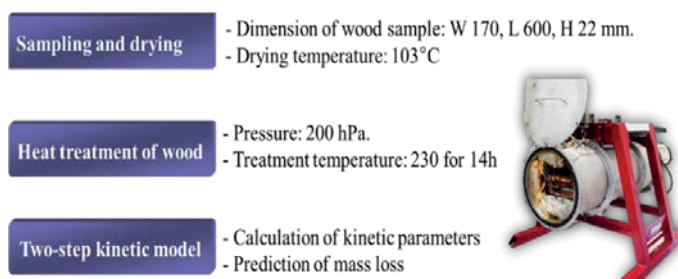


Figure 1: The flow chart of thermal modification of poplar and fir

The obtained results are encouraging for a future development of a numerical tool able to conduct performing the heat treatment of wood in industry. The experiments were carried out on wood boards (30 x 22 x 2.5 cm³) in a pilot scale system, under conditions close to the industrial ones (heat transfer by conduction with 0.2°C min⁻¹ heating rate under vacuum). Two different wood species, a hardwood: the poplar (*Populus nigra*) and a softwood: the fir (*Picea abies*), were used. The heat treatment was conducted at 230 °C for 14 hours under low air pressure (200 hPa) with less than 5% oxygen content. 14 hours duration is examined to obtain kinetic profiles required for the modelling, while usual treatment duration in the industry is close to 1 to 5 hours. Results of the present work are shown in Fig. 2. They indicate that the mass loss of poplar (14.21 wt%) is higher than fir (10.45 wt%). The difference of thermal sensitivity between poplar and fir is due to the hemicelluloses composition of hardwood and softwood (Chaouch et al. 2010). Moreover, if the target of the wood modification is to reach 10 wt% of mass loss, the duration for the poplar is closed to 750 min and 1200 min for the fir. This first observation allowed selecting the hardwood specie in priority to limit the heating energy consumption and carbon footprint, as well as optimize the economical balance.

A two-step kinetic model (Di Blasi and Lanzetta 1997) is adopted to predict the mass loss dynamics of poplar and fir. The kinetic parameters are calculated from experimental data by curve-fitting. A good agreement between modelled and experimental data is achieved for both two species. This model can be integrated in the development of a numerical tool able to give recommendations to the industry by the prediction of the treatment time to modify wood specie for reach required properties.

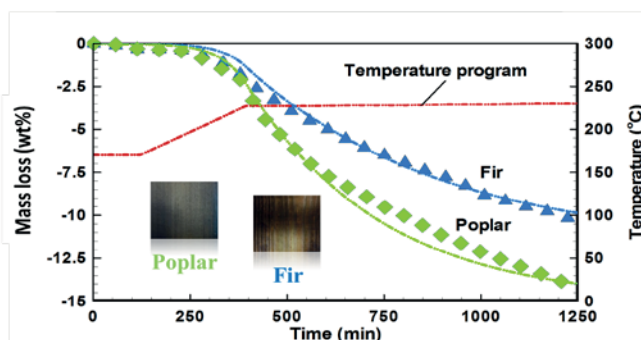


Figure 2: Modelled (lines) curves for poplar (green) and fir (blue) during heat treatment.

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Acknowledgments:

The authors gratefully acknowledge the program ANR-11-LABEX-0002-01 (Lab of Excellence ARBRE) for their financial support.

Mechanical properties of densified and thermally modified timber

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Keywords: densification, thermal modification, hardness, bending strength

Densification and thermal modification change wood properties in different ways depending on the treatment conditions and the wood species. In the presented investigations, densification and thermal modification were applied consecutively. The primary objective of this treatment combination was the compensation of reduced mechanical properties due to the thermal modification by densification. The combined processes were applied to five European wood species: poplar (*Populus nigra* L.), beech (*Fagus sylvatica* L.), English oak (*Quercus robur* L.). Depending on the mean density of the specie, a thermo-mechanical densification of 43% or 50% was imposed to improve mechanical strength parallel to the grain. Subsequently, the densified material was thermally modified in the so-called Vacu³-process at 230 °C and 20 % or 80 % vacuum and at 240 °C and 20 % vacuum. The thermal modification resulted in changing wood colour, mechanical strength, hardness, dimensional stability and durability (Welzbacher et al. 2008). All the wood modification processes were carried out at industrial scale after pre-tests at laboratory scale. The modified material was characterized regarding density profiles, bending strength, static and dynamic hardness. In further investigations were tested structural integrity, abrasion resistance, moisture dynamics, dimensional stability, and durability against white, brown and soft rot fungi. The modulus of rupture (*MoR*) and the modulus of elasticity (*MoE*) were determined in a three-point bending test according to DIN 52186 (1978) with a span of 300 mm. Densification of wood improved MOR and MOE significantly. This is causally connected with increased density. As expected thermal modification led to a strength reduction, but combined with densification the initial strength properties were sustained. Brinell hardness transverse to the grain was determined using a universal testing machine with a ball indenter with a diameter $D = 10$ mm according to EN 1534 (2010). Meyer *et al.* (2011) described the tests of dynamic hardness.

Densification of wood improved *MoR* and *MoE* significantly and increased with density as shown in Tab. 1. The maximal increase of *MoR* of 99 % compared to the reference was determined for densified oak followed by beech, poplar. Thermal modification led to mass loss and decreasing

mechanical properties. The Brinell hardness of the not densified and not thermally treated reference specimens are in the magnitude of values as reported in the literature. The densification results also in an increasing hardness. For poplar, the hardness is doubled; for the other species, the increase is even higher. Heat treatment led to a decrease in hardness compared to the untreated, densified specimens. However, the influence of the heat treatment depends on the wood specie. For beech, each of the heat treatments resulted in decreased hardness compared to the densified untreated specimens.

Table 1: test results of bending strength (MoR) and Resistance to Brinell hardness *HB* and dynamic hardness *HD* of differently densified and thermally modified materials (reference not densified and not thermally treated).

Wood	Process temperatur / vacuum	Density	MOR		HB		HD	
		[kg/m ³]	mean [%]	SD [%]	mean [kg/m	SD [kg/mm ²]	mean [kg/ms	SD [kg/ms]
Beech (43 % densificatio	reference	659	138	12	3.57	0.16	42116	3145
	only	1188	250	39	8.25	0.85	-	-
	230°C/80%	993	134	35	7.07	1.11	61076	7176
	230°C/20%	914	94	19	7.26	1.02	50119	10044
	240°C/80%	969	112	16	7.51	1.46	51701	8884
Poplar (50 % densificatio	reference	349	62	28	1.05	0.21	26804	6623
	only	692	107	6	2.06	0.16	-	-
	230°C/80%	624	121	9	5.11	0.76	46533	8081
	230°C/20%	609	119	20	4.85	1.01	57586	6173
	240°C/80%	611	101	16	4.33	0.42	48237	4480
Oak (43 % densificatio	reference	622	110	28	3.59	0.30	37919	2286
	only	1202	219	30	8.54	0.88	-	-
	230°C/80%	674	43	18	3.37	0.46	34616	3096
	230°C/20%	736	34	3	2.91	0.27	35589	2815
	240°C/80%	672	39	26	3.24	0.33	42301	15235

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Acknowledgments:

The authors gratefully acknowledge the financial support from German Federal Ministry of Education and Research for the Leading-Edge Cluster BioEconomy project VP 1.11 – DURAPRESSTIMBER under grant number 031A440A to 031A440D.

The ability to modification of pressing time of layered wood composites

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Keywords: wood, composite, layer, modification, pressing

There are several factors influencing the properties of wood layered composites like plywood or laminated veneer lumber (LVL). Some of them come from raw materials (like properties of veneers, resins, fillers etc.), while others come from production technology. The most important parameter that comes from the production process, influencing the properties of composite as well as influencing the efficiency of the process itself, is pressing time. Hoong and Paridah (2013) tested the mechanical properties of plywood depending on different pressing times. They produced three-layer plywood at constant temperature 140 °C and pressing times as follows: 14, 16, 18 and 20 min. The pressing pressure was 2 MPa during initial 5 min and was then increased to 5 MPa. The tests of produced plywood have shown that the modulus of elasticity (MOE) did not change significantly for pressing times between 14 – 18 min (5813 – 5914 N/mm²), but when the plywood was pressed 20 mins, the MOE was much higher (7797 N/mm²). In case of modulus of rupture (MOR) there was no clear linear dependence, but the highest value (60.1 N/mm²) was found for 20 min pressing time.

The similar phenomena of increased mechanical parameters of plywood were described by Bekhta *et al.* (2009). They investigated the properties of birch plywood pressed for 5, 8 and 11 min. The conclusion of these tests was that with the increase of pressing time the mechanical properties of plywood (MOR) were also higher.

The typical formulas to calculate the pressing time include: number of pressed veneers, thickness of the single veneer, experimentally established factors (e.g. Smirnov' formula). The other formulas are based on fixed coefficients, which consider time, plywood thickness, as well as the distance from the plywood surface to the bonding line nearest the centre of plywood thickness (e.g. Thoemen *et al.* 2010).

Above mentioned formulas provide significantly different results. When calculating the time for exemplary 16-layer LVL composed from 3 mm thick pine veneers, the calculated pressing time vary from 25 to 34 min. Such difference (36% from the shortest time) can result in production efficiency. Also, the composite properties can be different. Such long pressing time is mostly caused by the necessity to transfer the heat from the surface to the middle layers; in case of composites with higher thickness, this fact has a crucial impact. It is worth to mention that dry wood is quite good thermal insulation material, which efficiently inhibits the heat transfer inside the pressed composite.

The aim of mentioned on-going research is to try to shorten the pressing time of the layered wood composites by modifying the pressing system. The idea is to press at the beginning of the composite production process with as low veneer number as possible, and add the face veneers successively, to reach the final veneer number.

The above mentioned modified technique of layered wood composite pressing can lead to composites with modified properties. The expected change is e.g. different density profile (Figure 1). Outer layers in conventionally pressed composites are more densified, as the heat acts there earlier and for longer time, when in case of the modified pressing technique, all the subsequent outer layers will be exposed for heat for comparable time.

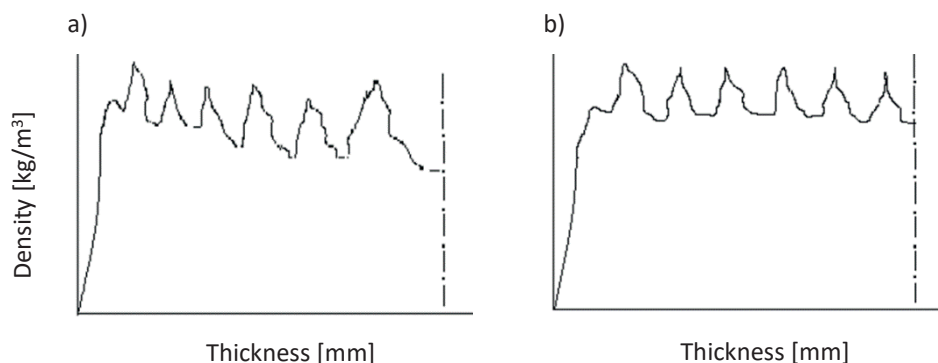


Figure 1: The expected differences in composites' density profile: conventional pressing technique (a) and modified pressing technique (b); half of thickness shown only.

The mentioned research will be preceded by preliminary tests, which will be focused on heat transfer dynamics through the veneers with varied parameters: moisture content, density etc. These data are crucial to assume the pressing time for subsequent veneers.

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Investigation of periodical effect on modified woods outdoor colour change

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Keywords: heat treatment, weathering, colour change, scots pine, beech

The different wood modification processes give the opportunity to improve the applicability of wood materials. The most significant effect of heat treatment is the colour change due to the high temperatures applied. This phenomenon is mostly taken as a positive effect of the treatment, as dark brown shades are always popular and sought. The wood material is often preferred in outdoor using, nevertheless because of its aesthetic value. The appearance of wood is composed from the texture and the colour. But the latter one can be changed during the service life of the product due to exposure to sunlight, rain and temperature changes. The consequence of outdoor exposure is the well-known greying effect. In order to prove the applicability of a material for using outdoors, weathering tests are recommended. The heat treatment modifies the colour of the wood favourable, but the stability of the colour is a major factor of a product's value, thus it calls for special emphasises. For the better understanding of outdoor colour change of the wood material, we need diverse information about the process. One of such information is the effect of periodical effect of the weathering. The aim of the presented work was the investigation of the colour change of heat treated and untreated wood material for a one-year long period in a monthly resolution. It was a short-term investigation of the colour change, related to the exposure period (1 calendar month).

Heat treatment was made at atmospheric pressure in air medium at 200°C, where treatment time was 10 hours. Beech and scots pine wood was treated, and untreated samples served as control. The treated laths and the controls were fastened onto stands made of poplar wood, rather than metal or other wood material, in order to avoid contamination of the surface with metallic oxides, or extractives leached out of the wood. The laths were inclined by 45° and faced to the south. The test area was free from any shadow, thus the sun could irradiate the test material directly. The colour of the samples was measured on the face by a Konica Minolta CM-2600d device, using the CIELab system. The colour was measured right after the treatment. These colour values were considered as initial reference data (Start). The colour was measured every 30th day during the test period at 5 spots on each lath (4x5=20 data for each treatment combination at each time, on the

same spots). To be able to determine the effect of the investigation period (calendar month), every month new samples were placed at the stands, thus every sample was weathered only one-month long. In this way, we have the information about the weathering impact of each calendar month on the colour change. Two set of samples were placed outdoors every month, one set unprotected and another one under a glass sheet (protected samples) to protect them from the effect of rain (leaching).

As expected, total colour change was higher during the summer months and decreased continuously to the winter months (Fig. 1. and 2.). Leaching does not affect the protected samples, this might be the reason for the bigger difference between the highest and lowest total colour change values in August and November respectively. In case of protected samples staining as a result of moisture condensation was a common phenomenon, which had a negative influence on the variability of the results. A negative effect related to the variability of the unprotected samples was observed as well, namely the surface was attacked/damaged by hornets continuously, and thus the weathered surface disappeared in small spots.

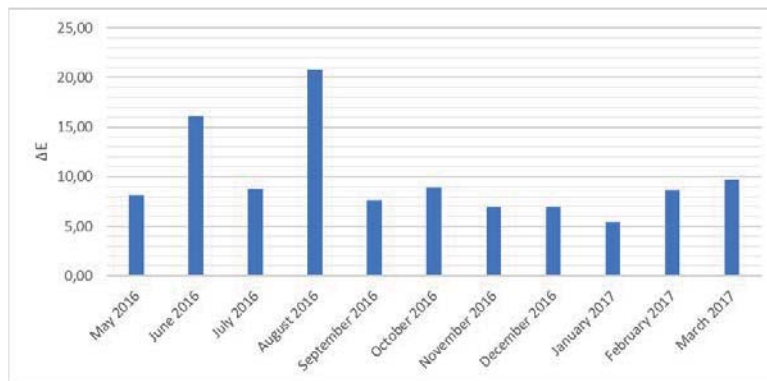


Figure 1: Effect of weathering impact of calendar months on the total colour change of heat treated beech wood.

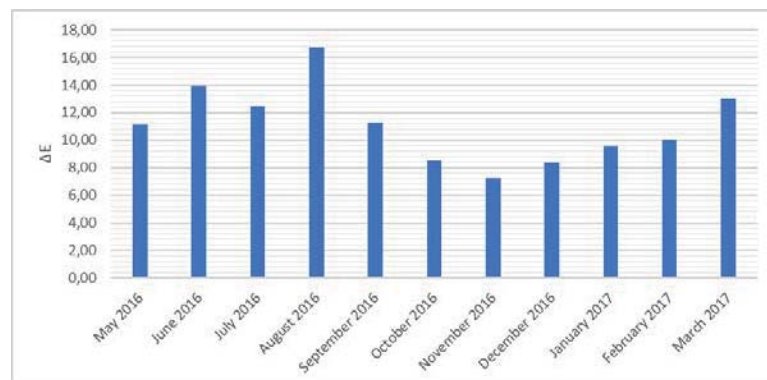


Figure 2: Effect of weathering impact of calendar months on the total colour change of heat treated beech wood under a glass sheet.

Nanoscale mechanical properties of wood: effects of heat treatment

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Keywords: nanomechanical properties, nanoindentation, atomic force microscopy, heat treatment

The wood cell wall is predominantly composed of cellulose microfibrils embedded in a matrix of lignin and hemicellulose. The cellulose microfibrils contain both crystalline and disordered or amorphous domains (Fengel and Wegener 1983). The size, arrangement of the cellulose fibrils and the proportion of crystalline cellulose are key factors determining the mechanical performance of wood materials (Lichtenegger *et al.* 1999). Hence, tracking the changes of wood cell wall constituents due to different modification procedures is essential to understand the mechanical response of the wood cell wall. We focus on environmentally-friendly thermal modification, which can significantly improve the physical properties of wood, including the durability and size stability (Bhuiyan *et al.* 2000).

Anatomical, structural and chemical properties of wood under different thermal modification procedures have been investigated (Navi and Heger 2004). However, the changes in the constituent behavior and the nanomechanical properties of wood cell wall have not been fully addressed, due to the rather complicated chemical and structural changes in the wood cell wall during modification.

In this study, *Eucalyptus nitens* species and Pine wood were heat-treated at 160°C and 210°C with different relative humidity conditions. Comparative analysis with atomic force microscopy (AFM) and nanoindentation were used to determine the variability in nanomechanical properties within the cellular structure before and after thermal modification. This comparative method allows us to identify and measure energy dissipation processes at the nanoscale (Garcia *et al.* 2013).

According to nanoindentation measurements, we observed that the wood samples become highly heterogeneous after heat treatment: In some regions, the Young modulus is high (~28 GPa), while in other regions it is low (~2 GPa). In comparison, the Young modulus only varies between 6 GPa and 18 GPa in the untreated state. A possible explanation is that the cellulose becomes spatially localized after heat treatment.

Based on our first results, we aim to use the capabilities of atomic force microscopy (AFM) and nanoindentation to obtain a deeper understanding of the effect of thermal treatment, relative humidity and compression on wood cell wall structure and also provide scientific guidelines for designing novel composites.

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Acknowledgments:

This research was supported by the German Ministry for Science and Culture of Lower Saxony (MWK), Georg-Christoph-Lichtenberg grant for 'Material Research Wood'.

Color and wettability changes of heat-treated wood finished with UV-rad cured coating after artificial weathering

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Keywords: heat-treated wood, visual appearance, wood surface wettability

Wood of European ash (*Fraxinus excelsior* L.) was thermally modified according to the state-of-the-art industrial production standards at 192 °C, 202 °C and 212 °C, which are referred as Mod-192, Mod-202 and Mod-212 respectively. Technical details regarding the modification procedures implemented are presented in detail in a previous work (Herrera et al., 2015). Wood surfaces were sanded before coating with an orbital sander in a sequence of sanding paper gradation from P150 to P320 to assure minimal surface roughness and improve adhesion with the coating. An industrial UV-hardened coating (C) was applied by using industrial processes. Coated wood samples after finishing and conditioning were exposed to the accelerated weathering test in order to simulate a combined effect of temperature, solar radiation and precipitation on the surface properties. Color changes were quantified with hyperspectral imaging by generating surface color maps in CIE Lab coordinates. The results point out an acceptable photostability of the heat-treated wood. Changes in wettability were determined by contact angle. Complementary parameters were extracted from the median values of hyperspectral histograms to summarize the overall color variation (ΔE) as well as to quantify the chromatic strength (C^*) and hue angle (h^*). The analysis of ΔE revealed a peculiar trend, in which the color stability varied according to the following sequence: Untreated > Mod-212 > Mod-192 > Mod-202 (Table 1).

Table 1: Optical parameters before and after weathering test.

UV-hardened (C)	Untreated			Mod-192			Mod-202			Mod-212		
	S_o	Δ	S_w	S_o	Δ	S_w	S_o	Δ	S_w	S_o	Δ	S_w
ΔE	4.76			9.64			17.48			7.41		
C^*	71.78	75.80		58.4	71.46		48.14	51.73		34.5	38.57	
h^*	18.19	25.96		13.33	13.48		11.72	11.88		6.64	6.12	
Gloss	26.79	31.28		28.58	27.92		15.32	15.72		7.91	9.34	

The interactions between reference liquids and uncoated surfaces were assessed by measuring a sessile drop contact angles. Three liquids of different molecular properties were used in order to determine the thermodynamic state of each sample configuration, including no-coated (control) samples (Table 2).

Table 2. Liquid-solid interactions before and after weathering test.

Coating product	Evaluation	Untreated		Mod-192		Mod-202		Mod-212	
		S_o	S_w	S_o	S_w	S_o	S_w	S_o	S_w
no-coated control	θ_w	66.31	-	99.96	-	99.19	-	97.20	-
	θ_E	29.40	-	30.10	-	37.90	-	35.20	-
	θ_D	19.70	-	27.60	-	23.50	-	25.30	-
	γ_{S1}^P	44.88	-	0.02	-	0.02	-	0.01	-
	γ_{S1}^d	6.78	-	52.79	-	52.36	-	51.72	-
	γ_{S1}^T	51.66	-	52.81	-	52.39	-	51.72	-
UV-hardened	θ_w	72.12	64.80	96.38	95.41	75.88	70.40	81.89	73.30
	θ_E	52.56	49.87	62.84	56.32	57.89	53.87	60.49	59.45
	θ_D	48.57	47.10	41.88	41.86	40.64	47.40	43.75	46.80
	γ_{S2}^P	31.40	10.99	38.25	0.31	34.50	8.23	34.12	6.76
	γ_{S2}^d	7.64	30.90	0.21	39.70	4.89	31.22	3.16	30.91
	γ_{S2}^T	39.05	41.90	38.47	40.01	39.39	39.45	37.29	37.67

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Acknowledgments:

The authors are thankful for the financial support from the Basque Government through project IT1008-15 and scholarship for young researchers training.

Surface behaviour of poplar and spruce wood after immersion in extractives solution achieved from thermally treated Hungarian oak

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Keywords: Hungarian oak, extractives, contact angle

The extractives change in quantity and quality composition after the thermal modification of wood (Esteves and Pereira 2008). Considering our scientific knowledge, the use of these compounds as natural preservatives to improve the surface behaviour of non-durable wood species, has been poorly investigated (El Bakali et al. 2014). To better understand the effect of the Hungarian oak extractives (*Quercus frainetto* Ten.) on the behaviour of colour and wettability of wood surface, samples of Poplar and Spruce were used. Two m³ of defect free H. oak boards with dimension of 35 mm by 250 mm by 2500 mm were supplied by a local manufacturer in Basilicata Region in Italy, and then thermally treated at 180°C and 200°C for three hours under vacuum condition. After thermo-treatment, random quantity of boards was milled and reduced in powder. Extraction was carried out with a mixture of ethanol:water (70:30 v/v) using a Dionex extractor by the extraction system ASE at 100 bars of the pressure in 2 cycles and 110°C. A total of ten samples of poplar and spruce wood with dimension of 5 mm by 50 mm by 50 mm were immersed for 10 seconds into each type of extractives solution previously prepared. Then, the samples were left for 168 hours under artificial accelerated weathering. The progress of surface wettability was monitored by contact angle measurements of water test liquid for 120 sec. The colour change of the specimens were measured using CIE L*a*b* colour space system. The analysis, performed before and after immersion in the extractive solution and accelerated weathering test, highlighted that the ΔE of spruce and poplar wood surface increased with extractives obtained at highest temperature (Fig. 1). Interestingly, after 168 hours of ageing the average value of ΔE , mainly for spruce surface, was

only slightly changed with extractives obtained at highest temperature and further investigations are being carried out to clarify this behaviour. In addition, an increase of hydrophobicity on both wood species was depicted when extractive solution was used. However, the effect of extractives disappeared after accelerated weathering highlighted a possible leaching of water soluble extractives compounds due to the water during aging test.

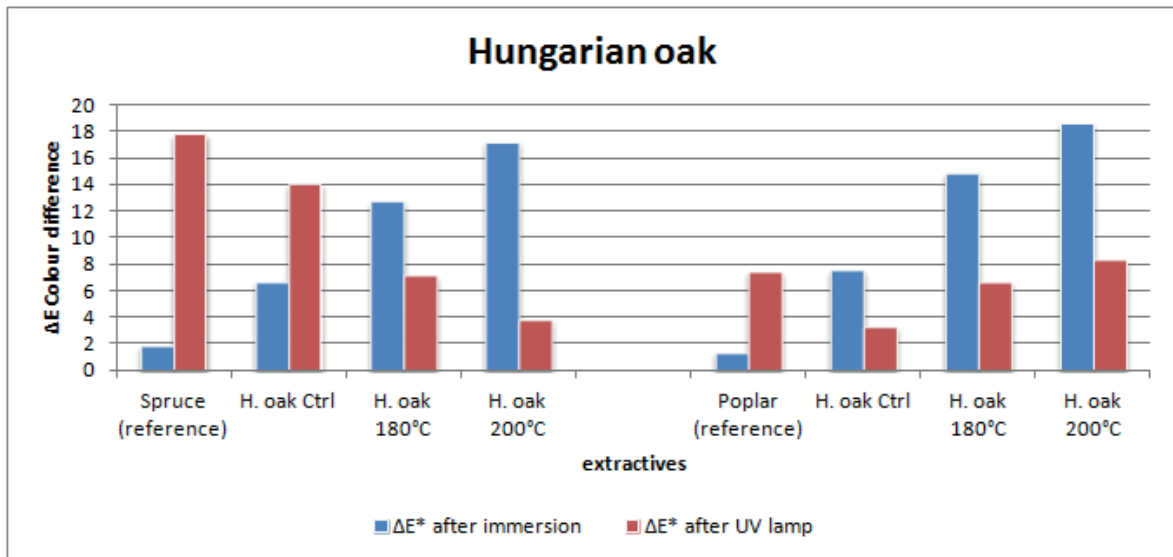


Figure 1: Total colour difference before and after accelerated aging



Figure 2: Hungarian oak extractives obtained at different temperature.

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Studies of the gluability of the pine wood veneers after TM modification with the use of PVAC adhesives

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Keywords: pine veneer, densification, PVAC adhesive, glue line, strength

Thermally compressed wood (TM) is being used to an increasing extent in furniture production, especially in the USA and in Scandinavian countries. There is interest in the use of thermally modified veneers in the production of high-quality plywood.

The manufacturers of the wood based materials e.g. plywood are under increasing interest to reduce formaldehyde emissions. From the initiative, first of the IKEA Comp. - the world's largest furniture producer – they expect the reduction of formaldehyde emissions less than 0.5 E1 level. These requirements can't be obtained with the urea-formaldehyde resins (UF) widely used for plywood and veneering processes (Bekhta, Niemz, Sedliačik 2012). Recent investigations by JOWAT Comp. from the Germany showed that these requirements are met to the greatest extent using dispersion adhesives in particularly PVAC binders (Hänsel, Linde 2012). Compared to other dispersion adhesives, glue lines of EPI adhesives can compensate for the high stresses of the materials.

The aim of this paper was the determination of the gluability of thermally modified pine wood veneers with the use of PVAC adhesives with the various water-resistance parameters (Table 1).

Table 1: Properties of the adhesives

Trade name of the adhesives	Base	Durability class [acc. to the PN EN 204 standard]	Density [g/cm ³]	Apparent viscosity [mPa·s]	Open assembly time [min]
Jowacoll 104.20	PVAC	D2	1.07	10.500	8 - 11
Jowacoll 103.05		D3	1.08	11.000	5 - 8
Jowacoll 107.20		D4	1.05	6.000	10
Jowacoll 102.49 (with filler)	PVAC + hardener (isocyanate)	D2	1.50	11.000	8 - 12
Jowacoll 102.49 + 195.60 (EPI)		D4	1.50	11.000	8 - 12

Pine wood veneer sheets were pressed using the anti-adhesion foils individually in the hydraulic press with the following parameters: unit pressure 1 MPa, temperatures 20, 120 and 180°C, time 2 min. The parameters of the thermal densification were taken from the investigations of Bekhta, Marutzky (2007). To calculate the compression degree of the veneer sheets, the veneer thicknesses were determined at 5 measuring points before and after pressing. After 24 hrs conditions adhesives with the use of roller in the amount of $180 \pm 5 \text{ g/m}^2$ were applied. Bonding process of the 3-layer plywood in the laboratory press under parameters: pressure 1 MPa, temperature 20°C and time 10 min was carried out. The samples were stored for 7 days under normal conditions at a temperature of $20 \pm 2^\circ\text{C}$ and $65 \pm 5\%$ relative humidity, and test specimens were then prepared acc. to the PN EN 314-1 standard). The shearing strength was determined using the Schopper ZDM 2.5/91 test machine at velocity of 5 mm/min was set. After measurements wood failure percentage coefficient (WFP) was estimated.

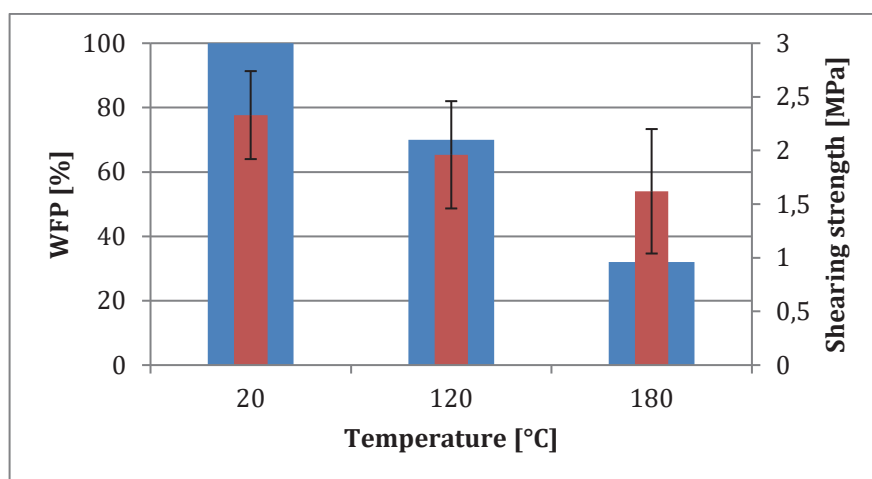


Fig. 1. Influence of the pine veneers thermal modification on the shearing strength of glue lines from PVAC Jowacoll 102.49 adhesive in plywood

It was stated among others, that relatively high shearing strength values were achieved with relatively high WFP factor (80-100 %) for gluing thermally modified veneers with the use of non-filled PVAC dispersion adhesives. The addition of fillers to the PVAC dispersion adhesives leads to a substantial reduction in the shearing strength of the plywood produced by thermally modified veneers. An addition of isocyanate crosslinker to the dispersion adhesive in a significant manner increase the shearing strength of glue lines in plywood.

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Impact of selected modification systems on elasto-mechanical properties of wood

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Keywords: chemical modification, elasto-mechanical properties, DMDHEU, melamine-formaldehyde resin, phenol-formaldehyde resin

This study deals with the impact of chemical modification on elasto-mechanical properties of Scots pine (*Pinus sylvestris* L.) and beech (*Fagus sylvatica* L.). The elasto-mechanical properties examined were impact bending strength, determined by impact bending tests (DIN 52 189-1 1981), tensile strength and work to maximum load in traction, determined by tensile tests (DIN 52 188 1979). The modification agents used were one melamine-formaldehyde resin (MF), one low molecular weight phenol-formaldehyde resin (Phenol 1), one higher molecular weight phenol-formaldehyde resin (Phenol 2) and one dimethylol dihydroxyethyleneurea (DMDHEU).

Special attention was paid to the influence of the solution concentration (0.5 %, 5 % and 20 %).

With an increase of the concentration of each modification agent, the elasto-mechanical properties decreased as compared to the control samples. Especially impact bending strength was decreased greatly by modifications with the 0.5 % solutions of each agent (by 37 % to 47 %).

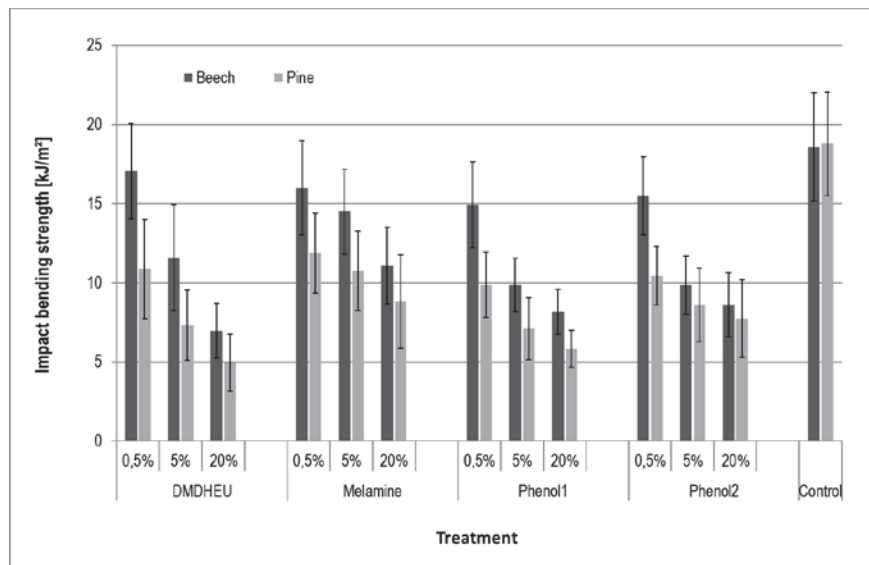


Figure 1: Impact bending strength [kJ/m²] of treated and untreated pine and beech

Modification with DMDHEU resulted in the highest over-all reduction of the elasto-mechanical properties examined. In general, the mechanical properties of pine were more negatively affected than beech.

Investigation of tropical wood modification under hydro-mechanical loadings with digital image correlation and X-ray microtomography

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Keywords: fracture, hydromechanical, X-ray microtomography

Wood-based materials are very sensitive to the effects of climatic loadings (temperature and hydric variations), during their service life. In this case, the wood modification due to these impacts can also compromise the durability of timber structures. The present work investigates the cracking of tropical species from the Gabonese forest, such as *Milicia excelsa* (Iroko), *Aucoumea klaineana* (Okoume) and *Pterocarpus soyauxii* (Padauk), in order to improve their sustainability. The experimental setup is composed of an electronical testing machine (Fig. 1 b), a wooden specimen (Fig. 1 a) with an Arcan steel system (the set constitutes the Mixed-Mode Crack Growth or MMCG developed by Moutou Pitti *et al.* 2011) is presented.

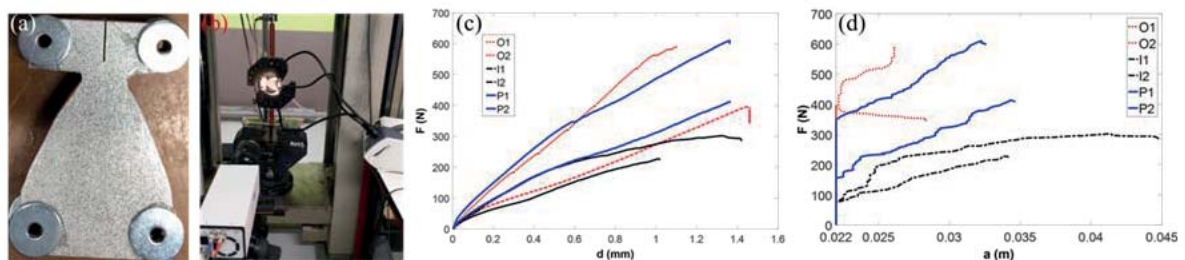


Figure 1: Wood MMCG specimen (a); experimental setup (b); load-displacement curves (c); load-crack length curves (d).

The displacement fields (Fig. 1 c) and deformation are measured by the Digital Image Correlation (DIC) method (Mété *et al.* 2013). The advance of the crack tip at different loading stages is recorded (Fig.1 d). These results are obtained from the displacement and strain maps (Fig. 2) given by the camera (Fig. 1 b). Preliminary results show important information about the fracture toughness of these tropical woods in constant and variable environments. Simultaneously, in order to follow the three-dimensional crack growth process of these tropical species, the X-ray microtomography

(XMT) method is performed (Mayo *et al.* 2009). Fig. 3 (a) and (b) show the 3D image acquisition protocol by MXT.

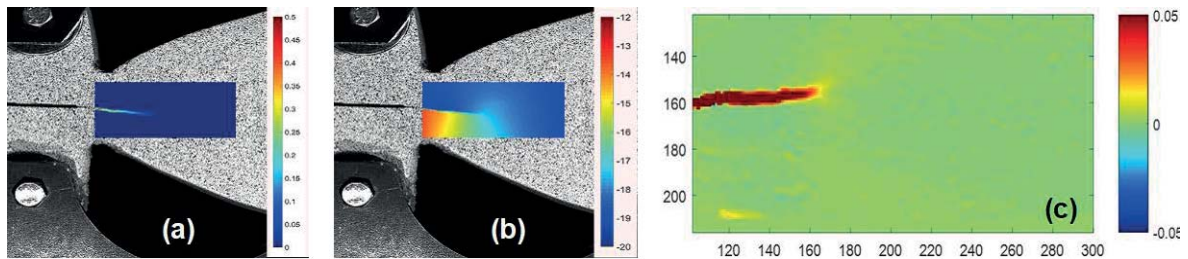


Figure 2: Displacement maps (a); strain maps (b); Crack evolution (c).

The tested specimen is cut according to the direction of cracking initiated by a pre-cut of 22 mm (b). The wood sample to be scanned is then introduced into the microtomograph chamber (b). After reconstruction, a 3D volume is obtained from several 2D scans at 4 μm resolution (Figs. 3 (c) and (d)). Images on healthy and cracked samples were taken under dry and wet conditions, respectively. Fig. 3 (d) shows the ability of the MXT to detect and track internal crack growth in tropical timber.



Figure 3: Image acquisition protocol by: (a) - scanned specimen; (b) - 3D cross-section of Padouk under wet conditions: specimen with a 22 mm pre-cut; (c) - crack path (d).

In the coming works, the MXT method will be applied to study the impact of the density and the spatial variability details observed on Fig. 3 (c) and (d) on the mechanical behaviour of wood and wood modification.

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Acknowledgments:

The authors wish to strongly acknowledge the French National Agency of Research for its financial support of this work through the project CLIMBOIS N° ANR-13-JS09-0003-01 labeled by ViaMeca, and the COST Action FP1407 for the conference.

Acoustic Emission Technique to monitor real-time wood fracture properties in room temperature

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Keywords: wood; fracture properties; acoustic emission; data clustering; crack tip growth

Nowadays, environment effects, and in particular the reduction of greenhouse effect, are one of the most important issues that are of great concern to the research community. As per the Kyoto protocol and the COP 21 meeting, the use of wood in civil engineering structures reminds one solution for the regulation of these effects by minimizing both CO₂ emission and the use of grey energy. Since the wood mechanical behaviour is quite complex due to its anisotropic constitution, the coalescence of various defects (nodes, orientation of annual rings, ...) during its service life, the increasing use of wood as structural material in civil engineering is a major challenge, and needs to increase scientific efforts in understanding the mechanical behaviour of timber structures.

In order to bring some responses to these scientific problems, the JCJC 2013 CLIMBOIS research project (Moutou Pitti *et al.* 2014) is dealing with the effects of climatic and mechanical variations on the durability of notched timber structures. Material cracking is one of the most important factors involved in the collapse of structures. However, if the crack initiation is detected earlier, and the monitoring of the crack propagation within the materials is under control, the structural integrity of buildings can be easily evaluated by sending alerts. Within the framework of this research project, the study carried out herein is devoted to both an identification of failure mechanisms in wood material, and an evaluation of the crack length evolution during fracture tests. The proposed methodology includes fracture tests under constant environmental conditions as well

as statistical and probabilistic analysis of the acoustic emission (AE) results (Diakhate *et al.* 2017). Fig.1 shows some results of mode I fracture tests.

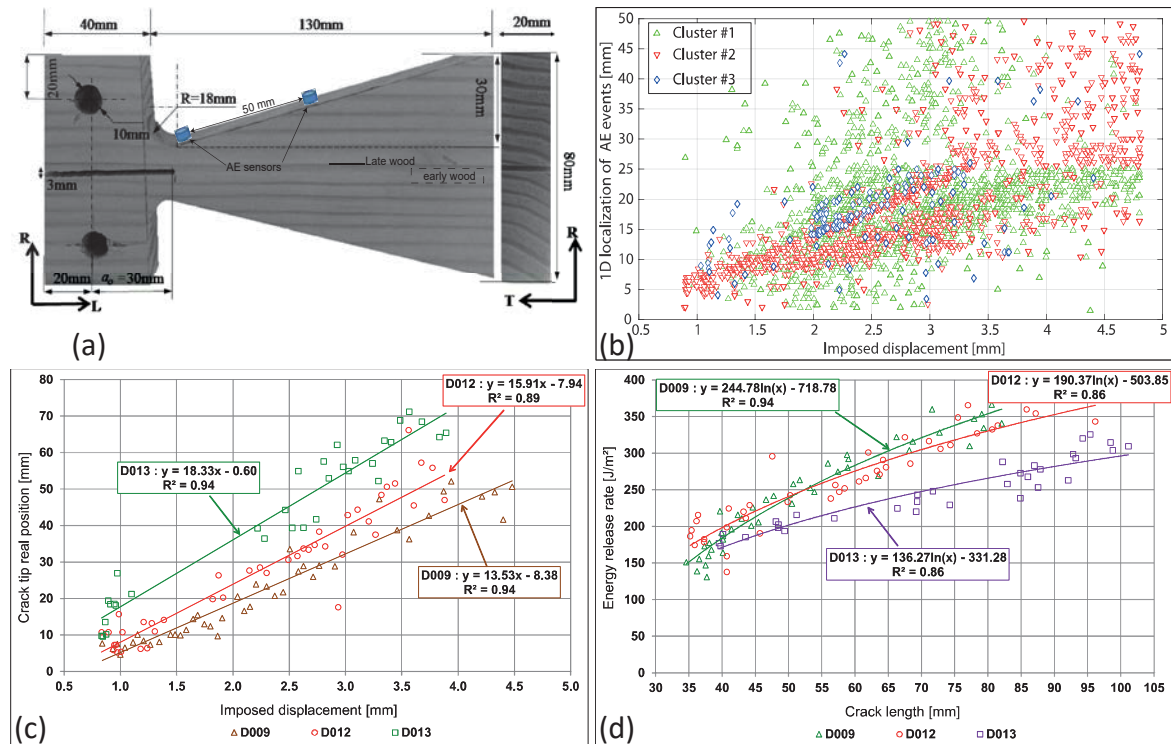


Figure 1: Mode I fracture test on wood material: (a) Modified DCB specimen – (b) Cluster analysis of acoustic emission activity within wood material – (c) Monitoring the crack tip propagation – (d) Energy release rate versus crack length

The results show the ability of the proposed technique to identify the crack tip advance. In the coming work, the proposed methodology will be generalised to different moisture content rates and mixed mode configurations in order to investigate the behaviour of timber structure submitted to outdoor conditions coupled with complex loadings.

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Acknowledgments:

The authors wish to gratefully acknowledge the French National Research Agency for funding this work through the research project CLIMBOIS N° ANR-13-JS09-0003-01 labelled by ViaMeca. Also thanks to the COST Action FP1407 for their financial support to attend this conference.

Performance of 3-layer composites with densified surface layers of *Nothofagus* species of Southern Patagonian forests

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Keywords: beech, densified, hardwood, mechanics, wood modification

The development of innovative, high added value wood products doesn't always start in the laboratory, but rather, in the forest. Today, Slovenian and Argentinian partners are finding new ways to produce high quality products from the forest. They have found that they are strongly compatible and complement each other in an effort to integrate forest management and wood processing technologies.

Currently, this effort is focused on the valorisation of *Nothofagus* wood of Southern Patagonia (Tierra del Fuego and Santa Cruz provinces) of Argentina. These trees are currently harvested from native, unmanaged forests (Gea-Izquierdo et al. 2004). However, foresters in the region are looking forward and promoting forest management as a sustainable way to produce high quality lumber (Pastur et al. 2009). The trees produced in these managed forests are encouraged to grow quickly, which yields a high percentage of low density wood. This, in combination with the traits of *Nothofagus* wood itself, leads to wood with mechanical properties that are often inadequate for many structural purposes (Puettmann et al. 2015).

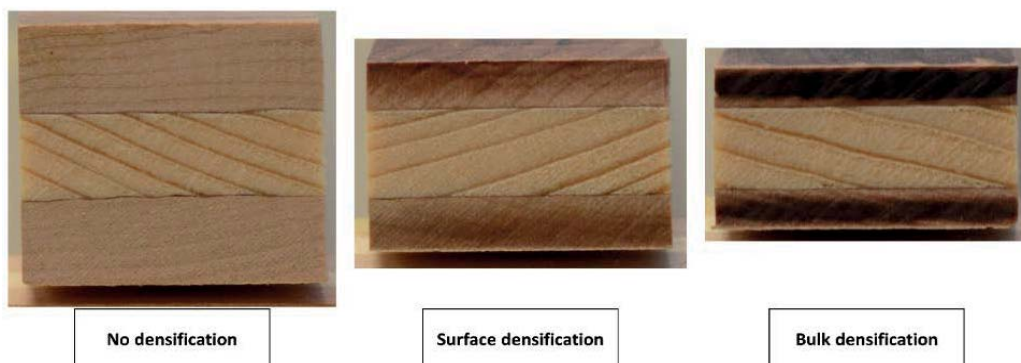


Figure 1: 3-layer composites made with *Nothofagus* outer laminates and a spruce core. Non-densified control composites (left), 3-layer composites using surface densified outer laminates (middle), and 3-layer composites using bulk densified outer laminates (right).

The poor mechanical properties of low-density wood can be improved by wood densification techniques (Kutnar et al. 2009). By using these techniques, low density and commercially less important wood species can be modified into high performance and high value products. In this study, a simple application of densified wood in a 3-layer laminated composite was created. The composites had densified wood in the two outer layers, and a layer of untreated low-density wood in the core (Fig. 1). The objective of this study was to apply these bulk and surface densification treatments to two species of *Nothofagus* wood from Southern Patagonia, improving their mechanical properties (Fig. 2).

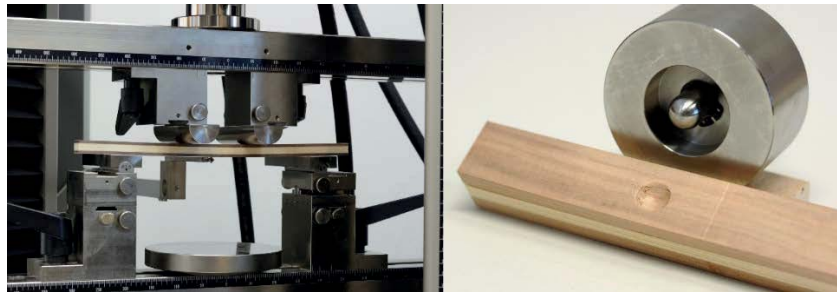


Figure 2: Four-point flexure test setup with extensometer arm engaged (left) and hardness test fixture and tested specimen (right).

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The activity of moulds on wood surfaces modified with laser

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Keywords: beech, laser, surface modification, moulds

A carbon dioxide (CO₂) laser irradiation method uses a mixture of three gases: CO₂, N₂, and He. It can be applied for technological treatments of wooden materials (cutting, engraving, drilling, marking, etc.) and also for modification of wood surfaces. The aim of this work was to use a CO₂ laser method for increase the resistance of wood against moulds. Laser modifications were carried out on the top surfaces 50 mm × 20 mm of beech (*Fagus sylvatica* L.) wood samples (50 mm × 20 mm × 5 mm = longitudinal x radial x tangential). Before starting the laser modification processes: (1) all four side surfaces of each sample were brushed with epoxy resin CHS-Epoxy 324 as a protection against water uptake from agar soil during the mould test, (2) the top surface of each sample was sanded with 60 and 120 grain-size sandpapers, and (3) each sample was conditioned at a temperature of 20 ± 1°C and a relative humidity of air 60% for constant moisture app. 12%.

Modification of beech samples was performed in a laser equipment – which consisted of: (1) a sealed CO₂ laser tube (Reci Laser Technology CO., Ltd, Beijing, China) with an outgoing beam diameter of 8.0 mm, wave length of 10.6 µm and a maximum output power of 100 W, (2) a CNC positioning table system (Formetal Piesok, Slovakia) which allowed laser head positioning and raster scans of the laser beam, and (3) a special PC control system. The samples were placed away from the lens focus at a distance of 127 mm below the focus point. The unfocused beam diameter on the top surface of each sample was 10 mm. The laser beam output power was set on 45 W in CW mode. Totally eight irradiation doses from 7.8 to 75 J/cm² were used.

The mould test of laser modified and reference beech samples was performed with microscopic fungi *Aspergillus niger* and *Penicillium brevicompactum* in Petri dishes according to the Standard STN 49 0604 (1980), testing four samples in each series. The growth activity of moulds (GAM) on the top surfaces of samples was determined on a scale from 0 to 4, such that 0 = no mould on the surface; 1 = mould up to 10 % of surface; 2 = mould up to 25% of surface; 3 = mould up to 50% of surface; and 4 = mould more than 50% of surface.

The activity of *Aspergillus niger* on the top surfaces of beech samples did not apparently drop due

to the increased laser irradiation dose from 7.8 to 18.8 J/cm². However, its activity significantly decreased at higher irradiation doses from 25 to 75 J/cm² – for example for the highest irradiation dose (75 J/cm²) the GAM in the final 28th day of test was a minimal, i.e. app. 1 (Fig. 1). In summary, on wood surfaces having an evident charring layer occurred only a slight and infrequent growth of this mould species. On the other hand, the growth of *Penicillium brevicompactum* was not apparently decreased even due to the highest laser irradiation doses (37.5 and 75 J/cm²) when the modified surfaces were apparently covered with a charring layer (Fig. 1).

Generally, the laser modification of beech wood surfaces had only a mild or no inhibition effect against the growth activity of moulds. The highest irradiation doses had inhibition effect against the mould *Aspergillus niger*, but no against the mould *Penicillium brevicompactum*.

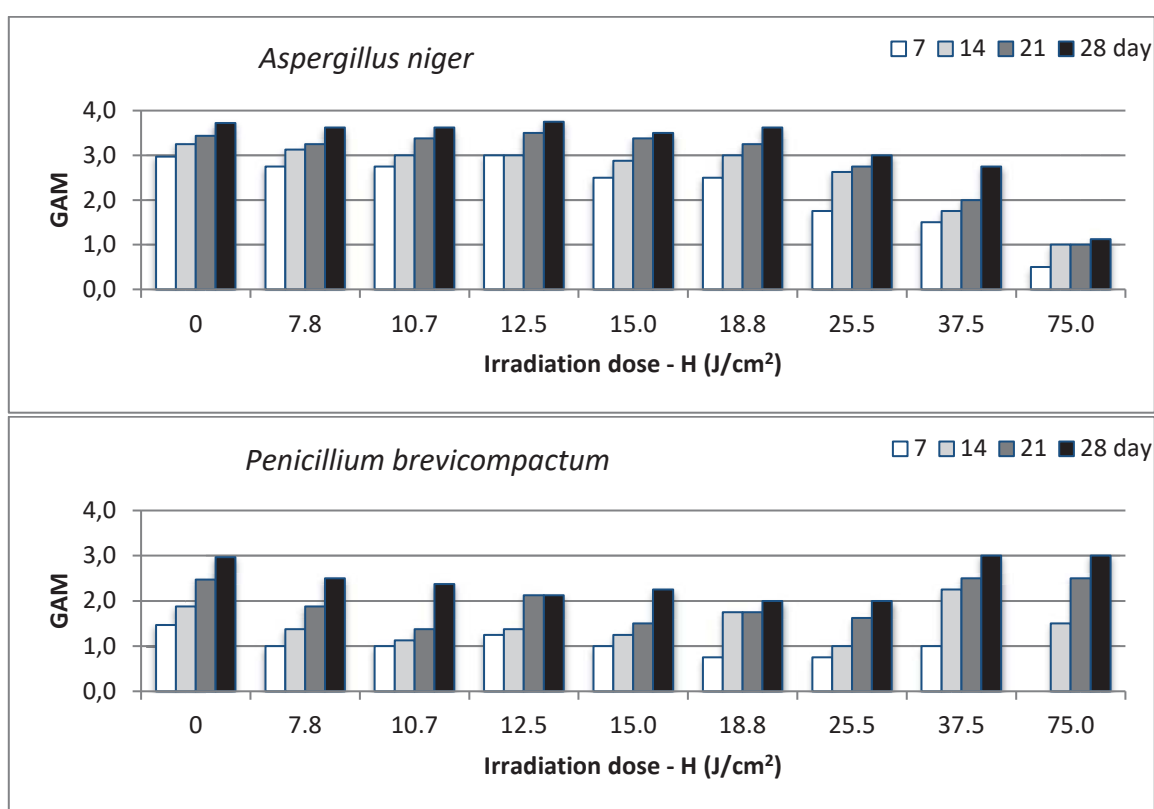


Figure 1: The growth activity of moulds (GAM) on surfaces of beech wood modified with CO₂ laser.

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Acknowledgments:

The authors would like to thank the Slovak Research and Development Agency under the contract No. APVV-0200-12 for financial support.

Do extractive compounds of thermally modified woods play an important role in the decay and termites resistances of these modified materials? A preliminary study.

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Keywords: antifungal activity, ash wood, extractive compounds, termite resistance, thermal modification

Thermal modification processes have been developed to increase the biological durability and dimensional stability of wood. However, the reasons of the decay resistance improvement of heat treated wood are still not very well known. This durability improvement of heat treated wood can be explain by several reasons, as the high hydrophobic behaviour (Weiland and Guyonnet, 2003), the polymer chemical composition modifications (Vallet *et al.*, 2001), the hemicelluloses degradation (Hakkou *et al.*, 2006) and the generation of new extractive substances (Lekounougou *et al.*, 2009).

The aim of this paper was to evaluate the anti-fungal and anti-termite activities of extractives compounds from heat treated ash woods according the modification process intensity. All of the tests were carried out in the laboratory with two different complementary research materials. The main research material consisted of ash (*Fraxinus excelsior* L.) wood thermally modified during 2 hours, at temperatures of 170, 200, 215 and 228 °C, under ThermoWood® Process. The reference material was untreated ash wood for decay and termite resistance screening tests. Each treated and untreated wood sample were extracted with water or acetone. The extractives contents were determined for each treated and control wood sample. One part of the obtained extracts was blended with malt agar block test [1200 µL, C= 2.5 % m/m in acetone, in 10 mL of Malt-Agar medium] in order to investigate their anti-fungal properties. To determine the inhibition

effectiveness of extractives, two different fungi were selected: *Coriolus versicolor* (white rot) and *Poria placenta* (brown rot). Fungal activity was carefully observed for the duration of seven days. The other part of extractives was impregnated within Whatman papers [70 µm, C= 2.5 % m/m in acetone, on a Cellulose paper of 2.5 cm-diameter] and expose to termite attacks. Finally, extractives were analysed by GC-MS and their number of chemical components and their respective quantity are related to their anti-termite and antifungal activity levels.

First results show that anti-fungal activity of heat treated ash wood extracts vary according to heat treatment intensity and the solvent used during the extraction process (Figure 1). It appears clearly that untreated and treated ash wood extracts are more efficient against brown rot than white rot growing.

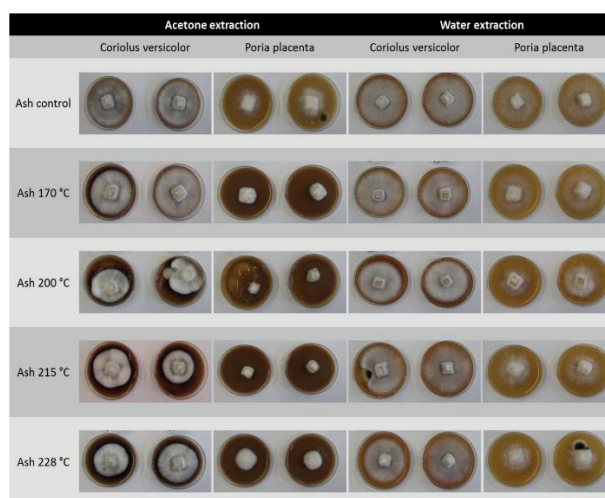


Figure 1: *Coriolus versicolor* and *Poria placenta* growing according to the inhibition of untreated and heat treated ash wood extracts (1200 µL), after seven days.

However, the anti-termite activity of heat treated ash wood extracts is not really significant. Finally, GC-MS analyse highlights that almost all of the original acetonic extractives disappeared and new compounds were formed resulting from degradation of hemicelluloses and lignin. These included monosaccharides and their dehydration products, as well as syringaldehyde, as the most prominent lignin derived compounds and generally the major detected component according to the TIC. Further studies involving investigation of toxicity and fungal inhibition properties are needed to draw conclusions on the specificity of such phenolic compounds.

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In-situ SEM / TEM fracture tests on (modified) tracheids of pine latewood

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Keywords: fracture, toughness, DMDHEU, heat treatment

There is evidence that intracellular failure occurs at the interface between the S₁ and S₂ cell wall layer (Delorme and Verhoff 1974; Côté and Hanna 1982; Gindl and Teischinger 2003). However, up to now, the fracture mechanisms of wood cell walls remain unexplored. Therefore, we have performed fracture tests on pine wood tracheid cell walls, because pine is one of the most common tree species in Germany and mostly made of tracheid cells. Our experimental setup enables us to create a crack in the cell wall and to observe the crack propagation in-situ with an electron microscope.

We have observed that crack propagation is not continuous, but intermittently starts and stops accompanied with a change in propagation direction resulting in a wave-like crack path (Fig. 1).

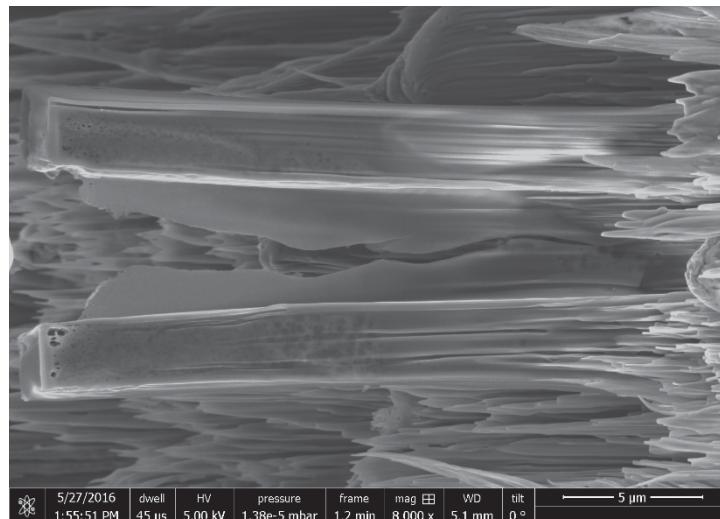


Figure 1: SEM image of an unmodified pine latewood sample after the in-situ SEM fracture test at the interface between the S₁ and S₂ tracheid cell wall layers.

We attribute this intermittent behavior to the abrupt change of the microfibril angle at the interface between the S_1 and S_2 cell wall layer and propose that the resultant increase in toughness is a driving force for the natural adaptation of the layered structure.

Modified wood

Heat-treated and DMDHEU modified wood have higher resistance against fungi but reduced tensile strength and fracture toughness relative to untreated wood (Boonstra *et al.* 2007, Majano-Majano *et al.* 2012, Verma *et al.* 2009; Xie *et al.* 2007). In order to understand the reasons for the poorer mechanical properties, we have tested pine sapwood that has been heat-treated or modified with DMDHEU. Based on our preliminary results we assume that both the heat treatment and the DMDHEU modification change the microfibril structure to such an extent that the crack inhibition as described above no longer occurs. In consequence the fracture toughness is reduced.

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Acknowledgments:

This research was funded by the German Ministry for Science and Culture of Lower Saxony (MWK), Georg-Christoph-Lichtenberg grant for 'Material Research Wood'.

Traditional wood finishing substances and their influence on surface roughness

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Keywords: traditional wood finishes, oil, wax, wood surface roughness

Aim: The purpose of the project was to assess the influence of traditional substances (Sandak *et al.* 2015) used for finishing wooden parquets and their influence on surface roughness. Tests were performed on 240 samples of oak and elm wood covered with natural beeswax, linen varnish (*Drewnochron*: 98% of linen oil and 2% drying agents) and contemporary synthetic parquet oil (*Bona Carl's 90*), additionally finished with wax oil (*Fiddes Hard Wax Oil*), in comparison with reference samples of antique wood of both species without finish.

Methods: The study of traditional finishing coatings included the assessment of their aesthetical effects and changes in wood surface properties. Therefore, macro and microscopic surface characteristics were considered, with the determination of Ra, Rz and Rmax with the Mar Surf PS1 device (in compliance with the requirements of DIN EN ISO 3274, measurement range from -200 µm to +150 µm). We studied the significance of finishing, wood species and measurement direction factors.

Results: Both visual assessment and roughness test results indicate that wax provides the best surface quality out of the traditional wood finishing methods (Rozanska *et al.* 2013). The influence of the finishing, wood species and measurement direction factors on roughness test results is significant (Fig.1-3). Roughness value is evidently higher for wood without finish and wood covered with varnish, when compared with wood finished with wax. The finishing of samples with wax significantly reduces roughness parameters (up to over 50%), as well as synthetic parquet oil and wax oil - to a lesser extent (by about 10-20%). The reduction of parameter values caused by varnish surface finishing is not statistically significant. The influence of the wood finishing substance on the differences between roughness measured along and across fibres (expressed in %) is most substantial in case of wax. In case of measurements across fibres, the percentage difference of change in parameters after covering with wax is two times higher than in case of oil coating, and the latter in turn is two times higher than after finishing with varnish. For wax finishing, the biggest

changes in surface roughness (in %) were observed for elm wood, and in case of other kinds of wood finishing, for oak.

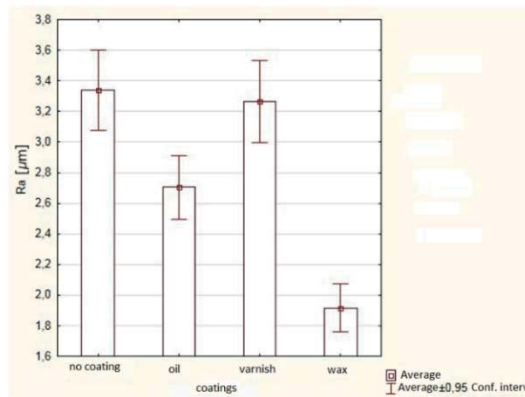


Figure 1: Influence of the finishing substance on the Ra factor values.

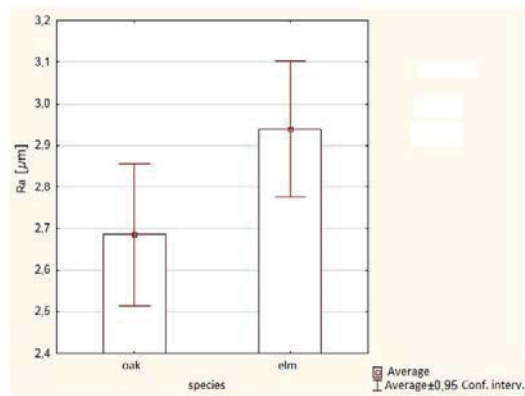


Figure 2: Influence of the wood species on the Ra factor values.

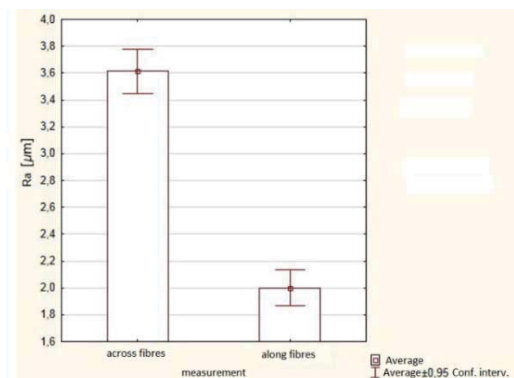


Figure 3: Influence of the measurement direction on the Ra factor values.

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Biobased phenolic resins for wood protection against fire

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Keywords: wood, lignocellulosic, resins, inorganic

Despite the desired environmental, mechanical and structural properties of wood and wood-based panels, they are vulnerable to failure during fire exposure. Hence, the search for new flame retardants currently, is gaining importance to reduce the fire hazards (Seo *et al.* 2016). In this context, the design of a new generation of appropriate fireproofing treatment of solid wood or based related products is a major issue. The addition of nanoscale additives is reported to produce a synergistic effect regarding the intumescent properties and features of the material. Besides, following the restrictions concerning certain halogenated flame retardants, the current market demand, tends to move towards non-halogenated inorganic compounds, such as silicates and clays.

On the other hand, lignocellulosic biomass has become a great economic issue in the context of the biorefinery, as feedstock of materials and polymers for the replacement for fossil resources (Fernández-Rodríguez *et al.* 2017). Among lignocellulosic products, lignin, which accounts for 10-30% of the lignocellulosic biomass (Maity 2015); and tannins, which is the second biggest natural source of phenolic compounds (Laurichesse and Avérous 2014), are attracting considerable attention lately.

Thus, the aim of this work was the elaboration of environmentally-friendly biobased phenolic resins for the replacement of phenol and formaldehyde present in traditional phenolic resins. The evaluation and comparison of their main thermal properties for the protection of wood and wood derivative products was carried out as well.

Different phenolic resins formulations were elaborated using lignin and tannins and glyoxal as phenol and formaldehyde substitutes. For the elaboration of the resins the lignin was chemically modified with glyoxal. On the other part a tannins solution was prepared as well using acetone (solvent) and hexamine (hardener). The introduction of an inorganic phase, by mechanical mixing, based on nanoclays, was tested as well. Following the resins synthesis, several thermal analyses

such as differential scanning calorimetry (DSC) and thermal degradation analysis (TGA) were performed.

Three different formulations of resins, namely resin A, resin B and resin C (table 1) were produced.

Table 1: Proportion of the mains resins solid components

RESIN	RATIO LIGNIN/TANNINS	INORGANIC PHASE (%)	MIXTURE CONDITIONS
A	1	0	12h, T=25°C
B	1	5	12h, T=25°C
C	1	5	12h, T=40°C

On the other side the TGA analysis was carried out and it proved that the addition of an inorganic phase to the resins provided with a lower thermal degradation (figure 1). Besides the mixture conditions seemed to have relevant importance in the introduction of the inorganic phase into the resins.

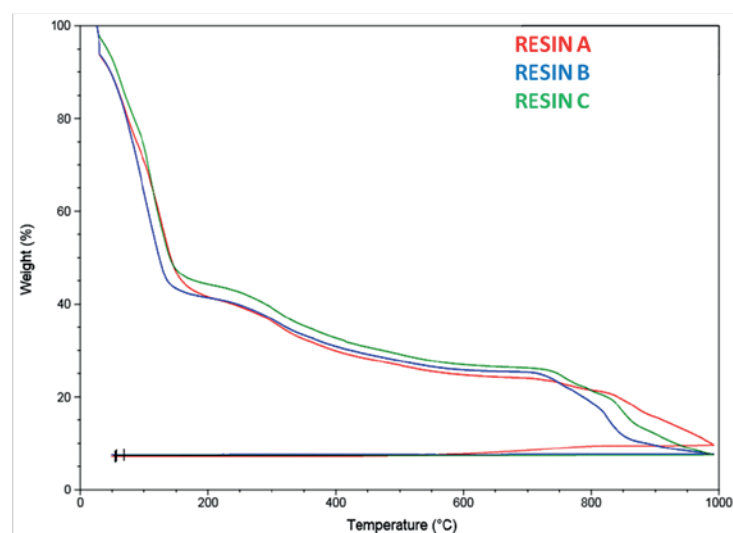


Figure 1: Thermogravimetric analysis (TGA) of the three resins formulations

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Acknowledgments: The authors thank the University of the Basque Country (UPV/EHU) and the University of Pau and Pays de l'Adour (UPPA), (Predoctoral fellowship- PIFPAU 15/01) for financially supporting this work.

The activity of bacteria on surfaces of wooden composites painted with acrylate coating with addition of silver nanoparticles

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Keywords: wooden composites, silver nanoparticles, bacteria, *Escherichia coli*, *Staphylococcus aureus*

Wooden composites with antimicrobial surfaces would reduce the risk of microbial infection spreading especially in healthcare facilities and public buildings. Commercial wooden composites without antibacterial treatment are often inhabited by bacteria (Vidholdová *et al.* 2015)

In this work we performed surface modification of three commercial wooden composites: (1) particleboard – PB (thickness 16 mm), (2) medium density fibreboard – MDF (thickness 18 mm), and (3) hardboard – HB (thickness 5.5 mm). Surfaces of these wooden composites (300 mm x 300 mm) were treated by sanding, and then with spraying using one-component acrylate water-soluble lacquer EM 1157-0050 (produced by Sherwin-Williams). It was applied in two spraying layers. The first layer was made with original lacquer, while the second one with lacquer treated with nanoparticles of silver in amount of 0.004, 0.012, or 0.02% w/w (weight of silver per weight of lacquer). Dispersion of silver nanoparticles (10 nm particle size “TEM”, 0.02 mg/mL in aqueous buffer, containing sodium citrate as stabilizer) was obtained from Sigma Aldrich GmbH., Germany. Painted wooden composites were placed into a curing chamber heated on a 50°C for 5 min.

The anti-bacterial resistance of the coated top surfaces of the PBs, MDFs, and HBs samples (50 mm x 50 mm) was evaluated against the gram-positive bacteria *Staphylococcus aureus* ATCC-25923 and the gram-negative bacteria *Escherichia coli* ATCC-25922. These bacteria we achieved from the Collection of Microorganisms at Department of Clinical Microbiology, Hospital in Zvolen. Samples of wooden composites were firstly cleaned with alcohol solution (8.8:1.2 mixture of ethanol and 2-propanol), again sterilized in autoclave, and then in Petri dishes inoculated with 0.1 ml of bacterial suspensions having density 0.5 of McFarland scale (1.5×10^8 CFU/ml). Incubation of the top surfaces of samples with bacteria at a temperature of 37 °C lasted 48 hours. Afterwards, bacteria were striped from tested surfaces using sterile swap and taken up in liquid culture medium for 48 hours.

Finally, bacteria were pre-inoculated from liquid medium into the sodium chloride diagnostic soil in Petri dishes. The anti-bacterial resistance of painted PBs, MDFs, and HBs in the diagnostic soil was assessed based on the bacterial activity (BA) valued from 0 to higher numbers in CFU/ml.

Results related to the increased anti-bacterial resistance of the top surfaces of painted wooden composites, having the silver nanoparticles in the top acrylate coating, are shown in the Table 1.

Table 1: Bacterial activity (BA) of *S. aureus* and *E. coli* on the top surfaces of commercial wooden composites (PBs, MDFs, HBs) painted with acrylate coatings containing different amounts of nano Ag particles.

Nano Ag [w/w %]	PB	MDF	HB
<i>Staphylococcus aureus</i> (CFU/ml)			
0	0.18	0.24	0.19
0.004	0.16	0.11	0.15
0.012	0	0.15	0.07
0.020	0	0.14	0.07
<i>Escherichia coli</i> (CFU/ml)			
0	0.10	0.17	0
0.004	0.16	0	0
0.012	0	0	0
0.020	0.03	0	0

The anti-bacterial properties of the silver-modified surfaces of all used commercial wooden composites were higher comparing to surfaces of the control wood composites without silver nanoparticles. Activity of the gram-positive bacteria *Staphylococcus aureus* decreased for PB from 0.18×10^8 up to 0.0 CFU/ml, for MDF from 0.24×10^8 up to 0.14×10^8 CFU/ml, and for HB from 0.19×10^8 up to 0.07×10^8 CFU/ml. Activity of the gram-negative bacteria *Escherichia coli* decreased for PB from 0.10×10^8 or 0.16×10^8 up to 0.0 CFU/ml, for MDF from 0.17×10^8 up to 0.0 CFU/ml, while for HB it was always 0.0 CFU/ml – *i.e.* also for the painted top surfaces without Ag biocide.

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Acknowledgments:

This work was supported by the Slovak Research and Development Agency under the contract No. APVV-0200-12. The authors would like to thank also to the COST FP1407, and to the company Sherwin-Williams Czech Republic.

Hydrophobicity of ϵ -caprolactone-modified wood materials

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Keywords: hydrophobicity, wettability, contact angle, ϵ -caprolactone, wood modification

Wood as sustainable biomaterial is used in many applications such as furniture, construction, interior design, decking, siding etc. However, there are still several undesired properties such as low dimensional stability and durability, which limit its end-use. Researchers have been used many methods such as chemical modification and thermal modification to improve dimensional stability of wood or wood-based materials (Hill 2006).

Surface quality of wood materials is a physical property influencing different processes including their finishing characteristics (Ozdemir *et al.* 2009). Wettability of wood-based materials is also an important issue and is affected by modification. During heat treatment, physical and chemical processes occur in layers near the surface that result in a modified surface with new characteristics (Follrich *et al.* 2006). Wettability is also an essential issue for adhesion. Wettability of wood based materials is usually evaluated by contact angle. Wetting quality of wood is influenced by many factors including macroscopic characteristics, surface quality of wood, processing temperature, and properties of adhesives (Lu 2003).

In this study, various wood materials were chemically modified with poly(ϵ -caprolactone) (PCL) which is one of the most promising synthetic biodegradable polymers. Wettability analysis was performed to evaluate hydrophobic characteristics of the materials. In addition, the color changes by the chemical modification was determined according to ASTM E1164-12 (ASTM 2017) standard. The results obtained in this study revealed that the chemical modification of the wood materials by PCL affected the wettability properties.

Fig. 1 shows chemically treated wood materials by ϵ -caprolactone. As can be seen from the figure, the chemical modification affected the colour of the wood materials. The modified wood materials were darker than that of the unmodified wood material.



Figure 1: ϵ -caprolactone-modified wood materials.

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Acknowledgments:

The authors would like to thank Istanbul University Research Fund for its financial support in this study (Project Number: 31014; 43150; 49525). The authors would also like to express thanks to Bursa Technical University Research Fund for its financial support in this project.

Natural adhesives from liquefied wood based resins and their applications

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Keywords: liquefied wood, adhesive, phenolic, epoxy, polyurethane

In the wood-based panel/composite industry, a wide range of adhesives is used. Particularly, the aminoplastic type adhesives, which are mainly used for the manufacturing of particleboards and medium density fibreboards, dominate the market. These products are obtained via polymeric condensation reactions of formaldehyde with aminic- or amidic-based chemicals (i.e. urea, melamine) (Čuk *et al.* 2015). Formaldehyde-based adhesives are harmful to human beings and environment due to the carcinogenic feature of formaldehyde. Thus, the scientists have investigated the possibilities of lowering the formaldehyde emission and producing formaldehyde-free adhesives due to strict environmental rules and increasing ecological awakening (Ugovsek *et al.* 2010). The eco-friendly adhesives from renewable resources could be a substitute for available synthetic adhesives. Several efforts were made by using feedstock such as lignin, animal proteins, soy, tannins and liquefied wood (LW) resins for the production of natural formaldehyde-free adhesives (Ugovsek *et al.* 2011).

Liquefaction/solvolytic of biomass is one of the thermochemical methods at which biomass dissolves in an organic solvent (e.g. phenol, polyhydric alcohols) in the presence of an acid or alkaline catalyst under atmospheric pressure. A variety of phenolic compounds such as guaiacol, coniferyl alcohol, vanillin, etc., were produced in the presence of phenol and acidic catalyst due to the cleavage of lignin (Acemioğlu and Alma 2002). Moreover, the cellulose and hemicellulose also undergo transglycosylation to form hydroxymethyl furfural compound in high yield. The furfural thus formed has been found to condense with phenol and formaldehyde through methylene bridges (Alma and Basturk 2006). The liquefied products can easily react with formaldehyde to form novolac type resin under acidic conditions or resol type resin under basic conditions. The condensation reaction of LW and formaldehyde is an efficient way to use the un-reacted free phenol remained after the liquefaction. The added formaldehyde did not react only with free phenol but also with LW components having reactive sites.

With the exception of the novolac and resol type phenol-formaldehyde adhesives from LW, several studies focused on the usage of LW based resin in different adhesives systems. Esteves *et al.* (2015)

used LW as an additive in the melamine-urea-formaldehyde and urea-formaldehyde resins. They found that the reduction of internal bond strength relatively low when 20% LW was used in formulation while there was a significant decrease in bond strength for 70% LW. They concluded that for lowering the formaldehyde content, a small amount of LW resin as a partial substitute could be used in the particleboard production. In another study, Ugovšek and Sernek (2011) applied pure LW resin alone as an adhesive system for the lap joints to investigate the influence of pressing time and temperature on the shear strength. It was found that the bond shear strength increased with increasing temperature while higher pressing times contributed to greater shear strength values of the bond. Kishi *et al.* (2006) prepared a novel epoxy resin from LW obtained into resorcinol with or without a sulfuric acid catalyst at high temperature. LW resin and epichlorohydrin were mixed under alkali conditions via glycidyl etherification to bring epoxy functionality to LW. They found that the flexural strength and modulus of elasticity of LW based epoxy resin were equivalent to those of the commercially available epoxy resin (i.e. diglycidyl ether of bisphenol A). Moreover, the shear adhesive strength of the LW based epoxy resin was higher than that of commercial one for plywood. Finally, in their study, Lee and Lin (2008) liquefied the two different wood species into polyethylene glycol/glycerol co-solvent with sulfuric acid. LW resin were mixed three types of isocyanate (poly diphenylmethane diisocyanate, adduct of toluene diisocyanate with trimethylol propane and trimer of hexamethylene diisocyanate), for the preparation of polyurethane (PU) type resins. The gel prepared PU resins and adduct of toluene diisocyanate with trimethylol propane had a proper gel time for processing. Moreover, the same isocyanate type showed better dry- and wet-bonding strength than others as a wood adhesive.

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Bio-based foams from renewable and sustainable polyols obtained via liquefaction of wood and other lignocellulosics

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Keywords: lignocellulosic, biomass, liquefaction, bio-polyol, bio-foam

Foams are one of the most useful three-dimensional materials with great versatility because they can be used in various forms in several applications such as packaging, cushioning and insulation. Polymeric foams involve polyurethane foam (PUF), polystyrene foam (PSF) and phenolic foam (PF). The production of these materials consists of varied processing conditions such as gaseous extrusion of molten polystyrene (PS) into foam while the reaction of selected polyols (polyether and polyester) and isocyanate with a blowing agent generate PUF. On the other hand, the PF manufacturing procedure needs the usage of a heat/acid reactive resole type phenolic resin, emulsifier, a volatile blowing agent and an acid catalyst (Pilato 2010). The major drawback of these petroleum-based foams is that they are typically manufactured from non-renewable, non-recyclable and non-biodegradable raw materials. Accordingly, lignocellulosics has become an attractive alternative for producing bio-based materials due to increasing environmental concerns about fossil sources.

Agricultural and forestry lignocellulosic wastes naturally involve biopolymers such as cellulose, hemicellulose, lignin and tannin which contain more than one hydroxyl group in the molecular chains. The liquefaction method is an effective way to convert these wastes into intermediate biopolyols which can be used as a raw material for the production of green polymers. Liquefaction of wood in the presence of organic solvents became a popular research area over the last decade. The polyhydric alcohols and phenol are the most used solvents while different organic solvents have been used in liquefaction reaction. So far, the liquid products obtained after liquefaction of wood have been applied to the preparation of novolac and resol type phenolic resins, Bakelite like molding materials, carbon fibers, polyesters, epoxy resins, resol type PF and PUF (Pan 2011).

It has been proven by many scientists that the liquefied biomass obtained using polyhydric alcohols such as polyethylene glycol (PEG), glycerol or their mixtures as a solvent could be used directly as polyols for the manufacturing of PUFs without additional treatment. These foams were produced in three different structures comprised the rigid type using polymeric methylene diphenylene

diisocyanate (PMDI) (Alma and Basurk 2003), semi-rigid type using polyaryl polymethylene isocyanate (PAPI) (Gao *et al.* 2010), and flexible type using toluene diisocyanate (TDI) (Zhang *et al.* 2012). Polyurethane foams are typically produced from polyols with hydroxyl numbers ranging between 300-500 mg KOH/g while biomass typically has hydroxyl numbers around 1500 mg KOH/g. Therefore, the amount of biomass used have to be kept under 33% to avoid the re-condensation reactions. The strong hydrogen bonding contained in lignocellulosics is a result of many hydroxyl groups present in the molecular chains of biopolymers. During liquefaction process, the polyhydric alcohols and acid catalysts lead the disrupting the hydrogen bonding with additional hydroxyl groups to struggle with the cellulose inter- and intra-chain hydrogen bonding and their large size impel the chains apart. These phenomena reduce the efficiency of the process causing a re-condensation reaction (Yaoguang *et al.* 1996).

PFs have attracted great attention due to its perfect fire resistant, low fire toxicity, high dimensional stability, and low thermal conductivity comparing the other foam types. There are a few studies regarding the preparation PF from liquefied lignocellulose-based resol resin. Lee *et al.* (2002) liquefied the wood into phenol in the presence of the sulfuric acid catalyst at a temperature of 150 °C under constant stirring and reflux. The liquefied wood was used to prepare resol resin by the reaction with formaldehyde under alkaline conditions. Then, the obtained resol resin was applied for the preparation of the phenolic foam. The resin mixed with the poly (ethylene ether) of sorbitan monopalmitate as a surfactant, hydrochloric acid as a catalyst, and diisopropyl ether as a blowing agent. The obtained foams showed satisfactory densities and compressive properties, comparable to those of foams obtained from conventional resol resin. The resol-type resin was also prepared from the liquefied products of the walnut shell into phenol by Huang *et al.* (2011). They successfully applied the biomass-based resol resin to produce phenolic foam with diisopropyl ether as the blowing agent, Tween 80 as the surfactant and hydrochloric acid as the catalyst. The foams showed adequate mechanical properties and a uniform fine cellular structure.

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Analysis on the quality of vegetable charcoal derived from Sapucaia's endocarp (*lecythis pisonis*)

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Keywords: biomass, endocarp, charcoal

Using biomass as a biofuel for power production appears to be an alternative to supply energy world demand. In 2015, Brazil consumed about 4,6 million tons of vegetable charcoal, and a considerable amount of this input was employed by steel companies. Given the rising demand for vegetable charcoal, it was necessary to find alternatives that could replace the use of wood in the production of this input, since this use increases the pressure for the extraction of forest natural resources in Amazon, region that accounts for one third of the national production of vegetable charcoal. This paper aims at qualifying charcoal obtained from the chestnuts (endocarp of the fruit) of Sapucaia (*Lecythis pisonis*), which were gathered in Altamira county, Pará state. The charcoal was prepared in accordance with the following temperatures in degrees Celsius: 300°, 350° and 400°. The samples were characterized by fixed carbon content, a quantity of volatile materials, ash content and gravimetric performance according to the methodology described in NBR (Brazilian Regulatory Standard) 8112 of ABNT (Brazilian Association of Technical Standards). Results obtained from the analyses indicate that the charcoal produced from Sapucaia's endocarp possesses an

acceptable quality, also presenting, at 300 degrees Celsius, a better gravimetric performance, 61,44%.

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Spectroscopic characterisation of bio-based filaments for the Fused Deposition Modeling proceeding

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Keywords: 3D printing; Raman spectroscopy; Infrared spectroscopy; Wood filament; Polylactic acid; Additive manufacturing

In the last few years, the utilisation of additive manufacturing (AM), also known as layer manufacturing, for producing three-dimensional objects has increased significantly in the industrial and private sector, which is shown in Fig. 1a. In addition, the number of scientific publications as well as patents concerning the topic additive manufacturing has more than tripled in the period from 2000 to 2012 (see Fig. 1b). Both histograms indicate that there is a real interest in additive manufacturing.

The ASTM an international standards organisation defined additive manufacturing in the following way: *“A process of joining materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies.”* (ASTM, 2012). Furthermore, layer manufacturing is a collective term for a multitude of different processing methodologies, and one of them is called fused deposition modeling.

Fused deposition modeling (FDM) is a common and versatile technique, in which a thermoplastic material, referred to as filament, is pressed through a heated nozzle and is afterwards deposited layer upon layer until the three-dimensional object is completed. The nozzle of the 3D printer moves along the x-axis and y-axis while the build plate slides downward once a nouveau layer has been deposited. A wide range of thermoplastic materials like PLA, ABS, Polyamide, PP and so on can be processed with the aid of this revolutionary manufacturing technique.

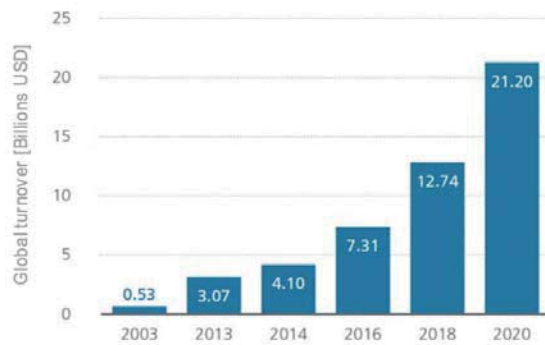


Figure 1a: Global turnover generated through the sale of goods and services in the area of additive manufacturing (EFI, 2015 & Wohlers Associates, 2014).

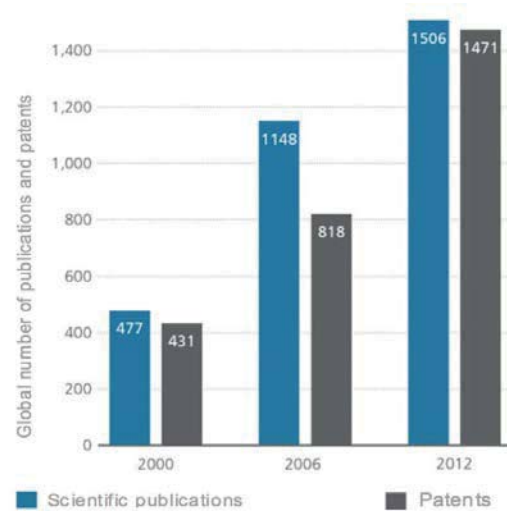


Figure 1b: Global number of scientific publications and patents, dealing with additive manufacturing, submitted and approved between 2000 and 2012 (EFI, 2015).

A major objective of this project is a comprehensive characterisation, with the aid of Raman spectroscopy and Infrared spectroscopy, of different bio-based 3D printing filaments. Furthermore, several of these filaments are filled with wooden particles or fibres. The measurement results generated by means of confocal Raman microscopy and Infrared spectroscopy allow a detailed estimation of the polymers as well as the polymer interfaces, which in turn significantly influence the mechanical properties of the printed objects as well as the formability.

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Acknowledgments:

The authors would like to thank the Interreg Austria - Bayern for financing the research project AB 97 TFP-HyMat.

Friday 15th September,
COST Action FP1407: 3rd meeting
“Wood modification research and
applications”

Session 4:

**Modified wood in sustainable built
environment**

From 11:50 to 12:55

Chair: C. Hill, F. Dolezal

Common themes in Wood Modification and Environmental Impact Assessment of Wood

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Keywords: bibliometrics; network analysis; wood modification; environmental impact assessment; meta-analysis

Bibliometric analysis utilises data mining techniques to capture selected aspects about a set of publications. For instance, researchers can quickly assess how research themes change over time, identify new trends in research, gaps in the knowledge base, assess collaboration patterns, identify key researchers, and other aspects of the research base. Our objective in this study was to identify common aspects of two research areas important to COST Action FP1407: wood modification and environmental impact assessment of wood.

To assess the common themes in these areas of research we retrieved all publications in the Web of Science database related to wood modification and environmental impact assessment of wood. The dataset included 922 publications in the environmental impact assessment of wood publication record (from 1977 onward), and 695 in the wood modification publication record (from 1955 onward). The publications were screened to for topical relevance. The data were then cleaned to equate similar keywords (e.g., removing plurals). Using this dataset, we analysed the keywords used to describe the publications and assessed the most frequently used terms in both groups, and the terms each group publications shared. For this study, keywords were processed for both datasets then merged to find the most common keywords for each group separately and the most common keywords used in both groups. The data were processed in R and the network graphs built in Gephi. This work extends our previous work in bibliometric analysis of the COST Action FP1407 themes and research work (Burnard *et al.*, submitted 2017).

There were only 5 publications that were in both datasets. 417 keywords were shared out of 3023 keywords used across all publications in the dataset. In Figure 1, a network graph shows the 15 most common keywords for each topic, and the 15 most common keywords used in both topics.

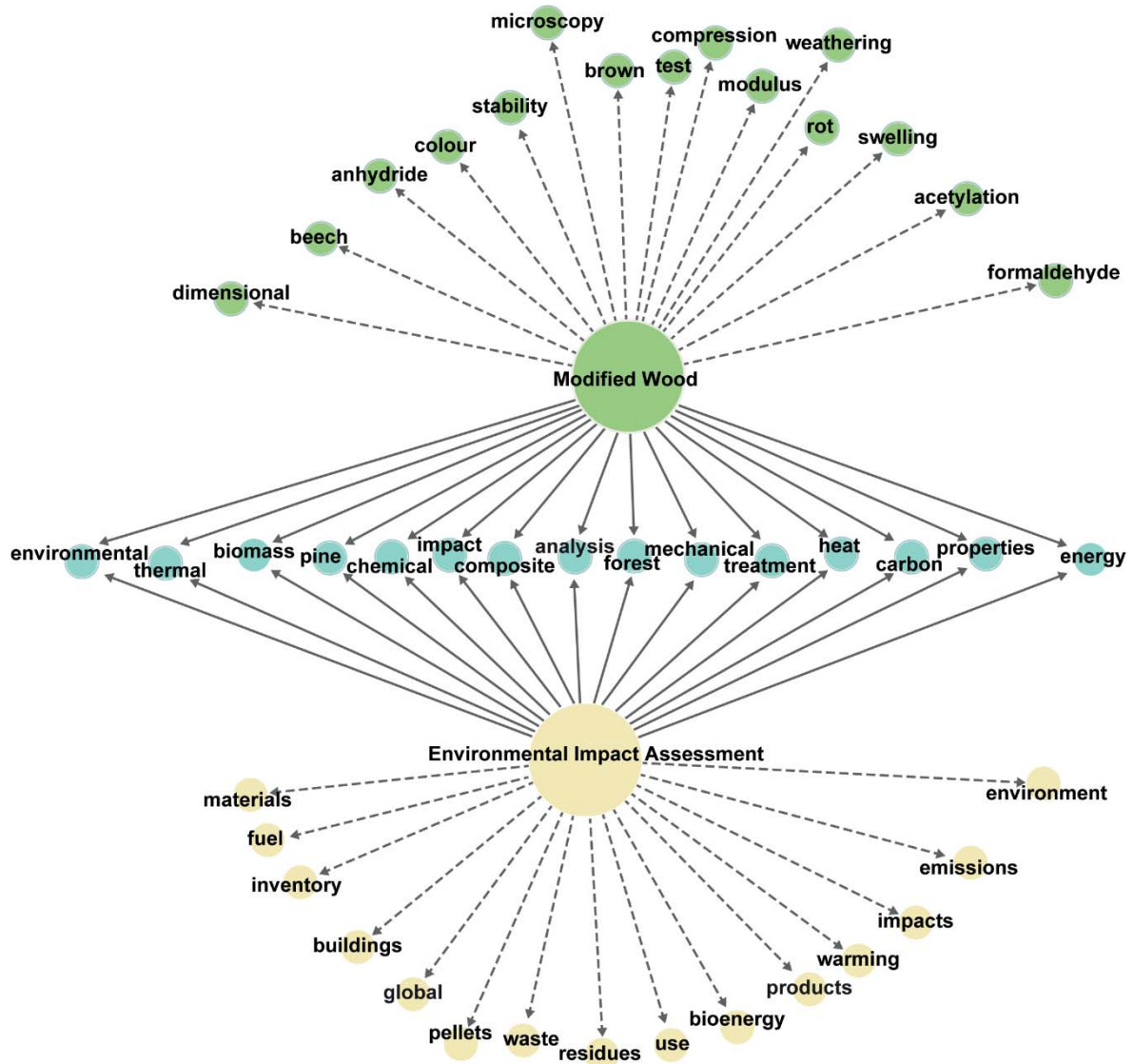


Figure 1: Network of the 15 most common keywords for each topic (yellow, green), and the 15 most common keywords in both dataset (teal).

The shared themes indicated here, changes in research topics over time, and collaboration networks will be presented.

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Carbon Sequestration in the Built Environment – The Role of Harvested Wood Products

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Keywords: carbon sequestration, life cycle assessment, built environment, embodied energy

The use of timber in construction can have a significant role as part of an energy reduction and carbon storage strategy for the built environment. The paper gives attention to issues surrounding carbon sequestration in forests and how atmospheric carbon can be stored in long life products in the built environment. There have been numerous studies of this subject and the general consensus is that although carbon storage certainly has a role to play in climate change mitigation, the effects of material substitution (where timber substitutes for higher embodied energy and GWP impact materials) and energy recovery from timber processing wastes and end of life demolition wastes are of more importance. This paper reviews the scientific literature of published LCA studies of commonly-used building materials (timber, cement/concrete, aluminium, steel, poly(vinyl chloride)). It is shown that the outcomes of the LCAs are very heavily dependent upon the assumptions made and the system boundaries used. It is not possible to arrive at definitive values of (for example global warming potential, GWP) that is characteristic for a material, but rather there is a range of values. The methodology used to determine the environmental impacts is complex and many studies are not readily amenable to comparative studies. This is because of differences in functional unit, supporting databases, assumptions regarding material life, maintenance, end-of-life scenarios, etc. In addition, most studies lack sufficient transparency to allow for proper verification of the results obtained. LCAs also inevitably contain simplifications, which may affect the accuracy of the data. Most studies do not employ a sensitivity analysis to show how the assumptions and variabilities affect the results. There is considerable variability in the methodology applied for LCAs, hence the task of making comparative assertions is extremely difficult. However, there has been some degree of consensus reached with the introduction of environmental product declarations (EPDs) and standardisation of procedures; known as product category rules (PCRs). Nonetheless, there is still concern that inter-product comparisons are not reliable, due to uncertainties and variations in the assumptions made, the use of different databases, etc. The main advantage with EPDs which are produced in conformity with the European standard EN15804, is that the impacts have to be reported separately for different life cycle stages. Of these, the cradle to factory gate life cycle stage is likely to be the most reliable, since this part of the life cycle involves the least assumptions and the most accurate data. This study has largely focussed on data concerned with the embodied energy associated with materials and the global warming potential

(GWP) environmental impact category, because these have the lowest uncertainties. GWP data is strongly influenced by the time-frames of the study and by a range of different factors that have to be taken into account when making comparative studies:

- Greenhouse gas (GHG) emissions associated with the manufacture of construction materials, maintenance, replacement and disposal
- GHG emissions associated with operational energy requirements, if these are relevant and realistic and have not been introduced to favour one material over another
- Carbon emissions and storage from forestry operations and sequestration by growing biomass
- Substitution effects associated with the use of timber in comparison to other building materials
- End-of-life scenarios, such as landfilling, incineration with energy recovery

A variety of factors can affect the carbon dioxide and energy profiles of building materials over their lifetime, which can be divided into uncertainties and variabilities. Uncertainties arise from lack of precise knowledge regarding processes or the use of assumptions. Variabilities can arise due to different choices being made regarding the use of materials, such as frequency and type of maintenance, different disposal methods, transport distances, etc. Combinations of uncertainty and variability can be difficult to separate. There is considerable scope for uncertainty to affect the data, especially when the in-service and end-of-life stages of the life cycle are included. However, in the majority of studies analysed there is agreement that there are environmental advantages associated with the use of timber in construction from a climate change mitigation perspective. One of the advantages of using timber in construction is the potential for the storage of biogenic carbon (derived from atmospheric carbon dioxide) in long-life structures. Although this does have a role to play in climate change mitigation, this literature review has revealed that most studies show that the effects of substitution for high embodied energy materials and for fossil fuels for energy production are much more significant. All of the LCAs of timber products have shown that the amount of atmospheric carbon stored in the wood is always larger than the GHG emissions associated with the processing of the material. Additional benefits arise when the wood is incinerated at the end of the life cycle, with substitution of fossil fuels. The highest fossil fuel substitution benefits arise when coal is replaced with timber wastes/by-products. In a Norwegian context, the highest benefits will arise if wood is used as a fuel for cement kilns, or as a carbon-source for aluminium anodes, followed by a replacement of oil for heating then natural gas for heating or electricity production. The embodied energy used to produce construction materials is an important consideration when analysing the environmental impacts. This initial embodied energy is to be distinguished from the recurring embodied energy which arises due to maintenance of the materials and the operating energy, which is energy consumed due to the operational requirements (e.g., heating) of the building. As the operating efficiency of buildings improves, the embodied energy will be a larger proportion of the overall energy requirements. The embodied energy also represents a greater proportion of the overall energy consumption of the sector in a growing market.

Comparative assessment of carbon uptake and release of wooden and concrete building materials

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Keywords: carbon uptake, carbon release, carbon sequestration, carbonisation, biogenic carbon

The building industry plays a major role in climate change issues. Especially production of concrete, as the most important building material, contributes 5% to global carbon dioxide emissions (Xi *et al.* 2016). On the first glance it seems to be obvious to replace concrete by renewable materials if procurable. But taking the whole life cycle of the building products into account, results may change. Comparative assessments usually are not satisfying since outcomes mainly depend on the chosen methodology. However, climate change mitigation is the major issue nowadays and building materials with the ability to sequester atmospheric carbon dioxide are the ones that have to be chosen. Therefore, two different, important building materials with ability of carbon sequestration, but with completely different performance are analyzed. This survey aims to clarify methodological differences, but also amounts of stored carbon dioxide in common building elements in order to get a clearer picture.

Storage of biogenic carbon in wooden building products

Among the major building materials, wood is the only one with the ability to store a significant amount of biogenic carbon dioxide. Starting with the germination of the seed, carbon dioxide assessment of a wooden product is not only time dependent, it requires an accurate tracking through the whole life cycle until its release into the atmosphere through combustion or decay (Kuittinen *et al.* 2013). The important life cycle phases (Fig. 1) for the carbon stock of wooden building products according to EN 15804 are A1 (the growth of the tree), when carbon is stored by photosynthesis, and C3, where the carbon stock is released, usually by combustion of the timber.

Storage of carbon in concrete building products – (re)carbonation

Concrete stores carbon as well, but in a completely different way than wood. When limestone is burned during cement production, calcium carbonate CaCO_3 is transformed to calcium oxide CaO by releasing carbon dioxide CO_2 . So on one hand cement production needs a lot of energy which is covered to a significant amount by fossil fuels with related CO_2 emissions, on the other hand, the process itself emits around 90% of global GHG emissions from industrial production (Xi, *et al.* 2016). These latter emissions are partly reversible in a process called recarbonation or simply carbonation. In this physiochemical process, CO_2 diffuses into cement based materials throughout their entire

life cycle and is reabsorbed to an amount of nearly 50% of the one that has been released during production process (without fuel emissions). Hence, important phases for CO₂ storage and release (Fig. 1) for concrete are phase A3 (production) where large amounts of CO₂ are emitted, B1 (use phase, with contact to the atmosphere) and C4, when exposed concrete surfaces have the opportunity to absorb CO₂ after having reached waste status. Calculation is carried out according to FprEN 16757 on the basis of a variety of different studies e.g. Lagerblad 2005 and Andersson *et al.* 2013.

Carbon uptake and release in the life cycle of wooden and concrete building products

Fig. 1 shows the significant differences in CO₂ storage and release of wooden and concrete building products. Uptake and release takes place in different life cycle phases with subsequent methodological impacts. Relevant quantities are indicated by the size of the arrows.

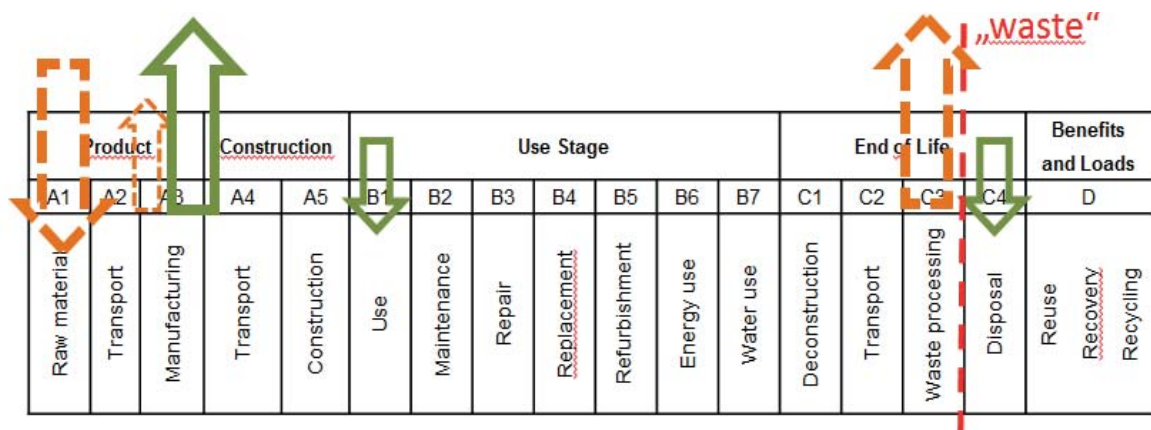


Figure 2: CO₂ uptake and release in different life cycle phases - wood (dashed) and concrete (solid line)

Conclusions

Calculation of CO₂ storage of wood is based on simply considering carbon share in the product, and described in detail in EN 16449. Methodology of assessment of carbonation is already normalized in FprEN 16757, but is not based on natural law and contains a lot of controversial assumptions and scenarios which need to be discussed. Especially carbonation scenarios during use phase, when concrete is covered with different materials and end of life phase, when used as a secondary material or landfilled in deep layers, still need further investigations.

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Performance of thermally modified radiata pine facade, gallery and decking in a passive house in Spain after six years exposure. Research and applications in a real case

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Keywords: facade, thermally, radiate, performance, Spain

This abstract describes a Spanish project with thermally modified radiata pine (*Pinus radiata*) used in the façade, gallery and decking as friendly building material in a passive house located in Asturias (north Spain), after six years of exposure.

There is a dose of degradation, performance and service life in exterior wooden uses, implicit to each national geographical location, local climate and inherent wood material variables as well as design details such as: sheltering, distance from ground, moisture traps, exposure to wind-driven rain, physical protection and coatings, and maintenance. In short, all these factors could generate changes to the wood material due to biological and abiotic agents.

Independently of the problems regarding beetle and termite attacks, that normally don't cause problems in exterior applications, the most important problems in exterior wood uses are the attacks of wood destroying fungi as well as aesthetics (colour changes, moulds, wood disfiguring fungi, etc.).

The paper analyses the decision-making process between the architects and the technical assistance given by the consultancy (David Lorenzo, Alfonso Lozano and Josu Benito) about the project decision of using thermally modified radiate pine (*Pinus radiata*) in some parts of the façade,

gallery and decking; design details and coating applied to the thermally modified wood as well as its maintenance program.

This paper also shows the performance of thermally modified wood after six-year exposure, considering the main factors to assess the real wood performance status, focusing on: thermally modified wood species selected, local climatic conditions and the influence of design details. It is important to identify the parameters influencing in the performance and service life, from simple aesthetic defects to serious problems affecting the thermally wood elements.

As a result, is the perfect performance after six years exposure in severe exposure climatic conditions as Asturias (north Spain) with a wet and warm climate and exposure wooden elements to driven rain and sun.

This example shows the cooperation between different key actors involved in the project, since its performance strongly depends on the selected material, exposure conditions and construction design.



Figure 1: View of thermally modified Radiata Pine wood elements in a passive house.

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Architects' perception of modified wood: a parallel study in selected countries in Europe and selected regions in USA

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Keywords: EWPs, wood modification, architects, construction, promotion

The market, especially in Europe, for new durable products of modified wood has increased substantially during the last few years. This increased interest depends partly on the restricted use of toxic preservatives due to an increased environmental concern and partly on the need for products regarding maintenance. Figure 1 shows the wide variety of engineered wood products (EWPs) nowadays are available on the market for both structural and non-structural uses, but many of these EWPs are still underutilized (Sandberg 2016).

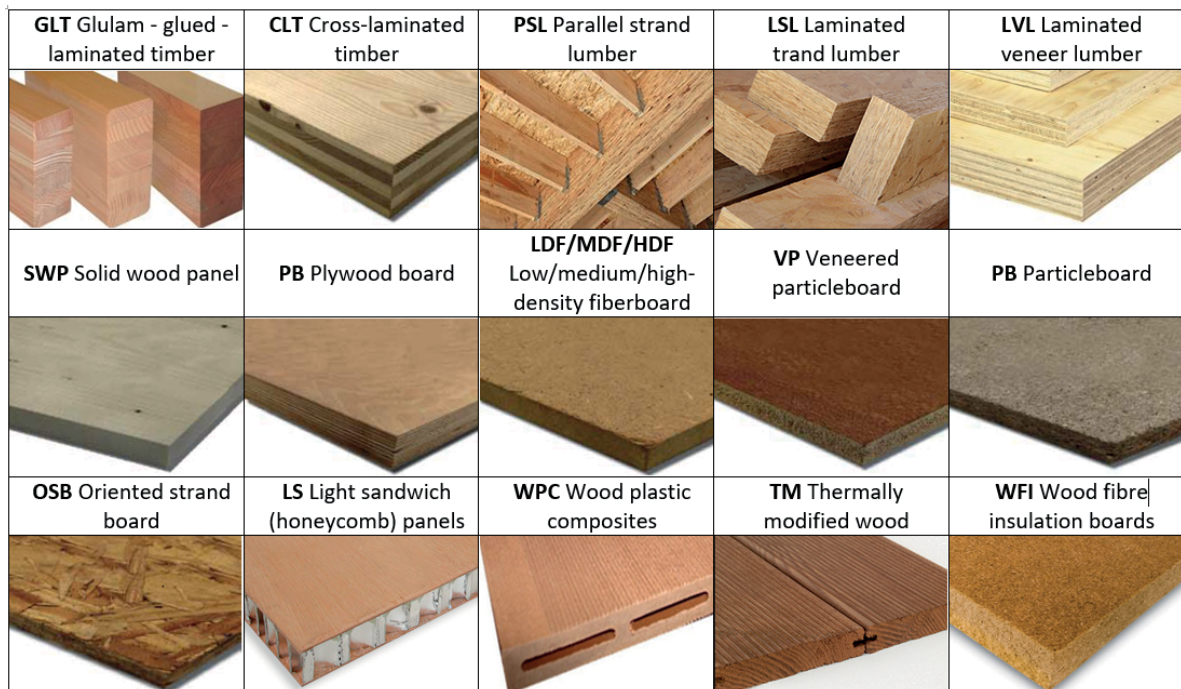


Figure 1: Only recently has wood been developed to form a range of products that are increasingly functional, based on a combination of performance and sustainability requirements. The result is a wide range of materials known as EWPs in contemporary architectural design

To better understand this situation, a global research project has been undertaken in selected countries of Europe and selection regions in the United States to determine how architects specify EWPs in their profession. Architects are key decision-makers determining material selection in the construction sector (Lähtinen *et al.* 2017). Their perception of wood and EWPs as building materials is therefore of great importance if non-renewable and fossil-based building materials are to be replaced by wood and EWPs. As a part of this project, the study provides a preliminary study of architects' perception of modified wood.

Data were collected through an on-line survey (Dillman 2000), and a survey questionnaire was developed by an international group of architects. The study methods included a two-stage survey; in the first stage, personal interviews with a selected group of architects from the architects' professional organization were conducted. Based on the information given by these in-person interviews, an exploratory web-based survey was subsequently designed.

The specific goals of the study were: (1) to identify the use of modified wood in architectural planning, and (2) to clarify the architects' knowledge of modified wood and its advantages. The study provides an updated overview of the perceived identity of modified wood products among the architects. New environmentally friendly technologies present designers and architects with new tasks and challenges, as well as opportunities to contribute to the creation of a sustainable environment.

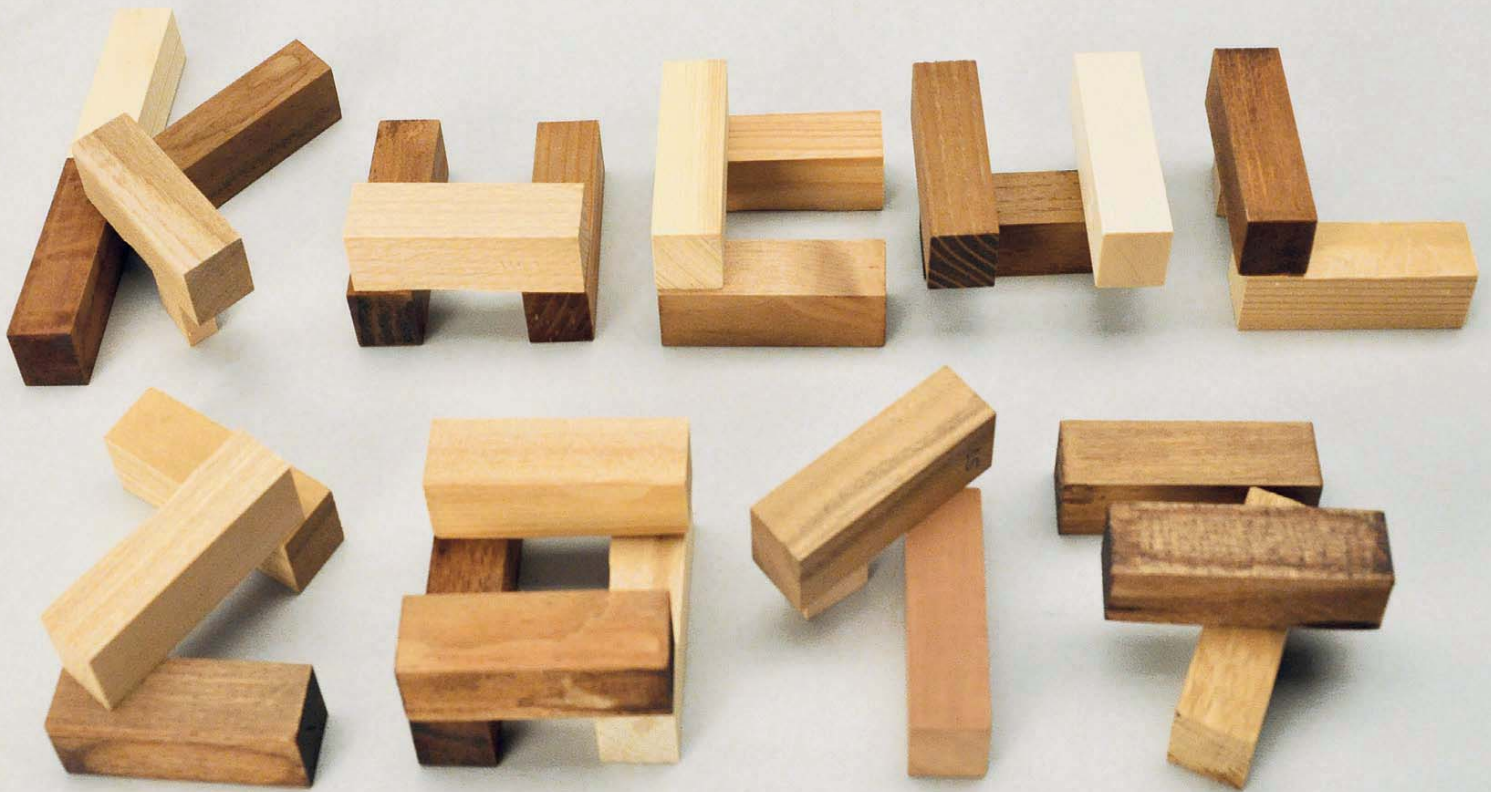
Using information obtained in this and similar studies, region-specific promotion campaigns could be developed with the aim of increasing the use of modified wood in various applications. Long-term cooperative programs, multidisciplinary approaches, including lobbying efforts and promotional campaigns, are needed to ensure that material specifiers have the knowledge and training to be able to use traditional and new wood products that are ideally suited for the building sector.

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Acknowledgments:

STSM COST FP1407, Slovenian Research Agency for financial support (P4-0015 program), and Swedish Research Council Formas (P. Nos. 2014-172 & 942-2016-64) are gratefully acknowledged.



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Pictures: Gianluca Tondi, Hermann Huber, Salzburg University of Applied Sciences

<http://costfp1407.iam.upr.si/en/events/cost-fp1407-3rd-conference>
ISBN 978-3-200-05255-0