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Editor:
Maria Fredriksson
Preface

The Northern European Network for Wood Science and Engineering (WSE) had its first annual meeting in 2005. The purpose of the organisation is to promote collaboration between northern European researchers within wood science and engineering. Young researchers are especially encouraged to participate at the meetings and present their work. To put additional focus on young researchers, the meeting has during the last years been preceded by a course for PhD-students and early stage researchers. This year, the course has the theme “Measuring moisture related properties of wood”.

Nordic Forestry Research (SNS) and North European Regional Office of European Forest Institute (EFINORD) are gratefully acknowledged for their financial contributions to WSE2019. We would also like to thank Swedish Wood, who sponsored this year’s awards for best student oral presentation and poster presentation.

Division of Building Materials at Lund University is pleased to host the 15th Annual Meeting of the Northern European Network for Wood Science and Engineering – WSE2019.

Welcome to Lund, Sweden!

Maria Fredriksson
Coordinator of WSE2019
Lund, Sweden

List of previous WSE meetings:
2005 Honne, Norway
2006 Stockholm, Sweden
2007 Helsinki, Finland
2008 Riga, Latvia
2009 Copenhagen, Denmark
2010 Tallinn, Estonia
2011 Oslo, Norway
2012 Kaunas, Lithuania
2013 Hannover, Germany
2014 Edinburgh, United Kingdom
2015 Poznan, Poland
2016 Riga, Latvia
2017 Copenhagen, Denmark
2018 Tallinn, Estonia
This year’s sponsor of the awards for best oral presentation and best poster presentation is Swedish Wood.

About Swedish Wood
Swedish Wood’s aim is to increase the size and value of the market for Swedish wood and wood products in construction, interior design and packaging. Through inspiration, information and education, we promote wood as a competitive, renewable, versatile and natural material. Swedish Wood also lobbies on behalf of its members on key industry and trade issues.

Swedish Wood represents the Swedish sawmill industry and is part of the Swedish Forest Industries Federation. In addition, Swedish Wood represents the Swedish glulam and packaging industries, and collaborates closely with Swedish builders’ merchants and wholesalers of wood products.

About Nordic Forest Research (SNS)
Nordic Forest Research (SNS) is a cooperating body, financed with Nordic funds under the auspices of the Nordic Council of Ministers that strives to enhance benefits for the Nordic region and contribute to a sustainable society. The members are Iceland, Norway, Sweden, Finland, Denmark and the independent areas of Åland, Faroe islands and Greenland.

About North European Regional Office of European Forest Institute (EFINORD)
North European Regional Office of European Forest Institute (EFINORD) promote and facilitate research collaboration and interactions between science and policy in forestry issues that arises in the northern region. A particular focus is given to the bioeconomy research field in combination with natural- and social sciences for a world where forests significantly contribute to sustainable well-being across disciplines. The EFINORD currently has around 30 partner organizations.
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Background

The ability to see inside solid objects in 3D and non-destructively with x-ray tomography is providing great new opportunities in many areas of research for enhanced multi-scale understanding of materials and structures. Such imaging can be performed in medical-CT type devices for large samples and, for more detailed analysis, in laboratory devices or at large-scale synchrotron facilities. Regarding applications to wood and wood-based materials x-ray tomography has been employed to characterise structures from the bulk (trunk) scale down to the fibre scale. Furthermore, extension of 3D imaging to 4D (3D + time) can provide new understanding of evolving processes at the heart of material behaviour, e.g., during environmental, chemical, mechanical, humidity or thermal loading. In this presentation, the possibilities of using 3D and 4D x-ray imaging to understand structures and processes in wood and wood-based materials will be discussed and illustrated through a number of different applications.

Experimental

From medical-CT to synchrotron x-ray tomography, the principal of measurement is the same; samples are rotated relative to the x-ray beam and detector (either the sample is rotated relative to the imaging system, in synchrotron or lab micro-tomographs, or the source and detector system is rotated around the sample/person, as in medical CT systems). Medical-CT type systems allow 3D imaging of objects the size of a person with spatial resolutions in the range of 100’s μm, whilst synchrotron x-ray tomography allows resolutions down to the 10’s nm, for samples in the mm to 10’s μm range; i.e., the possible spatial resolution scales with the sample size.

Results and Discussion

In our work we use both laboratory and synchrotron tomography combined with image analysis to characterise the 3D structure of samples. In some studies, such imaging is performed before and after some processing of the samples or of “sister samples” processed to different levels to investigate the structural evolution. A more effective approach is to analyse the structural
evolution during processing, which involves performing experiments “in-situ” in the tomograph with adapted experimental set-ups. Figure 1 shows examples of imaging on different wood samples performed with a Zeiss XRadia XRM520 at the 4D-Imaging Lab at Lund University. These examples range from individual wood fibres imaged with an image voxel size of 300 nm and a field of view of 300 microns to a 75x75x107 mm³ bulk wood specimen.

The outputs from x-ray tomography are 3D images of the test specimens with intensity values related to the amount of transmission of x-rays through the material. Such images can provide significant qualitative information, but, generally, 3D image processing and analysis are required to extract useful quantitative data on sample structure. Figure 2 shows an example of such 3D image processing where the cells in a cellulose-based foam have been segmented and measured in 3D.

Figure 1. Examples of tomography imaging of wood samples from the 4D-Imaging Lab at Lund University. Clockwise from top left: 3D rendering of 3 joined wood fibres imaged with 300 nm voxel size (sample provided by A. Kulachenko, KTH); slice through tomographic volume of a mahogany wood sample; 3D rendered tomography image of a spruce wood specimen treated in a soda pulping reactor for 150 min (collab. A. Wagih & M. Hasani, CTH); cross-section through a tomographic image of a large spruce wood sample containing a knot (collab. M. Hu & A. Olsson, Linnaeus Univ.); 3D rendered tomography image of a spruce sample deformed under uniaxial compression at 10° to the grain (collab. M. Dorn, Linnaeus Univ.); slice through a tomography image of a spruce wood sample treated with high pressure and steam (collab. P. Kvist & A. Rasmuson, CTH); slice through a tomography image of a partially saturated spruce wood specimen under controlled humidity conditions with water and air-filled lumens (collab. M. Fredriksson & J. Engqvist, Lund Univ.).

Figure 2. Example of 3D image segmentation and quantification of tomography data. The specimen was a cellulose-based foam (see Gordeyeva et al. (J. Colloid & Interface Science, 2016), for more details). Left three images are example orthogonal slices through the image volume after running 3D watershed segmentation – colours indicate the “label” numbers of each identified cell. The right two images show examples of the quantification in the form of histograms of the maximum and minimum diameters of the identified cells.
SMART SELF-FORMING WOOD FOR ARCHITECTURE AND CONSTRUCTION

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Background

The anisotropic swelling and shrinking of wood can be taken as a basis for utilizing wood in a smart way. By manufacturing wood bilayers in a cross-ply manner, the dimensional changes can be translated into reversible shape changes such as bending, twisting or a combination of both (Reichert et al., 2015; Rueggerberg and Burgert, 2015). The curvature of such bilayers can be predicted by analytical as well as numerical simulations using appropriate material models (Hassani et al., 2015; Grönquist et al., 2018; Grönquist et al., 2019).

Here, we show the dimensional upscaling of wood bilayers to metre scale and demonstrate their use as an autonomous, solar controlled and driven motor element for climate adaptive building shells (Vailati et al., 2018) as well as basic element within an alternative manufacturing of highly curved cross-laminated timber (CLT) (Grönquist et al., 2019). Furthermore, we demonstrate the first application of this technology at building scale.

Experimental

Manufacturing of wood bilayers

Wood bilayers with cross-ply structure were manufactured from beech or spruce wood for achieving single curved structures in the centimetre to metre size range with thicknesses from a few to up to 40 millimetres. Bilayers were glued at high moisture content in flat state using polyurethane adhesive. Drying to the target moisture content induced curvature, which can be calculated using analytical or FE modelling. For form-stable, curved CLT, two curved bilayers and a covering layer are stacked and glued in a minimum formwork.

Inducing and recording of shape change and wood moisture content

Drying or wetting was induced in climate chambers at room temperature or in an industrial kiln-drying chamber. Shape changes were monitored by time-lapse photography and standard measuring devices while the wood moisture content of the demonstrators was determined by either weighing or by electrical resistance.
Results and Discussion

Wood bilayers reversibly bend in response to changes of ambient relative humidity. However, the rate of shape change is rather low for bilayers with a few millimetre thickness due to low diffusion rates of water in wood. Hence, for their use in autonomous shading devices, which require higher rates of shape change for quickly adapting to changing weather (and illumination) conditions, bilayers were coupled, which induces fast rotation of one bilayer by the bending of the other bilayer. Figure 1a and b show a prototype of such an autonomous shading system with four coupled elements. The rate of rotation can be adjusted by the distance between the two bilayers.

Figure 1. Self-shaping of wood bilayers and structures, a, b) Autonomous shading device with coupled bilayers in (a) open, straight configuration (night time, clouds, rain) and (b) closed, curved configuration (sunny times); c) curved bilayers, 5m x 1.2m, thickness 40mm; d) 5-layer curved, formstable cross-laminated timber (5m x 3.6m) made up of 2 bilayers (c) with an additional covering layer (inset: layer built-up); e) Urbach tower, 14m high, made up of elements of (d), on display at the Remstal gardenshow, Urbach, Germany.

The alternative manufacturing of curved CLT utilizes the self-forming capacity of bilayers once (Figure 1c). After the self-forming step induced by the usual industrial kiln-drying, two curved bilayers and an additional cover layer are stacked and glued in a minimum formwork for obtaining form-stable curved CLT (Figure 1d). This smart manufacturing does not require heavy machinery and offers more freedom in size, geometry, as well as in curvature and layer thickness compared to conventional manufacturing, which is beneficial in terms of efficiency of material usage. Such self-formed CLT has been employed for the first time at building scale for the 14m high Urbach Tower, which is installed in the framework of the Remstal Gartenschau at Urbach, Germany (Figure 1e). Here bilayers of 5mx1.2m size with 40mm thickness have been manufactured and combined to curved CLT-elements of 90mm thickness and 15m length with a radius of curvature of 2.4m. The tower consists of twelve of such elements.

Conclusions

Re-thinking the material intrinsic capacities of wood opens up new possibilities in utilizing wood and in manufacturing of complex shaped wood parts. The unique combination of anisotropic responsiveness, mechanical stiffness and strength and good workability facilitates upscaling of self-shaping wood structures to metre scale, which is mostly prevented for other materials due to complicated synthesis. While the Urbach tower is the very first example of using self-shaping manufacturing at building scale, the autonomous shading devices have not been implemented at that scale. As wood at the same time represents a sustainable building material, the self-shaping capabilities may further promote the use of wood in climate-adaptive and performative architecture.
References


MODELLING OF SELF-SHAPING WOOD COMPOSITES

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Background

Wood can be assembled as a bi-layered cross-laminated composite, so called wood bilayers (Rüggeberg and Burgert, 2015). Hereby, the swelling and shrinkage anisotropy enables wood bilayers to act as hygromorphic self-shaping composites that display large deformations upon changes in moisture content. Application at both the small-scale, e.g. as climate adaptive actuators, and at the large-scale, e.g. as curved cross-laminated timber are possible (Grönquist et al., 2019). However, the complex mechanical behaviour of bulk wood challenges both the fundamental understanding of the self-shaping mechanism and the accurate shape-prediction in function of given boundary conditions. We address these issues by the combined study of experimental data and results from wood-specific mechanical models such as analytical and numerical models.

Experimental

Wood bilayer strips are produced in wet state by cross-lamination using 1cPUR adhesive. The bilayer strips are then dried and their curvature and moisture content is recorded until reaching equilibrium. The experimental wood bilayers are modelled using a linear-elastic analytical formula, which was adapted to the orthotropy and moisture-dependency of the wood elastic properties (Grönquist et al., 2018). In addition, a more complex rheological constitutive material model for wood is used in combination with the Finite Element Method (Hassani et al., 2015). The model uniquely accounts for deformation mechanisms such as hygro-elasticity, visco-elasticity, plasticity, and mechano-sorption in a coupled and moisture-dependent numerical implementation.

Results and Discussion

Experiments did successfully validate the developed analytical linear elastic model for prediction of shape in function of change in moisture content of beech wood bilayers (Grönquist et al., 2018). The model can be used to simulate a range of possible configurations; examples are shown in Figure 1. Furthermore, application-relevant design aspects such as minimization of layer internal axial stresses (Figure 1b)), e.g. to avoid delamination, can be assessed. In the case of beech wood, numerical FE models accounting for anisotropic moisture diffusion and complex mechanical behaviour were equally able to validate the linear elastic model (Grönquist et al., 2019). Additionally, such models provide insight into specific large-scale effects such as the relevant influences of moisture diffusion on mechanics of self-shaping.
Conclusions

Modelling of hygromorphic self-shaping bi-layered wood composites, with both analytical and numerical models, enable efficient application-based design for innovative concepts such as dynamic actuator elements or curved mass timber elements in construction.

References


Sorption Hysteresis in Wood Cell Walls Beyond the Fibre Saturation Point

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Background

Moisture affects most physical properties of wood. Like other porous materials, wood exhibits sorption hysteresis, i.e. the equilibrium moisture content depends on both ambient climate and moisture history. If equilibrium is attained by desorption, the moisture content is higher than if it is reached by absorption to the same ambient climate. This difference in moisture content between desorption and absorption increases with increasing relative humidity (RH) in the hygroscopic range (0 to ~95 % RH) (Fredriksson and Thybring 2018). In the over-hygroscopic range (~95 to 100 % RH), both moisture content and sorption hysteresis increase due to capillary condensation in the wood structure. According to traditional wood literature, the presence of significant amounts of capillary water indicates that the wood is above the fibre saturation point and cell walls are saturated. However, it is unknown if cell walls are indeed water-saturated or whether sorption hysteresis persists in the over-hygroscopic range.

Experimental

Douglas-fir wood were extracted first in 1:2 ethanol:toluene and secondly in 9:1 acetone:water to remove extractives. Circular specimens with diameter ~4 mm and a thickness of 2 mm (longitudinal direction) were produced. These were conditioned in both absorption from dry state and desorption from water-saturated state in the full RH-range: saturated salt solutions were used for generating 33-95 % RH, and the pressure plate technique for the range 99.65-99.98 % RH. The latter technique was modified for conditioning in absorption. After conditioning for 2 months, specimens were transferred to DSC pans and hermetically sealed with a press. The amount of capillary water was subsequently determined with a DSC which measured the total heat of melting after quenching specimens to -20 °C and slowly ramping the temperature to 20 °C. After final determination of the specimen dry mass, the total moisture content could be divided into capillary water and cell wall water.

Results and Discussion

The DSC measurements enabled separation of capillary water and cell wall water, and therefore the absorption and desorption isotherms of each of these types of water could be constructed, see Figure 1. As expected, capillary water was not detected for specimens conditioned at 95 % RH and therefore DSC measurements were not performed for specimens conditioned to relative humidity levels below 95%.
Figure 1. a. Cell wall sorption isotherms (o) and capillary water isotherms (x). The y-axis is cut, but the capillary moisture content at water saturation was 0.792 g g⁻¹ with standard deviation 0.215 g g⁻¹. Note that moisture contents at water-saturation are arbitrarily placed at -10⁶ J kg⁻¹. b. Absolute sorption hysteresis evaluated from the data in a.

The results show that sorption hysteresis for cell wall water persists in the over-hygroscopic range, even as high as 99.98 % RH. Thus, it is seen that the absorption and desorption isotherms for cell wall water only merge in the fully water-saturated state. However, it appears that the hysteresis for cell wall water decreases in the over-hygroscopic range. Perhaps this effect is due to the gradually increasing amount of capillary water which was 3.0 ± 1.2 % moisture content at the two highest conditioning RH levels (99.93 and 99.98 %).

Additionally, Figure 1 clearly documents that the presence of capillary water is not an indication of water-saturated cell walls, since the cell wall moisture content was lower in the measured over-hygroscopic range 99.65-99.98 % RH than at water-saturation for specimens conditioned in both absorption and desorption. This contradicts the concept of the fibre saturation point (FSP) where cell walls are suggested to be saturated with water before significant amounts of capillary water are present in wood. Moreover, the results show that it is not possible to water-saturate cell walls by conditioning in water vapour.

Conclusions

The total moisture content in wood was successfully divided into cell wall water and capillary water with the DSC technique after conditioning in the full RH-range. The results show that sorption hysteresis for cell wall water persists in the over-hygroscopic range despite the presence of capillary water. Moreover, the amount of cell wall water is lower than at water-saturation even at 99.98 % RH contradicting the concept of the fibre saturation point.

References

OBSERVING MOISTURE-RELATED CHANGES IN WOOD NANOSTRUCTURE WITH X-RAY AND NEUTRON SCATTERING

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Background

The properties of wood as material and its usability in many applications depend on its moisture content and history. In spite of of extensive research (Engelund et al., 2013), the effects of moisture changes on the nanoscale structure of wood and the interactions of water with its various components are not fully understood. This is partly due to a lack of methods for characterizing the wood nanostructure at different moisture contents (Rongpipi et al., 2019).

X-ray and neutron scattering methods are non-invasive and allow structural characterization of wood and other cellulosic materials under varying external conditions (Martínez-Sanz et al., 2015). Small-angle scattering of X-rays and neutrons (SAXS and SANS) can be used to detect moisture-dependent changes in the organization and cross-sectional dimensions of cellulose microfibrils, which is facilitated by the newly developed WoodSAS model for the data interpretation (Penttilä et al., 2019). Wide-angle X-ray scattering (WAXS), on the other hand, yields information on the inner structure of cellulose microfibrils and the crystalline order of cellulose molecules, which are influenced for instance by moisture-related stresses.

We used X-ray and neutron scattering to observe the effects of drying and rewetting as well as relative humidity (RH) changes on the nanoscale structure of different woods (Penttilä et al., submitted). The structural changes were coupled with the actual moisture content of the samples, as determined by dynamic vapour sorption (DVS).

Experimental

Samples from European beech, including both normal wood and tension wood, as well as European silver fir and Norway spruce were collected fresh and stored in liquid water (never-dried) or dried in a desiccator at room temperature and rewetted in liquid water (dried/rewetted). Samples before and after drying/rewetting were characterized with SANS, SAXS and WAXS, and their water sorption properties were studied with DVS. SAXS and WAXS data were also collected at controlled RH conditions, from RH 90% down to RH 15% and back, with similar steps between measurements as used in the DVS experiments.
Results and Discussion

According to the SANS results, the interfibrillar distance (i.e., lateral distance between centre points of neighbouring cellulose microfibrils) decreased by 2-3% in normal wood due to drying at room temperature and rewetting in liquid water. The tension wood sample showed an opposite trend, with a 2% increase in the interfibrillar distance. DVS also showed that the drying affected the moisture sorption capacity of the wood samples, yielding lower values of moisture content at RH 90% after desorption from the dried/rewetted state as compared to never-dried state. However, this effect was cancelled in most samples when the RH was decreased to 45% and below.

The SAXS and WAXS measurements at controlled humidity conditions allowed *in situ* observations of the effects of moisture changes on the wood nanostructure. Based on the SAXS data (Figure 1), the interfibrillar distance decreased with decreasing RH and moisture content, and a similar trend was observed also in the microfibril diameter. Both values were typically highest when the sample was immersed in liquid water. According to the WAXS results, the lattice spacings and crystal size correlated with moisture content, but with different signs between the axial and lateral directions of the fibrillar crystallites. A general trend of increasing crystalline order at the presence of more water could be recognized in the WAXS results.

![Example SAXS data and fits for a spruce latewood sample at various moisture conditions, showing the variation of the interfibrillar distance $a$ and microfibril diameter $2R$ with changing humidity.](image)

Throughout the results, the softwood samples behaved rather similarly to each other, whereas the two types of beech samples often deviated from them and from each other. For instance, the wet-state interfibrillar distance based on both SANS and SAXS was highest in beech tension wood (6 nm), second highest in the softwoods (4 nm) and lowest in beech normal wood (3 nm). On the other hand, especially the coherence length of crystalline order (004 crystal size) along the microfibril axis correlated with moisture content in a similar way in all samples.

Conclusions

X-ray and neutron scattering methods proved very suitable for real-time observations of moisture-related changes in the wood nanostructure. The nanoscale moisture behaviour of wood depended slightly on the wood species and the presence of reaction wood, which might be related to the properties of the non-crystalline matrix polymers.
References


TARGETED ACETYLATION OF NORWAY SPRUCE TISSUE AND ITS EFFECT ON MOISTURE STATES IN WOOD

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Background

Due to the inherent biological nature, wood is susceptible to microbial degradation. Traditionally, broad toxicity biocides have been used to protect wood against decay, but these are gradually being phased out for environmental reasons. Instead, non-toxic modification methods, e.g. acetylation, are increasingly used to improve the durability of wood. Acetylation is a chemical wood modification method involving the substitution of accessible hydroxyl groups of the cell wall polymers with acetyl groups. The acetylation is well known to reduce the cell wall moisture content of wood, improve its dimensional stability as well as the resistance to microbial degradation (Rowell, 2014). How the acetylation affects the location and state of water in wood is, however, less known. In this study, thus, we investigate acetylated wood-water interactions by employing targeted cell wall acetylation.

Experimental

Materials and modifications:
Norway spruce (*Picea abies* L.) Karst.) specimens with dimensions 10 (longitudinal) x 5 x 5 mm³ were vacuum dried at 60 °C for 24 h prior to acetylation. The specimens were then vacuum saturated with acetylation solutions; 10 g of acetylation solution (either pure acetic anhydride or 1:4 mixtures of acetic anhydride and pyridine) was used for each g of wood. Atmospheric pressure was re-established and acetylation reaction was carried out at elevated temperatures for a defined period of time. The detailed acetylation conditions used are listed in Table 1. After reaction completion, the specimens were extensively washed in acetone and finally in water.

LFNMR

A Bruker mq20-Minispec NMR instrument equipped with a 0.47-Tesla permanent magnet was used to identify location and state of water in vacuum saturated specimens (Fredriksson and Thygesen, 2017).

Raman microscopy

A WITec alpha300R Confocal Raman Microscope equipped with a 532 nm laser and an oil immersion objective (100x) was used to identify the extent and location of the acetylation in wood. Raman spectra analysis was carried out using true component analysis (TCA).
Results and Discussion

The weight percent gain (WPG) of spruce acetylated under the different reaction conditions is shown in Table 1. Notably, the acetylation with pyridine (swelling agent) resulted in higher WPG values than acetylation with acetic anhydride alone. The Raman images in Figure 2 show how the acetylation is distributed within the spruce cell walls. The cell walls were acetylated homogenously in the presence of pyridine (treatment B), but the acetylation of wood with a pure acetic anhydride, on the other hand, allowed targeted modification of lumen surfaces. Noteworthy, the longer reaction time (24 h) resulted in a deeper acetylation of spruce cell walls adjacent to the lumens. Due to the significantly thinner cell walls, a higher proportion of the earlywood cell walls was acetylated compared to the latewood cell walls (treatment D).

![Figure 2. Raman images of acetylated Norway spruce cross sections based on TCA. The acetylation of cell walls is visualized by a green color. Yellow areas represent un-acetylated cell wall. Blue color indicates lignin rich middle lamella, while a red color – lumen.](image)

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Acetylation solution</th>
<th>Time (h)</th>
<th>Temp. °C</th>
<th>WPG (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Untreated</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Pyridine</td>
<td>pyridine</td>
<td>3</td>
<td>80</td>
<td>-2.3</td>
</tr>
<tr>
<td>A</td>
<td>1:4 acetic anhydride:pyridine</td>
<td>1</td>
<td>80</td>
<td>14.2</td>
</tr>
<tr>
<td>B</td>
<td>1:4 acetic anhydride:pyridine</td>
<td>3</td>
<td>80</td>
<td>20.9</td>
</tr>
<tr>
<td>C</td>
<td>acetic anhydride</td>
<td>5</td>
<td>75</td>
<td>3.4</td>
</tr>
<tr>
<td>D</td>
<td>acetic anhydride</td>
<td>24</td>
<td>75</td>
<td>9.9</td>
</tr>
</tbody>
</table>

The LFNMR measurements give information about the interaction between wood and water present in cell walls, water in small voids as well as in lumina, where shorter $T_2$ indicates a stronger interaction. The continuous $T_2$ distributions in Figure 3 indicate, thus, that acetylation in the presence of pyridine led to reduced water interactions with wood both in cell walls, lumina and small voids (peaks in between cell wall water and water in lumina). Lumen surface acetylation with an acetic anhydride, in contrast, substantially reduced wood-water interactions in small voids and lumina, whereas water interactions within the wood cell walls primarily remained unchanged. In general, higher degree of acetylation resulted in weaker wood-water interactions (Figure 3).

![Figure 3. Continuous $T_2$ distributions](image)

Conclusions

By varying duration of the acetylation reaction it is possible to distinctively acetylate lumen surface and control the depth of acetylation. The LFNMR analysis clearly demonstrated that acetylation not only affects state of the water within cell walls and voids present in wood, but also that targeted acetylation opens up new opportunities to specifically modify water interactions within wood.
Acknowledgements

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References


EFFECTS OF ACETYLATION ON MOISTURE IN WOOD

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Background

Wood is a naturally hygroscopic material, and the atmospheric water molecules interact with sorption sites in the cell wall, causing changing moisture contents depending on the relative humidity of the air (Skaar, 1988). Many properties of wood, such as dimensional stability (Kong et al., 2018), mechanical properties (Wagner et al., 2015) and resistance towards biological degradation (Thybring et al., 2018) are affected by the moisture content in wood. Acetylation is an effective method to reduce the hygroscopicity of wood by substitution of hydroxyl groups with acetyl groups as well as by bulking of the cell wall (Popescu et al., 2014; Beck et al., 2017). Wood-water interactions are related to the biopolymer composition of the cell wall, as the number of accessible hydroxyl groups differ between cellulose, hemicellulose and lignin (Thybring et al., 2017), and we hypothesize that biopolymer composition also affects the acetylation result. The objective of this study is to change the relative biopolymer composition in wood prior to acetylation and investigate the effects of these changes on the moisture in acetylated wood as studied by low field nuclear magnetic resonance (LFNMR) and by measurements of hydroxyl accessibility.

Experimental

Materials

Specimens (20 (tangential) × 20 (radial) × 4 (longitudinal) mm) were cut from the sapwood of a five-year-old poplar (Populus euramerica Cv.) harvested from the Greater Khingan Mountains in China. The average air-dried density was 0.36 g cm⁻³ and the average ring width was 3.5 mm.

Methods

The specimens were vacuum-dried at 65 °C for 48 h, and then either hemicelluloses or lignin were partially removed according to the procedures of Yang et al., 2018. The lignin content was reduced by approximately 9.0 %, while the hemicellulose content was reduced by approximately 8.6 %. Then specimens were subjected to 15 min vacuum treatment at about -0.1 MPa, and subsequently a mixture of pyridine and acetic anhydride (volume ratio 5:2) was injected into the reaction flask. For the pyridine controls neat pyridine was injected. After leaving specimens in the liquid for 1 h at ambient temperature, the reaction flasks were heated in an oil bath at 80°C for 1 h. Afterwards, residual chemicals were removed by washing, first in pure acetone and then in distilled water before the specimens were air dried at ambient conditions. Finally, the specimens were vacuum-dried at 65 °C for 48 h. After acetylation, the specimens were characterized with low field nuclear magnetic resonance after water saturation
(Beck et al., 2018) and hydroxyl accessibility was determined using a sorption balance (Thybring et al., 2017). The weight percent gain and moisture content were also determined for each specimen.

Results and Discussion

The effects of acetylation on mass and volume changes of the native wood are shown in Table 1. After pyridine treatment, the mass decreased by 2.5% and the volume by 0.2%, while acetylation led to 15.7% weight percent gain and 9.7% volume increase.

Table 1. Weight percent gain (WPG), volumetric change (VC) and moisture content (MC) for cell walls of wood under water-saturated conditions derived from the LFNMR data.

<table>
<thead>
<tr>
<th></th>
<th>WPG</th>
<th>VC</th>
<th>MC (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control (C)</td>
<td>0</td>
<td>0</td>
<td>30.0 (2.4)</td>
</tr>
<tr>
<td>Pyridine Control (PC)</td>
<td>-2.5 (0.4)</td>
<td>-0.2 (0.6)</td>
<td>27.9 (0.2)</td>
</tr>
<tr>
<td>Acetylated wood (AC)</td>
<td>15.7 (0.4)</td>
<td>9.7 (0.6)</td>
<td>19.7 (0.9)</td>
</tr>
</tbody>
</table>

Data provided as the average (standard deviation) from five replicates.

Four peaks are visible in the example T2 distribution of a control specimen (Figure 1a). After pyridine treatment, a peak disappears for the bound water. This might be due to merging of the two bound water peaks as a result of the pyridine induced cell wall swelling. Acetylated wood exhibits five peaks and T2 becomes longer for the bound water compared to untreated wood, as seen earlier (Beck et al., 2018). Results for the specimens with reduced lignin or hemicellulose content will be given in the presentation.

Conclusions

As expected, acetylation in pyridine made the wood cell wall less accessible to water, while pyridine treatment alone seemed to have little influence on the accessibility.

References


Background

The utilization of biocides to prevent biological degradation of wood in outdoor situations is facing increased challenges. Environmentally motivated legislation is restricting the use of biocides and thus incentivizing the search for new, low-cost wood modification technologies (Hill 2006). This research highlights the potentials for esterification of citric acid and sorbitol in wood by an aqueous modification process. Citric acid and sorbitol are both low priced and readily available feedstock chemicals, an essential pre-requisite for a commercial modification process. Limited research has been performed on the utilisation of sorbitol for wood modification. Bateson (1938,1939) conducted experiments on using sorbitol for dimensional stabilisation of the wooden matrix. More recent studies have been performed by Larnøy et al (2018).

Experimental

Chemicals and wood treatments

Powdered citric acid (VWR Chemicals, CAS 77-92-9) and D-Sorbitol (Ecogreen Oleochemicals GmbH, CAS 50-70-4) were used in a 3:1 molar ratio to achieve complete esterification of the citric acid. Ten grams of these solids were dissolved in 8 grams of water by stirring at 20°C. The liquid solution exhibited a pH of 2 and a density of 1.284 g cm$^{-3}$. All pine (Pinus Sylvestries sapwood samples, except for the untreated controls, were impregnated with the solution by performing a 30 min pre-vacuum of 40 mbar followed by a 1, 2 or 3 hour pressure phase at 8 bars. The samples were then cured for 18 hours at a temperature of 140°C.

Tests

To this date the authors have investigated wood treated with polyesterification using sorbitol and citric acid under aqueous conditions against; decay fungi; staining fungi; termites; marine organisms and tested dimensional stability, water uptake, leaching, heartwood impregnability, fire resistance and pilot scale impregnation.
Results and Discussion

The ongoing research of polyesterification of wood using sorbitol and citric acid under aqueous conditions at NIBIO, shows very promising results for a future wood modification system. Both citric acid and sorbitol are low-priced and readily-available feedstock chemicals, an essential prerequisite for a commercial modification process. Moreover, the chemicals are bio-derived; citric acid is mostly produced by microbial fermentation using *Aspergillus niger* (Show et al. 2015) and sorbitol is industrially manufactured from starch by enzymatic hydrolysis to dextrose and catalytic hydrogenation of dextrose to sorbitol (Young and O’Sullivan 2011).

Ongoing trials are now undertaken to investigate the possibilities of fresh wood impregnation. By achieving this, one drying stage would be avoided, and the planed wood shavings would be used for wood based panels with enhanced properties.

Table 1. Tests conducted with sorbitol and citric acid under aqueous conditions

<table>
<thead>
<tr>
<th>Tests performed by NIBIO</th>
<th>Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>Decay resistance (Miniblock)</td>
<td>The treatment shows decay resistance until a solution concentration of 55%</td>
</tr>
<tr>
<td>Decay resistance in soil contact (EN 807)</td>
<td>The test is still running</td>
</tr>
<tr>
<td>Colonization of staining fungi</td>
<td>The treatment prevents staining by discolouring fungi</td>
</tr>
<tr>
<td>Dimension stability</td>
<td>The treatment improves anti-swelling efficiency</td>
</tr>
<tr>
<td>Leachability</td>
<td>Curing at 140°C is necessary to prevent leaching</td>
</tr>
<tr>
<td>Resistance against subterranean termites</td>
<td>Resistance against subterranean termites in non-choice and two-choice tests has been shown</td>
</tr>
<tr>
<td>Resistance against marine borers</td>
<td>The treatment prevented attack by wood borers such as <em>Teredo navalis</em> in field trials</td>
</tr>
<tr>
<td>Heartwood impregnability</td>
<td>Impregnation trials showed widely variable results</td>
</tr>
<tr>
<td>Fire resistance</td>
<td>No fire resistance was achieved. The treatment in combination with additives is ongoing</td>
</tr>
</tbody>
</table>

Figure 1. Treated wood pole (left), with 20cm diameter, treated wood boards with dimensions of 48x98mm (center), treated wood samples in soil contact according to EN 807 and wooden poles in marine tests(right).

Hover over the picture to enlarge

Conclusions

The research on polyesterification of wood using sorbitol and citric acid under aqueous conditions shows very promising results, however, further development is necessary, and some questions remain unanswered. Fire resistance may be improved with additives and reaction time and temperature may be lowered by the use of catalysts. Moreover, further research is needed to clarify the mode of action for biological resistance. Moisture relations seem to be unique compared to other wood modification technologies and this must be further explored. NIBIO will continue to investigate these topics in the years to come.
References


MOISTURE RELATIONS IN WOOD MODIFIED WITH SORBITOL AND CITRIC ACID

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Background

Susceptibility of wood to fungal degradation shortens service life and is one of the primary factors limiting the use of wood in constructions today. Traditionally, biodegradation has been mitigated by treatments with biocides, but the use of these chemicals is increasingly restricted due to environmental and health concerns. Alternatively, resistance to fungal decay can be improved by chemical modification which provides a nontoxic mode of action (Hill 2006). Although commercial chemical modification processes such as thermal modification, acetylation and furfurylation are gaining market share (Jones et al. 2018), growth is constrained by higher production costs compared to traditional wood preservation methods. Thus, a low-cost wood protection system with a non-toxic mode of action is needed. Polyesterification of wood with sorbitol and citric acid appears to be a promising technique. Larnøy et al. (2018) showed that polysorbitol (PS) modified wood cured at 140°C was resistant to leaching of reactant chemicals and provided increased decay protection against brown-rot and white-rot fungi. The exact mechanism behind the increased durability in chemically modified wood remains unclear, but it is generally acknowledged that a critical factor contributing to enhanced decay resistance is moisture content reduction (Thybring 2013, Ringman et al. 2019). This study assesses the moisture content in wood modified with sorbitol and citric acid. Various reactant concentrations provided different levels of modification and the modified samples were assessed for volumetric swelling and measured with low-field NMR (LFNMR).

Experimental

Scoots pine (Pinus sylvestris), birch (Betula pendula) and Norway spruce (Picea abies) sapwood samples were used for volumetric measurements. Sample dimensions were 25 mm in the radial orientation, 25 mm tangential and 15 mm longitudinal. Pinus radiata earlywood, sapwood was used for the LFNMR measurements. Samples were cylindrical with diameter of 6 mm and height of 10 mm. Powdered citric acid (VWR Chemicals, CAS 77-92-9) and D-Sorbitol (Ecogreen Oleochemicals GmbH, CAS 50-70-4) were used in a 3:1 molar ratio for the esterification reaction. Various solution concentrations were used which produced samples with a range of weight percent gains. Samples were impregnated with the solution by performing a
30 min pre-vacuum of 40 mbar followed by a 2-hour pressure phase at 8 bars. The samples were then cured for 18 hours at 140°C and leached according to EN 84. Swelling samples were dried at 103°C and dry weights and dimensions were obtained. They were then vacuum impregnated with water and the swollen samples were weighed again and dimensions obtained. The LFNMR samples were also vacuum impregnated with deionized water and a Bruker mq20 minispec with a 0.47 T permanent magnet (Bruker, Billerica, MA, USA) was used to perform the measurements. A CPMG pulse sequence was used to measure the spin-spin relaxation time ($T_2$) of the samples with a pulse separation ($\tau$) of 0.04 ms, 32,000 echoes, gain 76 dB, 16 scans and a recycle delay of 2 seconds.

Results and Discussion

Bulking coefficients were in the range of 10-20% for all wood species and increased with increasing weight percentage gain (WPG) (fig. 1a). This indicates penetration of the polymerized chemicals within the wood cell wall. Anti-swelling efficiency (ASE) was in the range of 40-50% and decreased with increasing WPG (fig. 1b). Other wood modification systems show a positive correlation between ASE and WPG (Thybring 2013). The negative correlation observed here was likely due to the increased wet volume of the saturated wood at higher WPG (fig. 1c). This super-swelling may be due to the fact that the modification polymer is also hygroscopic. LFNMR results show that at higher levels of WPG a new peak develops (fig. 2) which may represent moisture associated with the hygroscopic modification polymer. Peak 1, which represents water within the cell wall (Fredriksson and Thygesen 2017), does not change substantially with increasing levels of WPG. Other modifications, like acetylation, tend to decrease this peak with increasing WPG (Beck et al. 2017) and this reduction in cell wall moisture content has been thought to be responsible for decay protection in chemically modified wood (Ringman et al. 2019). Enhanced decay resistance in PS modified wood may be explained by another mechanism.

![Figure 1](image1.png)

**Figure 1.** Bulking coefficient (a), anti-swelling efficiency (b) and wet volume (c) of Norway spruce, birch and Scots pine samples.

![Figure 2](image2.png)

**Figure 2.** LFNMR results for radiata pine samples at different levels of WPG. White areas are the average spectrum for 3 replicates.

Conclusions

PS modified wood has a unique interaction with water compared to other wood modification systems. Further research on the wood water relationship in this material may provide broader insight into the mode of action for decay protection in modified wood.
References


APPLICATION OF CHEMICAL BUFFERS TO PREVENT AND REDUCE VOC-EMISSIONS OF DIFFERENT WOOD-SPECIES

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Background

In museums the installation of wooden display cabinets is prohibited due to the VOC’s and their corrosive effects on metals -especially lead. Regularly all materials have to pass the obligatory standard Oddy test which guaranties the material suitability, considering the sensitivity of museum area. Therefore, emission of VOCs of wooden display cabinets in museums need to be reduced and prevented.

In a previous study wood-specimens were sawn, impregnated with alkaline buffer and dried in either vacuum drying or conventional drying processes. The results of the Oddy-test showed variable corrosive decomposition of the lead-coupon which were part of the Oddy-test set up. In the present test series natural and kiln dried wood-specimens were impregnated with neutral buffers and dried after impregnation at indoor climate. Regarding the different drying-conditions of the raw materials, the aim was to determine adapted buffer capacities.

Experimental

Natural and kiln dried specimens of ash (*Fraxinus excelsior* L.) and beech (*Fagus sylvatica* L.) were impregnated with two different buffer systems.

Specimens

The natural dried specimens were sawn in 2017 and stored outside under natural conditions. The kiln dried material was purchased from a local wood supplier. All specimens were prepared with the dimensions of 10 mm x 10 mm x 10 mm.

Buffer systems

For the test series two different buffer systems (McIloaine and Sörensen) with the pH values of 6.5 and 7.0 were used. The specimens were impregnated with the buffer systems for 30 min at 50 mbar and 60 minutes at 6 bar before they were dried at room temperature.

Oddy Test

Beside the investigation of untreated specimens of natural and kiln dried wood, five specimens of each wood species, each treated with buffer systems were tested. According to the required set up one wood specimen, lead (Pb) coupon ( 10 mm x 10 mm x 0,1 mm) and 1 ml of destilled
water (stored in a separated small vessel) was enclosed in a 100 ml vessel (Figure 1). This vessel was stored for 28 days in a drying chamber with a temperature of 60° C. After the test period the degree of corrosion on the metal coupon was rated, differentiated according to Permanent = No corrosion; Temporarily = Light corrosion; Unsuitable = Heavy corrosion.

![Figure 1. Oddy Test Set Up.](image)

Results and Discussion

All samples, native as well impregnated, either natural or kiln dried, showed either no corrosion or light corrosion on the lead-coupon (Table 1). These homogenous results indicate that in contrast to the different drying and buffering procedures another parameter had a levelling influence. Thus the storage time after drying now is considered to have an influence on the rate of the VOCs emissions. This observation matches with Roffael and Uhde (2012) who discovered that the storage time of wood influence the emission-rate of volatile acids from wood.

The marginal differences that has been detected are: Sörensen buffer system provoked less corrosion than the McIloaine buffer system. Beech specimens showed better results at the buffering based on pH value of 7 and Ash specimens based on pH value of 6.5.

<table>
<thead>
<tr>
<th>Raw material</th>
<th>Drying of the raw material</th>
<th>Storage time before impregnation</th>
<th>Drying after impregnation</th>
<th>Oddy test results</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Native specimens</td>
</tr>
<tr>
<td>Natural</td>
<td>Natural drying</td>
<td>About two years</td>
<td>Drying at room conditions</td>
<td>Sörensen</td>
</tr>
<tr>
<td>dried</td>
<td></td>
<td></td>
<td></td>
<td>No corrosion/</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>light corrosion</td>
</tr>
<tr>
<td>Technical</td>
<td>Kiln drying</td>
<td>No information</td>
<td>Drying at room conditions</td>
<td>McIloaine</td>
</tr>
<tr>
<td>dried</td>
<td></td>
<td></td>
<td></td>
<td>No corrosion/</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>light corrosion</td>
</tr>
</tbody>
</table>

These results contradict the expectations, as due to the high drying temperatures of the kiln drying process a higher decomposition rate of the lead coupons were expected for the native kiln dried specimens compared to the native natural dried specimens. And compared to the native specimens the buffering of the specimens partially resulted in higher decomposition rates of the lead-coupons.

Outlook

The present test series showed that the storage time after drying of the raw material influenced the Oddy test results. These results indicate that a right set up for wood drying and storage will allow the passing of the Oddy test without any impregnation of any buffer. Further tests will have to confirm this presumption.
Acknowledgement

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References

MALEIC ANHYDRIDE AND SODIUM HYPOPHOSPHITE AS A POTENTIAL WOOD MODIFICATION SYSTEM

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Background

Modification with cyclic anhydrides forms an ester bond with wood component, whilst providing bound carboxylic moieties capable of undergoing further reaction (Hill and Mallon 1998). In studies of cotton cellulose, it was suggested that sodium hypophosphite (SHP) not only acted as a catalyst, but also seemed to react with the unsaturated carbon of maleic acid to realise a crosslinked maleic acid modified cellulose (Yang et al. 2011), resulting in better properties such as wrinkle resistance and fire properties after laundry (Wu and Yang 2007). As wood has a lower crystallinity, such reactions with hemicelluloses and other constituents could improve the performance of wood products at various conditions such as dimensional stability, durability etc..

In this study, the possibility of modifying wood using maleic anhydride (MA) combined with SHP was investigated.

Experimental

Scots pine (*Pinus sylvestris* L.) sapwood blocks with dimensions of 20×20×10 mm (R×T×L) and a oven-dried density of 583±7 kg/m³ were subjected to Soxhlet extraction using a mixture of acetone: water (4:1, v:v) prior to impregnation with solution of MA in acetone at 15 bar for 1 h. Treated specimens were placed in oven at a range of temperatures, followed by Soxhlet extraction using acetone for 6 h to remove excess MA. Weights and dimensions of extracted specimens were measured to calculate weight percentage gain (WPG) and bulking effect (BE) by MA treatment.

MA treated specimens were vacuum impregnated with aqueous solution of SHP for 0.5 h. For the reaction of SHP and wood-MA complex, impregnated specimens were placed at the corresponding temperature for 6 h.

To remove unreacted chemicals, all specimens were vacuum impregnated with water for 0.5 h and kept immersed under water for 72 h. Water was changed every 24 h. Weights and dimensions of specimens were measured to calculate WPG and BE.

To investigate the formation of the ester bond by MA treatment and reaction of unsaturated carbon after SHP treatment, specimens were analysed with Fourier-transform infrared
spectroscopy (FT-IR) with an attenuated total reflectance (ATR). Peaks at 1725 cm\(^{-1}\) and 1640 cm\(^{-1}\) were observed. All spectra were rescaled against strongest absorption band (1023-1030 cm\(^{-1}\)).

Results and Discussion

Based on bulking effect and FT-IR spectra, MA seemed to penetrate into the cell wall and form an ester bond with cell-wall components (Table 1 and Figure 1). The decrease of peak at 1640 cm\(^{-1}\) was observed after treating wood with SHP, which might be due to the reaction with C=C in MA. The similar tendency was observed in previous study of cotton treated with maleic acid and SHP (Yang et al. 2010). Treating specimens with SHP at low temperature brought not only lower WPG compared to only MA treated specimens (Table 1), but also a decrease of peak at 1725 cm\(^{-1}\) in FT-IR spectra, which indicated a decrease of ester bond present. On the other hand, spectrum of specimens treated with SHP at higher temperature showed a similar intensity of peak at 1725 cm\(^{-1}\). It is possible that the hydrolysis of the ester bond was preferred at lower temperature compared to crosslinking reaction between MA and SHP (Figure 1).

Table 1. Weight percentage gain (WPG) and bulking effect (BE) before and after leaching in water

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Without Leaching</th>
<th>After leaching</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>WPG %</td>
<td>BE %</td>
</tr>
<tr>
<td>MA 3.5M/103°C</td>
<td>16.4±0.39</td>
<td>5.9±0.11</td>
</tr>
<tr>
<td>SHP 3.5M/115°C</td>
<td>19.0±0.28</td>
<td>7.5±0.41</td>
</tr>
<tr>
<td>2M/130°C</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>2M/170°C</td>
<td>—</td>
<td>—</td>
</tr>
</tbody>
</table>

Figure 1. FT-IR spectra of specimens without treatment (untreated), treated with maleic anhydride (MA) and treated with maleic anhydride and sodium hypophosphite (SHP).

Conclusions

The results suggested that an increased reaction temperature favoured the modification of wood with MA and subsequent treatment with SHP. Further studies on the properties of treated material are needed to justify the importance of treatment with the catalyst.
References


DIGITAL TRANSFORMATION OF BIOLOGICAL PROCESSES AND BUILDING DESIGN THEORY – AN APPROACH TO FACILITATION OF SOFTWARE DESIGN FOR SERVICE-LIFE PLANNING OF TIMBER ELEMENTS

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Background

Design software has revolutionized the building design sector, with programs simplifying complex design procedures on a digital platform. The implementation of service-life planning (SLP) to this platform is growing in importance to facilitate more comprehensive architectural design approaches. Service life is defined as the period from installation to when a construction element fails to meet its performance level requirements (Brischke and Rapp, 2007). SLP requires information about material properties and its behaviour to environmental conditions (Brischke et al., 2006). Information on these processes are available throughout scientific literature; however, few attempts have been made to transfer this information and knowledge to the digital landscape (MacKenzie, 2010; Thelandersson et al., 2011). The goal of this paper is to translate material properties and behaviour information to engineering- and software design formats in an attempt to streamline the digital transformation process. Ultimately, an attempt is made to bring together and summarize the current state of service life research.

Experimental

A comprehensive survey of service life research has been conducted to incorporate all the facets of timber construction. All the available prediction tools (models) have been identified and summarized in a database format. An assessment of each model was conducted, using a criteria matrix developed for the purpose. The matrix assesses multiple aspects of each model, from the experimental design used to construct the model, to aspects of its accuracy and practicality. Based on the information captured from assessing each model, an integrated modelling design could be developed. The comprehensive nature of the integrated process retards digital transformation processes as its complex interactions are challenging to describe concisely. The use of flow- and Unified Modeling Language (UML) diagrams have been explored to express the integrated modelling processes in a simplified manner.
Results and Discussion

Dose-response modelling has been identified as the state of the art modelling approach for SLP. Expanding dose-response approaches to various decay mechanisms of timber accommodates the development of an integrated modelling approach. In turn, an integrated model structure optimises information capture and usage through the sharing of variables. It can also lead to the unification of research objectives and identification of missing aspects. Figure 1 illustrates the conceptualisation of SLP, incorporating a dose-response approach to modelling and prediction.

Figure 1: Conceptualising the incorporation of dose-response modelling to service life planning
Conclusions

Available service life models have been identified and assessed. The model assessment was conducted based on model performance and other modelling attributes. Flow- and UML diagrams have proven to conceptualise the dynamics of SLP concisely while integrating various aspects into an overall design structure. Ultimately, the principles of software development have been integrated with SLP in an attempt to facilitate digital transformation.

References


INOCULATION OF SOIL WITH BASIDIOMYCETE FUNGI FOR DECAY TESTS

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Background

Wood decay tests are important for testing different types of wood protection systems. Field tests in Scandinavian climate takes 2-3 years to failure for pine sapwood. Accelerated lab tests have been developed for faster determination of decay resistance. Single strain tests on malt agar or sterile soil is mainly used for basidiomycetes (e.g. EN 113, 1996). However, this type of test does not take the dynamics of the terrestrial microflora into account. Soil beds have been used for decay testing as accelerated field tests. However, the specifications in ENV 807 (2001) favours soft rot over basidiomycetes due to high soil moisture content (MC). Using soils from a field with a known basidiomycete rot type may facilitate an accelerated “field” test against these fungi; however, the exact type of decay activity will be unknown. The aim of this study was to inoculate soils with basidiomycete fungi and evaluate their potential in decay tests. The advantages with inoculated soils over forest soils were hypothesised to be: i) knowledge of presence of fungal species, ii) higher reproducibility due to a more controlled microflora, and iii) overcoming the need for access to forest soil.

Experimental

Two types of commercial organic compost soils, free from pesticides and artificial fertilizers, were used: planting soil (P) and sowing soil (S). Water holding capacity (WHC) was measured according to ENV 807 (2001) (table 1). pH was measured in 35 ml water which had been incubated with 5 g of soil for 2 hours (table 1). Part of the soils were dried in 103°C over night after which dried soil, fresh soil, sand and water were mixed to achieve similar WHC and MC as in forest soils with known BR and WR activity, respectively. The soils were kept in plastic boxes with a ventilated lid.

Five brown rot (BR) fungi and four white rot (WR) fungi were grown on agar plates with Pinus sylvestris and Picea abies sapwood (BR) and Fagus sylvatica (WR), respectively, for 8 weeks. Infected wood pieces were transferred to the designated soils and covered with soil.

Soil MC was measured regularly through weighing of the soil boxes. Water was added to maintain optimal soil MC when needed. P. sylvestris sapwood was used as wood MC controls, according to ENV 807 (2001). Fungal activity was evaluated using cotton cloth according to ENV 807 (2001). When soil MC, wood MC, level of saturation and fungal activity were estimated to be adequate, a decay test according to ENV 807 (2001) was started.
Table 1. Composition and characteristics of soils.

<table>
<thead>
<tr>
<th>Soil</th>
<th>Composition</th>
<th>pH</th>
<th>WHC</th>
<th>MC</th>
<th>Saturation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soils for WR</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>P-VRf</td>
<td>100% fresh</td>
<td>5.6</td>
<td>54%</td>
<td>77%</td>
<td>81%</td>
</tr>
<tr>
<td>P-VRt</td>
<td>90% dried/10% fresh</td>
<td>5.6</td>
<td>54%</td>
<td>77%</td>
<td>81%</td>
</tr>
<tr>
<td>Soils for BR</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>S-BRIWHC</td>
<td>90% dried/10% fresh</td>
<td>6.4</td>
<td>41%</td>
<td>43%</td>
<td>74%</td>
</tr>
<tr>
<td>P-BRIWHC</td>
<td>90% dried/10% fresh</td>
<td>5.7</td>
<td>36%</td>
<td>61%</td>
<td>104%</td>
</tr>
<tr>
<td>P-BRIII</td>
<td>90% dried/10% fresh</td>
<td>5.7</td>
<td>18%</td>
<td>12%</td>
<td>61%</td>
</tr>
</tbody>
</table>

Results and Discussion

In BR soils, wood MC was steady at 20-40% throughout the majority of the test. Wood MC increased to >70% in P-BRII at the end of the test, correlating with major mycelial growth and substantial wood mass loss (ML). Wood MC in the WR soils was fluctuating during the first 1.5 month but then reached a steady level around 40%.

Table 2. ML, wood MC, soil MC and soil saturation during the decay test.

<table>
<thead>
<tr>
<th>Soil</th>
<th>Pine sapwood</th>
<th>Beech</th>
<th></th>
<th>Soil saturation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>ML 16w ave.</td>
<td>ML 32w ave.</td>
<td>ML 16w ave.</td>
<td>ML 32w ave.</td>
</tr>
<tr>
<td>P-BRII</td>
<td>19% 9% 53% 13%</td>
<td>52% 10% 40% 21%</td>
<td>3% 1% 14% 5%</td>
<td></td>
</tr>
<tr>
<td>P-BRIWHC</td>
<td>1% 0% 5% 3%</td>
<td>8% 6% 24% 7%</td>
<td>6% 1% 16% 2%</td>
<td></td>
</tr>
<tr>
<td>S-BRIWHC</td>
<td>1% 0% 12% 14%</td>
<td>5% 3% 26% 2%</td>
<td>6% 1% 15% 3%</td>
<td></td>
</tr>
<tr>
<td>S-VRf</td>
<td>4% 0% 3% 0% 5% 2% 43% 11%</td>
<td>40% 1% 78% 2%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S-VRt</td>
<td>3% 0% 3% 1% 4% 1% 67% 41%</td>
<td>39% 2% 76% 4%</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

In BR-II, wood specimens reached ML values similar to those seen in ENV 807 with 19% ML in pine sapwood after 16 weeks and 53% after 32 weeks (table 2). Also beech samples in this soil reached high ML values, 52% after 32 weeks. However, even though the soil had been inoculated with five different brown rot fungi, only one seemed to grow based on the visual characteristics of the mycelium. Hence, this «microcosm» may have consisted of only one organism, presumably due to that the environment in the soil bed favoured this specie. In all other tested soils, wood specimens did not reach adequate ML.

The problem with obtaining adequate WR activity may be linked to the findings in Brischke and Wegener (2019), who showed that wood MC increased sharply at soil MC levels close to saturation. At insufficient soil MC, wood MC may have been too low for WR decay, while adding just a little bit more water may have led to a wood MC that favoured soft rot over WR.

Conclusions

The study showed that it was possible to inoculate BR fungi in a commercial soil adjusted to a WHC and MC close to that of a forest soil with known BR activity. However, apparent dominance of one of five inoculated BR species led to questioning of this method for creating microcosms. For WR fungi, the problem with obtaining a suitable wood MC was not overcome in this study. To draw conclusions on reproducibility, a more comprehensive test needs to be conducted.

References


STATIC WETTABILITY OF THERMALLY MODIFIED TEAKWOOD

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Background

In Brazil, fast-growth plantations of Tectona grandis L.f. are thinned prior to the final harvest (16 to 22-year cycle). Thinned trees have a small diameter, resulting in small dimension (transversal section) juvenile lumber, but with the formation of some heartwood.

Thermal modification is a suitable process to homogenize the color of sapwood and heartwood (Lopes et al., 2014a), as well as to improve the dimensional stability (Lopes et al., 2014b) and the durability of juvenile teakwood against rot-fungi (Pratiwi et al., 2019).

On the other hand, the process decreases surface wettability, mainly by reducing the number of primary sorption hydroxyl sites, which are degraded together with hemicelluloses (Hill, 2006). Wettability is a key factor regarding the quality of adhesion and finishings adherence, due to the interaction needed between these products and the wood surface (Tshabalala, 2005).

Discrepant results have been found in literature about the effect of thermal modification on surface wettability, due to the different processes and species studied (Jirouš-Rajković; Miklešić, 2019). The aim of this study was to assess the wettability of thermally modified sapwood and heartwood of teak.

Experimental

Material, process and treatments

Fifteen-year-old thinned trees planted in the state of Mato Grosso, Midwestern Brazil were broken down into flat sawn slats (25 mm thickness x 60 mm width and variable length), which were thermally modified in a company located in the state of Paraná, Southern Brazil. It was used a hygrothermal closed system process, carried out in five steps (around 16 h): initial heating up to 110 °C; holding 110 °C for 25 minutes; heating up to 160 °C; holding 160 °C for 45 minutes; cooling down to 65 °C. Twenty four slats were sampled, 12 from sapwood and 12 from heartwood, which were sawn into specimens of 25 x 25 x 50 mm (radial x tangential x longitudinal) and then conditioned in a climatic chamber (20 °C e 65% relative humidity).

Measurement of contact angle

Wettability was measured by the static contact angle formed between distilled water and the surface of the specimens. A Krüss Drop Shape Analyzer DSA100 version 1.92 (Hamburg, Germany) was used in a climate-controlled room, using a dosing syringe of 0.5 mm diameter and 100 μl capacity. The distance between the needle and the material surface was 3 mm.
Measurements were taken every 10 seconds for 120 seconds, totaling 12 contact angle readings for each water drop deposited on the surface. The specimens were measured on three points: the central part and at 10 mm from the tops.

Results and Discussion

In Table 1 are presented the averages of contact angle after 10 s and 120 s, respectively the first and last measurements. Higher contact angles mean lower wettability and vice versa. According to the results, the same behavior was noticed at 10 and 120 s, where heartwood (untreated and thermally modified) had the highest averages (p>0.05ns), followed by thermally modified sapwood and sapwood.

Table 1. Results of the statistical analysis

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Contact angle after 10 s (°)</th>
<th>Contact angle after 120 s (°)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Average</td>
<td>Kruskal-Wallis score</td>
</tr>
<tr>
<td>Untreated heartwood</td>
<td>86,03</td>
<td>97,53 a</td>
</tr>
<tr>
<td>Thermally modified heartwood</td>
<td>86,51</td>
<td>99,88 a</td>
</tr>
<tr>
<td>Untreated sapwood</td>
<td>37,98</td>
<td>19,43 c</td>
</tr>
<tr>
<td>Thermally modified sapwood</td>
<td>78,59</td>
<td>69,38 b</td>
</tr>
<tr>
<td>H-test</td>
<td>89,35*</td>
<td></td>
</tr>
</tbody>
</table>

Scores followed by the same letter in a column do not differ according to the H-test test of Kruskal-Wallis (p>0.05). *Significant at 5% (p<0.05).

Comparing untreated wood, heartwood was less wettable than sapwood, which can be explained by the highest content of extractives (Garcia and Marinonio, 2016) which can play a role as water-repellents. Thermally modified sapwood turned less wettable than untreated sapwood, probably due to the increase of extractives content (Brito, 2017) imparted by the mass loss caused by the process. These results were the same as those reported by Lopes, Garcia and Nascimento (2018), who also studied thermally modified (juvenile 12 years-old) teakwood, but with a different process and temperatures (180 and 200 °C, open system). However, thermally modified heartwood had the same wettability as untreated heartwood, differently from the reported by Lopes, Garcia and Nascimento (2018). According to Pratiwi et al. (2019), thermal modification (open system, inert atmosphere) can reduce the extractives content of mature heartwood (40 years-old).

Conclusions

Sapwood had higher wettability than heartwood. Thermal modification diminished the wettability of sapwood but non-significant results were found for heartwood. For further elucidation of the results, the next steps will be the determination of the extractives contents and to perform diffuse reflectance infrared Fourier transform spectroscopy (DRIFT) analysis of the chemical surfaces of wood.


COMPARISON OF DIFFERENT RECIPES FOR THERMAL MODIFICATION OF EUROPEAN TONE WOOD AND THEIR INFLUENCE ON THE ACOUSTIC BEHAVIOUR

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Background

Until today, a variety of instruments are made of wood. Especially in the field of string instruments, wood is the material of choice, due to its good resonance qualities. The requirements on tonewood however are very high compared to ordinary lumber. In order to be used in instruments, wood needs to be very dimensional stable, has to have sufficiently high mechanical properties, and of course its acoustic behavior needs to be appropriately high, depending on the component and the instrument in which the material should be used (Wegst 2006). Due to their superiority concerning most of these properties, tropical woods like indian rosewood (Dalbergia latifolia [L.]) or cedro (Cedrela odorata [L.]) are chosen over European wood species for the construction of string instruments in particular. Over the last few years however the research on thermal modification of wood have led to the development of processes for the creation of tonewoods derived from European wood species with improved acoustic behaviour (Pfriem 2006, Zauer et al. 2016). One aim of this research is to reduce the deployment of tropical woods in the instrument industry by substituting them with European species. In this study, the effect of three different thermal modification processes on the acoustic properties of four european wood species was examined.

Experimental

The four European wood species, wild service tree (Sorbus torminalis [L.]), common ash (Fraxinus excelsior [L.]), black alder (Alnus rubra [Bong.]) and sycamore maple (Acer pseudoplatanus [L.]), were thermally modified at 160 °C, 180 °C and 200 °C with a heating rate of 10K/h and 3 hours holding phase. The change in colour was measured in the CIE-L*a*b* colour space. The L*-Figure, which represents the brightness, serves as an indicator for the grade of modification according to Patzelt et al. (2003). The acoustic values, eigenfrequency and damping, were measured by means of experimental modal analysis. The dynamic E-Modulus was calculated according to the Euler-Bernoulli beam theory. Resonance quality was calculated from the ratio between E-Modulus and density, according to Ono and Norimoto (1983). The property changes due to thermal modification were determined by the comparison of modified specimen to unmodified twins.
Results and Discussion

Figure 1 shows the change of the dynamic E-Modulus and density in percentage-based relation to the change in brightness. An increase of the dynamic E-Modulus due to thermal treatment was measured for all species except maple, where the E-Modulus decreased with higher modification-temperature. At the same time, a loss of density occurred in relation to a decline of the brightness.

Figure 2: Change of dynamic E-Modulus $\Delta E_{\text{specimens}}$ and density $\Delta \rho_{\text{specimens}}$ in relation to the change in brightness $\Delta L^*$ due to thermal treatment

Figure 2 shows the absolute values for the resonance quality and damping of the different species, unmodified and after thermal treatment respectively. An increase of the resonance quality due to thermal treatment was detected for all species. For alder however, the optimal treatment temperature for the improvement of the resonance quality was 160 °C. At the same time, the damping decreased due to thermal treatment. Only exception was ash, where the damping increased at treatment temperatures of 160 °C and 180 °C.

Figure 3: Absolute values of resonance quality and damping Q-1 for the different species (unmodified 160°C 180°C 200°C)

Conclusions

Resonance quality and damping serve as the main variables for the assessment of resonance wood. The higher the resonance quality and the lower the damping, the better the acoustic behaviour. Therefore it could be shown, that thermal treatment led to an improvement of the acoustic behaviour for all species. However the results are closely related to the wood species and treatment temperature.
References


SOUND ABSORPTION COEFFICIENT OF THERMALLY MODIFIED AND UNMODIFIED WOOD SPECIES MEASURED IN AN IMPEDANCE TUBE

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Background

Different parts of the guitar body are made of certain wood species, selected by their specific properties. The sound board, which is mainly made of spruce or cedar, transfers the vibration of the strings to the resonance body by means of the bridge. The sides and the back of the guitar rather reflect the sound of the acoustic vibrations and are predominantly made of tropical hardwoods like palisander, mahogany or ebony species. These species have limited availability and are listed on the CITES Annex I and II. To substitute tropical wood species, thermally modified European wood species with improved acoustic properties and dimensional stability may be used (Pfriem 2007, Pfriem 2015, Zauer et al. 2016). Although the acoustic reflection of the sides and the back parts of a guitar body is an important property, there is not much knowledge about this reflection behaviour of wood. A few studies like the work of Hong (1989), Kang (2010) or Smardzewski et al. (2013) investigated the sound absorption properties of unmodified wood. Subsequently this study considers the sound absorption coefficients of unmodified and thermally modified European wood species in comparison to an unmodified tropical wood species.

Experimental

The sound absorption coefficients were measured in an impedance tube (measuring device AED 1000 Type II – Acoustic Engineering Dresden which works in a frequency range of 50 to 2000 Hz) according to DIN EN ISO 10534-2 at the acoustic lab of the Technical University of Berlin. Sycamore maple (Acer pseudoplatanus [L.]) which is used for manufacturing of side and back parts of guitars, black alder (Alnus rubra [Bong.]) which is not commonly used and Brazilian rosewood (Dalbergia nigra [Fr. All.]) as a reference for tropical wood species were investigated. The thermal modification was done in a nitrogen atmosphere with a heating rate of 10 K/h, a maximum temperature of 160°C, 190°C and 210°C and each treatment for 3 hours at maximum temperature. The cylindrical samples (diameter 100 mm, height 25 mm) were produced on a CNC machine and conditioned by 20°C and 50% RH. The test setup consisted of one sample for each wood species and modification treatment.
Results and Discussion

Figure 1 shows the sound absorption coefficient of unmodified and thermally modified maple (AH) and alder (ER) as a function of the frequency (octave bands range from 125 to 1000 Hz). The absorption coefficient describes the ratio of the absorbed sound-intensity to the total incident sound intensity. It can takes values from 0 (no absorption) to 1 (absolute absorption). The low absorption coefficients under 0,1 signify a reverberant behaviour of the wood species. For maple the highest modification type III caused the lowest absorption coefficient up to 0,02 across the whole frequency range and for both wood species maple and alder. For the unmodified type and modified type I and II a slightly higher absorption coefficients of 0,03 to 0,05 were obtained. For alder the lowest absorption coefficients between 0,01 and 0,04 within the range up to 500 Hz were detected for modification type I and III. The unmodified and modification type II alder specimens had slightly higher absorption coefficients in the range from 0,05 up to 500 Hz. In addition, the value improves to 0,03 compared to type I and III which increased up to the highest absorption coefficient of 0,06.

Figure 2 shows the sound absorption coefficient for the tropical reference wood species Brazilian rosewood. It is noticeable that the absorption behaviour is similar to the one of maple type III.

Conclusions

The study showed a particularly decrease of the sound absorption behaviour due to the thermal modification particularly for maple which is comparable with Brazilian rosewood. Also a thermal modification of alder achieved a decrease of the absorption especially in the frequency range up to 500 Hz. Even if wood in general is reverberant, the results indicated that slight differences between the three different wood species and the state of being thermally modified or unmodified occurred. Therefore further research needs to be done to get more evidence, concerning sound absorption behaviour of native wood and the influence of a thermal modification.
References


SURFACE COATING OF PYROLIZED AND NATIVE WOOD SURFACES WITH CALCIUM CARBONATE CRYSTALS BY BIOMINERALIZATION

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Background

In a former project pyrolized surfaces for wooden facades with defined surface depth have been developed. The evaluation of the mechanical performance showed an adhesion of the surface layers worthy of improvement with additional coatings (Gengnagel et al. 2017).

Using biomineralization, it could be shown that the durability of building materials can be improved (Gaylarde et al. 2003) or concrete cracks can be closed. Processes such as microbially induced calcium carbonate precipitation (MICP) are widely known (Vos et al. 2011) and have been developed for concrete and other mineral materials and surfaces (Jonkers 2011).

However, the knowledge about the targeted preparation and production as well as the application of biomineralization on wood and especially on pyrolyzed wood layers for the bonding of the layers is scarce.

Content of a research project was the study of biomineralization with Sporosarcina pasteurii as a biological surface coating of pyrolyzed wood surfaces.

Material and Methodology

The research had three main aspects: the selection of suitable species of wood, the comparison of suitable coating methods and the selection of formulations to trigger the induced biomineralization.

Selection of types of wood
For the test series, wood species with pH values between 8.5 and 9.5 were sought, since the biomineralization takes place in an alkaline medium at these pH values.

Surface coating methods
Surfaces can be coated by various techniques (dipping, painting, spraying). For applying of the solution, it was important to choose a low-loss and metered process.

Formulations to trigger induced biomineralization
Different formulas that create a precipitation environment allowing an induced biomineralization are described in the literature (Bhaduri et al. 2016, Ghosh et al. 2019). In terms of level of detail and cost-effective production a suitable formulation had to be found.
Results and Discussion

From the three randomly preselected types of wood which are highly available on the market, ash is selected. The ring-porous hardwood's pH forms a good basis for the pH increase in the biomineralization of calcium carbonate (Figure 1). A high proportion of wood rays and the absence of resin channels ensure a good absorption capacity at the surface. As a surface coating method, the application with a pipette is selected. This method provides a largely sterile and exact dosing application of the reaction solution. For the preparation of the reaction solution, Bhaduri's approach was chosen, and Ghosh's formula is used to ensure the initiation of induced biomineralization.

![Figure 1. Formation of calcium carbonate by Sporosarcina pasteurii on the surface of ash wood.](image)

The quantitative detection of the microorganisms was carried out with the aid of a Neubauer counting chamber. A cross-cut test according to DIN EN ISO 2409 may be used to test the adhesive strength of the applied coating.

Conclusions

A defined experimental setup for the surface coating of wood with biomineralized calcium carbonate could be developed. The specimens are coated with the two reaction solutions by the repeated application with a pipette. In a following research, the developed procedure will be carried out. The aim is the proof of concept of the found setup. In addition, it will be investigated to what extent ash wood shavings can be bound together using biomineralization.

References


DIN EN 2409: 2013-06, Paints and varnishes - Cross-cut test
Background
The UK has a British Standard for external timber cladding, BS8605-1, in which support battens are defined as timber components of a section, grade, and durability, suitable for supporting external timber cladding. The term includes both battens (which are used horizontally or diagonally) and counter battens (which are only used vertically). Clause 17 states that where the density of timber for support battens is required, this should be determined in accordance with EN384. This is because design of support battens might involve structural calculation, for which a characteristic density value (5th percentile, with appropriate adjustments) is necessary. This characteristic density is conservative on the side of underestimating real density, since this density value is used as an estimator for fixing performance and charring rate (fire resistance). Minimum mean density, which is calculated from characteristic density (a factor of 1.2 in EN384), is used as a threshold for reaction to fire classification without need for further testing.

This paper examines the effectiveness of grading by ring width for characteristic density, using UK and Irish grown British spruce as the case study. British spruce has relatively wide growth rings compared to other common timbers. There is a separate question as to whether ring width affects fastener performance and charring rate separately from density, but for this paper it is assumed that the equations given in EN384 and EN1995-1-1 are correct for any ring width.

Experimental
Timber was sampled from both the UK and Ireland. Both countries have a similar temperate maritime climate and manage their spruce forests in a similar way. The primary wood processing industry is active across the borders of the two countries. Approximately 92% of these spruce forests are Sitka spruce (Picea sitchensis), with the remaining 8% Norway spruce (Picea abies). Sawmills do not differentiate between these two species and they are processed and sold together. The sampling for this paper makes use of 878 battens collected as part of projects funded by Coillte, and the SIRT network (a combination of Forestry Commission and industry funding). All of this timber was sourced from sawmills, and it is representative of normal production. It was of saw-falling quality, kiln dried, and was kindly donated by Glennon Brothers Timber, Murray Timber Group, BSW timber and James Jones and Sons. The timber was not sorted for quality, except for removal by obvious EN14081-1 visual override criteria (excessive mechanical damage from transport and handing, severe wane, clearly apparent
strength reducing growth defects and decay). The specimens are all small cross-section (Table 1), broadly similar to those used for cladding support battens (see also BS5534).

The ring width was measured at both ends of each batten at approximately 12% to 18% moisture content. Due to the small cross-section dimension this measurement could not be fully compliant with BS4978, and while it does not include the pith, it may include rings next to the pith. Density was measured after testing in four point bending in accordance with EN408 and EN384. This is density of a portion cut from the test specimen rather than density of the whole batten, and was adjusted to 12% moisture content as in EN384. The average moisture content at time of testing was 11.6% (Coefficient of Variation, CoV, 9%).

Characteristic density was calculated in accordance with EN384 based on five subsamples (three locations in Ireland and two locations in the UK). The parametric method in EN14358 was used with 75% confidence, normal distribution for density, and the exact method for calculating \( k_{s(n)} \). However, instead of applying a ring width limit from a grading standard a range of ring width limits were applied to see how well they grade the timber for density.

Table 1. Details of the sampling (ungraded, only obvious visual override removed) [CoV]

<table>
<thead>
<tr>
<th>Nominal cross section</th>
<th>27 x 37 (mm)</th>
<th>22 x 47 (mm)</th>
<th>35 x 47 (mm)</th>
<th>44 x 47 (mm)</th>
<th>47 x 47 (mm)</th>
<th>All Without pith</th>
<th>With pith</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nominal length (m)</td>
<td>3.0</td>
<td>2.4 – 3.6</td>
<td>2.4 – 3.6</td>
<td>2.4</td>
<td>3.0</td>
<td>2.4 – 3.6</td>
<td></td>
</tr>
<tr>
<td>Number measured</td>
<td>106</td>
<td>418</td>
<td>204</td>
<td>50</td>
<td>100</td>
<td>878</td>
<td>726</td>
</tr>
<tr>
<td>Simple mean density (kg/m³)</td>
<td>419 [12%]</td>
<td>423 [12%]</td>
<td>395 [10%]</td>
<td>427 [11%]</td>
<td>429 [12%]</td>
<td>417 [12%]</td>
<td>416 [12%]</td>
</tr>
<tr>
<td>5th %ile density (kg/m³)</td>
<td>332</td>
<td>339</td>
<td>328</td>
<td>341</td>
<td>340</td>
<td>335</td>
<td>333</td>
</tr>
<tr>
<td>Mean / 5th %ile</td>
<td>1.26</td>
<td>1.25</td>
<td>1.21</td>
<td>1.25</td>
<td>1.26</td>
<td>1.25</td>
<td>1.23</td>
</tr>
</tbody>
</table>

Results and Discussion

Although the mean ring width of both ends of the batten is correlated with density (\( R^2 = 0.30 \)) and is known to affect density via the ratio of earlywood to latewood (Moore 2011), the ring width is of limited use when grading for characteristic density to EN384, as shown in Table 2.

Table 2. Density and yields above and below ring width limits (mean ring width of both ends)

<table>
<thead>
<tr>
<th>Ring width limit (mm)</th>
<th>Below &amp; = to limit</th>
<th>Above limit (bigger rings)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield (%)</td>
<td>Simple mean dens (kg/m³)</td>
<td>5th %ile density (kg/m³)</td>
</tr>
<tr>
<td>14</td>
<td>100</td>
<td>417</td>
</tr>
<tr>
<td>13</td>
<td>100</td>
<td>417</td>
</tr>
<tr>
<td>12</td>
<td>98</td>
<td>417</td>
</tr>
<tr>
<td>11</td>
<td>97</td>
<td>418</td>
</tr>
<tr>
<td>10</td>
<td>96</td>
<td>419</td>
</tr>
<tr>
<td>9</td>
<td>92</td>
<td>421</td>
</tr>
<tr>
<td>8</td>
<td>86</td>
<td>424</td>
</tr>
<tr>
<td>7</td>
<td>74</td>
<td>429</td>
</tr>
<tr>
<td>6</td>
<td>59</td>
<td>436</td>
</tr>
<tr>
<td>5</td>
<td>43</td>
<td>443</td>
</tr>
<tr>
<td>4</td>
<td>24</td>
<td>457</td>
</tr>
<tr>
<td>3</td>
<td>8</td>
<td>470</td>
</tr>
<tr>
<td>2</td>
<td>0</td>
<td>514</td>
</tr>
</tbody>
</table>

Conclusions

Ring width is not useful for grading British spruce cladding battens for characteristic density since it results in high reject for modest density increase. Instead it is appropriate to simply assume the characteristic density of the ungraded population. The density of these small cross-section boards is the same as that in the literature for UK-grown Sitka spruce (Moore 2011).
References

STRENGTH CLASSES OF SCOTS PINE WOOD GROWN IN THE PLANTATIONS OF DIFFERENT INITIAL STAND DENSITY

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Background

The objective of this study was to determine strength classes of Scots pine wood, depending on different initial stand density and thinning intensity in Scots pine experimental plots according to EN 338. The problematic question is how thinning intensity and different spacing could affect pine wood strength classes? This topic is important to get knowledge about influence of thinning on wood quality. Wood quality of Sitka spruce was determined of 5 different spacing in Northern Ireland (Moore and etc. 2009). Wood quality of Sitka spruce, Norway spruce and Douglas fir of different time’s thinned stands was determined in Republic of Ireland (Krajnc and etc. 2019). Douglas fir strength classes was analyse in Wallonia, Southern Belgium (Henin and etc. 2018). This study shows differences of wood quality in Scots pine experimental plots in forest land and agricultural land. Initial stand density and thinning intensity is the main parameters of differences of wood quality.

Experimental

Two experimental plots of Scots pine were chosen for wood quality determination. First plantation was established in agricultural land and second in forest land. Both objects grow on arenosols soil. Objects are divided in 10 experimental area plots and grouped in pairs. Material is taken from 3 different pairs with different initial stand density and thinning intensity. 12 model trees from each pair are taken to determine wood quality parameters: mean global modulus of elasticity (MOEg), 5% wood density (ρ) and 5% bending strength (f_m). All model trees are taken from thinning from below. 50x50x1000 mm battens were cut from model trees for MOEg and f_m determination according EN 408. Density (ρ) was determined of timber cut as close as possible to the fracture location and measured immediately after testing. Moisture content was determined using the oven dry method as per EN 13183-1. All tested properties were adjusted to 12% moisture content using the equations provided by EN 384.

Results and Discussion

The main parameters of different lands and thinning intensity area plots are show in Table 1. It was determined, that all measured samples from these experiment plots can’t be classified to
strength classes. It could be cause of high proportion of juvenile wood because of young stand age. Because of that we decide to calculate how many percent of samples we must reject to reach C14 and higher classes.

**Table 1. Main parameters of Scots pine plantations**

<table>
<thead>
<tr>
<th>Land</th>
<th>Stand age</th>
<th>Initial stand density</th>
<th>Thinning intensity</th>
<th>Thinning times</th>
<th>MOEg N/mm²</th>
<th>fₘ 5% N/mm²</th>
<th>ρ 5% kg/m³</th>
<th>Strength class</th>
</tr>
</thead>
<tbody>
<tr>
<td>Agricultural</td>
<td>34</td>
<td>4000-5000</td>
<td>30%</td>
<td>3</td>
<td>6418,65</td>
<td>12,89</td>
<td>372,81</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>34</td>
<td>2400-2000</td>
<td>50%</td>
<td>2</td>
<td>6147,35</td>
<td>11,52</td>
<td>372,67</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>34</td>
<td>1200-1000</td>
<td>66%</td>
<td>1</td>
<td>5352,28</td>
<td>11,58</td>
<td>358,00</td>
<td>-</td>
</tr>
<tr>
<td>Forest</td>
<td>27</td>
<td>4000-5000</td>
<td>30%</td>
<td>3</td>
<td>7240,70</td>
<td>11,00</td>
<td>379,90</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>27</td>
<td>2400-2000</td>
<td>50%</td>
<td>2</td>
<td>6789,16</td>
<td>11,49</td>
<td>396,76</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>27</td>
<td>1200-1000</td>
<td>66%</td>
<td>1</td>
<td>6406,34</td>
<td>12,48</td>
<td>361,92</td>
<td>-</td>
</tr>
</tbody>
</table>

Table 2 shows that in agricultural land plantation we need to reject from 89.29% to 72.29% samples for construction usage. In forest land we need to reject from 68.89% to 11.54% samples. From 7.41% to 19.28% of sample is in C14 class in first experimental plot. In second sample plot fall into C14 class from 19.4% to 58.97% of samples. C16 class is from 7.41% to 2.38% in agricultural land and in forest land from 10% to 21.8% of samples.

**Table 2. Rejected samples percentage of different area plots**

<table>
<thead>
<tr>
<th>Land</th>
<th>Stand age</th>
<th>Initial stand density</th>
<th>Thinning intensity</th>
<th>Number of samples</th>
<th>To reach C14</th>
<th>To reach C16</th>
<th>To reach C18</th>
</tr>
</thead>
<tbody>
<tr>
<td>Agricultural</td>
<td>34</td>
<td>4000-5000</td>
<td>30%</td>
<td>83</td>
<td>72,29%</td>
<td>91,57%</td>
<td>97,60%</td>
</tr>
<tr>
<td></td>
<td>34</td>
<td>2400-2000</td>
<td>50%</td>
<td>108</td>
<td>78,70%</td>
<td>86,11%</td>
<td>93,52%</td>
</tr>
<tr>
<td></td>
<td>34</td>
<td>1200-1000</td>
<td>66%</td>
<td>84</td>
<td>89,29%</td>
<td>97,62%</td>
<td>-</td>
</tr>
<tr>
<td>Forest</td>
<td>27</td>
<td>4000-5000</td>
<td>30%</td>
<td>78</td>
<td>11,54%</td>
<td>70,51%</td>
<td>92,31%</td>
</tr>
<tr>
<td></td>
<td>27</td>
<td>2400-2000</td>
<td>50%</td>
<td>67</td>
<td>59,70%</td>
<td>79,10%</td>
<td>92,54%</td>
</tr>
<tr>
<td></td>
<td>27</td>
<td>1200-1000</td>
<td>66%</td>
<td>90</td>
<td>68,89%</td>
<td>88,89%</td>
<td>98,89%</td>
</tr>
</tbody>
</table>

**Conclusions**

Despite of younger age, Scots pine plantation growing in forest land have higher wood quality values than plantation growing in agricultural land. The limiting parameter to strength classes is low value of mean MOEg. Lower thinning intensity and higher initial stand density cause higher quality of wood.

**References**

EN 384:2016 - Structural timber - Determination of characteristic values of mechanical properties and density.
EN 408:2012 - Timber structures - Structural timber and glued laminated timber - Determination of some physical and mechanical properties.
EN 338:2016 - Structural timber - Strength classes.
WOOD FUEL IN ENERGY PRODUCTION - FINLAND CASE STUDY

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Background

Concerns about the environmental performance of energy production from fossil fuel are increasing all over Europe. As a member of the European Union, Finland has committed to reach the climate and energy targets, such as reducing domestic emissions of greenhouse gases (by at least 40% by 2030 compared to 1990 level) and increasing the national share of renewable energy in the gross final consumption (to 27% by 2030). The Finnish government support electricity production based on wind power, biogas, forest chips and wood fuels (e.g. Finland’s national action plan for promoting energy from renewable sources pursuant to Directive 2009/28/EC). Wood is one of the country’s main natural resources – more than three-fourths of the land is forested. Therefore it is not surprising that the use of wood-based fuels is promoted (e.g. “heat bonus” allocated to CHP plants).

In order to meet national and European Union climate objectives Finland decided to phase out fossil-based energy. Recent government proposal approved by the Finnish Parliament states that the coal will be banned from use as an energy source by 2029, except in an emergency. Nowadays coal generates about 9% of the country’s power needs, whereas one-quarter of total energy supply is derived from a wood-based fuels (Figure 1.). The ban is expected to increase even more the role of wood biomass in energy production in Finland.

![Figure 1. The distribution of different energy sources in the total energy supply in Finland in 2017 (Finland’s Integrated National Energy and Climate Plan)](image)
Objectives

The aim of this study is to assess environmental impact of the Finland’s decarbonization plan. Expected outcome of this work is to provide a comprehensive cradle-to-grave analysis of coal and wood-based fuel. Access to the specific, objective data can deliver basis for a substantive discussion about the transition in energy production not only in Finland but also in Europe.

Technically speaking wood is a renewable source of energy, because trees can be replanted to repay carbon debt over time. However, intensified wood harvesting causes deforestation, is not fully climate-neutral and poses a threat to the biodiversity. For instance, some forest species depend on decaying wood to survive. Moreover the immediate CO₂ emission from wood biomass burning depends on the wood type and could be even higher than from coal burning (Table 1.). In principle, a molecule of CO₂ emitted to the atmosphere at present have the same impact on global warming whether they came from million years old coal or biomass grown just short time ago. When evaluating environmental impact of biomass as fossil fuel substitution the harvesting, processing, transport distance and supply chain emission should be considered.

Table 1. Lower heating value and CO₂ emission of coal and wood-based fuel

<table>
<thead>
<tr>
<th></th>
<th>CO₂ emission [kg CO₂/ GJ]</th>
<th>lower heating value (LHV) [MJ/kg]</th>
</tr>
</thead>
<tbody>
<tr>
<td>COAL</td>
<td>94</td>
<td>22.7</td>
</tr>
<tr>
<td>WOOD FUEL</td>
<td>112</td>
<td>15.6</td>
</tr>
</tbody>
</table>

In terms of energy the lower heating value (LHV) of wood-based fuel tends to be lower than from coal combustion (Table 1.). However, heating value of wood fuel depends on many factors i.a. tree species, resin content (considerably higher LHV), moisture content (lower MC result in higher LHV) and chemical composition. Typically, coniferous have more extractives and more lignin than do deciduous, which accounts for the slightly higher heating value. Moreover, different tree components may vary in the LHV parameter. While the heating value of bark has been reported to be higher than that of wood in many species, in Norway spruce however bark has a lower heating value than wood. The mineral content of clean wood is 0.1% to 0.6% and that of bark 3% to 5%. Mineral matter is transformed in ash during combustion and gasification. The ash content of wood grown in the temperate zones is 0.1-1.0%, whereas bark contains 3-8% of ash yield. Increased ash content may generate some troubles, i.e. more particulate emission and additional costs of adaptation or device maintenance. Coal has considerably higher ash content than wood (4-16% depending on its origin). Although the LHV parameter for the coal is high burning it in a power plant result in sulfur oxides and nitrogen oxides emissions, generating air pollution problems.

In replacing fossil-fuel by wood-based fuel it is supposed to use logging residues (like tree tops, needles/leaves, stumps, roots). They are plentiful and do not have any industrial use yet, since they are unsuitable as raw material. Roots and stump residuals could be more problematic not only because of the higher harvesting costs and CO₂ emission but also due to mineral soil content. It is crucial to find an efficient way to fire such a demanding fuels, as, for example, fluidised bed combustion (FBC) to create greater value out of residuals than in conventional processes.

In Finland phase-out of coal is expected to lead to an increase in use of wood-based fuel for electricity and heat. It has a positive climate impact if considered in a long time horizon, so that trees would be replaced and offset the primary emission. Otherwise, wood bioenergy may trigger ‘carbon debt’ because of the amount of CO₂ released in a short time. Hence wood
burning could be somewhat a hazardous alternative to coal. Active management should result in grow of net primary production (NPP) in forests while more energy is extracted from them.

References


Finland’s Integrated National Energy and Climate Plan (2018). Draft version submitted to the European Commission


Ministry of Employment and the Economy (2009) Finland's national action plan for promoting energy from renewable sources pursuant to Directive 2009/28/EC


STATE-OF-THE-ART OF WASTE WOOD IN FINLAND

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Background

The EU Waste Framework Directive (2008/98/EC) requires a five-stage waste hierarchy for waste management in the member countries of the EU. The objective of the waste hierarchy is to move the amounts of waste from the lowest level of the hierarchy (landfill waste) to the next level (recovery) and further upstream (recycle or reuse) to reduce waste (Ec.europa.eu, 2019). This study aims to assess waste wood management in Finland.

Finland generates about 3,638,000 tonnes of waste wood each year. Based on the characteristics of the waste wood (Alakangas and Impola, 2014), it can be classified in four (4) categories: A, B, C and D (Alakangas and Wiik, 2019). Energy recovery is almost only used for waste wood and sorting is done very limited, only for wood packages and D class wood.

Experimental

This study search for information from various recent reports regarding the sources, markets for, and management of wood waste. The study is based on literature and benchmarking. Material is compiled by statistics produced by Statistics Finland, Luke and Eurostat providing a mass balance approach based on input-output analysis of the entire system.

A goal is to give a good understanding of the state-of-the-art of wood waste in Finland with aspects: general goals of waste management, economic aspects, environmental aspects, and social aspects. The information will inform any future development of options aimed at ensuring wood waste is managed in a way that delivers the best outcome for the environment and economy.

Results and Discussion

Finland produced 3,638 million tonnes (Table 1) of waste wood during 2017 (Stat.fi, 2019). By industry, the most significant sources for waste wood were pulp, paper and paper products (2,08 million tonnes per year) and sawn timber and wood products (1,25 million tonnes per year). The Finnish Waste Plan (Ym.fi, 2019) sets a recycling target 70 % to construction waste being used as material until 2030 and 60 % to the municipal bio-waste where waste wood is also part of it.

In 2018, a total of 78.2 million cubic meters of timber was cut from Finnish forests (Stat.luke.fi, 2019). The number was higher than ever and higher than the sustainable production limit in
five provinces. The total use of waste wood as a material was only 3.7% and use as energy was 96.2% during the year 2017 (Stat.fi, 2019). In the Nordic countries, there is a constant need for heating energy. In a country with a large and sparsely populated area such as Finland, long distances easily raise transportation costs to a level where a business is not profitable. It is obvious that technically and economically best way to use waste wood is to burn it to energy (Heräjärvi et al., 2011) if it is not sorted or there is very little of it.

Table 1. Amount of waste wood in 2016 and 2017 produced in Finland, 1 000 tonnes

<table>
<thead>
<tr>
<th>Waste wood, 1 000 tonnes</th>
<th>2016</th>
<th>2017</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total</td>
<td>4 738</td>
<td>3 638</td>
</tr>
<tr>
<td>Sawmilling</td>
<td>1 818</td>
<td>1 252</td>
</tr>
<tr>
<td>Pulp and paper</td>
<td>2 387</td>
<td>2 077</td>
</tr>
<tr>
<td>Construction and demolition wood</td>
<td>264</td>
<td>192</td>
</tr>
<tr>
<td>Municipal waste wood</td>
<td>46</td>
<td>40</td>
</tr>
<tr>
<td>Reduction from an earlier year</td>
<td>+19.5%</td>
<td>-23.2%</td>
</tr>
<tr>
<td>Use as material</td>
<td>3.7%</td>
<td>1.5%</td>
</tr>
<tr>
<td>Use as energy</td>
<td>96.2%</td>
<td>98.2%</td>
</tr>
<tr>
<td>Use as landfill</td>
<td>0%</td>
<td>0.03%</td>
</tr>
</tbody>
</table>

Conclusions

1. Sorting methods should be improved to use different waste wood categories
2. Waste wood applications and business are needed in addition to energy production
3. Long distances easily raise transportation costs to a level where a business is not profitable.
   It is therefore important to generate recycling and re-use of wood waste where the raw material is produced.

References


BLUE STAIN ON SCOTS PINE (PINUS SYLVESTRIS) IN FORESTS NEAR EBERSWALDE, BRANDENBURG

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Background

Fungal discoloration of coniferous sapwood (primarily pine and spruce) is caused by blue stain fungi (sap stain) belonging to the Ascomycetes and Deuteromycetes. The dark colored, large diameter hyphae penetrate wood by entering tracheids by way of ray cells (Fig. 1) and extract nutrients from the ray parenchyma. Optimal temperatures for fungal growth are between 18 and 29 °C. The structure and strength of wood are minimally affected by invasion of blue stain fungi, however the cosmetic damage caused by discoloration(s) can lead to considerable economic loss, both domestically and in the export markets.

Primary blue stain (i.e., stain occurring in standing trees) is initiated by fungal spores introduced into wounds in the bark caused by wind or insects. Secondary blue stain occurs in wood after sawing and during storage. It is a problem in wood which is insufficiently dry and which has been poorly stacked. Some authors distinguish between “blue stain” and “sap stain;” the former referring to stain occurring in poorly stored sawn wood, with sap stain referring to discolorations in standing trees and felled logs.

A study was undertaken to better understand the occurrence, distribution, and economic consequence of blue stain fungi in the forest region near Eberswalde, Brandenburg. Blue stain fungi occurring on pine were specifically analyzed. Forest and lumber companies were consulted for information relating to economic losses caused by blue stain.

Experimental

Microscopical investigation of blue stained wood

Steffen Krause investigated the occurrence and distribution of blue stain with forest and lumber companies near Eberswalde (Fig. 3.). 60 samples of Pinus sylvestris were collected by means of a spatula and first examined with a hand lens. Samples were then taken to the lab and further examined microscopically.

Consulting forest companies and lumber mills

A mailed-out questionnaire was prepared by the Institution of Forestry Sciences Eberswalde, Brandenburg. The questionnaire requested information on the following: a) changes in the incidence and severity of blue stained wood, b) types of wood affected by blue stain, c)
economic consequences of selling blue stained wood, and d) receptiveness to the idea of using anti-blue stain chemicals.

Results and Discussion

Microscopical determination of blue stain fungi
In the Division Ascomycota, analysis of spot tests on pine (Pinus sylvestris L.) revealed the presence of the genus Ophiostoma, respective Ophiostoma piceae (MÜNCH) H. & P. SYD = Ceratocystis piceae (MÜNCH) BAKSHI. Fig. 4 illustrates morphological features of the fungus.

In the Division Deuteromycota, Krause detected the presence of the following fungi:

- *Alternaria tenuis* NEES = *Alternaria alternata* (FR.) KEISSLER
- *Cladosporium* spp.
- *Sphaeropsis sapinea* (FR.) DYKO & SUTTON = *Diplodia pinea* (DESM.) PETRAK

Conidia of *Alternaria tenuis* are pictured in Fig. 5. The hyphae and conidia are dark colored with the characteristic multi-septate conidia occurring in chains.

Economic consequences of blue stained wood in the forest region of Eberswalde, Brandenburg
Although blue stained wood is primarily a cosmetic (rather than structural) problem, it cannot be used for joinery and the economic consequences can be severe. In Brandenburg, harvested wood is divided into different quality classes A, B, or C according to economic desirability. The highest quality wood, grade A, demands the highest prices. Blue stained wood is placed in categories B or C. The results of the questionnaire from the forest companies and lumber mills are presented in Fig. 6. Approximately 50% of the wood produced in lumber mills in the region is affected by blue stain. The period of highest occurrence is April – September.

Conclusions

Quantitative analysis of the incidence of blue stained wood in the forest region of Eberswalde, Brandenburg revealed that approximately 50% of coniferous wood is affected by blue stain. This incidence of blue stained wood could result in an economic loss of about 25,600 EUR per year, or about 50 EUR per m³.

In the Division Ascomycota, *Ophiostoma piceae* – *Ceratocystis piceae* predominated. In the Deuteromycetes, *Alternaria tenuis*, *Cladosporium* spp., and *Sphaeropsis sapinea* were the primary fungi involved in the development of blue stain.

References


MECHANICAL PROPERTIES OF SINGLE-LAYER PARTICLE BOARD MADE FROM BY-PRODUCTS CHIPS

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Background

Climate changes can be observed every day and they encourage legislators or executing entities to change the manner of managing post-production wood wastes (Bojar and Miedziński 2018). So far, wood processing factories have used by-products (obtained during the manipulation of the raw material) to convert it into the heat energy, mainly as fuel for dryers (Guzenda and Świgoń 1997). Sawmills processing round, raw material for general-purpose wood products show an average efficiency of the main material at 50 - 60% (Wieruszewski 2019), i.e. over the 1/3 of the raw material is considered as a waste. In other branches of the woodworking industry this material can find more economically justified application than using it just as a fuel. Thus, the aim of the research was to investigate the size of wood particles obtained during wood processing and the possibilities of using particles characterized by different sizes in particle board manufacturing process.

Experimental

The material used for the research was purchased from a sawmill processing mainly pine wood in the amount of 150 thousand m³ per year. The wood wastes were obtained from the cyclone tank during its cleaning. In order to investigate the size of the particles, material was subjected to a sieve analysis using the screens with a mesh size of 6.3 × 6.3 mm, 4.0 × 4.0 mm, 2.5 × 2.5 mm, 2 × 2 mm, 1.4 × 1.4 mm and <1.4 mm. The single-layer particle board with the density of 800 kg/m³ and thickness of 11 mm were manufactured from each fraction obtained during the dimensional analysis. Wood particles were glued with urea-formaldehyde adhesive in an amount depending on the fraction from 9 to 12% Particle boards were produced using the following pressing parameters: unit pressure 1.2 N/mm², temperature 180°C, pressing time 25 s/mm.

In order to determine mechanical properties of the boards following tests were carried out: bending strength and modulus of elasticity according to EN 310.

Results and Discussion

The shape and size of the particles are among the primary parameters affecting the mechanical properties of manufactured panels. This relation results from the elementary construction of the board, and thus from the mutual position of the particles, their quantity and the total contact
surfaces of the particles with each other (Drouet et al. 1980). The particles used in the particle board manufacturing process are characterized by irregular shape, different thickness and length. Such diversity results from the use of various types of both machines and tools in the woodworking process from raw, round wood to the final product. Table 1 presents the results of bending strength test of manufactured panels. The best values were obtained by the boards made of fraction retained on a sieve with a mesh size of 2.5 mm × 2.5 mm. Using the fraction characterized by the smallest dimensions caused a deterioration of mechanical properties, despite using the largest amount of the adhesive.

Table 1. Bending strength of the particle boards depending on the size of the particles

<table>
<thead>
<tr>
<th>Mesh size [mm × mm]</th>
<th>Resination [%]</th>
<th>Density [kg/m³]</th>
<th>Bending Strength f_m [N/mm²]</th>
<th>Standard deviation σ [N/mm²]</th>
<th>Modulus of elasticity E_m [N/mm²]</th>
<th>Standard deviation σ [N/mm²]</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt;1</td>
<td>13</td>
<td>791</td>
<td>12,48</td>
<td>1,87</td>
<td>2444,36</td>
<td>451,13</td>
</tr>
<tr>
<td>1</td>
<td>13</td>
<td>787</td>
<td>12,64</td>
<td>1,06</td>
<td>2500,22</td>
<td>591,32</td>
</tr>
<tr>
<td>2</td>
<td>12</td>
<td>793</td>
<td>13,24</td>
<td>0,62</td>
<td>2487,33</td>
<td>91,19</td>
</tr>
<tr>
<td>2,5</td>
<td>11</td>
<td>791</td>
<td>14,89</td>
<td>1,81</td>
<td>2648,63</td>
<td>727,34</td>
</tr>
<tr>
<td>4</td>
<td>10</td>
<td>790</td>
<td>14,37</td>
<td>1,70</td>
<td>3116,82</td>
<td>896,1</td>
</tr>
<tr>
<td>6,2</td>
<td>9</td>
<td>788</td>
<td>13,87</td>
<td>2,39</td>
<td>3485,00</td>
<td>467,47</td>
</tr>
</tbody>
</table>

The best results of modulus of elasticity were obtained in case of panels made of the largest particles (6.2×6.2 mm² fraction). This relation confirms the general statement regarding increased stiffness of panels containing large particles (Drouet et al. 1980). Similarly, as in case of bending strength results, particle boards made of the smallest fraction had the lowest values of modulus of elasticity. The difference between the best and the worst results was 27.5%.

Conclusions

It is possible to manufacture particle boards made of particles which were sawmills by-products, usually considered as a waste intended to incineration. Mechanical properties of the boards depend primarily on the shape and size of the particles used in the production process. As a future work would be interesting to continue the research on the application of woodworking by-products in the wood-based materials industry.

Acknowledgments

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References


DIMENSIONAL STABILITY OF PARTICLEBOARDS INTENDED FOR CONSTRUCTION

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Background

The demand of the construction industry for wood-based materials is very high. A special place in the group of these products is occupied by those that are intended for use in humid conditions, or even load transferring in these conditions. OSB and MFP boards occupy top positions on the market of these materials. Numerous studies in the field of dimensional stability of OSB boards have already been carried out at the Department of Wood-Based Materials at the UP in Poznań (Mirsk et al. 2017, Mirski et al. 2015, Mirski et al. 2013, Mirski and Dziurka 2013, Mirski 2010 and 2009). Studies on MFP boards involved more changes in mechanical properties in conditions of variable humidity than their dimensional changes (Kociszewski et al. 2013). This issue is important because it first informs users what size expansion joints should use, and secondly determines the stability of the entire structure. Because of there is no information about the dimensional stability of MFP boards, and in addition this type of boards are characterized by high density, it was decided to assess the dimensional stability not only of MFP boards, but also boards manufactured in laboratory conditions with a much lower density, and these results refer to the properties of OSB boards.

Experimental

For this study industrial production of OSB/3 and MFP boards 15 mm thick and two types of laboratory boards of the same thickness, marked as RAM/1 and RAM/2 were used (Table 1). Laboratory boards were made of chips according to Mirski et al. 2018. A similar procedure was also used to manufacture these boards. The boards meet the requirements of the EN 312:2011, EN 300:2007 standards (excepted of RAM/1 type boards).

<table>
<thead>
<tr>
<th>Type of boards</th>
<th>( \rho ) (kg/m³)</th>
<th>( \text{MOR}_{II} ) (N/mm²)</th>
<th>( \text{MOR}_{II} ) (N/mm²)</th>
<th>( \text{MOE}_{II} ) (N/mm²)</th>
<th>( \text{MOE}_{II} ) (N/mm²)</th>
<th>IB</th>
<th>ST</th>
</tr>
</thead>
<tbody>
<tr>
<td>MFP</td>
<td>770</td>
<td>21.7 (1.66)*</td>
<td>25.1 (1.90)</td>
<td>3700 (240)</td>
<td>4170 (420)</td>
<td>0.79 (0.05)</td>
<td>8.9 (1.1)</td>
</tr>
<tr>
<td>OSB/3</td>
<td>570</td>
<td>20.3 (0.99)</td>
<td>11.4 (0.69)</td>
<td>4080 (220)</td>
<td>2140 (175)</td>
<td>0.36 (0.06)</td>
<td>12.1 (1.8)</td>
</tr>
<tr>
<td>RAM/1</td>
<td>525</td>
<td>12.9 (1.92)</td>
<td>2110 (360)</td>
<td></td>
<td>0.45 (0.01)</td>
<td>16.4 (0.7)</td>
<td></td>
</tr>
<tr>
<td>RAM/2</td>
<td>550</td>
<td>17.5 (0.99)</td>
<td></td>
<td>2750 (240)</td>
<td></td>
<td>0.67 (0.06)</td>
<td>16.1 (1.7)</td>
</tr>
</tbody>
</table>

*Standard deviation is given in brackets
The boards used in the study were evaluated for changes in dimensions caused by relative changes in humidity. Humidity levels were adopted in accordance with EN 318. The samples were therefore air-conditioned in a relative humidity of 30%, then 65% and the process was completed after air-conditioning in air with a humidity of 85%. In addition, it was decided to determine the test results in terms of % change in moisture content of the panels, as was presented in earlier publications.

Results and Discussion

The results of tests on the relative change in thickness and length of the tested boards are presented in Table 2. The obtained average values of relative thickness and length change for OSB/3 boards are comparable with the results of previous tests for this type of boards (Mirski et al. 2013). However, the relative change in thickness of MFP boards is close to the average, determined from both directions of OSB/3 boards. Laboratory boards show significantly lower values of thickness changes than the other two types of boards, which is confirmed by Post-hoc analysis (ANOVA F (4, 119) = 34.214, p = 0.0000). This is probably the result of a much lower density of these boards, especially in the middle layer. The middle layer probably suppresses the increase in the thickness of individual chips due to the loose structure. Relative changes in the length of the tested boards are statistically different (F (4, 35) = 7.4722, p = 0.00018). However, it results from small and very large changes, determined respectively for the major and smaller axes of OSB board. Laboratory plates intended to be a substitute for the industrial boards tested exhibit relative length changes similar to MFP boards.

<table>
<thead>
<tr>
<th>Property</th>
<th>Parametr</th>
<th>Samples</th>
<th>MFP</th>
<th>RAM/1</th>
<th>RAM/2</th>
<th>OSB/3 II</th>
<th>OSB/3 <em>I</em></th>
</tr>
</thead>
<tbody>
<tr>
<td>Relative change in thickness/ change MC, %/ %</td>
<td>Average</td>
<td>0.814</td>
<td>0.479</td>
<td>0.531</td>
<td>0.892</td>
<td>0.696</td>
<td></td>
</tr>
<tr>
<td></td>
<td>SD</td>
<td>0.16</td>
<td>0.12</td>
<td>0.10</td>
<td>0.22</td>
<td>0.11</td>
<td></td>
</tr>
<tr>
<td></td>
<td>HSD</td>
<td>a</td>
<td>c</td>
<td>c</td>
<td>a</td>
<td>b</td>
<td></td>
</tr>
<tr>
<td>Relative change in length/ change MC, mm/mm/%</td>
<td>Average</td>
<td>0.25</td>
<td>0.23</td>
<td>0.22</td>
<td>0.18</td>
<td>0.30</td>
<td></td>
</tr>
<tr>
<td></td>
<td>SD</td>
<td>0.056</td>
<td>0.035</td>
<td>0.025</td>
<td>0.058</td>
<td>0.062</td>
<td></td>
</tr>
<tr>
<td></td>
<td>HSD</td>
<td>ba</td>
<td>c.b</td>
<td>c.b</td>
<td>c</td>
<td>a</td>
<td></td>
</tr>
</tbody>
</table>

Conclusions

The relative change in MFP board thickness is similar to the average change in OSB/3 board thickness. Laboratory boards are characterized by a smaller change in thickness, which may resulted from the low density of their middle layer, compensating for moisture deformation of the chips. Boards made of fine chips exhibit similar relative length changes. OSB/3 boards are characterized by significant changes in the relative change in length depending on the orientation axis, which has already been demonstrated in previous studies. However, the relative changes in MFP board length are slightly smaller than the changes specified for the major axis of OSB boards.

Acknowledgements

The authors are grateful for the support of the National Centre for Research and Development, BIOSTRATEG3/344303/14/NCBR/2018.
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PN-EN 312:2011 Particleboards. Specifications
PN-EN 318:2004 Particleboards. - Determination of dimensional changes due to changes in relative humidity
INVESTIGATION OF FIRE-RETARDANT ADDITIVE ON PARTICLEBOARD AND FIBREBOARD PROPERTIES

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Background

Fire resistance of wood-based panels is in addition to mechanical and sorption properties, an important property when considering its usability for construction usage. Increased fire resistance is achieved through fire retardant addition during panel production or by spraying fire-retardant on already finished panel. The downside of fire-retardant addition during panel production is related to decreased mechanical properties. This work, the latest in studies into the use of a proprietary fire retardant, considers the impact of fire-retardant addition on mechanical, sorption and thermal properties of particleboard and fibreboard.

Introduction

This work, the latest part of ongoing studies has seen the investigation of the impact of fire-retardant addition on mechanical, sorption and thermal properties of particleboard and fibreboard. This followed on from previous work (Hastrup et al. 2015), where some of the properties of medium density fibreboard (MDF) was conducted. The fire-retardant was added to surface layer adhesive mixture only. As fire-retardant, a proprietary product, BurnBlock was used. The main reason for targeted application (surface layer) of fire-retardant is related to the origin of fire first contact point. This is usually with the material surface, hence its more important to additionally protect panel surface layer. Two sets of wood-based panels were produced namely three-layer particleboard (thickness 18 mm, target density 0.65 gꞏcm⁻³) and fibreboard for insulation purpose (thickness 50 mm, target density 0.125 gꞏcm⁻³) with and without fire-retardant in surface layer. The loading ratio of fire retardant was between 0 and 30 kgꞏm⁻³. The addition of fire-retardant influence properties of produced panels especially the fire resistance.

Experimental

In order to pre-treat wood fibres for MDF production, the fire retardant was sprayed on to the fibres using a closed loop system (Figure 1), after which a period of air-drying was necessary to minimise the risk of boards “blowing” during the pressing regime. The deposition of fire retardant on the fibres was clearly shown through the use of SEM-EDX (Figure 2).
Provisional experiments on MDF showed the potential of the fire retardant-treated boards being capable of reaching Euroclass B or C when testing with a cone calorimeter according to ISO5660, whereas conventional MDF (without any fire retardant) could only achieve Euroclass D. Similar improvements were found on testing insulation batts with or without treatment (Jones et al. 2016).

**Subsequent work**
Having established the potential of the proprietary fire retardant with MDF, further studies have been ongoing to ascertain performance in particleboard and lightweight fibreboard systems. In order to achieve this, alternative methods of introducing the fire retardant were considered, whereby the fire retardant was introduced to the resin mixture as a powder or as part of the aqueous system used in the resin preparation. A key benefit of the fire retardant is its pH neutrality, which in theory should not affect the adhesive properties of the resin.

**Results and Discussion**
Results will present how the methods of fire retardant addition affected the performance of the composites, as well as additional fire performance testing. Theses will be compared with results from earlier studies.

**Conclusions**
This poster will outline the potential of fire retardant treatment within composite manufacture.

**References**
STUDY OF POTENTIAL ON ADHESION CHARACTERISTICS FOR WOOD PLASTIC COMPONENTS IN INJECTION MOULDING

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Motivation

Based on current consumption trends, crude oil as a fossil fuel will only last until the middle of the 21st century (Shafiee and Topal, 2009). In contrast there is the steadily growing global demand for it of almost $1.6 \times 10^{10}$ litres daily in 2018 (BP, 2019). Petrochemical industry is the second largest oil processing industry with a share of 13 % in that consumption (OCDE, 2015). By providing basic chemicals such as monomers, petrochemicals represent the beginning of the value chain in the plastics processing industry. The availability of crude oil is therefore essential for the entire plastics industry according to its current state of development. In order to avoid the threatening shortage, the use of renewable cellulose-based raw materials such as wood in the plastics processing industry have been researched for some time (Mohanty, Misra and Drzal, 2002). The use of wood veneers as a substitute for flat, inorganically fibre-reinforced semi-finished products offers an approach. The structure of wood as a fabric structure, in which cellulose fibres are embedded in a matrix of lignin and hemicellulose, is similar to the structure of fabrics or layers embedded in a plastic matrix (Tsoumis, 2009). However, the monolithic use of wood veneers as load-bearing structures is limited by the lower mechanical properties compared to inorganic fibre-reinforced semi-finished products. Furthermore, the process windows set up for the processing of petrochemically based semi-finished products can only be transferred to the processing of wood veneers to a limited extent. In order to improve integration into these processes, a multi-material component made of wood veneer and a plastic application is first considered. The evaluation is initially carried out on the level of basic testing samples. For this purpose, an overlap joint between a wood veneer and a plastic component is produced by injection moulding. The bond is made by using a bonding agent added to the plastic granulate. The aim of this investigation is a first validation of the possibility of processing wood veneers suitable for large-scale production in plastics industry by creating the highest possible bond strength between wood and plastic.

Experimental

The aim of the investigation is to validate a process window to create the highest possible bond strength between a beech wood veneer with an initial thickness of 1.6 mm and an unreinforced polypropylene that is mixed with an adhesion promoter. For this purpose, a tensile shear test
based on DIN EN 1465 is used. Differing from the testing standard, the length of the specimen parts is only 90 mm (Figure 1). The specimen is produced by injection moulding.

<table>
<thead>
<tr>
<th>specimen dimensions (L x W x T):</th>
<th>materials</th>
</tr>
</thead>
<tbody>
<tr>
<td>167.5 x 25.0 x 4.3</td>
<td>Beech 90.0 x 25.0 x 1.6</td>
</tr>
<tr>
<td></td>
<td>plastic Borealis® BJ356MO</td>
</tr>
<tr>
<td></td>
<td>polypropylene PP 90.0 x 25.0 x 3.0</td>
</tr>
<tr>
<td></td>
<td>bonding agent ExxonMobil®</td>
</tr>
<tr>
<td></td>
<td>Exxelor™ PO 1020 MAH-g PP; MAH concentration: 1.1 mass-% (Reußmann, 2013)</td>
</tr>
</tbody>
</table>

Figure 1. Specimen dimensions and materials.

The proportion of the bonding agent varies in the steps 5, 10 and 20 wt-%. During production, a maximum injection pressure of approx. 500 bar was achieved. After that a holding pressure of 250 bar was applied over a period of 10 seconds. The melt temperature was varied in the steps 210 °C, 230 °C and 250 °C. The mould temperature was set to 40 °C.

Results and Discussion

The results of the tensile shear strength test show the influence and dependence of the parameters adhesion promoter content and melt temperature on the strength achieved (Figure 2). During testing the failure of the samples always occurs in the polymer component. There was no failure at any sample in the boundary layer. An increase in the percentage ratio of the bonding agent did not lead to an increase in the tensile shear strength and had no significant effect on the tensile shear strength. Only the injection temperature of the polypropylene shows a significant impact on the tensile shear strength. As the temperature of the melt has been increased from 210 °C to 230 °C, the tensile shear strength decreased by 13.03%. A further increase of the melt temperature shows the tendency of a positive effect, which is not significant.

Conclusions

With the tested material combination, it is possible to reach a bonding strength set with a low content of bonding agent in the melt. The failure does not occur in the boundary layer. Under the given conditions in a bond of beech and PP, the plastic part is the weakest joining partner and its strength depends significantly on the processing temperature. For future investigations, the correlation between the bonding agent percentage and the determined process window in the melt temperature must be investigated. The experiments can be carried out on 3-point bending specimens in order to characterise the boundary surface connection in a more specific way, e.g. as it was done with wood veneer prepregs (Siegel, 2015). The result shows that under
the given conditions both the boundary surface and the wood veneer are not the components that limit the strength of the structure. The proof for the mass production of plastic-wood-hybrid components is thus basically provided.

References


THE POSSIBILITY OF USING BIRCH BARK AS A FILLER FOR UREA-FORMALDEHYDE ADHESIVE IN PLYWOOD PRODUCTION

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Background

Plywood is a widely used material that finds many applications both as a construction and decorative material. In the last six years the production of plywood increased worldwide by 40% (Kawalerczyk et al. 2019a). The commonly used resins for plywood production are urea-formaldehyde adhesives. Despite many advantages, UF resin also have disadvantages such as formaldehyde emission and low water resistance. One of the most effective method for improving parameters of UF resin is the addition of various fillers (Pawlak et al. 2018). Many types of natural fillers have been the subject of ongoing research but the number of publications on UF resin modification with bark is very scarce. Birch bark is the waste product during wood mechanical processing. The woodworking industry generate great quantity of waste bark each year. The most common method of utilizing bark is to incinerate it, despite of its low heat production and high ash content (Ružiak et al. 2017; Réh et al. 2019). In Pedieu et al. (2009) studies the birch bark particles were used in the core layer of particle board with fiber-reinforced surface. They discovered that 70% of wood fibers could be replaced with birch bark while maintaining the required properties of the panels. Thus, the aim of the work was to investigate the possibility of replacing technical flour with birch bark in plywood production.

Experimental

The urea-formaldehyde resin used in this study had following characteristics: solid resin content of 68%, viscosity of 615 mPa s, gel time of 65 s at 100°C, pH 8.1, and density 1.28 g/cm³. Rye flour was used as a reference filler (REF) and ammonium nitrate (20 wt%) was added as a hardener. Bark was grinded and sieved to obtain the fraction with grains smaller than 0.4 mm. Adhesive mixtures containing 15(B15), 20(B20) and 25%(B25) birch bark were mixed mechanically to get a high level of homogenization. To determine properties of the adhesive mixtures, following tests were carried out: viscosity using a Brookfield DV-II + Pro viscometer, gel time at 100°C in accordance with PN-C-89352-3, pH, and solid content according to EN 1245 and EN 827 methods respectively. The prepared adhesive mixtures were applied at 170 g/m² on the surface of the veneers. The pressing process was conducted at 120°C for 4 min, with unit pressure of 1.4 MPa. The following properties of three-layered plywood samples were determined: shear strength both in dry conditions and after soaking in water for 24 h according to EN 314-1, free formaldehyde content using a flask method according to EN 717-3.
Results and Discussion

The results presented in Table 1 show that the replacement of rye flour with birch bark had an effect on the viscosity and pH of resin mixture. The viscosity of a mixture with 15% bark addition did not attain density values comparable to the control adhesive prepared by industrial methods. The most suitable viscosity results, similar to the reference mixture, were obtained with the addition of 20 g bark per 100 g of dry mass of resin (B20). However, further addition of the bark to the resin caused major increase of viscosity. During the production of plywood, the right viscosity of the mixture is extremely important because adhesives with too high or too low viscosity cannot be evenly spread on the veneer surface, which can affect the bonding quality and mechanical properties of plywood. Furthermore, the addition of bark led to decrease of pH values of the mixtures, probably because of the acidic character of the bark. Lowering the pH level may be beneficial due to the fact that the condensation of UF resin takes place under the acidic conditions (Kawalerczyk et al. 2019b). The bark fillers did not significantly affect the curing properties and solid content of the resin.

Table 1. Properties of adhesive mixtures

<table>
<thead>
<tr>
<th>Variant label</th>
<th>Viscosity (mPa s)</th>
<th>pH</th>
<th>Gel time (s)</th>
<th>Solid content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>REF</td>
<td>1421</td>
<td>7.91</td>
<td>83</td>
<td>63.87</td>
</tr>
<tr>
<td>B15</td>
<td>870</td>
<td>6.32</td>
<td>87</td>
<td>65.04</td>
</tr>
<tr>
<td>B20</td>
<td>1233</td>
<td>6.11</td>
<td>84</td>
<td>65.93</td>
</tr>
<tr>
<td>B25</td>
<td>1897</td>
<td>6.07</td>
<td>84</td>
<td>66.05</td>
</tr>
</tbody>
</table>

Studies revealed that in all variants of plywood glued with adhesive filled with birch bark, bonding quality were slightly lower in comparison to reference samples. The best results were obtain in case of 20% bark addition. Further addition caused a considerable decrease of bonding quality. However, all plywood regardless of the variant achieved good values exceeding 1.0 N/mm² and met the requirements of EN 314-2. The replacement of flour with bark led to significant decrease of formaldehyde emission. The reason of lowered free formaldehyde content are tannins contained in the bark (Medved et al. 2019).

Table 2. Shear strength and formaldehyde content

<table>
<thead>
<tr>
<th>Variant label</th>
<th>Shear strength (MPa)</th>
<th>Formaldehyde content (mg CH₂O/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>tested dry</td>
<td>tested after soaking</td>
</tr>
<tr>
<td>REF</td>
<td>1.872</td>
<td>1.529</td>
</tr>
<tr>
<td>B15</td>
<td>1.534</td>
<td>1.287</td>
</tr>
<tr>
<td>B20</td>
<td>1.815</td>
<td>1.503</td>
</tr>
<tr>
<td>B25</td>
<td>1.421</td>
<td>1.112</td>
</tr>
</tbody>
</table>

Conclusions

The replacement of rye flour as filler with birch bark had a significant effect on rheological properties and pH level of the adhesive mixture. However, the addition of bark did not affect curing properties of resin. The best results of shear strength were obtained in case of 20% addition of the bark and were very similar to the bonding quality of the control plywood. In case of 15% and 25% addition of bark shear strength was slightly lower, however all variants achieved values required by the standard. Studies confirmed that formaldehyde emission was significantly reduced by the addition of birch bark as a filler for UF adhesive.
Acknowledgments

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References


INVESTIGATION OF WOOD BOARDS FROM TEXTILE WASTE AND PLANT FIBRES

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Background

The focus of this work will be on changes in the properties of natural fibers through repeated processing in various ways. This involves the use of scientific information analyzing the physical properties of natural fibers and their evolution as a function of external influences and aging processes. This analysis is primarily of practical importance in the recycling of various organic materials, in order to obtain secondary raw materials for the production of various industrial products, and in the reduction of pollution and disposal of waste products. Continuous results of research on wood materials and textile waste composites are analyzed. The aim will be to set a limit on the number of recycling cycles after which the properties of the fibers will be degraded to such an extent that they are rendered inappropriate for recycling, expensive and non-organic.

Experimental

The process of wood waste composites and production took place in the Wood Technology Laboratory, Tallinn University of Technology. Material calculations were performed i.e. the amount of material needed to make the board, and the minimum amount of glue and hardener needed. The calculations made three panels of different compositions, each of which had different components.

The materials were mixed in a lab mixer, then layered in layers into selected size frames and compressed into press INFOR PM8 (see Fig. 1), which is exposed to high pressure and high temperatures, e.g. 200 °C and an average 4MPa pressing force were produced in the slab. For the calculations, we assume that the two outer layers use 9.5% of the adhesive and the two inner layers 8% of the total weight. Each board was different in appearance, quality and features. A testing machine H10KT was used to determine the bending strength.
Results and Discussion

The tests were carried out on a particle board made with hemp chaff in the inner layer (see Fig 3). Three specimens measuring 350x50x16,8 mm are used. The resulting bending strengths were 20.73 MPa, 21.82 MPa and 17.43 MPa. The resulting deflection averaged 5.8 mm. The most important characteristics are given in Table 1 and Figure 4.

Table 1. Calculations of bending strength of particle board from wood with hemp:

<table>
<thead>
<tr>
<th>SAMPLE NO.1</th>
<th>SAMPLE NO.2</th>
<th>SAMPLE NO.3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Deflection, mm</td>
<td>Force, N</td>
<td>Deflection, mm</td>
</tr>
<tr>
<td>1</td>
<td>8</td>
<td>1</td>
</tr>
<tr>
<td>2</td>
<td>50</td>
<td>2</td>
</tr>
<tr>
<td>3</td>
<td>258</td>
<td>3</td>
</tr>
<tr>
<td>4</td>
<td>479</td>
<td>4</td>
</tr>
<tr>
<td>5</td>
<td>679</td>
<td>5</td>
</tr>
<tr>
<td>6</td>
<td>820</td>
<td>5,79</td>
</tr>
<tr>
<td>6,02</td>
<td>820</td>
<td></td>
</tr>
</tbody>
</table>

bending strength  bending strength  bending strength

17,43 N/mm²  21,82 N/mm²  20,73 N/mm²
Conclusions

The properties of natural fibers, primarily composed of cellulose, degrade with the number of recycling cycles. Based on the analysis of information from various sources and the observations made with the waste paper and chipboard, it was found that after reprocessing, a raging effect begins. In the production of particle board from recycled wood, especially glued products, a large part of the chipped wood is sorted as dust / fines, the adhesive absorbency is reduced and the pressing cycle is slightly accelerated.

Using production waste: recycled wood and hemp chaff, we glued boards and investigated their properties using the methods specified in EN standards to determine the properties of particle boards. The wood and hemp composite showed good physical-mechanical characteristics compared to the requirements of EN 312 standard.

References

Дулькин Д. А., Спиридонов В. И., Комаров В. А. 2007. Современное состояние и перспективы использования вторичного волокна из макулатуры в мировой и отечественной индустрии бумаги./ Архангельск: Изд-во АГТУ, 2007: 599 – 806
Materials research for manufactur ing :an industrial perspective of turning materials into new products /editors: Lynnette D. Madsen, Erik B. Svedberg.. Cham : Springer, 2016.;
Background
Modification is a way to avoid or delay fungal degradation of wood. Exactly how and why different types of modifications function remains unclear, albeit it is established that a reduction in moisture content is involved (Thybring, 2013; Thybring et al., 2018). Acetylation and furfurylation are two different types of wood modifications. Recently it was found that brown rot fungi may deacetylate acetylated Radiata pine wood when enough moisture is available, and then proceed to degrade the cell walls (Beck et al. 2018). However, furfurylated Radiata pine wood appears initially to be somewhat degraded under the same conditions, but then degradation stops. In the present study we explored this difference with the aim of identifying degradation patterns and cell wall chemistries that covaried with these two dissimilar trends. Furfurylated wood contains furan polymers, a subset of which are fluorescent conjugated furan chains visible using Confocal Laser Scanning Microscopy CLSM (Thygesen et al., 2010).

Experimental
Specimens of Radiata earlywood (Pinus Radiata D.Don) were either furfurylated or acetylated to varying degrees and subjected to Rhodonia placenta degradation as earlier described (Beck et al. 2018). Specimens were collected before and after degradation and studied using light microscopy, infrared spectroscopy and CLSM using a Leica SP5-X instrument. CLSM was applied in a version where crude excitation-emission fluorescence landscapes were obtained for each pixel in the micrograph with 10-20 wavelength channels each in two directions. These data were thus 4-way structures (excitation wavelength × emission wavelength × image direction 1 × image direction 2). The data set presented here was analysed using Parallel Factor Analysis (PARAFAC).

Results and Discussion
Figure 1 shows the excitation and emission loadings of a 3-factor 4-way PARAFAC model of a single CLSM image of furfurylated pine. The model only explains 52% of the variation in the data, however appears sound as the emission loading peak occurs at a longer wavelength than the corresponding excitation peak wavelength for all three tentative fluorophore populations identified. Further, all loadings are positive and reasonably unimodal without these constraints being applied during the modelling.
Figure 1. Excitation and emission loadings of a 3-factor 4-way PARAFAC model of CLSM data obtained from a specimen of furfurylated Radiata pine.

Figure 2 shows a representation of the raw emission and excitation data and of the image direction loadings of the same model as shown in Figure 1. While the alignment with anatomical features is poor, this preliminary model seems to tentatively indicate that the three fluorophores are located in slightly different locations, with the fluorophores with the longest emission wavelength (Factor 1) located mostly in the polymer-filled cell lumina, as expected (Thygesen et al., 2010). More and less preliminary results will be included in the presentation.

Figure 2. A representation of raw CLSM data (left) and the image loadings from the PARAFAC model in Figure 1 with the same colour coding for the three factors (right).

Conclusions

No final conclusions can be drawn at this point, but it seems that a 4-way description of fluorophores in furfurylated pine is possible using a combination of CLSM and PARAFAC. Other deconvolution methods will also be tested, as refolding of loadings in the image directions did not align well with wood anatomical features using this approach.
References


Fungal Gene Expression in Furfurylated Wood – An Update

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Background
Predictable service life is crucial for wood as a construction material and preventing decay to maximise service life is beneficial. Hence, investigations of the mechanisms employed by decay fungi in their attempt to degrade treated and untreated wood is important. For wood in service brown rot fungi are of great importance since: 1) they preferentially attack coniferous wood species which are the primary species used in structural wood in the Northern hemisphere, 2) brown rot fungi cause significant strength reduction at low mass loss. “The full extent of feedback mechanisms regulating brown rot decay is not known. However, it is generally agreed that brown rot fungi utilize a nonenzymatic system that rapidly depolymerizes cell wall components in early stages of decay, prior to degradation by traditional cellulases and hemicellulases” (Skrede et al. 2019). It is accepted that modified wood provides increased service life compared to untreated wood, but the exact mechanisms employed by the different treatments against decay fungi is still under investigation. The aim of this review was to provide an update on gene expression studies on furfurylated wood. In addition supplementary methods to further understand fungal decay mechanisms are summarised.

Experimental
Papers that include gene expression studies of fungal decay of furfurylated wood are briefly summarised below.

Schmollerl et al. (2011), Pilgård et al. (2017): Scots pine sapwood, sample size 5x10x30 mm³, exposed to Rhodonia placenta for up to 26 weeks. Furfurylated wood WPG 37. Method: 10 selected genes for qRT-PCR.

Alfredsen and Fossdal (2009) and Alfredsen et al. (2016): Scots pine sapwood, sample size 5x10x30 mm³, exposed to R. placenta for up to 8 weeks. Furfurylated wood WPG 14. Method: 25 selected genes for qRT-PCR.

Skrede et al. (2019): Radiata pine sapwood, sample size Ø = 6 mm, h = 10 mm, exposed to R. placenta for up to 21 weeks. Furfurylated wood WPG 4, WPG 24 and WPG 37. Method: Illumina NextSeq sequencing.
Results and Discussion

Gene expression studies

Results from the reviewed studies support a two-step decay mechanism, with expression of genes related to initial oxidative depolymerisation followed by accumulation of transcripts of genes related to hydrolysis of cell-wall polysaccharides. However, the conclusions from the studies also highlight how test design influences the conclusions.

Schmollerl et al. (2011) and Pilgård et al. (2017) suggested “indications of increased expression of genes related to oxidative metabolism and reduction of genes related to the enzymatic breakdown of polysaccharides in modified wood”. Alfredsen and Fossdal (2009) and Alfredsen et al. (2016) reported “Among the findings based on gene profiles were indications of a possible shift toward increased expression, or at least no down regulation, of genes related to oxidative metabolism and concomitant reduction of several genes related to the breakdown of polysaccharides in furfuryl alcohol modified Scots pine sapwood compared to non-modified Scots pine sapwood.”

However, in Skrede et al. (2019) the entire gene expression pattern was followed in untreated and modified wood from initial to advanced stages of decay using Illumina NextSeq sequencing. They concluded that “a delayed and prolonged, but similar pattern was observed in the radiata pine and the modified experiments. This indicates that the fungus starts a common decay process in the modified wood, but proceeds at a slower pace as access to the plant cell wall polysaccharides is restricted.”

Suggestions for supplementary methods

In the search for fungal decay mechanisms it is obviously preferred to study the entire gene expression rather than just selected genes. But in order to fully understand the mechanisms additional methods should also be employed to view fungal decay activities over time. Thybring (2017) highlights that more details are needed on chemical composition and water-relations in modified (and untreated) wood under decay and the interplay between modification and decay resistance. Novel imaging techniques are also expected to bring new insight. According to Vogel and Marcotte (2012) “…~40% of the variation in protein concentration can be explained by knowing mRNA abundances”. Currently the “Holy Grail” when it comes to understanding fungal decay mechanisms are experiments on fungal secretome from solid wood.

Conclusions

- When studying the entire gene expression during fungal decay furfurylation does not seem to directly influence the gene expression as a delayed and prolonged, but similar, pattern was observed in radiata pine and furfurylated radiata pine.
- To better understand fungal decay mechanisms a set of methods should be used to profile every step of the decay process including: gene expression, water-relations, chemical composition, imaging analyses and secretome data.

References


MACRO BIOLOGICAL DEGRADATION OF WOOD MODIFIED WITH SORBITOL- AND CITRIC ACID

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Background

Modified wood, still being a niche product, is gaining more favour with architects, specifiers and end-users, which suggests a continued success (Jones et al. 2018). However, the commercialised wood modification systems are significantly more expensive than traditional alternatives containing biocides. Thus, a new, low cost and non-toxic wood modification system is urgently needed. Earlier studies on the utilisation of low-cost sorbitol for wood modification have been limited to describing solely the dimensional stabilisation of the wood matrix (Bateson, 1938, 1939). In recent studies, wood modification using polyesterification of sorbitol and citric acid (PS-modification) was shown to enhance not only dimensional stability, but also durability against decay fungi and reduced susceptibility to blue-stain fungi (Larnøy et al. 2018). The resistance of PS-modified wood against wood borers in the marine environment and against subterranean termites has not been studied earlier and is presented in this study.

Experimental

Two studies on PS-modified wood were performed in severe conditions according to European standards.

Resistance to subterranean termites

Termites (Reticulitermes grassei) were collected in a forest of maritime pine (Pinus pinaster) in Portugal. A termite test was performed in the laboratory according to EN117 using two-choice and non-choice tests with three different concentrations resulting in a weight percent gain (WPG) of 25%, 70% and 100% of PS-modified and untreated wood specimens. Termite resistance was described by visual examination after termite exposure using a rating system from 0-4 (where 0 = no attack and 4 = strong attack) and by wood mass loss (%).

Resistance to wood borers in the marine environment

PS-modified wood with a WPG of 50% was tested against wood borers in the Oslo fjord during one season (May to September 2018) according to EN 275. Resistance to wood borers was described by a rating system from 0-4 using x-ray photos from each wood sample.
Results and Discussion

Wood samples showed increased resistance against termites with increasing WPG. The highest WPG in PS-modified wood resulted in nearly no mass loss and no attack by termites. However, termite mortality in the non-choice test of modified wood samples was high already during the first week and 100% after the exposure period, while termite mortality was >50% after two-choice tests and non-choice tests with untreated wood samples.

Figure 1. Mass loss (in red) and attack rating (in blue) of modified- and untreated wood after termite attack in a choice- (left) and non-choice test (middle); mass loss-WPG correlation of wood samples from both tests (right)

PS-modified wood showed no attack by wood borers after exposure, while untreated Scots pine samples failed after attack by shipworm Teredo navalis (see fig 2). However, only one treatment level (50% WPG) was tested. PS-modified wood with other treatment levels were added in May 2019.

Figure 2. Wood borer attack on untreated Scots pine sapwood (upper sample) and PS-modified Scots pine sapwood (lower sample) after four months exposure in the Oslo fjord.

Conclusions

PS-modified wood with a high treatment level showed high resistance against the tested macro biological organisms. Further studies should investigate concentration thresholds, include additional marine test sites and provide insight into the mode of action.
References


COMPARISON OF METHODS TO INVESTIGATE INITIAL BROWN ROT WOOD DECAY

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Phone: +49 89 2180 6435

Background

Since brown rot fungi prefer conifers it is the most important wood decaying group in the boreal forest belt (Zabel and Morrell 1992, Goodell 2003, Vanden Wymelenberg et al. 2010). Especially for wood in service, brown rot leads to great problems (Zabel and Morrell, 1992). The reason for this is that brown rot fungi can cause massive strength loss even at low mass loss (Cowling 1961, Filley et al. 2002, Niemenmaa et al. 2008, Arantes et al. 2012, Arantes and Goodell 2014). Decay tests with model brown rot fungi (for example Gloeophyllum trabeum and Rhodonia placenta) are used for testing new and existing wood protection systems.

The current hypothesis is, that brown rot fungi degrade wood in two steps; oxidative processes followed by enzymatic hydroltic activities (Baldrian and Valášková 2008, Arantes et al. 2012, Arantes and Goodell 2014).

Previous studies have shown that the comparison of gene expression gives insight into fungal degradation mechanisms, when growing on untreated wood, compared to modified wood (Alfredsen and Pilgård 2014, Ringman et al. 2014, Alfredsen et al. 2016, Ringman et al. 2016). Difficulties have however been seen regarding the differentiation of the oxidative and the enzymatic degradation phase since the sample design was not capable of separating those two.

Zhang et al. showed in 2016 that the two decay mechanisms are in fact spatially segregated, therefore they suggested a new test design (wood wafer method), where the initial decay phase is better represented.

In this study we tested the wood wafer method on untreated and acetylated wood. This paper discusses advantages and disadvantages of the wood wafer method, compared to the traditional miniblock test (Bravery 1979) for decay tests to be used for further molecular and biochemical analysis.

Experimental

Wood wafer method

Wood wafers from Scots pine sapwood (Pinus sylvestris) were used (80 x 18 x 2,5 mm) and placed on previously with Rhodonia placenta (FPRL 280) inoculated and overgrown feeder strips (Zhang et al. 2016). Three different acetylation levels (10, 15 and 20 weight percentage gain (WPG)) and untreated samples were used. Each condition was represented by 16 glasses
with three samples in each, which were later pooled to be one biological replicate (n≥11). Samples were harvested when the mycelium had overgrown three quarters of the samples. Five mm pieces, including the hyphal front were cut out, RNA was extracted, cDNA synthesised, and qPCR was performed for ten genes, related to the oxidative degradation phase.

**Miniblocks**
According to Bravery (1979) mini-block samples (10 x 5 x 25 mm) were placed on agar or soil (modified version) plates and inoculated with *Rhodonia placenta* (FPRL 280). The modification methods varies between different studies (thermally modified, furfurylated and acetylated) as well as the test durations (Alfredsen and Pilgård 2014, Ringman et al. 2014, Alfredsen et al. 2016, Ringman et al. 2016). Whole samples were harvested, RNA was extracted, cDNA synthesized and different genes, related to brown-rot degradation processes were investigated by qPCR.

**Results and Discussion**

**Advantages of the wood wafer method**
The hyphal front can be used, and therefore the initial, oxidative, decay phase can be captured. There is also the possibility to study different time points in only one sample – according to the position in the mycelia of where the sample is taken from. With the miniblock test, different decay stages cannot be separated, or only with very early harvesting of the samples. This, however, still does absolutely not ensure that the initial phase is caught accurately.

**Disadvantages of the wood wafer method**
No long-term time studies are possible, because wafers are overgrown very fast. Miniblock studies could reveal changes in gene expression over a longer time period. Pooling of wafer samples is necessary, because the amount of extraction material is not enough when only using a 5 mm section. Therefore the standard deviation is artificially affected.

**Conclusions**
Fungal behaviour during the first steps of degradation, where no mass loss can be detected, might be of enhanced significance. The initial degradation phase seems to be the crucial point in brown rot fungi to overcome wood modifications. Transcriptome comparisons of samples of the different methods might give better insight in the exact differences between gene expression levels.

**References**


AN EXPERIMENTAL PLATFORM FOR FUNDAMENTAL STUDIES OF THE RATE OF COLONIZATION AND DEGRADATION OF WOOD BY DECAY FUNGI

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It is interesting to follow the gradual changes in unmodified and modified wood as it is attacked by decay fungi, so that it is possible to more fully understand how the degradation takes place and also get ideas for how to prevent decay. We are therefore developing an experimental platform in which we can continuously follow decay processes, and take out samples for analysis by different methods, e.g., optical microscopy, SEM, Raman spectroscopy and ergosterol analysis. The core method is isothermal calorimetry, that measures the heat production rate from processes. If the process is fungal degradation of wood, the heat production rate is closely related to the fungal respiration rate, i.e., the consumption of oxygen, and a true measure of the decay activity of the fungi. We have developed a calorimetric method to continuously follow the colonization and degradation of small samples of wood during long periods of time, typically our experiments last 45 days. The calorimetric method is interesting (its shows the kinetics of the decay process, different stages of decay and oscillating metabolism), but it is also the controlled way that we expose our samples, and the calorimetric result enables us to extract samples for other measurements at relevant points in the decay process.
STUDIES ON THE DURABILITY OF EUROPEAN BEECH WOOD IN GROUND CONTACT: UNDERSTANDING THE EFFECT OF BIOTIC AND ABIOTIC FACTORS

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Background

The most prominent factors affecting the durability of wood used in ground contact include exposure to moisture and temperature suitable for fungal proliferation and subsequent wood decay (Zabel & Morrell, 2012). In every application scenario, a cascade of relative importance of each biotic and abiotic factor to in-ground wood durability can be visualised. Climatic factors such as temperature and rainfall have shown sufficient correlation to wood decay for regional-level modelling resolution, but still lack the site-specificity for higher accuracy (local) in-ground wood durability determinations (Wang, et al., 2007). A series of semi-field experiments were undertaken to investigate soil-level characteristics influencing in-ground wood durability. The experiments sought to investigate the effect of soil water-holding capacity, soil moisture content, and soil temperature on wood decay and wood moisture content. This research feeds into a broader study further exploring a lack in the identification of soil-level characteristics affecting in-ground wood durability and modelling thereof.

Materials and Methods

Terrestrial Microcosms (TMCs) in accordance with CEN/TS 15083-2 (CEN/TS, 2005) were utilised in semi-field experiments. Soil was passed through a sieve with nominal aperture size of 12.5 mm. Soil water-holding capacity (WHC) was then determined according to the ‘Cylinder sand bath method’ according to ISO 11268-2 (ISO, 2012). Soil mixtures of 60% WHC were prepared from combinations of sand and compost, and wet to varying degrees to attain soil moisture content values in the range of 36 – 42%, and 54 – 57%, respectively. Wood specimens (5 x 10 x 50 mm³) were placed into the soil mixtures and the boxes incubated at 5°C fixed temperature intervals from 5 – 40°C, as well as fluctuating temperature of 10/20, 10/30, and 20/30°C, respectively. For fluctuating temperature, a temperature change was carried out after one full week of incubation. Wood decay as a measure of oven-dry mass loss was determined every two weeks over 16 weeks of incubation.

Results and Discussion

Figure 1 shows the decay of Beech wood measured as oven-dry mass loss throughout the range of incubation temperatures for soil of 60% WHC and 36% MC. Incubation temperature of 35 °C delivered the highest decay with a mean value of more than 40% after 16 weeks.
Figure 1: Graph of Beech wood decay measured as oven-dry mass loss at various soil temperatures every two weeks over 16 weeks of incubation in soil with WHC of 60% and moisture content of 36%.

Figure 2 below shows the decay of beech wood measured as oven-dry mass loss in conditions of fluctuating incubation temperature. The 20/30 °C incubation temperature delivered the highest decay with a mean value of more than 25% after 16 weeks.

Conclusions

In this study, control of incubation temperature was used as means to control soil temperature during TMC tests. As expected, lower incubation temperature, especially those outside of the optimal range of wood-decaying fungi (<15°C) delivered the lowest decay of beech wood after 16 weeks of incubation. The rule of thumb concerning reaction rate and temperature states that for every 10°C increase in reaction temperature, the reaction rate will double. This is true for the lower incubation temperatures (<15°C), but not for the higher temperatures. The fluctuating incubation temperatures delivered decay values lower than that of their mean fixed temperature counterparts (15, 20, and 25°C). This suggests that seasonal changes attributed to soil temperature influence in-ground wood decay rates.
References


Mapping the Decay Hazard of Wooden Structures in Topographically Divergent Regions

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Background

The service life of exposed wooden structures depends on a variety of endogenous and exogenous factors with moisture being key for fungal degradation. Climate parameters are therefore important input variables for modelling fungal decay in wood and the service life of wooden components. In recent years, different approaches aimed on modelling climate-induced dosage on the material climate (i.e. exposure models) and the effect of the latter on fungal decay (i.e. decay models). Based on maps for Europe, North America or Australia, the decay hazard can be assigned to zones and used for estimating the relative decay potential of an arbitrary location. However, especially in topographically divergent regions the climate-induced decay hazard can vary strongly within a small area. This study aimed therefore on quantifying and mapping the moisture- and temperature-induced risk for fungal decay in a mountainous region where topography-induced differences in local climate and corresponding exposure dosage can be expected. The area under investigation was Switzerland.

Experimental

Two exposure models were combined with two decay models and used to quantify the relative moisture- and temperature-induced exposure dose at 76 different weather stations in Switzerland and adjacent regions. In addition, the Scheffer Climate Index (SCI, Scheffer 1971) was determined for each location. Meteorological data were taken from the Meteonorm database (Meteonorm 7.3, 2018). The exposure was expressed as relative dosage with the city of Uppsala (Sweden) as reference location.

Exposure model I (EMI) according to Niklewski et al. (2016) accounted for relative humidity (RH), rain, and temperature. In contrast, exposure model II (EMII) according to Thelandersson et al. (2011) accounted for RH, temperature, and the wood moisture content (MC) at the previous day. Both models were combined with 1.) a logistic dose-response decay model (LM) according to Brischke and Rapp (2008) and 2.) a simplified logistic decay model (SLM) according to Thelandersson et al. (2011).

Relative dose values were calculated for locations between weather stations using an ‘inverse distance weighted (IDW) interpolation’. Continuous dose values were assigned to 12 classes between 0 and 3 in intervals of 0.25. Maps were generated with the help of ArcGIS (ESRI, USA). A more detailed analysis was undertaken for the Lötschental, which is the largest valley on the northern side of the Rhône valley in the canton of Valais. It lies in the Bernese Alps,
with the Lonza River running down the length of the valley from its source within the Long Glacier.

**Results and Discussion**

The effect of topography on local weather conditions and resulting moisture- and temperature-induced dosage became evident for many locations in Switzerland, but was dependent on the model combination applied. Differences in relative annual dose are exemplarily shown in Figure 1 for exposure model EMII combined with the logistic decay model (LM).

![Figure 1. Relative annual dose based on exposure model EMII and the logistic decay model (LM). Reference site is Uppsala, Sweden.](image)

Especially, in the Lötschental area the relative dose differed strongly within small areas. Elevation was well correlated with the average annual temperature and the resulting relative dose. However, further parameters such as differences in topography-affected differences in precipitation and RH were likely affecting the exposure dose, too. Again, the impact of the different climate parameters rain, temperature and RH was strongly dependent on the model combination applied.

**Conclusions**

Decay hazard mapping is a helpful tool for service life planning with wood and wood products. It became evident that small-scale mapping with high resolution is needed to fully reflect the impact of topography and other local conditions on the moisture- and temperature-induced decay risk in wooden components.

**Acknowledgements**

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References


Effect of Extractives on Brown Rot Degradation of Norway Spruce and Kurian Larch

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Background

Protection of wood against fungal degradation is crucial for its outdoor application. As traditional biocide-based protections systems are being banned, inspiration for environmentally friendly solutions may be found in tree species that form durable heartwood, such as Larix spp.

It is known that antifungal agents have a bigger impact if they cover several attack mechanisms of the fungus (Schultz and Nicholas, 2002). Most investigations focussed on the acetone- or water-soluble extractives; their distribution, effects on water sorption and resistance to degradation (i.e. Gierlinger et al, 2004, Venäläinen et al, 2006, Zule et al, 2015 and 2017, Skjövist et al, 2018, Nisula, 2018). The role of hydrophobic extractives, alone or together with hydrophilic extractives has not received much attention.

Identity and quantity of hydrophobic and hydrophilic extractives in Kurian larch and Norway spruce were assessed. The effects of removal of mainly hydrophobic and/or hydrophilic extractives on degradation by the brown rot Rhodonia placenta were tested. Additionally, the sorption isotherm of Kurian larch was determined for native and extracted specimens.

Experimental

Hydrophobic and hydrophilic extractives in two clones of Picea abies and Larix gmelinii x japonica were studied by total extraction using a sequence of four solvents: heptane, dichloromethane, ethanol and water (TOT), inspired by the procedures of Willför et al., 2003 and Giwa, 1973. Two more extractions consisted of using only heptane and dichloromethane (PHO), or only ethanol and water (PHI). Sticks (50 x 3 x 4 mm, L x T x R) were extracted with a Dionex™ Accelerated Solvent Extractor 350. Milled controls allowed the estimation of the extraction efficiency of the sticks. The composition of different fractions was determined by GC-MS-FID according to Örsta and Holmbom, 1994.

The sorption isotherm of differently extracted specimens of Kurian larch were obtained in the range of 65 – 99.99 % RH using several salt solutions and the pressure plate technique (Thybring et al., 2017, Fredriksson and Johansson, 2016).

Finally, the differently extracted and native sticks of both species were exposed to the brown rot Rhodonia placenta for 2 weeks using a method adapted from Zhang et al, 2016.
Results and Discussion

The heptane fraction was the most unique as compared to the other fractions and yielded similar amounts for both species (~7 mg/g dry wood). It contained the same chemical classes (fatty acids, resin acids, other diterpenoids and sterols), but different analytes. The dichloromethane fractions yielded the same constituents as the ethanol extracts, but in very low amounts (2-4 mg/g). The ethanol extracts yielded 2-3x more in larch (>30 mg/g) than in spruce (~10 mg/g). In both species, the extracts were composed of mostly a single chemical family – 14 lignans in spruce and 4 flavonoids in larch. The composition of the PHI ethanol fraction was a mixture of the heptane and ethanol fractions. The water fraction of spruce was not investigated by GC-MS, but the yields were low (10 mg/g) compared to the corresponding fraction of larch, which yielded very large amounts of arabinogalactan and some flavonoids (> 70 mg/g).

The sorption isotherm of Kurian larch was successfully determined, but no substantial differences were seen between the differently extracted samples. This suggests that moisture exclusion might not be a primary defence mechanism in larch.

Degradation for two weeks by *R. placenta* was more severe for spruce than for larch (Figure 1). Spruce lost almost twice the amount for all types of samples, except for the hydrophilic extraction treatment, by which larch was more affected. This is in accordance with literature, as the flavonoids are the main protection agent of larch (Venäläinen, 2006). Spruce was more affected by the hydrophobic treatment, indicating that lignans might not affect the resistance in the same way as i.e. resin acids do, but this explanation has to be taken with care, since the extraction of the sticks was not as efficient as for the milled controls (30-60 %).

![Figure 1. Average weight loss percentage of 2 clones of each *Picea abies* and *Larix gmelinii x japonica* after 2 weeks degradation by the brown-rot fungus *Rhodonia placenta*. NAT – native, PHI – hydrophilic extracted, PHO – hydrophobic extracted, TOT – total extracted (i.e. hydrophilic and hydrophobic solvents).](image)

Conclusions

We found that the heptane extracts of Norway spruce and Kurian larch yield similar amounts and chemical classes, while the hydrophilic fractions yielded very different amounts and types of molecules (i.e. lignans in spruce and flavonoids in larch).

The moisture content of Kurian larch sticks was not affected by removal of different portions of extractives, which might be due to a lack of extraction efficiency.

Finally, the removal of hydrophobic extractives lowered the resistance to degradation by *Rhodonia placenta* more for Norway spruce than for Kurian larch, while the latter was more affected by the removal of hydrophilic components.
References


UNDERSTANDING HEARTWOOD FORMATION IN LARCH BY USE OF SYNCHROTRON INFRARED IMAGING COMBINED WITH MULTIVARIATE RESOLUTION ANALYSIS AND ATOMIC FORCE MICROSCOPE INFRARED SPECTROSCOPY

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Background

The formation of durable wood tissue (heartwood) is linked to the occurrence of non-structural substances called extractives, which play an important role in the resistance of wood to fungal decay. However, the exact formation and distribution of these extractives within the xylem tissue at the cell and the cell wall level is one of the unsolved questions in plant science. Obtaining this knowledge could facilitate design of environmentally benign, bioinspired wood protection systems. Recently, the potential of Confocal Raman Microscopy to follow the extractive distribution in sapwood (SW) and heartwood (HW) of pine (a moderate durable specie) was shown (Felhofer et al. 2018; Belt et al. 2017). The novelty and objective of this work is to obtain a detailed overview of the HW formation in larch (a highly durable specie) at the micro-and nanoscale level by combining the use of Synchrotron FTIR images (SR-FTIR), Atomic Force Microscopy IR spectroscopy (AFM-IR) and advanced chemometric tools.

Experimental

The study was carried out using nine areas across the transition zone between SW and HW and along a single ray of a single Larch tree (Larix gmelinii x Japonica) at a specific height in the stem. Tangential and cross sections of 10 µm thickness were obtained using a Leica microtome.

SR-FTIR

Eight images in the tangential direction of the ray cells (DT2-DT8) and eight images in the cross section of the surrounding tracheids (DX1-DX8) were acquired. All images were imaged at the infrared beamline MIRAS of the ALBA synchrotron (Cerdanyola del Vallés, Spain, proposal 2018022761). The infrared measurements were acquired with a Bruker system (Hyperion 3000 microscope coupled to a Vertex 70 spectrometer). Images were collected with a spatial resolution of 3 µm.
AFM-IR

For the AFM-IR study, we used a NanoIR2 (Analysis). Two of the extreme samples of the cross-sections (DX1 and DX8) were used for the AFM-IR investigation. The spatial resolution was about 40 nm per pixel.

Results and Discussion

Multivariate Curve Resolution Alternating Least Squares (MCR-ALS)(Tauler et al. 1995; Jaumot et al. 2005; Juan et al. 2009) was used to analyse simultaneously all SR-FTIR images acquired in either the cross or tangential direction. MCR-ALS provided the distribution maps and the related IR spectra of the compounds involved during HW formation in larch (figure 1).

We were able to distinguish between extractives (component III) and lignin contributions (component IV and V). Extractives were detected only within the lumina and the S3 layer of tracheid and ray cells. The main feature is the emergence of a new band at 1640 cm\(^{-1}\) and the decrease of the band at 1660 cm\(^{-1}\) in component V. This change takes place in the transition zone of the heartwood formation. The reduction of the band at 1660 cm\(^{-1}\) could be likely explained in terms of the condensation or oxidation reactions of lignin molecules. The same behavior of IR bands was observed in AFM-IR measurements. Furthermore, AFM-IR spectra in HW ray cell wall suggest the formation of o-quinone structure on taxifolin (one of the most abundant extractives in larch) due to the appearance of new bands around 1676-1692 cm\(^{-1}\) (Kocábová et al. 2016).

Figure 2. AFM-IR image and spectra of the ray cell wall (point 1 and 2) and tracheid cell in larch heartwood (point 3).
Conclusions

SR-FTIR combined with MCR-ALS multiset analysis and AFM-IR spectroscopy allowed identifying and following the distribution of extractives during HW formation of highly durable tree species. The results indicate the presence of taxifolin within the lumina and the S3 layer of tracheid and ray cells in larch. Moreover, both techniques suggest possible condensation or/and oxidation reactions in lignin molecules during HW formation.

References


COMPARISON OF DIFFERENT THERMALLY MODIFIED WOOD RESIDUES FOR PRODUCTION OF WOOD PLASTIC COMPOSITES

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Background

Several thermal modification methods are developed, which all involve subjecting wood to high temperatures ranging from 160 to 260 °C for several hours in low oxygen environment (Esteves and Pereira, 2009). The methods differ between each other in one or more following aspects: treatment environment (steam, oil, nitrogen or vacuum), reactor system (closed or open) and number of process stages. The most widely used thermal modification (Thermowood®) is three-stage treatment in an open reactor system in steam environment (Hill, 2006; Sandberg and Kutnar, 2016, Teng et al. 2018). WTT has developed a similar process, where the only difference is that a closed reactor system is used. Such difference significantly change the technological parameters: lower process temperature, higher pressure and shorter modification time. Besides, in the closed reactor system all of the thermal degradation products are left in the reactor (Militz & Altgen, 2014). Previous studies have concluded that TM wood particles are suitable for production of wood plastic composites (WPC) with improved properties (Segerholm, 2012; Kuka et al., 2016). However, the influence of different thermal modification methods is less studied. The main objective of the present study is to compare and determine how wood residues from open and closed thermal modification processes influence WPC properties.

Experimental

TM wood from open reactor system (Thermowood®) was obtained from industrial-scale TM wood producer. The modification was performed for pine (Pinus sylvestris) according to treatment class D for softwoods (212°C/3h). TM wood from closed reactor system (WTT) was produced by using WTT multifunctional pilot device. Pine was modified by three modification regimes: 160°C/1h, 170°C/1h, 170°C/3h. Wood particles were obtained by milling and fractionation. For all wood specimens chemical composition was determined by using wet chemical analysis methods (Zakis, 2008). WPC were made by mixing 50 wt. % wood particles and 50 wt. % polypropylene in a two-roll mill. Injection moulding was used to prepare bar samples for flexural tests (EN ISO 178), impact tests (EN ISO 179) and water resistance tests.

Results and Discussion

Wood filler characteristics highly affect WPC properties and therefore should be analysed (Klyosov, 2007). The results characterizing wood particles are presented in Table 1.
Table 1. Mass loss and chemical composition of unmodified (UM) and thermally modified pine

<table>
<thead>
<tr>
<th>Wood modification</th>
<th>Mass loss, %</th>
<th>Extractives, %</th>
<th>α-cellulose, %</th>
<th>Hemicelluloses, %</th>
<th>Lignin, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>UM</td>
<td>0</td>
<td>2.6 ± 0.0</td>
<td>44.9 ± 0.3</td>
<td>21.7 ± 0.4</td>
<td>30.8 ± 0.4</td>
</tr>
<tr>
<td>Thermowood®-D</td>
<td>6.7 ± 2.0</td>
<td>3.4 ± 0.2</td>
<td>46.4 ± 0.1</td>
<td>13.1 ± 0.1</td>
<td>37.1 ± 0.2</td>
</tr>
<tr>
<td>WTT 160/1</td>
<td>3.1 ± 1.4</td>
<td>7.1 ± 0.0</td>
<td>41.2 ± 0.1</td>
<td>14.9 ± 0.1</td>
<td>36.8 ± 0.0</td>
</tr>
<tr>
<td>WTT 170/1</td>
<td>7.0 ± 2.4</td>
<td>6.4 ± 0.2</td>
<td>48.7 ± 0.6</td>
<td>8.0 ± 0.7</td>
<td>36.9 ± 0.3</td>
</tr>
<tr>
<td>WTT 170/3</td>
<td>10.2 ± 2.6</td>
<td>8.3 ± 0.1</td>
<td>48.4 ± 0.8</td>
<td>6.1 ± 1.0</td>
<td>37.2 ± 0.0</td>
</tr>
</tbody>
</table>

The relative content of α-cellulose and lignin differs only slightly between the chosen thermal modification methods and regimes with exception of WTT regime 160/1, where significantly smaller relative content of α-cellulose is present due to relatively weaker thermal treatment. The largest difference between the methods is for the results regarding extractives. WTT wood has twice as many extractives compared to Thermowood®-D. Hemicelluloses relative content is highly dependent on modification regime that decrease with increase of thermal modification severity (Esteves and Pereira, 2009). Based on mass loss and wood chemical composition Thermowood®-D is the most comparable with WTT regime 170/1.

Table 2. Mechanical (A – impact strength, MOR – modulus of rupture, MOE – modulus of elasticity, ε - elongation at maximum strength) and physical properties (W abs – absolute moisture content and ΔV – volume change after 100 days in water) of WPC with unmodified and thermally modified wood (50 wt. %)

<table>
<thead>
<tr>
<th>Wood modification</th>
<th>A, kJ/m²</th>
<th>MOR, MPa</th>
<th>MOE, GPa</th>
<th>ε, %</th>
<th>W abs, %</th>
<th>ΔV, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>UM</td>
<td>5.4 ± 0.4</td>
<td>37.1 ± 0.9</td>
<td>3.53 ± 0.15</td>
<td>1.86 ± 0.10</td>
<td>16.1 ± 0.5</td>
<td>12.1 ± 0.6</td>
</tr>
<tr>
<td>Thermowood®-D</td>
<td>3.7 ± 0.3</td>
<td>39.7 ± 1.1</td>
<td>4.18 ± 0.13</td>
<td>1.18 ± 0.06</td>
<td>10.3 ± 0.8</td>
<td>6.2 ± 0.2</td>
</tr>
<tr>
<td>WTT 160/1</td>
<td>5.1 ± 0.4</td>
<td>37.1 ± 0.8</td>
<td>3.72 ± 0.09</td>
<td>1.69 ± 0.08</td>
<td>10.0 ± 0.5</td>
<td>6.2 ± 0.4</td>
</tr>
<tr>
<td>WTT 170/1</td>
<td>4.4 ± 0.5</td>
<td>37.9 ± 0.9</td>
<td>3.70 ± 0.13</td>
<td>1.60 ± 0.09</td>
<td>10.7 ± 0.5</td>
<td>6.2 ± 0.3</td>
</tr>
<tr>
<td>WTT 170/3</td>
<td>4.7 ± 0.4</td>
<td>39.5 ± 0.9</td>
<td>4.20 ± 0.13</td>
<td>1.34 ± 0.09</td>
<td>6.7 ± 0.7</td>
<td>4.5 ± 0.5</td>
</tr>
</tbody>
</table>

The effect of different TM wood particles on WPC properties are presented in Table 2. The results clearly show that extractives in TM wood play a major role in WPC properties. Larger amount of extractives (see Table 1) in WTT 160/1 and 170/1 compared to Thermowood®-D cause smaller flexural strength and stiffness, larger impact strength and for WTT 170/1 relatively low water resistance despite significantly smaller hemicelluloses amount. The results comply with the study where the effect of unmodified (UM) wood extractives is analysed (Kim, Harper and Taylor, 2009). The most severe regime WTT 170/3 show comparable flexural properties to Thermowood®-D despite twice as much extractives. The results regarding water resistance show that Thermowood®-D has similar properties to WTT 160/1 and 170/1. These composites compared to WPC with UM wood particles have smaller moisture content and volume change by 36 % and 49 %, respectively. For WTT 170/3 the reduction is even more significant: 58 % and 63 %, respectively. The results suggest that wood residues from more severe thermal treatment have larger potential to achieve better overall WPC properties.

Conclusions

From the results it can be concluded that TM wood residues from open and closed reactor system does influence WPC properties differently and mainly it is due to differences in the amount of wood extractives. Wood from closed reactor system has twice as much extractives than wood from open reactor system. Such differences results in less stiff and less durable in bending, however more impact resistant WPC. In addition, it can be concluded that the most severe commercially used open reactor system regime Thermowood®-D is relatively weak to achieve the full potential of WPC that are made with thermally modified wood particles.
References


NEW INSIGHT REGARDING THE MODE OF ACTION OF CYCLIC N-METHYLOL COMPOUNDS IN WOOD

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Background

Wood modification with glyoxal resins such as 1,3-dimethylol-4,5-dihydroxyethyleneneurea (DMDHEU) has been studied extensively since the early 2000s (Emmerich et al. 2019). Originally developed for textile finishing, reactions of DMDHEU monomers in cellulose-based fabrics are cross-linking of cellulose molecules and self-condensation of DMDHEU via the reactive hydroxymethyl groups (Schindler and Hauser 2004). The same reaction types of the resin may be expected when utilizing DMDHEU in wood modification processes. However, recent results indicate that a reduction in maximum swelling is nearly entirely based on cell wall bulking, without a significant reduction in the water-saturated sample dimensions that would indicate a cross-linking effect (Krause et al. 2003). In this study, we used DMDHEU and methylolated DMDHEU molecules to analyze how cell wall bulking and cross-linking affect the water vapor sorption behavior and the hydroxyl accessibility of the modified wood.

Experimental

Scots pine sapwood (Pinus sylvestris L.) was treated with 10 % and 20 % solutions of DMDHEU, a methylolated DMDHEU (mDMDHEU) and mDMHEU with addition of diethylene glycol (Figure 1, a-c). Magnesium nitrate hexahydrate was added as a catalyst in a concentration of 2 % related to the amount of DMDHEU monomers. Impregnated specimens were cured at 120 °C for 48 h. Prior to testing, untreated and modified specimens underwent a leaching procedure according to EN 84 (1997). Changes in dry mass and dimensions were determined before and after leaching. Pure resins were prepared by heat-curing (120 °C, 48 h) the respective stock solutions without catalyst. Sorption isotherms of wood samples and pure resins were determined at 25 °C using a dynamic vapor sorption (DVS) apparatus. Changes in hydroxyl accessibility of wood specimens were determined by hydrogen-deuterium exchange.

Results and Discussion

The absorption isotherms of cured resins are characterized by a low moisture uptake at low RH and a steep increase in moisture content (MC) above ca. 50 % RH (Figure 1). The methylolation of DMDHEU and the addition of diethylene glycol increased the moisture absorption at elevated RH, which may be the result of fewer covalent bonds between adjacent DMDHEU molecules within the cured resin.
Figure 1. Sorption isotherm at 25 °C of various cyclic N-Methylol compounds (mDMDHEU with diethylene glycol (circles, a), mDMDHEU (triangles, b) and DMDHEU (rhombus, c)) after heat-curing at 120 °C.

Adsorption isotherms of modified wood specimens (Figure 2) differed significantly from the sorption isotherms of the cured resins. An increase in MC<sub>r</sub> at low RH levels was assigned to the moisture uptake of the resins in addition to the moisture uptake of the wood. This was in line with an increase in the (WPG-corrected) hydroxyl accessibility by the modification. At 95 % RH, the modification with a 10 % solution resulted in a reduction of the MC<sub>r</sub> compared to the reference value, which may indicate a cross-linking effect, similar to the effect of a formalization of wood (Himmel and Mai 2015). The modification with a 20 % solution increased the MC<sub>r</sub> compared to a 10% solution. This was presumably caused by a higher proportion of resin in the cell lumen. In line with this hypothesis, the lower cell wall bulking of DMDHEU resulted in a larger MC<sub>r</sub>.

Figure 1. Absorption isotherms (MC<sub>r</sub>) at 25 °C of Scots pine sapwood (Pinus sylvestris L.) after treatment with DMDHEU (rhombus), mDMDHEU (triangles) and mDMDHEU plus diethylene glycol (circles) at 10 % (blank symbols) and 20 % (filled symbols) treatment level and untreated controls (cross).

The OH accessibility also differed depending on the modification agent used. In line with differences in cell wall bulking, mDMDHEU with glycol resulted in the lowest OH accessibility. This may have been caused by the blocking of accessible OH groups by steric hindrance when increasing amounts of modification agent are present in the cell wall (Beck et al. 2017). The ratio of absorbed (deuterated) water molecules at 95 % RH per accessible OH group was decreased by the modification. In contrast to the results of the OH accessibility, DMDHEU was more effective in reducing this ratio than the other modification agents.

Conclusions

This study confirmed indications for both, a cell wall bulking and a cross-linking effect caused by the modification. The OH accessibility differed in the order of DMDHEU > mDMDHEU > mDMDHEU with diethylene glycol, while the ratio of adsorbed water per accessible OH group differed in the opposite order. Pure resins showed a strong moisture uptake at elevated RH, which may affect the sorption of the wood significantly if resin is located in the cell lumen.
References


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POTENTIAL OF BIRCH SHAVINGS BONDED WITH BIO-BASED ADHESIVES FOR FOOD PACKAGING APPLICATIONS

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Background

Europe produces 64.4 million tons of plastic every year while 40 % is used for packaging (The European Commission, 2018). 1.5 % to 4 % of global plastics production, which is approximately 12.2 million tons (Sherrington, 2016), pollute the oceans every year (Jambeck, et. al., 2015). According to the European strategy, all petrochemical-based packaging on the EU market should be either reusable or recycled in a profitable manner by 2030 (The European Commission, 2018). Also, manufacturers need to account for the increasing environmental awareness of consumers in order to meet the consumer demands. Using wooden packaging with its sustainable and natural character provides a potential solution to these problems (Aviat, et. al., 2016).

Birch is commercially underutilized in the majority of its distribution compared to other softwood tree species nowadays. In Norway, there is no tradition, industry, or established value chains for utilization of birch. Some birch is used for firewood, pulp wood, and in very small volumes in furniture and cladding (Bunkholt & Eikenes, 1993) as well as plywood (Grosser & Teetz 1998). In 2016, birch constituted less than 2% of commercial roundwood removal in Norway (SSB, 2019).

Some wood species are already used for food packaging, but birch is hardly used. The objective of this study is to investigate new product applications for birch which can replace plastic packaging and single-use items and to evaluate the potential of birch in contact with food. The end of life uses of the products will be composting in the summer and fire-starters for ovens in the winter. Various bio-based adhesives are assessed for their compatibility with birch shavings and the strength and moisture resistance of the composite materials are tested.

Experimental

Downy birch (Betula pubescens Ehrh.) planks were sawn and subsequently planed with a feed rate of 10 m/min and a cutting depth of 1.5 mm per round. The lengths of the shavings were approximately 12.3 % < 10 mm, 36.30 % < 8 mm, 14.1 % < 4 mm and 37.3 % < 2 mm and the thickness of the shavings was 0.1 – 0.25 mm.

Anhydrous citric acid powder (VWR Chemicals, CAS 77-92-9) was purchased from Weifang Ensign Industry Co., Ltd. (Shandong, China) and used without further purification. It was dissolved in de-ionized water to a concentration of 1 Mol. It was used as a dispersing agent with a cross-linking function for all adhesives and as an adhesive itself with a 15 % wt/wt and 20 % wt/wt resin content based on the oven-dried shavings.
Commercial wheat gluten powder was obtained from Kinsarvik naturkost (Nesttun, Norway) and soy protein isolate powder from Star nutrition which is traded from Health and Sports Nutrition Group (Oslo, Norway) were mixed with the 1 Mol citric acid solution, in order to unfold the proteins, at room temperature with 15 % wt/wt wheat gluten powder and 7,5 % wt/wt soy protein isolate, respectively. The solution was used as an adhesive with 20 % wt/wt and 25 % wt/wt resin content based on the oven-dried shavings.

In order to gelatinize the granules of commercial potato starch powder from Hoff SA (Gjøvik, Norway), 7,5 % wt/wt of the powder was dissolved in warm 1 Mol citric acid solution (70 °C ± 2 °C). The warm mixed solution was then sprayed onto the shavings with a 20 % wt/wt and 25 % wt/wt resin content based on the oven-dried shavings. 15 % wt/wt arabic gum powder from Urtegaarden which is traded from Sunvita AS (Nyborg, Norway) was dissolved at room temperature in the 1 Mol citric acid solution and used with the same resin contents as mentioned for starch.

Commercial sucrose from Nordic Sugar (Oslo, Norway) and honey from Honningcentralen SA (Kløfta, Norway) were used in a ratio of 50 % wt/wt dissolved in the 1 Mol citric acid solution. While the former was mixed at room temperature, the latter was heated to 35 °C ± 2 °C in order to guarantee a quick solubility. The solutions were sprayed with a 40 % wt/wt and 50 % wt/wt resin content based on the oven-dried shavings.

All solutions were stirred with a magnet stirrer and mixed and spray-applicated at room temperature on the same day. 200 g of the planed shavings with a MC of 7 % were used per board. A spinning welded bucket was used as a mixing container. A commercial spray gun was filled with the pre-weighed different adhesives according to the resin content and was then held through the transparent lid and the adhesives applied by a fine mist which occurred due to the high pressure.

In order to reduce the MC of the adhesive coated shavings to 4-6 %, the sprayed shavings were dried in a chamber at 75 °C. After drying, the shavings were hand-formed into a mat using a wooden forming box (35 cm x 35 cm) on a baking paper covered metal plate and then manually pre-pressed with another metal plate. The lifted forming box left a pre-formed mat which was covered with baking paper and with a metal plate on top.

All boards were hot pressed at 160 °C for 9 minutes and 15 seconds under a max. pressure of 350 kN and a ramp duration of 15 seconds. During the last 30 seconds the heating was removed and the press opened stepwise while reducing the pressure, 5 seconds 200kN, 5 seconds 90 kN, 15 seconds 10 kN and 5 seconds 0 kN, in order to introduce a degassing phase and to avoid abrupt steam pressure release.

The boards were cut into samples based on to the standards EN 323, EN 322, EN 311 and EN 310 and labelled. Six replications per adhesive and resin content were pressed. Three for climate A (20 °C ± 1 °C and 45 % ± 3 % RH), three for climate B (20 °C ± 1°C and 65 % ± 3 % RH), leftovers out of these boards were used for climate C (4 °C ± 1 °C and 90 % ± 3 % RH). The samples were conditioned in an upright position until a stable moisture content occurred.

Results, Discussion and Conclusion

Results, Discussion and Conclusion are not available by now, since the different tests start on the submission date of this paper. What will be done are quick checks for mass stability in every climate and on every adhesive and resin content. We will assess the RH by datalogger, the MC and the weight of prepared samples after 24 hours every day during the conditioning period. All samples in the three different climates will be tested based on the standards EN 323, EN 322, EN 311 and EN 310 once the conditioning is finished.
What can be expected is that the mechanical properties decrease with higher humidities. Further investigation is then necessary in order to clarify the bonding mechanism of the adhesion system and to improve the water resistance. Also, to investigate different biobased coatings and the degradation of the food packaging material.

References


PRESSURIZED HOT WATER EXTRACTION OF SCOTS PINE SAPWOOD: EFFECT OF SAMPLE SIZE

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Background

In hot water extraction (HWE) wood is treated in high-temperature water (above 100 °C) that is kept in liquid phase with elevated pressure. Previous studies on HWE-treatment have focused on wood powders, particles or chips and its potential as a pretreatment method for lignocellulosic materials prior to pulping and for extracting wood components for the conversion into value added products (Ragauskas et al. 2006; Amidon and Liu, 2007). HWE may potentially be applied to larger wood products (e.g. sawn timber) as well, which can then be utilized as modified wood. However, previous studies have demonstrated that the wood chip size affects the extraction rate of hemicelluloses significantly (Krogell et al. (2013) and Li et al. (2013)). Our research investigates how this phenomenon applies to larger samples of solid wood. Therefore, Scots pine sapwood samples of different sizes were hot water extracted at different temperature-time environments and the changes in chemical composition of the solid residue and the process water were analyzed.

Experimental

Scots pine (Pinus sylvestris) sapwood was cut into two sample types with different dimensions: small 10×10×20 mm³ and large 50×50×100 mm³ (R×T×L). HWE-treatment was performed in closed vessels that were heated in an air-bath reactor. The sample batches were first vacuum impregnated with deionized water and then treated in pressurized hot water at different temperatures (130-170 °C), durations (40-200 min) and liquid/solid ratios (4-20). Severity of each HWE-treatment was determined with equation by Overend and Chornet (1987). After HWE, the extraction liquid and the solid wood residues were separated with a filtering cloth. Mass loss (based on dry wood mass) and corrected mass loss (based on dry and extractive-free wood mass) were measured for the solid residue. Measurement methods for composition of solid residue were adapted from standards EN-13183:2002 (SFS, 2002), TAPPI T 211 (TAPPI, 2002), TAPPI T 204 cm-97 (TAPPI, 1997) and NREL/TP-510-42618 (Sluiter et al., 2012). Measurements for composition of extraction liquid were adapted from standards EN 15216:2007 (SFS, 2008) and SFS 3008 (SFS, 1990), NREL/TP-510-42623 (Sluiter et al., 2008) and NREL/TP-510-42618 (Sluiter et al., 2012). The analyses were completed by an observation of selected samples after water leaching and after extraction with acetone by scanning electron microscopy (SEM).
Results and Discussion

The mass loss of the samples increased as a function of the treatment severity, but a stronger increase in mass loss was evident for the small samples (Figure 1). The differences between the two sample types decreased when the mass loss was calculated based on the dry and extractive-free mass (corrected mass loss). Interestingly, the liquid/solid ratio did not influence the change in mass loss.

Figure 1. a) Mass loss [%] and b) corrected mass loss [%] in dependence on the hot water extraction severity (log [Ro]). The liquid/solid ratio during HWE was 20. Error bars show the standard deviation.

The changes in chemical composition in dependence on the mass loss confirmed a preferential removal of hemicelluloses and an accumulation of cellulose and lignin in the solid residue (Figure 2). While the loss in hemicelluloses and the accumulation of celluloses as a function of the mass loss were very similar for both sample types, a higher data scattering for the changes in lignin and a higher amount of acetone-soluble compounds was evident for large samples. The presence of acetone soluble compounds was also observed by SEM when comparing the outer surfaces of large samples before and after acetone-extraction. We speculate that increasing the sample size hindered the diffusion of degradation products from the wood blocks into the process water. Thereby, a higher amount of degradation products remained and potentially precipitated within the wood after cooling and was only removed by extensive acetone extraction.

Figure 2. Changes in amount of a) cellulose (estimated by glucose content) b) hemicelluloses c) lignin and d) extractives in the solid residue in dependence on the mass loss. The chemical composition was calculated on an as-received basis. SEM-images taken from surface of e) water leached and f) water leached and acetone extracted small sample and g) water leached and h) water leached and acetone extracted large sample. Red line shows 10 µm scale bar.

The chemical composition analyses of the solid wood were supported by analyses of the process water. Although the pH of the process water decreased by HWE, a poor correlation with the measured mass loss was found. In line with the mass loss results, the amount of carbohydrates in the process water as a function of the treatment severity was higher for the small samples. For both sample types, a continuous increase in the monosaccharides content with increasing treatment severity was found in the process water. In contrast, the amount of polysaccharides increased first, but decreased for high treatment severities due to their hydrolytic cleavage.
Conclusions

Our research demonstrated a decrease in the mass loss rate and the hemicellulose yield in the process water when increasing the wood sample size. This was at least partially caused by the accumulation of acetone-soluble degradation products within the large samples due to an incomplete diffusion of degradation products from the wood into the process water. Such an incomplete diffusion is a major challenge for the application of HWE to larger wood products such as full-sized timber boards.

References


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INVESTIGATION OF SPRINGBACK EFFECT OF LAMINATED BEECH STACKS

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Background

The springback effect of materials is well known but there are no reliable formulas to predict it (Jones, 2018). Mostly a FEM model is used which complicates the solution for simple forms. Wood lamellas can be bent and laminated into quite complex forms. The simplest form would be the shape of an arc. Gluing a number of wooden layers together the result differs from the original radius of the die. A preliminary study on beech wood was performed to investigate the springback effect as a function of the number of layers, the MOE of the sheet stack, the thickness of the lamella or/and the residual radius. Obtained correlations could be transferred into a formula to calculate the springback on wood.

Experimental

Paramters and material

The thicknesses of the beech (Fagus silvatica L.) lamella were 1 mm, 2 mm and 4 mm while the total thicknesses of the stacks were 8 mm, 12 mm and 16 mm. This allows the calculation of the number of lamellas. With each of the above mentioned combinations. Samples with three different radii (250 mm, 300 mm and 350 mm) were produced with identical procedure and parameters.

Results

Figure 1 shows the correlation of the count of lamellas in a stack and the springback in %. The trend line is a power function and is quite accurate considering that stacks consist of lamellas with different thicknesses. The higher the count of lamellas in the sample the lower the springback effect.
While there was a limited maximal springback due to the count of two lamellas, it was obtained that the radius size had no impact on the springback effect of the different samples of the entire test-series. Since the measurement of the MoE is very elaborate to be implemented in industrial processes and workshops, a formula would remedy the situation. Obtained results showed persuasive accurately determined correlation of the springback effect and the MoE (Figure 2).

Figure 1. Exponential correlation of springback effect in % and count of lamellas within the stack

Figure 2. Linear correlation of springback in % and mean MoE of stack in N/mm²

Conclusions

Our preliminary tests showed that the radius has no influence to the springback [%]. To determine the springback effect in laminated form parts the MoE stated the best option, since results showed a linear correlation. To validate the results, additional tests with identical counts of lamellas in each stack and different thicknesses of lamella need to be performed. Additionally, different wood species and grain orientations have to be investigated.
References

STUDY OF SHEAR CUTTING MECHANISMS ON WOOD VENEER

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Background

Due to environmental changes industries are looking for lightweight renewable and degradable material solutions to solve weight and therefore CO₂. One highly prioritized topic is the combination of wood materials with plastics. To take advantage of the mechanical behaviour of the wood structure it is necessary to investigate the combination of veneer lumber with plastic (Buchelt et al., 2015, p. 356). Regarding large-scale production an injection moulding process with integrated cutting is addressed in this study. For the process development, it is necessary to comprehend how different wood materials behave in the process step of cutting within an integrated injection moulding process. Recently there are no findings on favourable shear cutting parameters on wood veneers, due to the uncommon application of shear cutting and stamping on these materials (Kollmann, 2017, p. 781; Wagenführ and Scholz, 2018, p. 353). Hence, it is necessary to investigate the behaviour and cutting edge quality of veneer lumber in shear cutting process as a function of process parameters. This work investigates experimentally the cutting force under influence of cutting angle, cutting speed, wood materials and moisture content. Results will be analysed in the context of a subsequently following injection process after cutting.

Experimental

The shear cutting process is realized on a self-designed shear cutting tool following DIN 8588 (3.1.1.1 single stroke shear cut). Different single layer veneer lumber specimens were shear cut to investigate the cutting edge while documenting the cutting force \( F_c \) in kN as target variable. The processed specimen size was width of 60 mm and 20 mm in length. The investigated cutting edge is therefore 60 mm long. All process parameters and values can be found in Table 1.
Table 1. Used process parameter of experiments and their variation steps

<table>
<thead>
<tr>
<th>Process parameter</th>
<th>Symbol &amp; Units</th>
<th>Variation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cutting Speed</td>
<td>$v_c$ in mm/min</td>
<td>10; 50; 100; 200; 300; 400; 500</td>
</tr>
<tr>
<td>Shear Gap</td>
<td>$s$ in mm</td>
<td>0.016</td>
</tr>
<tr>
<td>Cutting Angle</td>
<td>$\alpha_c$ in °</td>
<td>0; 45; 90</td>
</tr>
<tr>
<td>Wood Material</td>
<td></td>
<td>beech (BE); oak (OA); birch (BI); maple (MA)</td>
</tr>
<tr>
<td>Veneer Thickness</td>
<td>$t_{xx}$ in mm</td>
<td>$t_{BE}: 1.3$; $t_{OA}, t_{BI}, t_{MA} : 2.0$</td>
</tr>
<tr>
<td>Water content (EN 13183-1) after 5 days water immersion at RT</td>
<td>$\omega$ in %</td>
<td>12 (at 65% RH); 90BE; 90OA; 70BI; 65MA</td>
</tr>
<tr>
<td>Quantity of specimen per series</td>
<td></td>
<td>10</td>
</tr>
</tbody>
</table>

Results and Discussion

Influence of cutting angle $\alpha_c$ to grain orientation
The highest cutting force could be detected at $\alpha_c = 90^\circ$ for all materials at all investigated parameters. This is caused to the cellular structure of the wood (tracheids, growth rings). The highest declination was explored for wet maple veneer $F_c 0^\circ$-$90^\circ = -85.89\%$ (Figure 2).

Influence of cutting speed $v_c$
The cutting force $F_c$ lowers with higher cutting speeds $v_c$ (regressive trend). This effect is mainly detected for $v_c = 5 – 200$ mm/min. A higher $v_c$ doesn’t take a significant effect on the cutting force but speeds up the whole cutting process without taking negative influences on the cutting edge behaviour (Figure 1 - right).

Figure 1. Influence of Cutting Velocity $v_c$ on Cutting Force $F_c$; Beech $t_{BE} = 1.3$ mm; $\alpha_c = 90^\circ$

Influence of Wood Material
In Figure 2 there is shown that birch induces the lowest $F_c$ at $\alpha_c = 90^\circ$ while maple shows the highest value. For all materials there is a significant effect on $F_c$ between $\alpha_c = 90^\circ$ and $\alpha_c = 0^\circ$. Here again maple specimens show the highest reduction on $F_c 0^\circ$-$90^\circ = -79.54\%$. The highest standard deviations are detected for maple veneers as well.

Figure 2. Influence of Wood Material and Moisture Level $\omega$ on Cutting Force $F_c$; $v_c = 500$ mm/min

Influence of Moisture Level
In Figure 2 there is also the influence of moisture content in the wood veneers shown. The specimen of $\alpha_c = 90^\circ$ wet birch and maple show an upward trend of $F_c$ but with regards to
standard deviation the effect is not significant. Anyhow the \( F_c \) on \( \alpha_c = 90^\circ \) of wet beech (- 34 %) and oak (- 23 %) lowers more in comparison to birch and maple. This can be referred to the high moisture content of beech and oak (< 90 %). All the specimens show a decrease of \( F_c \) on \( \alpha_c = 0^\circ \). The most significant decrease for \( \alpha_c = 0^\circ \) can be detected on birch veneer (- 75 %).

Conclusions

Basically shear cutting is a suitable process to cut veneer lumber. Cutting edge quality depends on all parameters investigated. Especially birch veneer is a potential material for the injection moulding process with integrated cutting by showing low cutting forces, small influence of the cutting angle and low deviation value. Future investigations will show if the mechanical properties of birch veneers are suitable for the application in hybrid veneer lumber-plastic parts.

Reference list


THE IMPORTANCE OF COLD TACK OF UREA FORMALDEHYDE
IN PLYWOOD PRODUCTION

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Background

Pre-pressing veneers to activate cold tack of urea-formaldehyde-(UF)-resin is essential in plywood production to ensure the veneer layers fit into the openings of a hot-press. Several studies on the cold tack of UF focussing on particleboard production were carried out and various influencing factors were identified by Neumann and Müller (1979) on scarf joints. Although cold tack is of great importance in the production of plywood, information based on systematic investigation was rare. Also the influence of cold tack on the behaviour of the cured glue joint has also not been investigated so far.

In this study, a better understanding of cold tack of UF in plywood production was sought and the gap between the influence of various factors on cold tack and the cured bondline was closed. Influencing factors were defined and tensile shear strength was chosen as a parameter to assess the quality of cold tack.

Experimental

Industrial UF adhesives of various ages were applied in different quantities to veneers with specific moisture content (MC) and temperatures. A second veneer was then applied to the overlapping surface without external pressure. The veneers were stored in climatic chambers at defined humidity and temperature for the duration of the lay-up time. The samples were then pre-pressed with a manual press, exposed to the same controlled climatic conditions as in the previous step. Tensile shear strength (TSS) was subsequently tested with a universal testing machine. The defined factors and their combinations were investigated and statistically analysed. It was therefore possible to calculate the effects of numerous combinations of different factors, including those that were not experimentally investigated. In order to assess the influence of cold tack on the performance of the cured bondline, the veneers were pressed in a hot press and TSS and wood failure percentage were evaluated.

Results and Discussion

The lay-up time, veneer moisture content (MC), veneer temperature and pre-press time, as well as interactions between factors showed a significance in the statistical evaluation of the results.
obtained from the cold pressed samples. Therefrom, the MC, followed by the veneer temperature showed the highest impact on the tensile shear strength as is depicted in Figure 3. Higher tensile shear strength and therefore a better cold tack could be reached with a lower moisture content as well as with a higher temperature. This results can be explained by the need for the resin to dry in order to develope cold tack (Dunky and Niemz, 2002).

In contrast to the case with the cold pressed samples, the median values of the TSS for samples with cured UF are quite similar (in a range of 6.1 – 6.4 N/mm²) and strong influences of the selected parameters are not discernible Figure 4.

![Figure 3: Tensile shear strength as the result of cold tack of birch veneer samples in dependence of veneer MC and veneer temperature.](image1)

![Figure 4: Tensile shear strength of hot pressed birch veneer samples in dependence of veneer MC and veneer temperature.](image2)

**Conclusions**

Cold tack is an important property of UF resin, used in the production of plywood panels. It was possible to evaluate the cold tack behaviour in relation to several factors important for plywood production (Hogger et al., 2018). The strongest influence on cold tack was shown to be MC. This effect was not detectable for cured glue joints, where no influence of the cold tack on the TSS could be observed (Hogger et al., 2019).

**References**


Background

Acetylation has proven successful in limiting the hygroscopicity of wood, and improves both durability and dimensional stability (Rowell, 1996). It provides an environmentally friendly way to protect wood in outdoor applications. Several studies have been conducted in order to determine basic mechanical properties of acetylated wood. Less research has been done regarding the impact of acetylation on fracture characteristics. Fracture characteristics are important to regard e.g. when designing mechanical joints in structural applications (Gustafsson, 2003). A previous study (Reiterer and Sinn, 2002) indicated a reduction of the fracture energy by 20% for acetylated spruce.

The aim of this study is to investigate fracture characteristics of acetylated young Scots pine and birch. The fracture energy, the modulus of elasticity and the tensile strength have been determined experimentally. Moreover, a numerical study has been conducted to calibrate proper material models, that will be used in analyses of structural applications.

Experimental

Material

The examined material consisted of unmodified and acetylated Scots pine and birch. The modified samples originated from boards acetylated in an industrial scale process, reaching an acetyl content of approximately 20%. Clear wood specimens consisting of sapwood were examined. After conditioning at a relative humidity of 60% and a temperature of 20°C, unmodified and modified samples reached a moisture content of approximately 10-11% and 2-4% respectively.

Methods

The fracture energy was determined for Scots pine and birch, according to the standard NT-BUILD 422 (1993). For Scots pine, also the modulus of elasticity in compression along the grain and the tensile strength perpendicular to the grain were determined. Figure 1 illustrates the test-setups.
Results and Discussion

The impact of acetylation on the examined characteristics is presented in Figure 2. Mean values for acetylated samples are related to mean values for unmodified samples, which are set to 100% as a reference. Differences are specified in percentage and error bars indicate confidence intervals with a level of significance of 95%. The result demonstrates a significant decrease of the fracture energy for acetylated samples. The mean fracture energy decreased with 50% and 57% for acetylated Scots pine and birch respectively. No significant differences were observed for the modulus of elasticity or the tensile strength.

The increased brittleness could be an adverse effect of acetylated specimens exhibiting less fibres per cross section area, or due to degradation of the cell wall polymers caused by the high temperatures or the chemicals involved in the modification process. Another possible explanation could be that the lower fracture energy is a consequence of acetylated samples having a lower moisture content. Previous studies have shown that a lower moisture content correlates to a decreased fracture energy (Majano-Majano, Hughes and Fernandez-Cabo, 2012; Reiterer and Tschegg, 2002). To further analyse why acetylation impairs the fracture energy, additional research is needed to separate the above-mentioned effects. An ongoing study aims at finding a correlation between moisture content and fracture energy for both modified and unmodified Scots Pine and Birch. Moreover, to enable the use of acetylated wood in outdoor load-bearing structures, the effect of acetylation on the structural behaviour of mechanical joints will be investigated.

Conclusions

Based on this study, an increased brittleness of acetylated wood was confirmed. To gain understanding of why the fracture energy is impaired, further studies are needed. These could e.g. be focused on finding the correlation between moisture content and fracture energy. Moreover, to facilitate the use of acetylated wood in outdoor load-bearing applications, the effect on structural members and mechanical joints should be assessed.
References


A MINIATURISED PRESSURE PLATE CELL FOR IN-SITU X-RAY IMAGING OF WATER DISTRIBUTION IN WOOD

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Background

Wood-based materials for construction purposes show great potential to lower the global emissions of greenhouse gases, but the use of these materials is limited by its degradation when exposed to high moisture levels. The durability of wood-based materials can be improved by chemical modification that decreases the moisture content. While the lowered moisture content is important for the increased durability, the location of the water is also believed to play an important role. The novel experimental method presented here combines the use of high resolution 3D X-ray tomography and the pressure plate technique conditioning (Fredriksson 2016) to measure the location of water in small wood samples.

Experimental

A small humidity cell in Figure 1 has been developed by the authors for in-situ 3D X-ray tomography measurements of the water distribution in wood samples. The cell is based on the pressure plate technique and enables in-situ measurements at well-defined high levels of relative humidity. In order to get good quality images in terms of resolution and noise while maintaining a short acquisition time, the cell has been scaled down as much as practically possible.

Results and Discussion

The X-ray images presented here was acquired with a voxel (3D pixel) size of 1.5 micron using the Zeiss Xradia XRM520 lab tomograph at the 4D Imaging Lab, Division of Solid Mechanics, Lund University. Figure 2 shows X-ray images of a wood samples at close to water saturated state and after 4 hours at 99.9% relative humidity (not at equilibrium). The images show how the distribution of...
capillary water changes with time. Already after a few hours, most of the water filled cell lumina appear empty.

Conclusions

The miniaturised pressure plate cell presented here can be used together with 3D X-ray tomography to measure the distribution of capillary water in wood under well controlled humidity conditions. Thanks to the small size of the device, and its transparency to X-rays, it is possible to image wood on a cell level while still keeping the acquisition time low enough to follow the evolution of water distribution during, e.g., wetting and drying.

References

WATER CAPILLARY UPTAKE OF SPRUCE WOOD

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Background

For purposes of numerical modelling of hygrothermal processes, it is necessary to have all important material properties. Regarding wood and its specific structure, it is usually more complicated to get a suitable value compared to the other building materials, especially in terms of moisture behaviour. One of those is the transfer coefficient for liquid water $D_{ws}$. To obtain this value, it is necessary to know the absorption coefficient $A_{cap}$.

Evaluating the absorption coefficient for scotch pine has been used by Sedighi-Gilani et al. 2014. For spruce wood, the water absorption coefficient has been measured in a dissertation by W. Zillig (2009).

This work deals with the measurement of water capillary uptake of spruce wood in three principal directions - radial, tangential and axial. The computational models based on this experiment can be used e.g. for predicting the moisture content of wood exposed to rain in varying climatic conditions.

Experimental

The experimental procedure is based on EN ISO 15148. However, due to the specific behaviour of wood, some changes are proposed in order to develop a suitable measurement method to get appropriate value for numerical simulations.

The most important changes compared to the standard:

1) In this work, smaller samples (cubic samples 25 ±0,3 mm) were used compared to the standard.

2) The samples were dried at laboratory temperature with a desiccant. This approach appears to be more suitable for many other purposes.

3) In the case of the axial direction, the measurement was performed until the full saturation was reached (for radial and tangential direction until equilibrium moisture content). According to the standard, if water appears on the upper surface of the sample, the experiment has to be terminated.

Measurement has been repeated to see the differences among capillary suction courses.

The results have been compared with the study by Zillig (2009).
Results and Discussion

The graphs show that the results in each direction are almost identical to those of W. Zillig's dissertation. During the first month, two different slopes of the capillary process are visible. As it was mentioned above, the capillary uptake differs from many other building materials where only one slope is usually present. Among the measured water absorption curves, there is a noticeable difference in liquid water absorption in each direction. In the axial direction, the wood sucks up to 5.0 times more water on the first day than in the radial direction. While the absorption coefficient in the radial and tangential direction are very similar.

The moisture distribution is clearly related to the layers of earlywood and latewood. Latewood shows more water absorption than earlywood layers.

Also, it was revealed that by the repeating experiment, completely different suctions has been recorded.

Conclusions

The experimental measurement has been performed in three directions according to fibre orientation - radial, tangential and axial. Faster transport of liquid water is evident 1) in the axial direction compared to the others directions; 2) in latewood compared to earlywood.

The results in each direction were compared with results from Zillig (2009) and they are almost identical.

By the repeating experiment, completely different suctions has been recorded.
References


Background

Wood is an important engineering material. However, wood surface is susceptible to moisture and biological degradation during natural weathering process that occurs both indoors and outdoors due to the hydrophilic nature of its cell walls. This significantly reduces the durability and service lifetime of wood products. Preparing superhydrophobic surfaces on a wood substrate has shown a great potential to address these problems and functionalize the wood (Teisala and Butt 2018). Recent research has also shown that liquid repellence of oils and other low surface tension liquids is possible and is called superamphiphobicity (Tuominen et al. 2016), which would enhance the applicability of such wood surface treatment.

In this work, we prepared superamphiphobic wood surfaces based on a solvent-free method, i.e., gas-phase silicone nanofilaments reaction and followed by gas-phase fluorine alkylsilane silanization (Zimmermann et al. 2008). The surface morphology, chemical compositions and wettability of the modified wood surfaces were studied.

Experimental

Materials

Rotary cut birch (Betula pendula) veneers (wood tangential section) from Finland with dimensions of 50×30×1 mm³ in the longitudinal, tangential and radial directions (L×T×R), respectively, were used as substrates. Trichloromethylsilane (TCMS, 99%), ethylene glycol, hexadecane, decane and potassium carbonate were purchased from Sigma-Aldrich. 1H,1H,2H,2H-perfluorodecyltrichlorosilane (PFDTS, 96%) was purchased from Alfa Aesar.

Method

Silicone nanofilaments reaction was performed under controlled humidity conditions at room temperature. A vial of 300 µL TCMS was placed in a sealed desiccator with a relative humidity of 42-43% using saturated aqueous potassium solution. The reaction time was 24 hours. Chemical vapor deposition of PFDTS was applied to further reduce the surface energy after silicone nanofilaments reaction. The liquids used in the contact angle measurement were water, ethylene glycol, hexadecane and decane, which had surface tensions of 72.8, 48.3, 27.5 and 23.8 mN m⁻¹, respectively.
Results and Discussion

Surface morphology
The wood surface has an inherent roughness as a result of micro- and nano-structures of different wood cells (Figure 1a), but still lack the nanoscale roughness that is needed for superhydrophobicity. The silicone nanofilaments were covered on the wood surface after the modification process as shown in Figure 1b-c. The diameter of the silicone nanofilaments was between 10 to 60 nm. A wood surface with both micro- and nano-scale roughness was obtained by silicone nanofilaments coating.

![Figure 1. SEM images of (a) the unmodified wood, (b-c) the modified wood and (d) the distribution of the diameter of silicone nanofilaments.](image)

Wettability
Table 1 gives contact angles for the surface modified wood. Water droplets could not pin on the surface and it rolled off immediately from the surface once dispensed. Thus, no water contact angle was given in Table 1. The modified surface also showed contact angles higher than 150° towards the low surface tension liquids such as ethylene glycol and hexadecane. The roll-off angles for ethylene glycol and hexadecane were lower than 10°. These results show that the modified surface is superamphiphobic.

<table>
<thead>
<tr>
<th>Liquid</th>
<th>CA(°)</th>
<th>θad(°)</th>
<th>θre(°)</th>
<th>CAH(°)</th>
<th>Roll-off Angle(°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>Very high</td>
<td>Very high</td>
<td>Very high</td>
<td>Very low</td>
<td>&lt;1</td>
</tr>
<tr>
<td>Ethylene glycol</td>
<td>153</td>
<td>160±5</td>
<td>140±12</td>
<td>20±9</td>
<td>4±3</td>
</tr>
<tr>
<td>Hexadecane</td>
<td>150</td>
<td>154±5</td>
<td>130±15</td>
<td>23±13</td>
<td>7±4</td>
</tr>
<tr>
<td>Decane</td>
<td>143</td>
<td>150±4</td>
<td>83±10</td>
<td>67±11</td>
<td>39±10</td>
</tr>
</tbody>
</table>

Conclusions
A superamphiphobic wood surface was successfully prepared by growing silicone nanofilaments on the wood surface and followed by fluorosilanization. The modified surface also showed excellent self-cleaning properties.

References
PROPERTIES AND STRUCTURAL FEATURES OF SUPERHYDROPHOBIC SCOTS PINE WOOD

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Background

Even though wood is a widely used material in different applications, it has a number of disadvantages, such as: low dimensional stability, susceptibility to biological attack and weathering (Hui et al 2015). Therefore, many efforts have been made to expand its potential applications or to increase the life time of wooden products by chemical modification or impregnation (i.e. Evans et al 2000, Mai & Militz 2004, Hill 2006, Hui et al 2015, Jia et al 2018, Li et al 2019). Recently, attempts have been made to create composite functional materials by mimicking biological structures and architectures (lotus and Salvinia leaf, rose petal, butterfly wings effect) that cause excellent functions and performance on which the water drops roll off the surface (Guo et al 2017, Jia et al 2018, Xing et al 2018, Li et al 2019, Lin et al 2018). Such superhydrophobic surfaces can minimize the wood-water interactions and thereby avoid damages associated with the water adsorption. Applying and understanding the underlying principles for the design of composite materials with special properties may guide materials science towards the use of renewable, sustainable, smart and more environmentally friendly materials. To date, significant effort has been developed to fabricate hydrophobic/superhydrophobic surfaces, but most of them exhibit poor durability and tend to lose their anti-wetting properties even upon small stresses, giving low wear resistance and reduced durability, or they use harmful solvents for their preparation. The aim of this research was to create a superhydrophobic wood surface by using modified silica nanoparticles in combination with a water soluble polymer.

Experimental

Materials

For the study, Scots pine (Pinus sylvestris L.) wood specimens were used. Tetraethyl orthosilicate (TEOS) was purchased from Aldrich. Ammonium hydroxide (NH$_4$OH), 1H,1H,2H,2H – perfluorodecyltriethoxysilane (FAS) and poly(acrylic acid) were purchased from Alfa Aesar, and the ethanol (EtOH) from VWR Chemicals.
Different solutions were used to impregnate the wood: S1 – modified nanoparticles with FAS, S2 – silica nanoparticles in combination with the polymer, and S3 – modified nanoparticles with FAS in combination with the polymer.

**Methods**

*Weight percent gain (WPG)* of the wood samples was determined according to the following equation:

\[ WPG\% = \frac{W_f - W_o}{W_o} \times 100 \]

where: \( W_o \) – oven dry weight before the impregnation and \( W_r \) – oven dry mass after the impregnation process.

*Scanning electron microscopy (SEM)* – the wood samples were investigated using a scanning electron microscope (Tescan Vega 3-LMU, Czech republic) equipped with Energy dispersive X-Ray spectroscopy (EDS, X-Max 20, Oxford instruments, UK). Each sample was attached to a SEM aluminium stub before being Pd coated for 3 min using a sputter coater (Polaron equipment limited SEM coating unit E5100).

*Attenuated total reflection infrared spectroscopy (ART-FT-IR)* – the spectra were measured at 4 cm\(^{-1}\) resolution and in the 4000-500 cm\(^{-1}\) spectral range on a Brucker ALPHA FT-IR spectrometer using a diamond crystal. Five recordings were performed for each sample, the evaluation being made on the average spectrum obtained from these recordings.

*Contact angle measurements* - the static contact angles were determined by the sessile drop method, within 30s, after placing 2 μL drop of water on the surface, using a CAM-200 instrument from KSV, Finland. Contact angle measurements were taken in five different locations on the surface and the average values were further considered. The pictures were recorded immediately after placing the water drop on the substrate surface, for a period of 30 seconds, to evaluate the water absorption rate.

**Results and Discussion**

The WPG% values were calculated for each sample and each type of solution (10 replicates for each type). The average values are as follows: 3.43, 3.30 and 4.34 % for the wood samples impregnated with S1, S2 and S3, respectively.

The ATR-FTIR spectra and SEM analysis demonstrated that the silica nanoparticles and modified nanoparticles without or with the polymer used as matrix deposited on the wood surface, and the resulted coating induce highly hydrophobic properties of the wood surface.

The initial contact angle of the wood samples was of about 64\(^o\) for the control, of about 149\(^o\) for the wood impregnated with S1 solution, 121\(^o\) for the wood samples impregnated with S2 and about 143\(^o\) for the wood impregnated with S3. The water droplet disappeared from the wood surface after about 2s in the case of the control sample, while in the case of the treated samples the contact angle remained constant over a period of 20s.

**Conclusions**

The control and impregnated samples were evaluated by SEM, ATR-FT-IR spectroscopy and contact angle measurements, and it was observed that the wood impregnation was successful realized and highly hydrophobic surfaces were obtained.
References


INCORPORATION OF ORGANIC BIO-BASED PHASE CHANGE MATERIALS IN WOOD FOR ENERGY STORAGE PURPOSES

Authors: Meysam Nazari*, Mohamed Jebrane, Nasko Terziev and Nadine Herold

Background

“Green” buildings constructed by bio-based materials have been considered as a strategy to battle the climate changes. In this regard, engineered wood such as cross-laminated timber or glulam is increasingly used in building applications (Asdrubali et al., 2017). In order to take more advantage of its attractive properties, wood as a natural porous material could be used as a substrate to encapsulate organic bio-based phase change materials (OBPCMs) to improve the thermal mass of buildings. Various OBPCMs have been tested to store and release solar energy (Mathis et al., 2018, Mathis et al., 2019). However, in Nordic countries there is deficiency of sunny days in winter when the demands for energy is high and thus, using OBPCMs incorporated into wooden materials to control indoor temperature fluctuations can be presented as an alternative. Permeable wood and wood-based materials could be used to cost effectively shape stabilize the OBPCMs to enhance its thermal and chemical capabilities of using these materials in building envelopes (Yang et al., 2019).

Experimental

Fatty acids/esters including linoleic acid (LA), oleic acid (OA), vinyl α-eleostearate (VE), fatty acid glycidyl ester (FAGE) and their mixtures were tested as potential OBPCMs for energy saving. Some other types of fatty acids and their homologue esters will also be investigated. Screening tests using differential scanning calorimetry (DSC) were performed to select the appropriate fatty acid/ester or mixture for the energy saving purposes. The aim is to use the wood features (wood cells) as microcapsules to incorporate OBPCMs into wood to control the indoors temperature fluctuations. OBPCMs will be incorporated into wood and wood fibers using vacuum-pressure impregnation to enhance thermal mass of the building and hence controlling inside temperature fluctuations leading to reduction in the energy consumption. Chemical, morphological, thermal stability and behavior of the composite will be studied using FTIR spectrometry, microscopy, guarded hot plate and DSC.

Preliminary results

DSC screening results for OA, LA, VE, FAGE and their mixtures are presented in Figure 1. In the coming stage of the project, mixtures and esters of fatty acids will be prepared to make these materials suitable for building application. These materials will be then incorporated into wood fibres by impregnation to make composites for construction applications. Porous wood species like poplar and beech will be used through the study.
Figure 1. DSC curves of the melting and freezing process of LA, OA and VE (1a) and eutectic mixture curves of OA/FAGE, OA/VE (7/3), OA/LA (1/1) and OA/LA (7/3) (1b).

It can be seen that there is a considerable temperature difference between melting and freezing temperatures. For the targeted application in this project, the ideal case is that the difference between the melting and freezing process should be as small as possible. Another important result is that by mixing two fatty acids/ester, an increase in the melting temperature is observed (Fig. 1b). As compared with the pure fatty acids/esters, the melting curves of the mixtures shows a shift to higher temperatures and present two-phase change peaks on both melting and cooling process. It is expected that by mixing and modifying the chemistry of OBPCMs and their incorporation into wooden fibres, a promising composite with suitable working temperature will be achieved. Chemical functionalization and optimization of the eutectics expected to minimize the effect of non-congruent melting of the OBPCMs. Moreover, carbonized wood and carbon-based materials coming from wood sources are expected to enhance thermal conductivity and thermal performance of the composite considerably.

Conclusions

The possibility of using wood features to incorporate OBPCMs for energy saving is studied. The resulting bio based composite is expected to be used in construction to enhance thermal mass of buildings and controlling inside temperature fluctuations and thus use energy more efficiently. The preliminary results showed that fatty acids derivatives such as esters are very interesting to be used as OBPCMs. Therefore, various fatty acids derivatives will be synthesised and tested as potential phase change materials.

Acknowledgement

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References

Background

Although the world has come to the realization that exploitation of non-renewable materials are not sustainable, (Petrillo et al, 2018) there has been a limited interest in fully utilizing natural, renewable resources such as wood and its products because of service durability concerns. One of such issues is the impact of weathering on facade materials causing aesthetical degradation (Sandek et al, 2019). Thus, this research was carried out as part of BIO4ever international project to determine the weathering resistance of bio-based façade materials in Estonian climate. 120 bio-based façade materials obtained from 17 countries were investigated by subjecting to accelerated natural weathering conditions on a 45° inclined outdoor weathering stand located at (59°23'50.6"N 24°39'24.0"E). Results from this research are to be used to optimize a software simulating changes in appearance of facade materials in outdoor conditions.

Experimental

Specimens were supplied by 31 companies based in Europe, Central America and New Zealand. The weathering test was carried out in Estonia and evaluation was done in Tallinn, San Michele (Italy) as well as Oleron and Guadeloupe (France) were more attention was paid to the effect of weathering on material durability (EN 350-2). The specimens were grouped into 7 categories; Natural (Wood and Bamboo), chemically modified (Acetylation & Furfurylation), Composites (Panels, bio-ceramics, tricoya, WPC), Coated and surface treated (carbonized woods, nano-coated), Impregnated (DMDHEU, Knittex, Madurit, Fixapret), thermally modified (Vacuum, saturated steam & oil heat), Hybrid (combination of aforementioned).

Method

The evaluation was done in line with COST Action FP1303. Weathering parameters such as precipitation, UV index, Average air temperature, minimum and maximum air temperature and average relative humidity were considered. Colour measurements was performed according to CIELAB. (Jones & Brischke, 2017). Checks were measured on the specimen surface using Avongard Check Width Gauge. Checks on the sides or under were not considered and only checks more than 5 mm based on mean maximum width and total length were analysed. Photographic observation was also done using the Canon EOS 450D.
Results and Discussion

Figure 1 shows the colour change occurring in the specimens during and after the 2 years period. Most samples already turned grey after 6 months of weathering in Estonia, with no changes in colour after 12 months in Italy. This is partly because of the difference in weather conditions and the vertical stand used in Italy compared to 45° inclined stand employed in Estonia. From visual observation, only 20 materials had retained their colour in Estonia at the end of the test.

![Figure 1: Colour change in specimens](image)

**Figure 3. Samples during natural weathering in (a) Tallinn, Estonia and (b) San Michele, Italy**

The colour change measured for natural wood (spruce) and acetyl treated beech are shown in figure 2. Using the L*a*b colour measurement scale, Spruce had a decrease in dark-light (L*) over time. Relative stability was observed in green-red (a*) colour scale while the material turned more yellow in the first weathering period as shown by b* (yellow-blue). The yellow colouration is synonymous with the degradation of lignin. (Oberhofnervá *et al.*, 2017). Results for acetylated beech however, showed relative stability in colour after slight changes shown by L*, rise in yellow scale a* and bluish colouration b* after the first months.

![Figure 2: Colour change of natural spruce and acetylated beech](image)

**Figure 4. Colour change of (a) natural spruce and (b) acetylated beech in L*a*b* coordinate scale**

Depending on weather conditions, checks on the specimens was observed every 3 months. At the end of the 2 years weathering period, 63 of the tested materials had developed checks. 

Conclusions

Aesthetical stability performance was more improved for surface, chemical and hybrid treated façade specimens. Almost all the tested materials developed checks which was more pronounced in impregnated pine, non-treated oak and thermally modified specimens. To ensure adequate resistance to weathering of wood based materials in building façades during service, surface and chemical treatment is the best approach.
References


FIRE PERFORMANCE AND LEACH RESISTANCE OF PINE WOOD IMPREGNATED WITH GUANYL-UREA PHOSPHATE (GUP)/BORIC ACID (BA) AND MELAMINE-FORMALDEHYDE (MF) RESIN
Chia-Feng Lin, Olov Karlsson, George I. Mantanis and Dick Sandberg

Background
Fire retardant treated (FRT) timber can be produced by impregnation under high pressure with aqueous guanyl-urea phosphate (GUP) and BA (Gao et al., 2006). However, neither GUP nor BA is strongly attached to the wood polymers and normally leach out during weathering; thus, wood loses its fire retarding property (Mantanis, 2002). Wood modified with melamine-formaldehyde (MF) resin can provide an increased dimensional stability (Inoue et al., 1993), thermal stability (Deka et al., 2002) and to some extent improve fire performance (Xie et al., 2016). Blending MF resin and the conventional fire retardant (FR) for decreasing leaching from the FRT timber has been poorly researched, up to date. The aim of this work was to evaluate the leachability of the FR by treating pine wood with MF resin/GUP/BA, and followed by an analysis of the fire behaviour of the treated material. The cured MF resin is thus expected to form a hydrophobic polymeric network and incorporate the FR within the wood cell wall.

Experimental
Scots pine (Pinus Sylvestris L.) sapwood specimens measuring 10x10x150 mm (TxRxL) were impregnated (30 min vacuum followed by 15 bar pressure for 1 h) with GUP/BA solution. The impregnated specimens (10 replicates) were dried in an oven at 103°C for 2 days, followed by conditioning at 20°C/65% RH for one week. Leaching tests were performed on 5 replicates, according to EN 84, replacing water 10 times during the 14-day leaching period. Fire tests were carried out on 5 replicates, using the limited oxygen index (LOI) method according to ISO 4589. Preparation of the FR solution was done by dissolving GUP/BA (weight ratio 7:3) in deionised water, before mixing it with the MF resin solution. The resin was provided by NTL Chemical Consulting (MF polymer: 50%; melamine content: <15%). Different amounts of MF resin in the GUP/BA solution were applied. For description of the treatments see Fig. 1 a.

Results and Discussion
Limiting oxygen index (LOI) is the minimum concentration of oxygen (expressed as a percentage) that will support combustion of a polymer, e.g. wood. It is typically measured by
passing a mixture of oxygen and nitrogen over burning wood specimen until a critical level is reached which corresponds to LOI value (White, 1979). A higher LOI value indicates enhanced fire retarding performance. The flammability of FR treated wood was obtained as a single numerical value. To simulate the FR performance after weathering, LOI values before and after leaching in water, according to standard EN 84, was determined. Wood treated with 10 wt% of FR only had similar LOI after leaching as the untreated wood, since the FR itself was not fixed within the wood structure (Fig. 1). Lower weight percentage gain (WPG) loss and higher fire retardant performance of treated wood (after EN 84) was attained after introduction of 10 to 30 wt% of MF resin into the GUP/BA solution. The possibility of incorporating this FR is supported by the fact that the MF/GUP/BA treated wood specimens exhibited superior FR performance, even after water leaching (EN 84), as compared with results with the samples treated only with MF resin (0-30MF). In addition, the presence of the MF resin in the FR system showed no significant influence on the FR performance of the wood samples; even when 10 to 30 wt% of resin was added in the FR solutions (Fig. 1c). To investigate which chemicals were leached out from the wood impregnated with the MF/GUP/BA, leached water fraction was evaporated, and the solid residue was analysed using ATR-FTIR. The material from 4-days leaching fraction of 10-30MF was mainly composed of BA (Fig. 1d). Similar spectra were observed in other dried fractions. Consequently, it was concluded that GUP (but not BA) had been efficiently trapped in the cell wall, by the cured MF resin network.

\[\text{Figure 1. Test results: a) The naming of the treatments, b) Weight percentage gain (WPG), c) WPG loss, d) limiting oxygen index (LOI) before and after leaching (EN 84) and, e) FTIR spectrum of leached water of the 10-30MF treated wood (after 4 days), as compared to pure BA.}\]

**Conclusions**

The resistance to leaching of Scots pine wood treated with GUP/BA can be improved, while maintaining a high fire retarding performance, by the incorporation of MF resin. A reduction of the WPG loss, following water leaching according to EN 84, up to 90 wt% was achieved, in presence of 30 wt% MF in the solution, probably by enclosing the FR in the cured polymeric MF network. Overall, we suggest that such treatment could be a good methodology for producing exterior-use FRT pine wood.
References


THE SWEDISH NYCKELHARPA WITH THERMALLY MODIFIED SOUNDBOARD

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Background

The modern nyckelharpa is a bowed string instrument with 4 melody strings 12 sympathetic (resonance) strings. Wooden keys with tangents are used to stop the strings, producing different tones. The origin of the nyckelharpa is not known but the oldest evidence (about 1350), a stone relief of a very similar instrument can be found on the island of Gotland (Sweden). The tradition of playing and making the nyckelharpa has existed unchanged in Sweden for more than 400 years. The modern Swedish nyckelharpa was developed during the early 1900’s and is the most common type used today. The body of the modern nyckelharpa is normally made from spruce. Improved sound properties of thermally modified instruments (e.g. guitars and violins) has been shown in earlier studies. In this project some properties of a thermally modified soundboard for a nyckelharpa were tested for improved acoustic properties. Figure 1 shows two examples of nyckelharpas before and after surface treatment.

![Figure 1. Examples of two nyckelharpas; in the front one almost finished (year 2019), and in the back one finished, treated with wood stain and shellac (year 2018) (Photo and maker: Esbjörn Hogmark).](image)

Experimental

Two spruce soundboard material from Mittenwald, Germany were used in this study. One of the soundboards was thermally modified and the other, half modified and half unmodified, was used for testing various properties. The mild thermal treatment was performed at a max temperature of 135°C, 2 h in nitrogen at the Faculty of Wood Engineering, Eberswalde University for Sustainable Development in Eberswalde, Germany. The density, moisture sorption (3 months in desiccators), dimensional change (radial direction), damping and dynamic
modulus of elasticity ($E_{dyne}$) were measured for thermally modified and unmodified wood. The dimensional change was measured in the radial direction as the shrinkage between wet and dry state. A modal analysis was performed by exciting wood samples with an impact hammer and the vibrations were recorded with a sensor from where eigenfrequencies could be determined. From analysis of the eigenfrequencies, the $E_{dyne}$ could be obtained.

Results and Discussion

Table 1 shows the results of the density, dimensional change and acoustic properties. Soundboards with high quality require low density and high elastic stiffness. The results showed a small decrease in density and also an increase of the $E_{dyne}$ due to thermal modified. The dimensional change in the radial direction was about 15% lower for the thermally modified wood compared with the unmodified wood. The damping was slightly decreased for the thermally modified wood compared with the unmodified. The moisture sorption is shown in Figure 2 and was lower for the thermally modified than the unmodified wood, as expected. Since this was the first time testing a thermally modified soundboard for a nyckelharpa, the thermal treatment process used was very mild. Despite that, the bending of the soundboard in hot water was more difficult than expected, compared with an unmodified soundboard.

**Table 1. Results showing the density (22°C, 40% RH), dimensional change in radial direction, damping, and the dynamic modulus of elasticity in the longitudinal direction. The average of six samples (with standard deviation) is shown.**

<table>
<thead>
<tr>
<th>Material</th>
<th>Density kg/m³</th>
<th>Dim. change %</th>
<th>Damping $10^{-2}$</th>
<th>$E_{dyne}$ N/mm²</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermally modified</td>
<td>394 (19)</td>
<td>3.2 (0.1)</td>
<td>0.59 (0.24)</td>
<td>8850 (904)</td>
</tr>
<tr>
<td>Unmodified</td>
<td>403 (20)</td>
<td>3.7 (0.2)</td>
<td>0.65 (0.12)</td>
<td>8331 (834)</td>
</tr>
</tbody>
</table>

**Figure 2. Moisture sorption for thermally modified and unmodified wood samples. The average of six samples (with standard deviation) is shown.**

Conclusions

A mild thermal treatment of spruce soundboard for nyckelharpa showed some improved acoustical properties (slightly lower density, damping and higher dynamic modulus of elasticity) which can be beneficial for the sound of the instrument.
References


THE STAINING EFFECT OF IRON (II) SULFATE ON NINE DIFFERENT WOODEN SUBSTRATES

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Background

Wooden facades without coatings have become popular in Norway, especially for large buildings like multi-story houses (Hundhausen, 2013). To obtain a quick and even greying, a one-off treatment with iron (II) sulfate is often applied. Iron (II) sulfate, also called iron vitriol, has been used as colorant for centuries in many applications, e.g., to manufacture iron gall ink (e.g. Krekel, 1999, Klöckl, 2015) and not least to stain wood (e.g. Canevari et al., 2016, Edlund et al., 1997). The latter is commonly ascribed to a reaction with phenolic wood extractives, particularly hydrolyzable tannins (Sandermann and Lüthgens, 1953). The Fe$^{2+}$-ion reacts with gallic acid to ferrous gallate, which subsequently oxidizes to a dark ferric pyrogallate complex (Krekel, 1999). This does however not sufficiently explain vitriol’s pronounced staining effect on wood species containing only marginal amounts of phenolic extractives. Moreover, little is known about the influence of the wooden substrate and light conditions on the color development of vitriol-treated facades. Against this background, we investigated the influence of wood extractives, light and 9 different wooden substrates treated with vitriol on the staining effect.

Experimental

The set-up comprised three sample sets:

Set 1: Two of 4 veneers (40 x 30 x 1 mm$^3$/l x r x t) of Norway spruce (P. abies) were Soxhlet-extracted with a mixture of toluene:acetone:methanol (4:1:1) for 6 h. Subsequently, a vitriol solution (4% wt/wt) was applied by brush on the front side of 1 extracted and 1 non-extracted veneer. All 4 veneers were then vertically exposed to sunlight behind window glass (g-value = approx. 0.85, τe = approx. 0.75).

Set 2: Six specimens (40 x 30 x 1 mm$^3$/l x r x t) of spruce were surface-treated by brush with a vitriol solution (4% wt/wt). Three of the specimens were exposed to light behind window glass under the same conditions as the veneers of Set 1. The other 3 specimens were stored in a ventilated box to protect them from light next to the light-exposed specimens.

Set 3: A total of 72 specimens (40 x 30 x 1 mm$^3$/l x r x t) of 9 different wood materials (n=8) were manufactured. The front side of 4 of the 8 specimens per material were brushed with a vitriol solution (4% wt/wt). Three treated and 3 untreated specimens per substrate were exposed
outdoors at 45° to the south on Treteknisk’s roof in Oslo while the remaining two specimens were kept as references, i.e., they were not exposed.

The CIELab color was measured on all specimens based on scans taken with a color-calibrated scanner (ScanMaker 9800XL plus, Mikrotek, Hsinchu, Taiwan).

Results and Discussion

Both, the non-extracted and extracted veneers became darker over time (Figure 1 Left). Those treated with vitriol developed a grey-brownish color short time after exposure. The extraction did not influence the color; thus, wood extractives are not necessary for staining with vitriol. Sample set 2 showed only significant staining under light influence (results are not displayed in this abstract). We explain this by complex formations between Fe²⁺-ions and phenolic compounds resulting from photooxidization of the spruce lignin.

In the outdoor test, spruce, pine, larch and WRC showed a strong decrease in lighness during the first 2 months of exposure (Figure 1 Right A); this artificial greying turned gradually into a lighter natural greying over the next months. Oak darkened only slightly after exposure as it was the only substrate that had shown an immediate pronounced color change when vitriol was applied due to a dark-blue/black iron-tannin reaction. A staining effect of vitriol in terms of artificial greying on acetylated wood was not seen (Figure 1 Right B). One reason might be that the Fe-ions are unable to penetrate into the cell wall matrix of acetylated wood due to a reduced pore size by cell wall bulking. This theory is supported by a study from Hosseinpourpia and Mai (2016) who found a resistance of acetylated wood to Fenton’s reagent. Another possible reason might be the low pH-value of the acetylated wood as chelation requires a pH above the pKa of the phenolic group (Hagerman, 2002). This could also explain vitriol’s little effect on thermally modified pine and ash (Figure 1 Right B). In addition, both substrates had already a dark color when they were exposed outdoors, thus, any staining reaction with vitriol became not prominent. Vitriol’s poor effect on Cu-salt impregnated pine is explained by a phototabilizing effect of the lignin aromatic system with Cu-HDO.

Conclusions

Staining of wood with iron (II) sulfate is based on complex formations between Fe-ions and phenolic compounds of wood extractives and/or phenolic compounds resulting from photooxidization of lignin. The latter explains i) vitriol’s staining effect on wood species containing only marginal contents of phenolic extractives, and ii) color variations in artificial greying on vitriol-treated facades. In terms of artificial greying, iron (II) sulfate does not work on acetylated pine, thermally modified pine and ash, and Cu-HDO preserved pine.
References


SHRINKAGE, CUPPING AND CRACKING OF MULTI-LAYER PARQUET AT LOW RELATIVE HUMIDITY – EMPIRICAL DEVELOPMENT OF A CALCULATION BASIS

Authors: Karl-Christian Mahnert*

Background

Multi-layer parquet (MLP) is a laminated construction consisting of a top layer and additional layers of wood or wood-based panels. Excessive drying of MLP elements in relative humidity (RH) below the manufacturers' minimum requirement, usually 30 % RH, is a common phenomenon in Scandinavian homes because the RH can reach a minimum of 20 % over longer periods during heating season (Berge and Mathisen, 2016, Bøkenes, et al., 2011, Kjellberg, 2005). The most frequent distortions of MLP and reason for customer complaints resulting from this drying are temporary gaps between the elements, temporary cupping of the elements and permanent cracking of the top layer. The Norwegian Institute of Wood Technology is often assigned to trace back the extent of drying based on the current situation of an MLP floor or to assist in drafting of product requirements. These tasks require a calculation basis for gaps and cupping as well as a critical moisture content of MLP elements to avoid cracking of the top layer.

Since product development happens internally at the MLP manufacturers, the most relevant technical documentation on the behaviour of MLP is assumed to be confidential. Some studies have investigated the influence of the composition of MLP (Blanchet, 2008), of different top-layer properties (Blumer, et al., 2009), of top layer finish (Blanchet et al., 2003), different types of base materials (Barbuta, et al., 2012, Bouffard and Blanchet, 2009, Bouffard, Blanchet and Barbuta, 2010), and thickness and density of selected base materials (Blanchet et al., 2003, 2006, Bouffard and Blanchet, 2009) on the cupping behaviour of MLP-elements. Numerical modelling of the cupping behaviour was conducted by Blanchet et al. (2005), Blumer, et al. (2009) and Deteix et al. (2007). However, concrete figures such as differential shrinkage and cupping, could not be found in the literature. In this abstract the results from climate test of 31 different MLP with oak as top layer conducted at Treteknisk are analysed to suggest a calculation basis.

Experimental

To test one type of MLP, 10 samples of 450 mm length are prepared from MLP-elements straight from the original package. During the 1. phase of the test, the samples are exposed to 20°C and 65 % RH for 14 days. During the subsequent four week long 2. phase of the test, the
samples are exposed to 23 °C and 17 % RF to resemble dry winter climate. Mass, width of the top layer, cupping of the samples and potential cracking is recorded prior to the test and at the end of each phase. Additionally, the samples are visually inspected once a week during the 2. phase. The moisture content (MC) of the samples before testing, after the 1. phase and after the 2. phase is determined according to EN 13183-1 (2002) at the end of the test. Differential shrinkage in width is calculated based on sample width and MC before the test and after its 2. phase. Cupping is measured according to EN 13647 (2011) and expressed in percent of the sample width. Differential cupping is calculated as the quotient of the maximum cupping during the test and the change in MC from the 1. to the 2. phase.

Results and Discussion

The total shrinkage of the MLP-samples was found to be dependent on the MC at delivery (Figure 1), the correlation between the sample width and the total shrinkage, however, yielded an R² of 0.02 only. Cupping after the 2. phase of the test was best explained by the total shrinkage (Figure 2) and the MC at delivery (R² = of 0.31).

![Figure 1. Total shrinkage of the MLP-samples between the end of the 1. and 2. phase of the climate tests as a function of the moisture content at delivery.](image1)

![Figure 2. Regression between concave cupping of the MLP-samples at the end of the 2. phase of the tests and the moisture content at delivery.](image2)

The average differential shrinkage of the MLPs was calculated to 0.07 %, compared to an average differential shrinkage of oak of 0.22 % (Wagenführ, 2007). This means that the combination of the MLPs different compounds’ anisotropic behaviour reduced the shrinkage by about two thirds.

The average differential cupping was calculated to 0.08 %.

Despite the solid calculation bases for differential shrinkage and cupping of MLP with oak as top layer, a validation based on other datasets is desirable.

At a MC at delivery of below 6.8 % the risk for unacceptable cracking of the top layer is very little. It increases with increasing MC.

To maintain the total annual MLP consumption of about 13.6 million m² in Scandinavia, the performance of MLP under the challenging climatic conditions should be optimized, for example by adjusting the required MC for MLP intended for the Scandinavian market to the lower end of today’s requirement of 7 ± 2 % (EN 13489, 2017). Another approach could be to reduce the thickness of the top layer. However, EN 13489 (2017) requires a minimum thickness of 2.5 mm for MLP.
References

INFLUENCE OF KNOT AREA INDEXES ON TENSION STRENGTH OF SITKA SPRUCE

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Background

It is well known that knots can reduce the strength of timber, and the selection of “critical section” for testing to EN 408 and EN 384 is typically based on the size and position of knots. In Ireland, the visual strength grading Standard IS 127 specifies the permissible limits of MKAR (margin knot area ratio) and TKAR (total knot area ratio) knot indexes for Irish softwoods. The limits are the same than those in the UK visual grading standard BS 4978.

A previous study in Ireland using density and the largest TKAR measured over a length of 150 mm for prediction of tension strength found a correlation with $R^2 = 0.50$ (Gil-Moreno, 2019). Another study using TKAR as sole predictor found a correlation of $R^2 =0.39$ (Raftery and Harte, 2014). The study presented here also explores the use of the MKAR index given in IS 127 for tension strength prediction. The research is ongoing, but there is already enough data to make some preliminary recommendations that could help to obtain better predictions of timber quality. Grading for tension strength is particularly important for glued laminated timber production.

Experimental work

The knotiness of thirty-two pieces of 44 x 100 mm of Irish Sitka spruce was assessed over a length of 500 mm. This corresponds with the gauge length for the determination of tension strength parallel to the grain according to EN 408. This assessment was carried out centred on the region judged to be the critical section, and the tension strengths measured were adjusted to a reference depth of 150 mm according to EN 384. The relationship between tension strength and knot indexes was examined using linear regression. This paper is concerned with the largest total knot index (TKAR) and marginal knot index (MKAR) over a length of 150 mm, where overlapping knot areas just count once. These indexes were calculated using the online software Web Knot Calculator v2.2 (Microtec, Italy), which has been used at European level in the Gradewood Project (Ranta-Maunus, 2009).
Results and Discussion

Table 1 summarises the properties measured in the study.

Table 1. Summary table of timber measurements

<table>
<thead>
<tr>
<th>Tension strength N/mm²</th>
<th>TKAR</th>
<th>MKAR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Minimum</td>
<td>7.5</td>
<td>0</td>
</tr>
<tr>
<td>Average (St.dev)</td>
<td>21.2 (6.36)</td>
<td>0.39 (0.18)</td>
</tr>
<tr>
<td>Maximum</td>
<td>32.5</td>
<td>0.73</td>
</tr>
</tbody>
</table>

The MKAR indicates a higher ratio of knots in the margins than on the full section. The linear relationship of tension strength with knot indexes (Figure 1) investigated how this influenced the prediction.

![Figure 1. Relationship between knot indexes and tension strength parallel-to-the-grain.](image)

In this study, the MKAR index explained 40% of the variation in tension strength (RMSE = 5.0 N/mm²) using linear regression, a slightly stronger relationship than that using the TKAR that explained 25% (RMSE = 5.6 N/mm²).

Conclusions

The results of this study show that the use of the MKAR index could offer better predictions of tension strength parallel-to-the-grain than the TKAR index. In addition, to improve the prediction power, this knowledge can help to visually determine the critical section (weakest part) for testing of mechanical properties. This could result more important for bending tests, where the tension edge must be randomly selected.

References


EFFECT OF DIFFERENT WOOD SPECIES AND LAY-UP ON THE MECHANICAL PROPERTIES OF PLYWOOD

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Background

Plywood is valued for its uniform strength in all directions. This gives plywood an upper hand against solid wood in terms of cracking and warping. In Estonia, only birch is used for plywood production. On the other hand, birch logs prices are rather expensive compared to alternatives. This leaves us few species that are much cheaper than birch logs and at the same time promising to be comparable match by mechanical properties. Therefore, topic is very actual, to consider cheaper species in value adding manufacturing.

The aim of the research is to determine the effect of replacing some birch layers with alder wood and how mechanical properties are changed due that. As plywood structure is even, then changing plies direction is having impact on mechanical properties. Second aim of the research is to determine the effect of adding consecutive plies in same direction. For sustainable manufacturing, there is strong demand to remove formaldehyde from resins, as it is toxic and carcinogenic material. Therefore, in this research lignin substituted phenol formaldehyde adhesive is used for comparison to traditional phenol formaldehyde glue.

Experimental

Four different wood species were used to produce the plywood: Birch, Grey alder, Black alder and Aspen. Plywood’s were made either only birch or birch with one other species at the same layup. Six different layups were used: standard, combi, combi mirror, twin, direction face and direction core. In this research, lignin phenol formaldehyde adhesive (LPF) is used and compared to traditional phenol formaldehyde glue (PH). Firstly, logs were cut with chainsaw into 140 cm length blocks, what is maximum limit for veneer lathe, as cutting knife length is 1450 mm and then soaked in water bath for at least 24 hours at temperature 40°C. After that, the logs are peeled with Raute veneer lathe and the veneer is cut with clipper. Then, the veneers are dried in the Raute roller type veneer dryer at 180°C with 2.5 min and moisture 500-600 g/kg. Dried veneers are then glued with LPF and PF glues using the roller coating machine. After gluing, the layups are hot pressed at 130°C, 1.5 MPa for 9 minutes. For the analysis, the veneer tensile strength, plywood density, bonding quality and bending strength are measured.
Results and Discussion

In this research, the lignin phenol formaldehyde adhesive had extremely low results in bonding quality. This weak point was mentioned also in the previous research, that LPF has low bonding strength and insufficient water resistance (Ferdosian et al., 2017). These were considered as main reasons, why it will not replace PF in near future. PF on the other hand is highly rated, because of its great mechanical and adhesive strength together with high moisture resistance. All the LPF Direction core products had shear strengths under 1.0 N/mm² and as they all have wood fibre failure less than required, they failed to get to class 2 by bonding quality, which is meant for humid conditions. Direction face bending strength in cross-wise direction had clearly the lowest results, as two plies in cross-wise direction will not stand tension. Direction face on the other hand had higher results in parallel direction. Birch Direction face had the topmost compressive extension with 27.4 mm at maximum compressive load. Despite so high compressive extension in cross-wise direction, product placed in fifteenth position out of eighteen by bending strength in that direction.

![Figure 1. Shear strength of plywood’s with different glues and layups.](image1)

![Figure 2. Bending strength of plywood’s with different glues and layups.](image2)

Conclusions

Other than lay-up directions, all the plywood’s were relatively equal. Direction face proved to better in parallel direction than core, but with this product should be kept in mind to use it only in forces parallel to direction. By this research aspen and birch combined plywood’s are strongest match for all birch layered plywood’s.
References
Background

The design of moulding tools can affect the properties of laminated veneer products (LVPs) (Stevens and Turner 1948, 1970, Lind 1981, Srinivasan et al. 2008, Blomqvist 2015). In particular, the poor shape stability of LVPs, a common cause for their rejection and customer complaints, may stem from variated surface pressure in time for moulding. Therefore, we sought to determine whether surface pressure in time for moulding affected the shape stability of LVPs.

Experimental

Samples of 5 peeled veneers of beech (Fagus sylvatica L.), measuring 340 × 140 mm² and conditioned at 20 °C and 20% RH, were bonded together (Table 1) in a half-circle mould (Figure 1) with a urea formaldehyde adhesive (resin 1206 and hardener 7501, Casco Adhesives Inc., Sweden). All veneers had their loose sides oriented outwards. During moulding for 4 hours at 20 °C, various load levels were evaluated in Group A (i.e. 12 kN), Group B (i.e. 70 kN) and Group C (i.e. 70 kN, relieved to 12 kN after 5 minutes), each containing 4 samples. Samples were exposed to various levels of RH at 20 °C, and distances between points on the outer curve were measured after each round of exposure.

Table 1. Thickness, lengthwise (L) and transverse (T) orientation and placement of shims in the sample of veneers. *The L orientation has a longitudinal direction parallel to the direction of the arc.

<table>
<thead>
<tr>
<th>Orientation of veneer</th>
<th>Shim</th>
<th>Top</th>
<th>Transverse</th>
<th>Middle</th>
<th>Transverse</th>
<th>Bottom</th>
<th>Shim</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness (mm)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.9</td>
<td>0.5</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>0.5</td>
<td>0.25</td>
<td></td>
</tr>
</tbody>
</table>
Results and Discussion

The starting distance of 161.4 mm increased immediately after pressing due to spring-back. As expected, the distance decreased during dehydration but increased during humidification. A marked difference in distance emerged among the groups, although the standard deviation was large (Figure 2 and Table 2). The higher pressure applied to the samples in Groups B and C in time for moulding resulted in their being compressed more than the samples in Group A, whose curved shape could mean reduced shape stability due to permanent collapse of cells.

![Figure 2. Moulded samples under different configurations of vertical load over 105 days.](image)

Table 2. Standard deviation of the measured distance (mm) over 105 days

<table>
<thead>
<tr>
<th>Group</th>
<th>0 days</th>
<th>0–58 days</th>
<th>59–71 days</th>
<th>72–105 days</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>1.6</td>
<td>4.2</td>
<td>3.9</td>
<td>6.5</td>
</tr>
<tr>
<td>B</td>
<td>1.4</td>
<td>2.3</td>
<td>1.8</td>
<td>3.7</td>
</tr>
<tr>
<td>C</td>
<td>1.9</td>
<td>0.9</td>
<td>2.9</td>
<td>0.6</td>
</tr>
</tbody>
</table>

Conclusions

Although the impact of different amounts of surface pressure seems to be significant, our study was limited, and the standard deviation was large. The best dimensional stability coincided with the lowest pressing pressure. To clarify their relationship, a larger study involving the real-time measurement of surface pressure in time for moulding and bond-lines could provide insights into other ways of applying pressure.

Acknowledgement

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References
MITIGATING CLIMATE CHANGE. CREATING VALUE. UTILISING RESOURCES EFFICIENTLY

THE CHARTER FOR WOOD 2.0 FROM THE FEDERAL MINISTRY OF FOOD AND AGRICULTURE IN GERMANY

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Background of presentation

Within the objectives of mitigating climate change, creating value and utilising resources efficiently, the Charter for Wood 2.0 focuses on quality growth to support central international, European and national political objectives. The objective of the former Charter for Wood launched in 2004 was to increase the average wood consumption in Germany by 200% per inhabitant within 10 years. This goal was reached. The focus now is on ensuring that there is a continuous supply of raw wood and on easing the use of wood and improve its resource efficiency in order to mitigate climate change and create value at the same time. The Charter is a milestone in the German Climate Action Plan 2050.

As a member of one of the working groups in the Charter process I will introduce you to the Charter for Wood 2.0, its principles and guidelines, activities and outcomes up to now.

References