WG2 TRAINING MATERIAL ON LESSER-USED WOOD SPECIES

1. Understanding of the Lesser used wood species

Lesser used Wood Species in Europe and their possible usage by P. Rademacher (MENDELU).

Keywords: climate change; change in forest site conditions and tree composition; bio- and material-diversity; lesser used wood species (luWS); wood property improvement

Introduction

For several decades, regionally also centuries, European forests are influenced or changed by both, the sylvicultural management activities of foresters as well as environmental, chemical and climate change processes of the individual or industrial processes.

These influences resulted in a change in forest site conditions and structures as well as tree species composition with a strong impact on the grown wood assortments and produced materials. Due to intensive forest management activities, the productivity of European forests is high and of economic importance, with a similar quantity of wood products in Asia or North America, and higher than in South America or Africa.

In Europe, this wood production is focused on a few tree species only, often concentrated on spruce, pine, beech and oak. In many European countries, the sum of only these 4 species covers 70-85 % of the national wood production. This resulted in high productivity and optimization in the forest and wood industry, but the bio- and material diversity in both sectors were dramatically decreasing during the last centuries. For many wood applications, these main species are not or not sufficiently suitable for higher value or exterior use, resulting in an increasing amount of imported assortments from outside Europe or treatment and improvement of lesser suitable species with oils, biocides or wood modification processes. Not all of them are suitable for high-value materials or environmentally friendly, sustainable processes, leading to increasing criticism and exclusion of wood products, including also the exchange of bio-based materials and products by competitive, non-renewable materials like plastic, aluminium, concrete etc. with a much lower sustainability index compared to bio-based materials.

Nevertheless, a lot of additional tree species exist in European forests, often better adapted to changing climate conditions, like drought, higher temperature, or storms. A lot of industrially non-wished tree species had been depressed or eliminated by forest management and sylviculture, leading to low stable and less resilient forest structures. Especially man-made monoculture forests are strongly suffering under the influences of these changing climate parameters, leading to decreasing productivity and increased exposure to diseases, calamities or forest fires and endangering the sustainability of high-productive, stable forest systems in many regions. A lot of lesser-used wood species (luWS) show an environmental optimum, which is, partly already now or in future, much better adapted to the expected climatic and site condition scenarios, possibly appearing subsequently under the climate change processes. Kölling (2014) and Thurm et al. (2018) showed for special model regions in Germany the potential correspondence of the ecological spread of selected tree species (Fig. 1, left and middle: yellow area) and their possible overlap with the actual site conditions (red- and blue-rimmed areas) as well as their running or future change under climate change conditions (expected scenario +2.3°C, decreasing precipitation during the growing season [red arrow]). The additional and supported enclosure of these up to now lesser-used wood species, better adapted to the future climate, for example, alder, hornbeam, ash, poplar, mountain ash or other Sorbus species, or also

This project has received funding from the European Union’s Horizon 2020 research and innovation programme under grant agreement N°952314.

species with an origin outside the local distribution of our native forests, like Robinia, Eur. chestnut, walnut, Platanus, or further cherry or oak species, can help to stabilize the forest environment and the bio-based sector, enabling an ongoing CO_2-fixation, leading to a decrease in global warming.

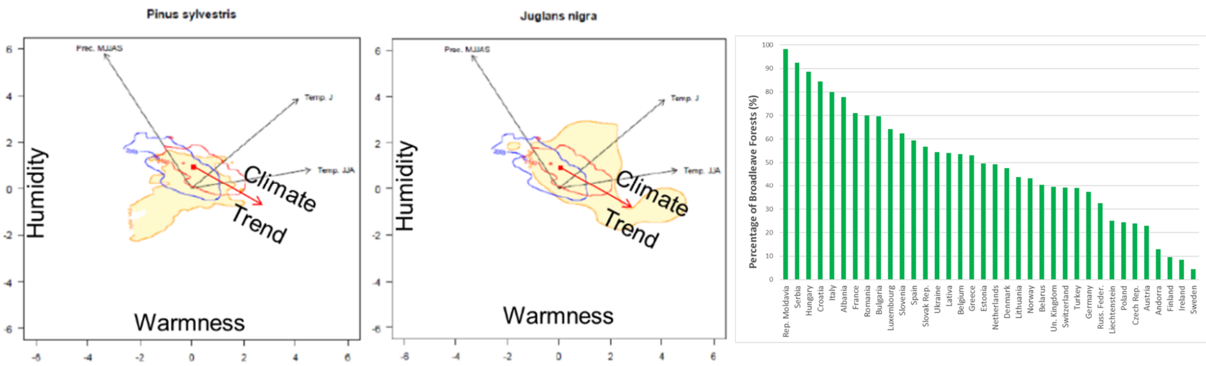

Fig. 1: Left (Pine) and mid (Walnut): Climate-Similarity of actual (blue and red circles) and future climate (+2.3°C, red arrow) and compare of their ecological optimum (yellow area) for a Test-Area in Germany for local and non-local tree species. Kölling 2014 (modified). Right: Percentage of Broadleaved trees in European Forests (%). Data Source: ICP Forests (2012). Forest Condition in Europe. 2012 Technical Report of ICP Forests.

In this paper examples of additional broadleaf- and lesser-used tree and wood species, their existing and potential ecological site optimum on regional, national and European scales (Fig. 1, right, Fig. 2) and their possible prosperity and availability are investigated, also including the widened property range and new applications on the bio-based market.

Methodology

Site conditions, ecological optimum, and climate change scenarios: The presented examples and results are based on the investigations of Kölling (2014) and Thurm et al. (2018).

Availability of wood and share of wood species in European Pilot-Studies: The presented data (Fig. 1-2) is published in several European and national Forest-Reports, commissioned or prepared e.g. by the European Commission (2018) or national institutions, dealing for example with State Forests in Poland (2018), BWI (2012) Dritte Bundeswaldinventur, Pro-Holz Webpages for Hungary or Croatia (2019) or Czech (Ministry of the Czech Republic [2018a+b]) and Slovakian forests (Ministry of Agriculture of the Slovak Republic [1997]) or authors compiling data about Romanian (Ionascu 1999), Austrian (Teischinger 2019) or Slovenian forests (Poljanec 2019).

Properties of selected native wood species: Basic data for general wood properties delivered by Wagenführ (2006) in his compendium. Own investigations about chemical, physical and mechanical wood properties were executed following the EN standards. Details are published in Pařil et al. (2014), Rademacher et al. (2017) and Rademacher (2019).

Findings

Availability of Wood in Europe:

The productivity of European forests is traditionally high compared with other worldwide important centers of wood production. Each with about 19-20% the USA and EU28 is leading the ranking list of a total 1.8 Bio m^3 of actual (5-year average global production 2010-14) yearly produced industrial round wood (energy wood not included), followed by total South America, Russia, China and Canada with about 13% down to 8% of the worldwide production (European Commission 2018).

Share of Wood Species in European Pilot-Studies:

The share of tree species and the added up percentage of coniferous and broadleaf species in Europe are, partly due to their site conditions, but highly due to man-made forestry management interventions, extremely different. Following an overview of the ICP-Forest Report (2012), the

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broadleaf forest share is ranging from 98% in Moldavia down to less than 5% in Sweden, with a focus on South-East European countries with >>50% broadleaf species and Scandinavian and Alpine countries with <<50% broadleaf species (Fig. 1, right).
In eight selected countries, focussing on E-, Central- and SE- Europe, the forest share of single tree species or species groups were chosen to show the range in different forest species compositions (Fig. 2).

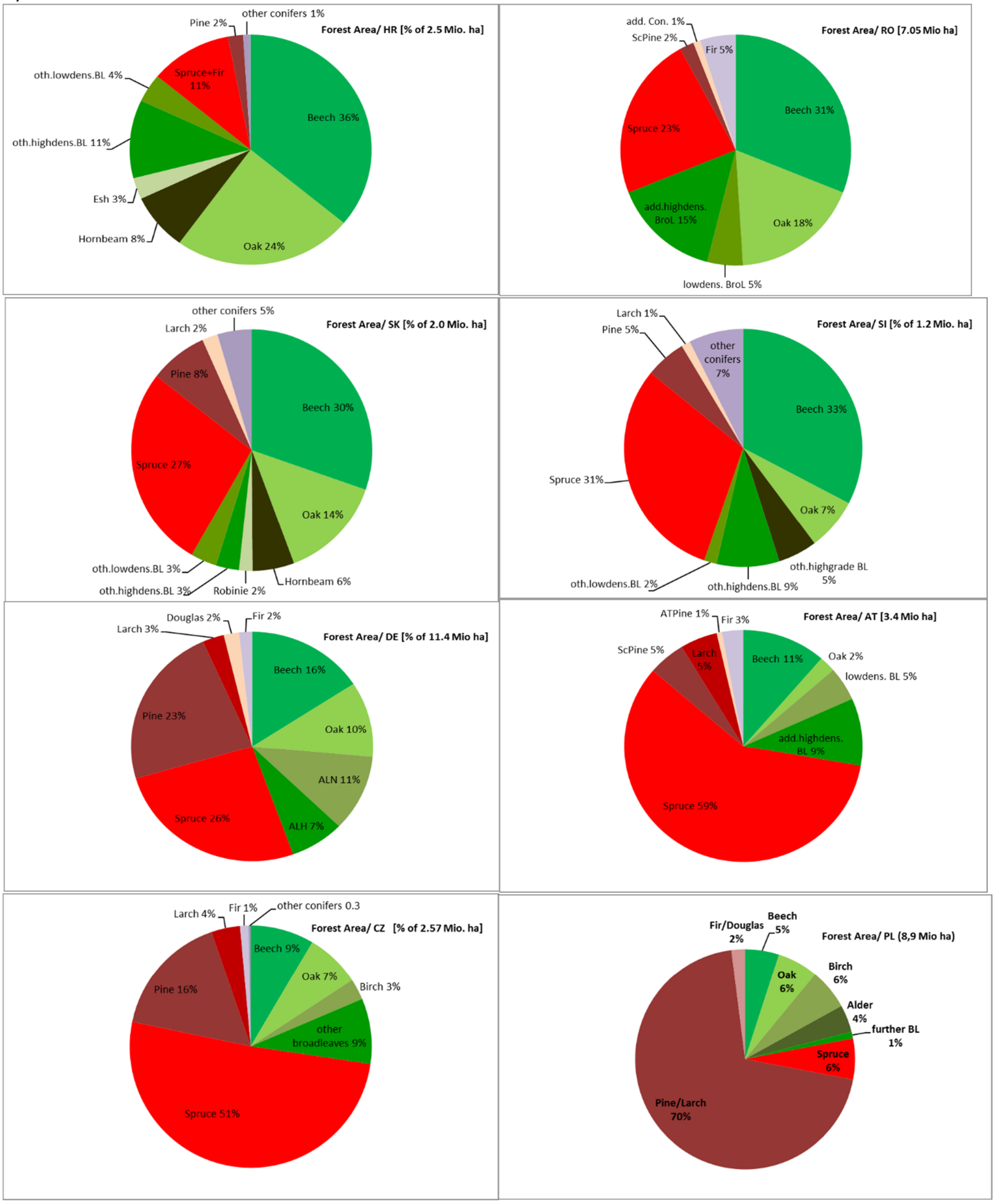

Fig. 2: Percentage of single broadleaved (BL) and coniferous trees (Con) in Polish (PL), Czech (CZ), Austrian (AT), German (DE), Slovenian (SI), Slovakian (SK), Romanian (RO) and Croatian (HR) forests (%). Data sources in the text.

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Selected countries with a higher share of coniferous trees (>70% Con.) were: Poland, dominated by Pine (State Forests in Poland 2018) as well as the Czech Republic (Ministry of the Czech Republic 2018a) and Austria (Teischinger 2019), both dominated by >50% spruces with partly low oak + beech share (sum ca. 10-15%) and a very low share of non-oak or -beech species (Fig. 2). In case of Slovenia (Poljanec 2019) and Germany (BMEL 2014) the share of both groups was quite balanced (DE [2012] 55% Con. and 45% Bl [broadleaved], SI [2018] 45% Con. and 55% Bl). In Slovakia [1997] with 42% Con. and 58% Bl the Bl-share was already higher (Min. of Agriculture of the Slovak Republic 1997). In these last mentioned three Central-European countries the share of non-oak and non-beech broadleaved species reached already 13-18%. For Slovakian forests information about a higher share of individual luWS are available, showing already much higher biodiversity in the SK forests: Besides the omnipresent dominating spruce, pine, fir, beech and oak with a sum of approx. 83% 7 lesser used broadleaved species (hornbeam, maple, ash, Robinia, birch, alder, poplar) showed a single share between 1-5%, several species (elm, tilia, willow, others) between 0.1-0.3% (Fig. 3, right).
In the case of Romania (69% [1998]; Ionascu 1999) and Croatia (86%; Pro-Holz 2019), the broadleaved species were dominating the forests, and the share of non-oak or -beech reaches 20-26%. Especially hornbeam or Robinia showed appreciable amounts of each up to 6-8% in these forests. Locally, outside these selected countries, the share of single lesser-used wood species was even higher (e.g. Hungary: 22% robinia, 10% poplar, 10% additional Bl, flanked by 32% oak and 12% beech [Pro-Holz 2019]).

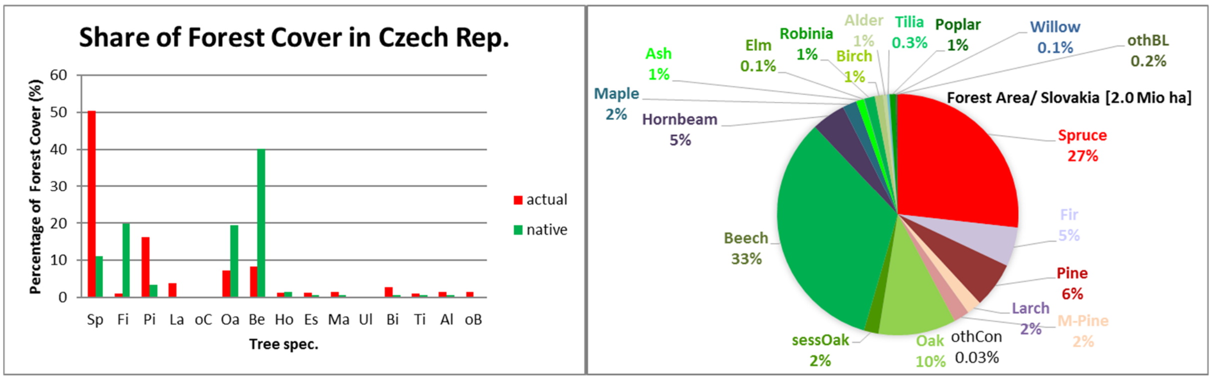

Fig. 3: Percentage of single tree species in the Czech Republic (left) for the actual and potential situation in forests (%; Forest Inventory 2017, Czech Republic [left]) and of single tree species in Slovakia (right; Ministry of Agriculture of the Slovak Republic, Forestry Section, 1997 [right]).

In the case of the Czech Republic the national Inventory (Ministry of the Czech Republic 2018b) delivers a comparison between the actual share of single tree species and the former potential native share, showing in the case of spruce, pine and larch an extreme 5 to 7-fold surplus in the actual forests, whereas, in case of fir, oak and beech the actual forest composition is 3 to 15-fold lower than in the potential forest community (Fig. 3 left). For further, also lesser used wood species, the share between actual and native situation is approximately balanced, sometimes a little bit lower, sometimes higher.
For Germany, the third national forest inventory (BWI 2012) separated about 50 different tree species in the German forests (Fig. 4 left). Here only spruce and pine already showed a share of more than 50% of the forest area, including beech and oak this share was already >80%. Additional 6 tree species, already much lower in the share (birch, black alder, Douglas and white fir, Eur. larch and ash) already exceeded the 90% area line. Additional 20 species, reaching from sycamore to elm, already covered 99.5%, and additional further 20 species, reaching from Eur. chestnut to sorb trees are sharing the remaining 0.5% of the forest area in Germany.
From the viewpoint of their potential existence these additional, lesser-used tree species could enrich the biodiversity in forests and stabilize impacted forests due to the wide range of their environmental optimum or amplitude, in many cases better adapted to warmer and growing season drier climate

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periods. Also, their contribution to additional material properties, urgently required for new innovative applications and products, could help to improve the market position of locally produced, renewable bio-based goods. However, due to decades or centuries of depressing luWS and preferring faster growing, easily processable species, often from the coniferous sector, the actual potential of luWS in many European regions is low and needs rethinking and technical diversion of all stakeholders, foresters, wood industry, landscape architects and end consumers.

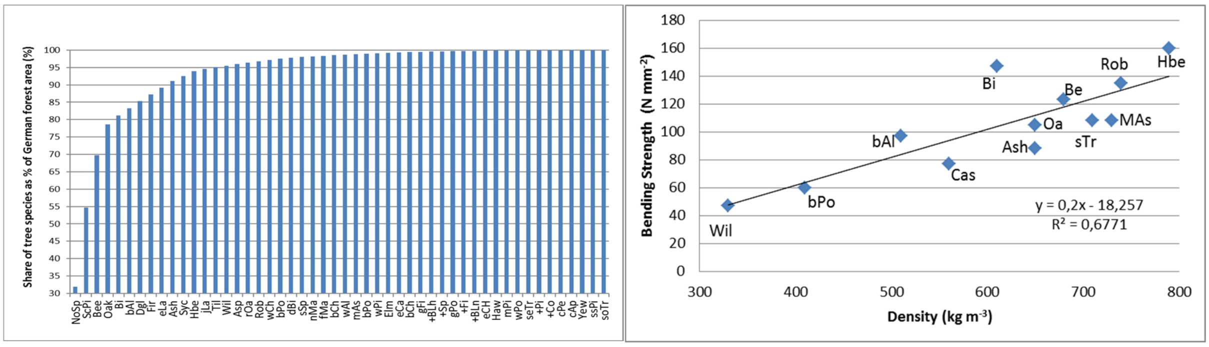

Fig. 4: Left: Percentage of single tree species in German forests ([%], Source: BWI 2012, Germany. Data compilation T. Mette, LWF Freising). Right: Bending strength <-> Density relationship of 12 Central European broadleaved species (Willow [Wil], black Poplar [bPo], black Alder [bAl], Castanea [Cas], Birch [Bi], Ash, Oak [Oa], Beech [Be], sorb Tree [sTr], Robinia [Rob], Mountain Ash [MAs], and Hornbeam. Data source: Wagenführ 2006.

Properties of selected native broadleaf Wood Species:

As already mentioned, broadleaved or hardwood species can play an important role in future forest biodiversity and applied wood material diversity. Whereas broadleaf species in general cover a wide range of the potential wood species properties (for example density, mechanical strength, dimensional stability, thermal or acoustic insulation, and durability), the dominating broadleaved species oak and beech are located in the median-high to higher density range, connected with higher strength, but also lower dimensional stability or thermal insulation potential (Fig. 4 right; all values from Wagenführ 2006). Oak and beech show a density average of about 650-680 kg m^{-3} and a strength of approx. 90-120 N mm^{-2} in case of bending strength and 60 N mm^{-2} in case of compression strength. Their related relatively low dimensional stability is shown for example in a volume swelling percentage of 14% in oak and even 18% in beech (Wagenführ 2006).

To widen this narrow field of wood properties of oak and beech, a selected set of additional 10, mainly central European tree species with up to now only minor (higher value) utilization was chosen to examine their potential applications. On the lower density range, willow or poplar are delivering density values of about 330-420 kg m^{-3}, related with a bending strength of only 40-60 N mm^{-2} and a better volume swelling of about 10-12%. The median-to-median-high range is covering the 500-650 kg m^{-3} density range, with black alder and castanea in the median and birch, ash and oak in the median-high range, connected with a bending strength of already about 80-120 N mm^{-2} and a volume swelling of approx. 13-14%. In the highest density range the selected species beech, sorb tree, mountain ash, robinia and hornbeam cover a density range of about 680 to more than 800 kg m^{-3}, related with a bending strength of 110-160 N mm^{-2} and a volume swelling of up to 19%.

Conclusions

The results demonstrated that the increase in forestry cultivation and the wood-industry application of lesser-used wood species (luWS) can improve the bio- and material diversity to a great extent. The frame of climate change influences on forests and the produced wood locally dramatically damage the forest health and the wood growth occurred, visible in bark beetle calamities, reduced growth

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conditions, or forest fires after drought or forest dieback. Additional luWS can help to reduce or replace the dominant species and diminish several observed negative influences.

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Lesser known wood species - material for research and final usage by M. Merela, K. Čufar (UL)

Keywords: less known wood species; properties; wood anatomy; wood utilization; wood textures; innovative products

Introduction

Alien (non-native) species are species that have been intentionally or unintentionally introduced by humans outside their original range. Invasive alien species are those that also have negative impacts on nature, the economy, or human health. More than 12.000 alien species have already been detected in Europe, of which about 15% are invasive and pose a serious threat to native species in Europe. According to EU reports, invasive species cause more than 12 billion euros of financial damage in Europe each year (damage to infrastructure and losses in agriculture, forestry, fisheries and tourism, as well as impacts on human health).

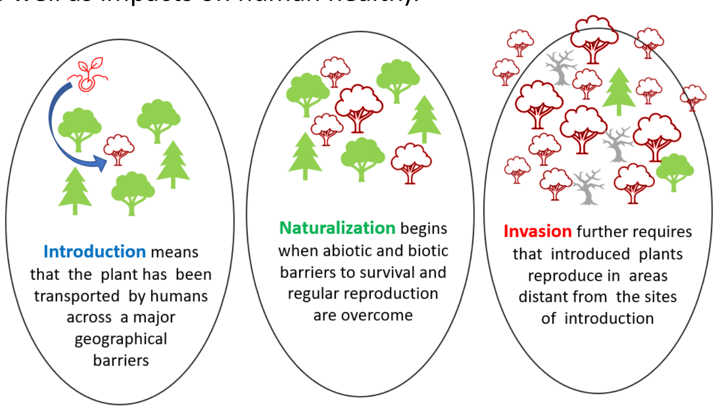

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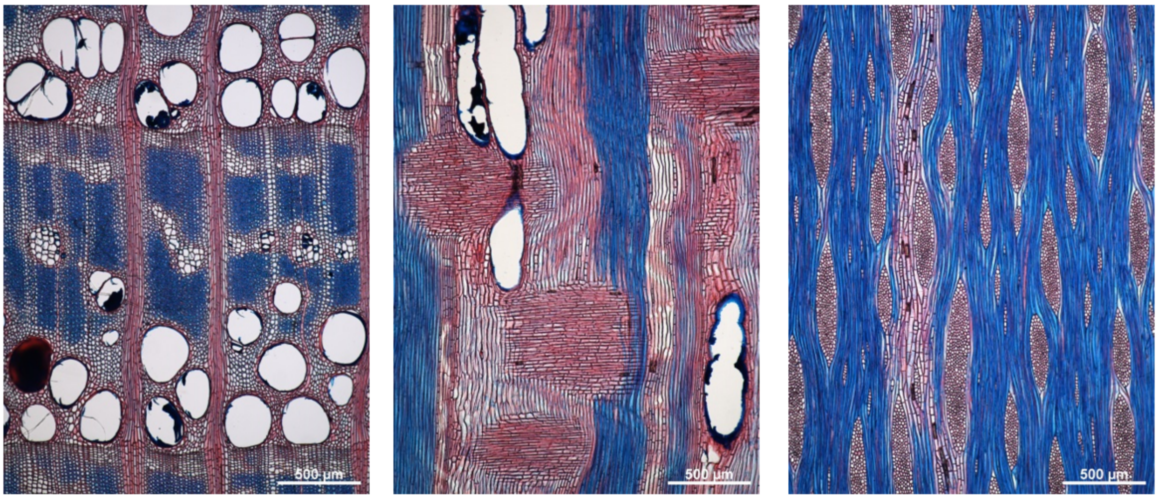
Fig. 1. From non-native to invasive plants.

The main goal of the project APPLAUSE - Alien plant species from harmful to useful with citizens' led activities was to teach citizens how to identify, remove, and innovatively use invasive plant material for social benefit (paper, wood products, food, dyes, pesticides, pharmaceuticals, etc.). The project included a total of 25 plant species (17 woody and 8 herbaceous).
The main tasks of the research group of the Department of Wood Science and Technology at the Biotechnical Faculty of the University of Ljubljana, Slovenia, were the analysis of basic wood properties, such as anatomy (Figure 2) and chemical composition, relevant properties for wood use, the development and design of new innovative products including solid wood and bio-wood composites, and finally the production of prototypes and their testing.



a) b) c)

Fig. 2. Tree of heaven (*Ailanthus altissima*), light microscopy: a) cross, b) radial and c) tangential section.

Methodology

Our work started with the selection of suitable trees in the urban environment from the tree species classified as invasive. After felling the trees, approximately 60 m^3 of logs were cut into planks and boards at a sawmill (Figure 3). All material was sorted, seasoned at outdoor conditions, and finally dried in kilns. Samples of each species of wood were prepared for different types of analyses. The drying properties, anatomy, chemical, physical and mechanical properties, machinability, bonding, and durability of the wood were investigated.

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Figure 3. Manipulation of the log and primary processing in the sawmill.
After the experimental work and the evaluation of the results, we proposed potential optimal wood use for each of the wood species. Finally, we designed and manufactured the final wood products.

Findings

Numerous results of the investigations of wood properties have already been published (e.g., Gorišek et al., 2018; Medved, et al., 2019; Plavčak et al., 2019; Merela et al., 2020; Merhar et al., 2020; Horvat et al., 2020; Vek et al., 2020; Plavčak et al., 2021; Zekič et al., 2021). We were particularly surprised by the uniquely beautiful wood texture of some of the species studied. Since we were dealing mainly with urban trees, many of them were injured, their branches were pruned several times and some trees were also topped. All this damage resulted in a response of the tree to the injury, sometimes accompanied by a fungal infection. All of these secondary changes in xylem growth and other changes in wood tissue result in unique textures that offer great potential for highly decorative wood products (Figure 4).

Fig. 4. A few examples of designed products made of wood of different less known and invasive species.
Since the project was designed for zero waste, we also found some solutions to use remains/residuals from wood processing. One very promising way to use scrap residuals was to make 3D-moulded WPC (wood-plastic composite) (Figure 5).

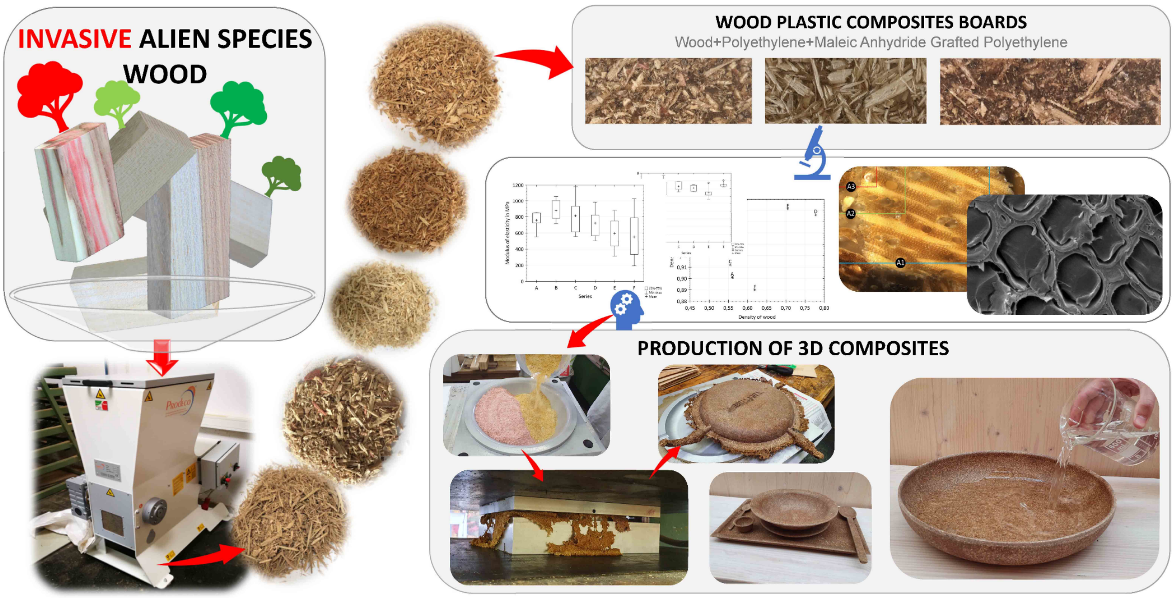

Fig. 4. WPC (wood-plastic composite) production from remains of invasive wood species processing.

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When processing wood residues from invasive alien plant species, it is the reuse of the harmful invasive alien plants that adds particular value to these products. The use of raw materials derived from lesser-known invasive species contributes directly to the protection of the environment and the conservation of biodiversity.

Funding: The research was supported by the project APPLAUSE - Alien plant species from harmful to useful with citizens' led activities (UIA02-228), co-funded by the European Regional Development Fund), and additionally supported by the Program P4-0015, Wood and lignocellulosic composites funded by the Slovenian Research Agency (ARRS) and Asforclic project (N°952314).

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Not used wood species. Analyses of museum objects, historical literature and modern testing by M. Grabner (BOKU)

Keywords: lesser-used wood species; historical wood utilization; wood quality

Introduction

In Austria, the choice of commercially available native wood species includes approximately 24 species (Fellner et al. 2006) – considerably less than what is indigenous in the forests. It is common knowledge that, based on different material properties, different wood species for example oak (*Quercus* spp.) and poplar (*Populus* spp.) cannot be used in the same field of application.

Not even 100 years ago, most of the daily used items were made of wood. Iron was a sign of wealth and was not affordable to everyone (Blau 1917). Wood had to meet all requirements, in particular those of people living in the countryside. Different parts of a wooden object had to deal with different loads (for example a ceiling beam needs high bending strength). This led to high complexity concerning the choice of the wood species.

At the beginning of the 20th century, the folklorist Josef Blau investigated a bohemian household and counted 27 wood species. He emphasized that all species were chosen according to their specific wood properties (Blau 1917). This indicates that knowledge about the proper utilization of wood and the selection of wood species was sorted out at some point in history - knowledge that might be usable today.

Many valuable wood species are not in use anymore, which led to a decline in the possibilities of wood utilization. The present study aims to outline the huge potential of the wide range of wood properties covered by today's rarely or not-used wood species, to meet the demands of modern wood utilization.

Methodology

The historic inventory of five Austrian museums has been analysed. Sampling implied the identification of the wood species used. Determination was done by analysing anatomical features (Gregus 1959,

Wagenführ and Scheiber 1985, Schweingruber 1990). Some species were detectable by just using a magnifying glass to enlarge the wood structure. For others, a transmitted light microscope had to be used. Most diffused porous species and today rarely used species had to be sampled by sectioning by hand.
The different loads, technological as well as special demands of every single piece, were discussed with handicraft men and museum staff knowing the objects in use. Based on these analyses up to six wood properties (like strength, and natural durability) were assigned by the authors.
Sampling in the museum was expanded by a search for historical literature. On the one hand, books dealing with the descriptions of different wood species were analysed focusing on today's rarely used species. On the other hand, folkloristic literature was searched to extract which wood species had been mentioned there. The year of publication of the 122 analysed books varied between 1690 and 1985.
Extensive sampling of shrubs, small trees and commercial wood species ended in several thousands of test specimens. Besides characterization, according to standards (like wood density, and bending strength), new testing procedures were developed (for example abrasion resistance, and dynamic friction).

Findings

All data presented here are published in Grabner (2017). In this investigation, 4335 objects and thereof 8985 object parts have been sampled. In total 48 different wood species could be identified. Figure 1 shows a bar chart with all species put in order, starting with the most frequently identified ones, which was spruce (*Picea abies*) followed by beech (*Fagus sylvatica*), ash (*Fraxinus excelsior*) and birch (*Betula pendula*). Within these 48 wooden species, 17 species can be classified as a shrub. The most frequently used shrubs were hazelnut (*Corylus avellana*), cornelian cherry (*Cornus mas*) and barberry (*Berberis vulgaris*). Furthermore, there are ten different fruit-bearing trees included, such as pear (*Pyrus communis*) or apple (*Malus domestica*), cherry (*Prunus avium*), plum (*Prunus domestica*), rocky cherry (*Prunus mahaleb*) walnut (*Juglans regia*) and four different species of genus *Sorbus* spp. Apple and pear were anatomically not distinguished and grouped.

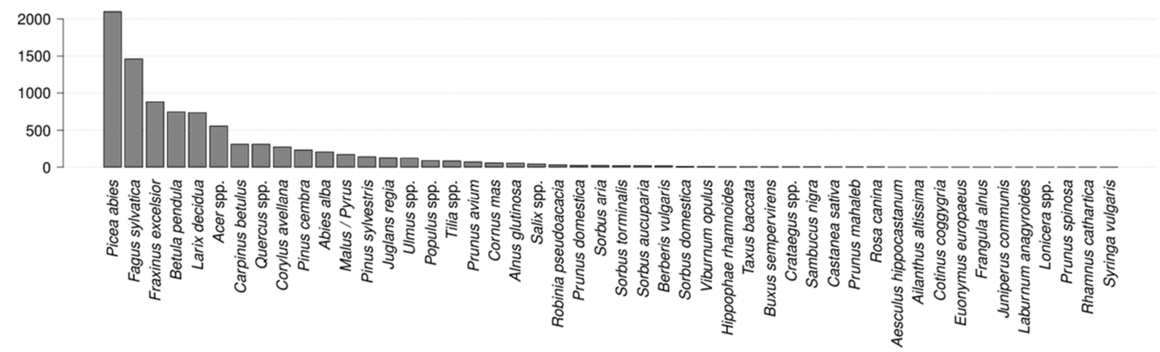

Fig. 1: The bar chart shows the frequency (total number of objects) of the 48 in museums identified wood species.

Due to the literature review, the number of species was increased to 60. For parameters that are easy to determine, such as wood density, it could be seen that the historical values lie within the range of variation of modern values. More complicated test setups (such as for strength values) are often no longer comprehensible. Therefore, these values are partly not further usable.

Modern testing showed, that several today not used species have very special properties – for example, an air-dry wood density of almost 1000 kg/m³ (lilac (*Syringa vulgaris*) and cornelian cherry), or tensile strength of more than 200 MPa (black locust (*Robinia pseudoaccacia*) and wayfaring tree

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement N°952314.

(*Viburnum lantana*). The impact bending test showed the highest values for the Cornelian cherry and the wayfaring tree – two shrubs. Comparable results showed the Brinell hardiness – the highest values for lilac (*Syringa vulgaris*) and boxwood (*Buxus sempervirens*) – once again two shrubs. Shrinkage and swelling were high for the wayfar tree and snowy mespilus (*Amelanchier ovalis*). The natural durability estimated by soil contact showed good results for some shrubs – within the group of well-known highly durable wood species like Douglas fir (*Pseudotsuga menziesii*) or black locust. These are buckthorn (*Rhamnus cathartica*) and the common spindle tree (*Euonymus europaeus*).
Giving an example of a possible substitute of ash wood (*Fraxinus excelsior*), which will get partially lost due to the ash-die-back, for tool handles: The strength, stiffness and impact strength should be comparable, which is for example the case for Cornelian cherry. The size of these big shrubs would be big enough to produce tool handles. Interestingly, often Alder (*Alnus glutinosa*) was mentioned by handicrafts to be used for tool handles. Of course, the strength is much lower. But it was described as "it lies well in the hand" – meaning, a lower risk of blistering.
The goal was to present comparable data for 60 Central European species, which were in use in former times and could be – at least partially – used in future as a kind of niche-raw material.

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2. Wood anatomy

Fibre characteristics and species determination for CITES and EUTR inspection by A. Olbrich, S. Helmling and J. Sieburg-Rockel (THUENEN)

Keywords: Wood species identification, fibre material, EUTR, CITES, machine learning

<u>Introduction</u>

To strengthen the trade with legal raw materials, it needs to be possible to determine the used wood species in all wood products on market. Also, fibre materials like pulp, paper and fibre boards have to be tested. Therefore, references have to be characterized and published (Schmitz et al., 2020).

Methodology

By macerating the fibre material and staining the cells wood anatomists can compare the cells under a light microscope with well-described references (e.g. "fiber atlas" Ilvessalo-Pfäffli, 1995). Since in fibre materials mostly a mixture of different timbers is included and the 3D structure of the tissue is destroyed we mainly use it for identification of hardwoods the cell type with the most remaining characteristics – the vessel elements. Individual cells are routinely examined by experts at the great expense of time. To automate this work light micrographs of reference material is needed as a database for training image recognition systems by machine learning.

<u>Findings</u>

References for the most important Asian wood genera were prepared, documented and published as an "atlas of vessel elements – identification of Asian timbers" (Helmling et al., 2018). The latest research project is being carried out in cooperation with Fraunhofer Institute ITWM. Therefore, light microscopic images of reference material are taken continuously and annotated. This growing database is the basis of the ongoing training of the automated image recognition system.

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Anatomical traits affecting moisture performance in *Q. pubescens* by Angela Balzano[1]*, Davor Kržišnik[1], Samo Grbec[1], Jožica Gričar[2], Boštjan Lesar[1], Viljem Vek[1], Miha Humar[1] (UL)
Keywords: pubescent oak, wood anatomy; durability; moisture performance

Introduction

Models (Hanewinkel et al. 2013) predicted that in the future mean 34% of European forests will be suitable only for a Mediterranean oak forest type. Pubescent oak (Quercus pubescens Willd.) is particularly important since has developed various mechanisms to survive in a drought-prone environment (Vodnik et al. 2019). Since the amount of pubescent oak is likely to increase in the future (Hanewinkel et al. 2013), it is of great commercial interest to determine the relevant properties of this wood species. Moisture performance predominantly depends on wood anatomical features, such as tyloses, presence of resins and pit aspiration. In some cases, pores can become filled with gums, resins, or other deposits, as well, what affects capillary uptake as well. All these anatomical features limit water uptake to wood. Pubescent oak wood was thus subjected to various decay and wetting ability tests and its anatomical traits were studied. In this paper, we will focus on the results of anatomical traits affecting water performance.

Methodology

The research was performed on pubescent oak wood originating from sub-Mediterranean Slovenia. Samples consisted of sapwood, mature and juvenile heartwood. Before cutting, the wood logs were air-dried for 6 months in a controlled climate. For anatomical investigation, we applied light and Scanning Electron Microscopy (SEM). Light microscopy was performed on a thin section (20 µm) stained with a water solution of Safranin and Astra blue, while SEM specimens were cut into 1 cm^3 cube, ensuring that they were oriented in all three anatomical planes and coated with gold. The envelope density of oven-dry wood was determined with GeoPyc 1365 (Micromeritics, Germany). Various moisture performance tests were performed. The sessile drop method was used to define the contact angles of distilled water on the surfaces of oak wood specimens, using a Theta optical tensiometer from Biolin Scientific Oy (Espoo, Finland). A short-term capillary water uptake test was performed under laboratory conditions (T = 23 °C; RH = 50%) on a Tensiometer K100MK2 (Krüss, Germany).

Findings

Pubescent oak is a ring-porous wood (Figure 1) with large earlywood vessels specialised for water transport (Lavrič et al. 2017). A typical feature of earlywood vessels in heartwood is tylosis which partially or completely blocks vessels' lumina (Figure 2). In pubescent oak, the first tylosis in solitary earlywood vessels appears the new ring is created in May. Earlywood vessels have much higher water-conducting capacity than narrow latewood. However, narrow latewood vessels with a lower risk of cavitation are an important stress avoidance strategy of pubescent oak in the case of drought. Latewood in pubescent oak consists of narrow vessels alternate with thick-walled libriform fibres, which provide mechanical support for the tree. Rays are of two types: uni and multi-seriate. Since wood density is closely related to anatomical traits, the different structures of earlywood and latewood result in their differences.
The anatomical composition of pubescent oak heartwood, predominately the presence of tyloses, affected positively its water performance.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement N°952314.

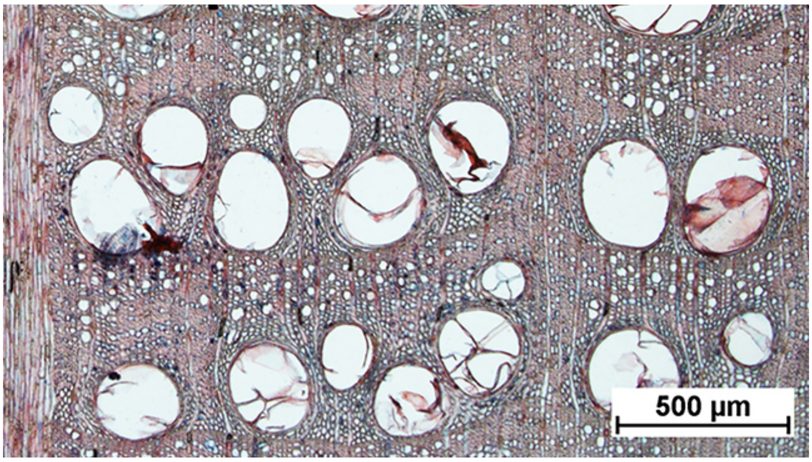

Figure 1: Tree rings and tylosis in vessels of *Q. pubescens*

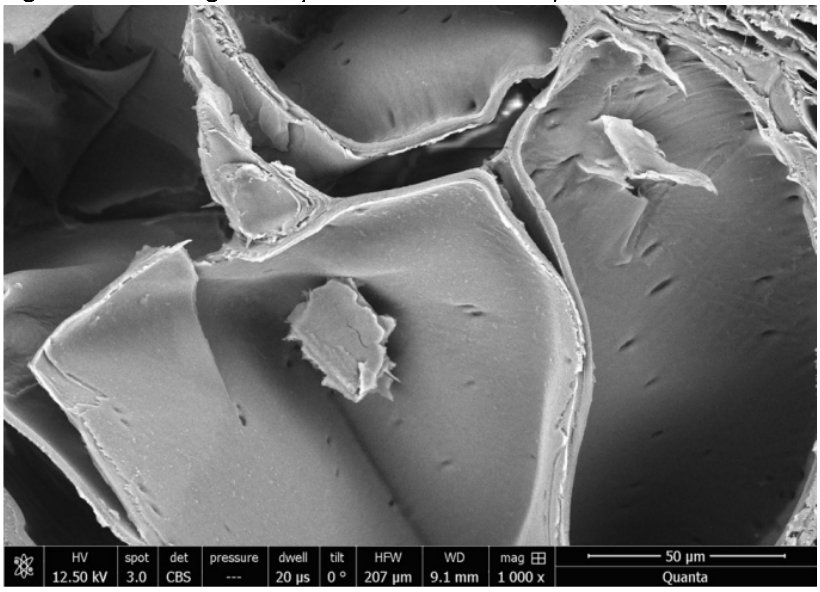

Figure 2: SEM micrograph of tylosis in vessel of *Q. pubescens*

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Natural durability of oak wood – influence of stand conditions and position along the stem radius by J. Baar (MENDELU)

Keywords: extractives, phenolic compounds, Sessile oak, *Trametes versicolor*, yield class

<u>Introduction</u>

The natural durability of wood is one of the most important properties, which determine the lifetime of outdoor used products. The knowledge of wood's natural durability is a great benefit if we considered the increasing pressure against the use of chemical preservative agents to protect the wood. The natural durability of European oak wood was for a long time classified as durable (durability class 2) according to EN 350-2. However, results from different studies have clearly shown its lower

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durability, especially for in-ground exposures, which led to revaluation to lower durability classes in the EN 350 (Brischke and Rolf-Kiel 2010). Hart and Hillis (1972) demonstrated that tannins are responsible for oak heartwood durability and many other later studies have also shown that phenolic compounds content is positively correlated with wood durability. Moreover, the presence of tyloses in vessels, which limited the leaching of extractives, or growth ring width was also considered a factor influencing the durability of oak heartwood (Ayadi et al. 2001, Humar et al. 2008). Despite the knowledge of the importance of phenolic compounds content, so far we do not exactly know the conditions, which impact their production during the growth of trees.

Methodology

Six trees were cut at each of the four forest plots, which differed by yield class (1 – the highest productivity, 5 – the lowest productivity). Trunk discs at 90 cm tree height were used for the preparation of samples. Samples for natural durability (5 × 10 × 30 mm) were cut along the radius from the sapwood to the pith (Figure 1). Another sample, corresponding to the durability sample position, was milled and used for chemical analysis. The natural durability was determined according to the modified EN 113 when samples were exposed to *Trametes versicolor* in Petri dishes for 6 weeks.

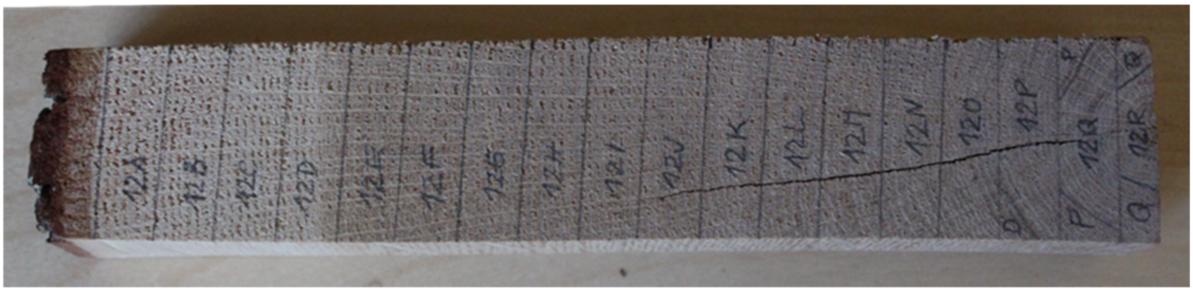
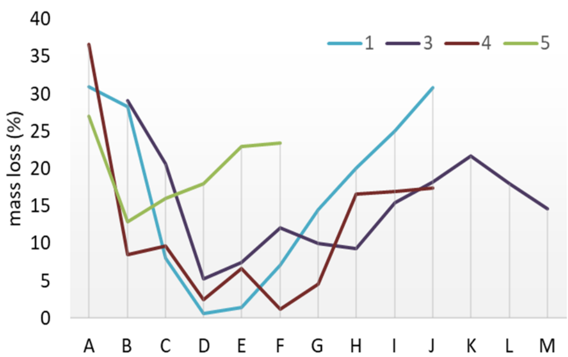


Figure 1 Sample position and marking along the stem radius

The oven-dried wood powder (5 g) was extracted in water: methanol solvent (60 ml, 50:50, V: V) by use of an ultrasound water bath for 20 min. The extract was decanted and wood powder was again extracted in the next 40 ml of solvent for 20 min. 2 ml was used for spectrophotometric analysis of phenolic compounds content, which was determined using the Folin-Ciocalteu method and expressed as gallic acid equivalents (mg GAE g^{-1} of extract). After the evaporation of solvent from the remaining extract in the laboratory oven, the total extractives content was expressed as a percentage of the dry mass of wood powder.

Findings

The mass losses of oak heartwood samples ranged from 0 to 27%, which means that oak natural durability covered all durability classes. Durability classes 1 and 2 were represented the most and their share ranged from 62% in yield class 3 to 90% in yield class 4. The natural durability increased from the pith towards the sapwood and was the highest in the transition zone. The trend was similar in all yield classes (Figure 2).



This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement N°952314.

Figure 2 The mass loss change along the stem radius of selected trees in individual yield classes

No significant differences between yield classes were observed in total extractives content and phenolic compounds content. All forest yield classes were characterized by high variability in total extractives content ranging from 7 to more than 20 % (Figure 3). An identical trend to natural durability was found also for extractives content, where the lowest content was extracted from heartwood close to the pith and then it continually increased towards sapwood, which responded to the positive correlation between these two parameters.

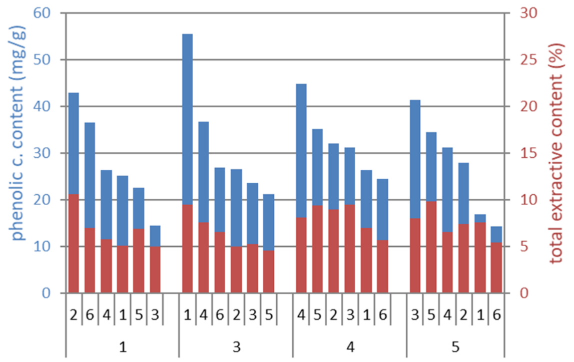


Figure 3 The average total extractive content and phenolic compounds content of individual trees from all yield classes

<u>Conclusion</u>

It seems that site conditions, which influence the production of oak wood (yield class), are not related to the formation of heartwood extractives and are closely linked to natural durability. Regardless of forest yield class, both parameters showed high variability and increase from the pith to the sapwood.

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3. Wood properties and applications

Willows in Czech lowlands: variability of density and shrinkage by Vladimír Gryc[1], Kyriaki Giagli[1], Marek Fajstavr[1], Hanuš Vavrčík (MENDELU)

Keywords: *Salix alba*, tree-ring width, radial shrinkage, tangential shrinkage, variability of wood properties.

<u>Introduction</u>

Genus *Salix* represents about 450 species worldwide distributed mostly in the North hemisphere (Argus 1997). White willow (*Salix alba* L.) is native to Europe and Asia (western and central). Nowadays, white willow has expanded beyond its original area, e.g. to North America and Australia. In the Czech Republic, the species grows in floodplain forests in warmer regions (Úradníček et al. 2001). Willows and willow clones are fast-growing species preferred for many reasons i.e., environmental restoration work, biomass production for energy purposes as well as timber for the wood industry (Kuzovkina and Quigley 2005, Leclerq 1997).

Wood is an exceptional raw material because it is renewable, very strong and elastic despite its low density, easily shaped, ecologically recyclable etc. Nevertheless, wood is not homogenous and it is a

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement N°952314.

highly hygroscopic material. Wood density is a fundamental property featuring the rest of the wood properties. Wood density and shrinkage depend on the genus, the locality type, wood defects and mainly on the position in the stem. Moreover, shrinkage manifests the anisotropic character of wood through different values in individual directions (Vavrčík and Gryc 2012, Gryc and Horáček 2007).

Methodology

The sampling material was taken from white willows growing in three plots in South Moravia, Czech Republic. All selected plots were located in a floodplain forest near Židlochovice (180 m a. s. l.). Plots 1, 2 and 3 were classified as *Ulmeto-Quercetum alluviale* (*Brachypodium sylvaticum*), *Saliceto-Alnetum* and *Ulmeto-Quercetum alluviale* (*Aegopodium podagraria*), respectively. Six healthy trees were randomly chosen per plot (mean height 23–25 m, diameter 26–32 cm). Logs (1 m) were cut at breast height (1.3 m from the ground) from each tree. Tree-ring widths were measured on the transversal section by using the Leica S6D stereomicroscope and the VIAS TimeTable (Vienna Institute for Archaeological Science, Vienna, Austria) measuring system (with an accuracy of 0.01 mm). Samples for density and shrinkage (20 × 20 × 30 mm) were prepared uniformly from the entire log.

Findings

Variability of tree-ring width; The selected trees showed a very similar amount of tree rings, between 17 and 19 at breast height. The tree rings were wider (10–12 mm) during the first years, followed by a gradual decrease in tree-ring width (2–6 mm) along the stem radius from pith to bark (Fig. 1). The average tree-ring width calculated from all three plots was 7.51 mm, ranging between 6.62 and 8.03 mm (Table 1). Our results on tree-ring widths were following Sacré (1974), who reported similar values (6.6–8.6 mm) depending on the logs.

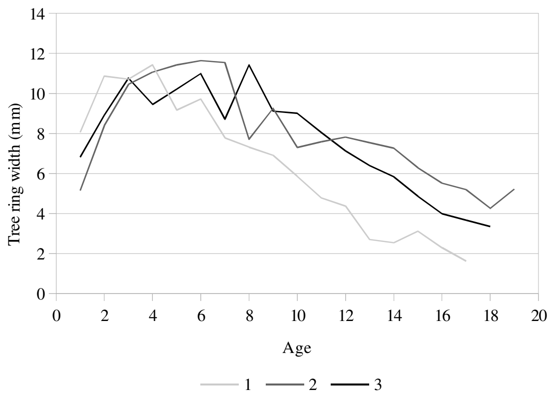

Fig. 1. Variability of tree-ring width along stem radius calculated per plot. All curves represent the mean values of six trees.

Variability of properties – density and shrinkage; We found significant differences in both average green density and average dry density among plots (Table 2). The differences in the case of green wood density among plots were higher (81.46 kg·m-3) than in the case of dry wood density (20 kg·m-3). The average dry wood density from all three plots was 390.8 kg·m-3 ranging from 278.6 to 631.6 kg·m-3. Kollmann and Côté (1968) reported a similar average dry wood density of 365 kg·m-3 ranging between 320 and 420 kg·m-3. Nevertheless, Wagenführ (2000) stated lower dry wood density of 270–330–380 kg·m-3 (minimum–average–maximum), while Wani et al. (2014) stated that basic density in Salix alba growing in Pakistan is influenced by locality. In our study, we noticed higher variability of dry wood density among individual trees in each plot than in average dry wood density among the plots (Fig. 2).

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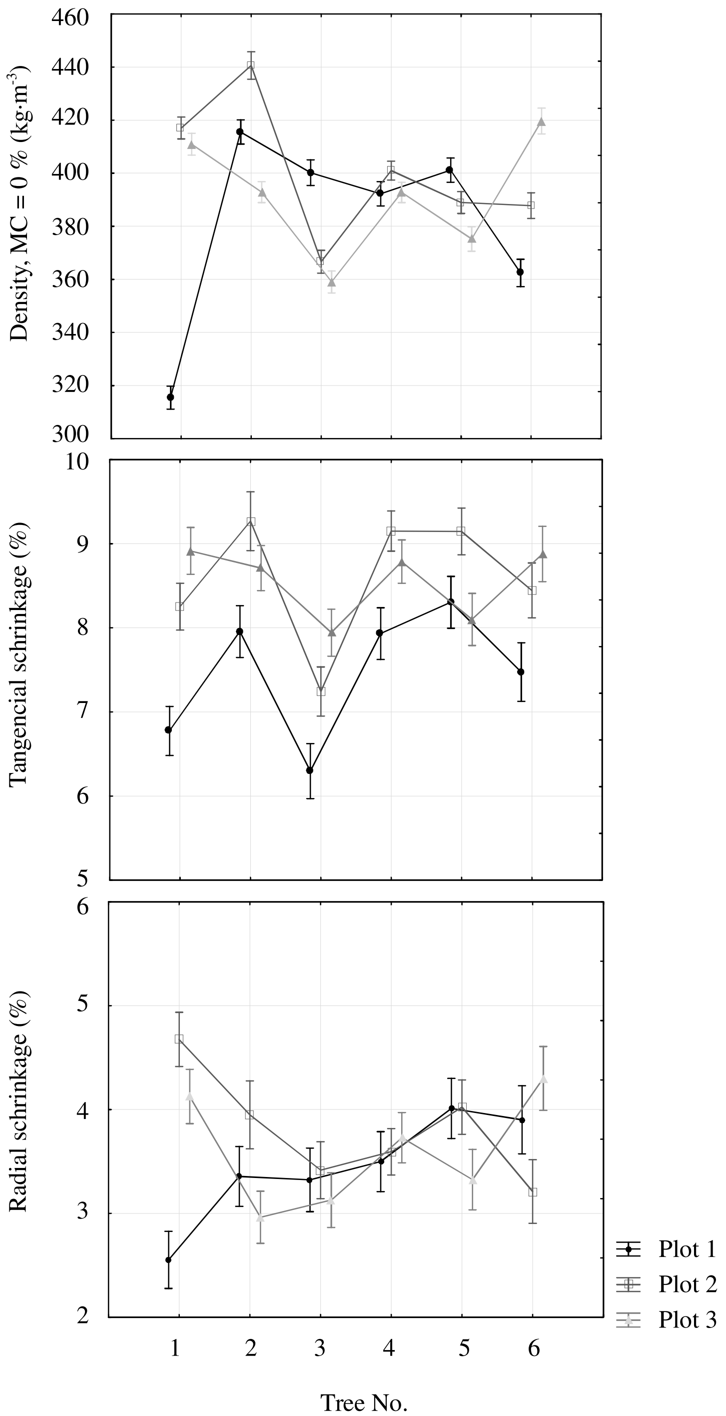

Fig. 2. Variability of wood density and shrinkage among trees

Conclusions

Our results indicated that white willow trees growing in the Czech lowlands produce wood of higher average density compared to the literature, while the radial and tangential wood shrinkage were found to be in line with previous studies. We noticed significant differences among the plots. Nevertheless, the variability of the examined properties was high among trees growing in the same plot.

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Adhesive based on liquefied wood prepared from lesser used wood species and hexamethylenediamine - Curing monitoring and wood bonding characterization by J. Žigon, V. Šeda, P. Čermák, M. Šernek (UL, MENDELU)

Keywords: adhesive, bonding, hexamethylenediamine, liquefied wood

Introduction

In the search for a replacement for the polluting petroleum-based adhesives used in wood bonding, researchers have developed many interesting solutions in recent decades. Substitutes based on natural resources, such as tannin, lignin, proteins, carbohydrates, starch, and others, have been extensively studied, and many have been reported to be very promising in terms of their mechanical and physical properties. Another natural substitute for synthetic adhesives intended for bonding wood is liquefied wood (LW). The findings from the literature inspired the authors of this to combine pure LW with hexamethylenediamine (HMDA) as a crosslinker. The HMDA was used to improve the adhesive properties of LW and the resistance of the adhesive joints to degradation by increased relative humidity.

Methodology

The liquefaction of the tree of heaven (*Ailanthus altissima* (Mill.) Swingle) wood was carried out according to the procedure developed at the Department of Wood Science and Technology, University of Ljubljana. The basic properties (non-volatile-matter content and viscosity) of liquid LW and LW+HMDA adhesives were determined. The physical transitions and chemical reactions that occur during the curing of adhesives were determined with differential scanning calorimetry (DSC), and the cured adhesives were analysed with Fourier transform infrared (FTIR) spectroscopy. The bonding performance of LW and LW+HMDA adhesives was evaluated with an automated bonding evaluation system (ABES) and tensile-shear strength test.

Findings

The effect of HMDA addition on the performance of pure LW as an independent adhesive for wood bonding was evaluated. In addition to the improved thermal stability of LW+HMDA adhesive, DSC analysis showed that HMDA caused improved reactions in LW. A similar finding was confirmed by FTIR analysis of the cured LW and LW+HMDA adhesives; the possible interaction between LW and HMDA could be confirmed by the enhanced presence of amide linkages with C–N bonds in the LW+HMDA adhesive. Monitoring the evolution of adhesive strength development by ABES showed that higher pressing temperatures resulted in stronger bonds in both adhesives (Figure 1). In addition, the HMDA in the LW adhesive notably improved the bond strength of the joints and accelerated the cross-linking of the adhesive. However, the tensile-shear strength test showed that both the LW and LW+HMDA adhesives were not suitable for interior non-structural use, as none of them met the standard requirements. The relatively high wood failure (70%) in the tested joints bonded with pure LW was related to the degradation of the wood in the adhesive joint by the acidic LW (Figure 2). The addition of HMDA to LW notably improved the resistance of the LW+HMDA adhesive joints to water

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement N°952314.

degradation. It can be concluded that HMDA improves the bonding performance of LW, which could be further enhanced by optimizing the LW+HMDA adhesive formulation.

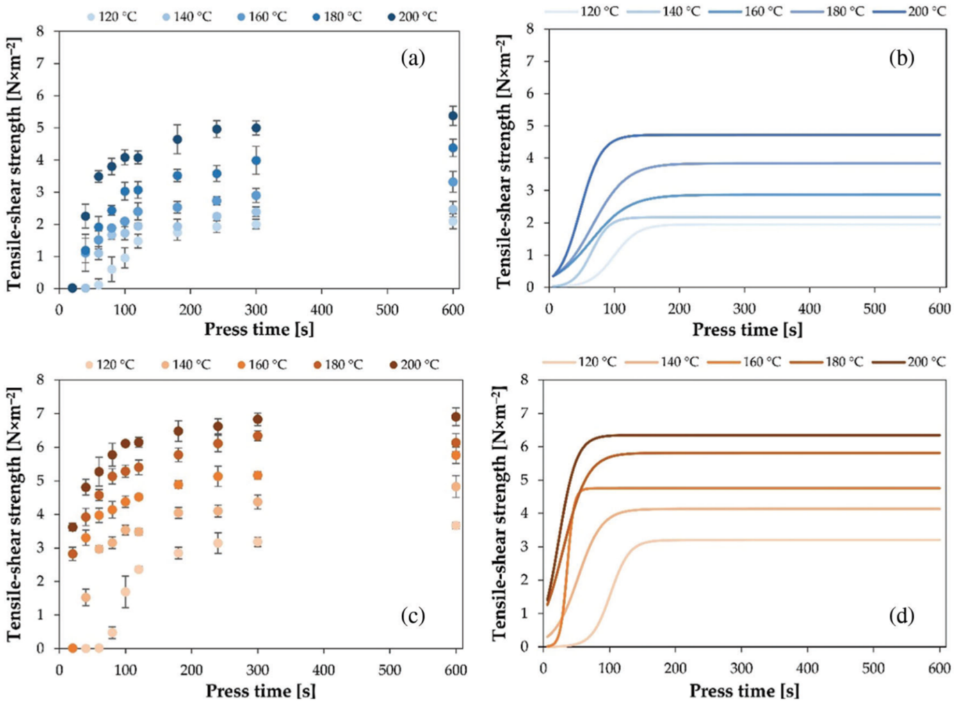

Figure 1: Actual (markings) and modelled (lines) developments of the tensile-shear strength in adhesive joints bonded with LW (a and b) and LW+HMDA (c and d)

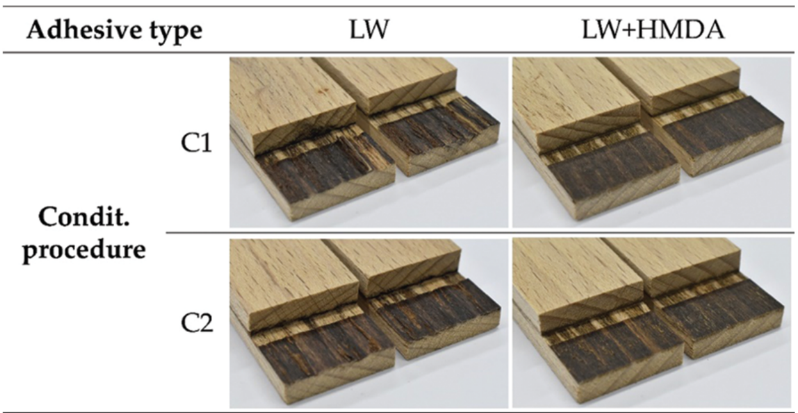

Figure 2: The appearance of the lap joints bonded with LW and LW+HMDA adhesives, tested after different conditioning procedures

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Surface finishing of selected invasive lesser-used wood species by J. Žigon, M. Pavlič (UL)

Keywords: coating, finishing, invasive tree species, oil, wood

This project has received funding from the European Union’s Horizon 2020 research and innovation programme under grant agreement N°952314.

Introduction

Invasive alien species is an alien species that has stabilized and is causing environmental change, threatening human health, the economy and/or native biotic variety. Due to their strong renewal power the invasive tree species are spreading rapidly, especially in abandoned agricultural and forest areas and along traffic routes and water courses. One way of their restriction is the promotion of their use. The research was focused on the surface finishing of the following species, from which we were able to obtain a sufficient quantity of wood for carrying out the tests: Box elder (*Acer negundo* L.; AcNe.), Black locust (*Robinia pseudoacacia* L.; RoPs), Horse-chestnut (*Aesculus hippocastanum* L.; AeHi), Honey locust (*Gleditsia triacanthos* L.; GlTr), Chinese sumac (*Ailanthus altissima* (Mill.) Swingle; AiAl), and European beech (*Fagus sylvatica* L.; FaSy) wood as a control. The interactions between wood substrates and selected finishes were investigated by the determination of several properties.

Methodology

As finishing agents were selected unpigmented semi-gloss one-component waterborne coating on the acrylate-polyurethane basis (WPU), unpigmented glossy one-component solvent-borne finish on polyurethane basis (SPU) and tung oil (TO). In addition, the following properties of coated wood were investigated according to European standards: colour, gloss, dry coating film thickness, coating adhesion, finished surface resistance to cold liquids, coated surface hardness and resistance to impact. On the oiled surfaces, the drying stage was also monitored.

Findings

Interactions between the substrate and the finish did affect the drying speed of TO and dry coating film thickness (WPU and SPU). Wood species with higher density had lower oil uptake and consequently, oil on that surface had a longer drying time. Wood species with higher density also had lower uptake of used coatings which donated to a higher thickness of the coating films.

The final appearance evaluated by colour and gloss measurements was very much dependent on the type of substrate and finish being used. On the RoPs and GlTr the yellowing of the coating film due to the most possible migration of wood extractives was noticed.

The lower coating hardness of SPU compared to WPU resulted in higher flexibility and higher resistance to the impact of SPU systems. It was also shown that surface hardness (determined by scratch test), resistance to impact (Table 1) and coating adhesion (Table 2) are very much related to interactions between the coating and the substrate, while resistance to cold liquids was dependent only on the type of the finish.

In general woods of the selected less used invasive species are not problematic for finishing. Nevertheless, it is reasonable to consider the findings of our study which also showed that TO cured very slowly on RoPs wood and that the coating film may turn yellow on RoPs and GlTr wood.

Table 1: Impact resistance of the coated surfaces

Substrate	WPU		SPU	
	Dropping height	Grade	Dropping height	Grade
FaSy	10 mm	2	100 mm	3
AcNe	10 mm	2	100 mm	1
RoPs	10 mm	3	100 mm	3
AeHi	10 mm	2	100 mm	1
GlTr	10 mm	3	200 mm	3
AiAl	10 mm	3	200 mm	1

This project has received funding from the European Union’s Horizon 2020 research and innovation programme under grant agreement N°952314.

Table 2: Coating adhesion (σ) and the type of the fracture (A – adhesive fracture between coating and the substrate, K – cohesive fracture of the substrate)

Substrate	Adhesion (MPa)					
	WPU			SPU		
	$\bar{x}$	σ	Fracture	$\bar{x}$	σ	Fracture
FaSy	4.95	0.36	70 % A, 30 % K	5.45	0.33	80 % A, 20 % K
AcNe	4.09	0.29	20 % A, 80 % K	4.11	0.30	85 % A, 15 % K
RoPs	4.47	0.48	100 % A	4.58	0.41	100 % A
AeHi	3.98	0.33	25 % A, 75 % K	4.01	0.29	20 % A, 80 % K
GlTr	4.56	0.45	100 % A	4.89	0.44	100 % A
AiAl	4.05	0.43	100 % A	5.12	0.42	100 % A

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Adhesive bonding of lesser-used invasive wood species by M. Šernek, M. Kariž, B. Šega[1], J. Žigon[1], M. Merela (UL)

Keywords: adhesive, bonding, lesser used invasive wood species, shear strength, laminated wood composite

Introduction

Invasive alien plant species displace local vegetation, destroy agricultural land, and harm the economy. Many of them are removed daily and mainly burned. However, some of them produce lignocellulosic material that could be used instead of domestic wood species. The objective of this study was to test the bonding properties of lesser-used invasive wood species that were available in suitable sizes to perform bonding tests with two cold-setting wood adhesives. In addition, laminated wood composites were made from lesser-used invasive wood species in combination with domestic wood species.

Methodology

The study was divided into two parts. In the first part, the shear strength of adhesive bonds was tested for five lesser-used invasive wood species that were available in suitable element sizes for testing: *Ailanthus altissima, Aesculus hippocastanum, Robinia pseudoacacia, Gleditsia triacanthos,* and *Acer negundo*. *Fagus sylvatica* was used as the reference wood species. The moisture content of all wood species was 10 -12 %. Two cold-setting adhesives were used: polyvinyl acetate adhesive Mekol D3 (PVAc) and polyurethane adhesive Mitopur E45 (PU), both manufactured by Mitol Sežana, Slovenia. The application rate was 180 g/m^2. Bonding was performed in a hydraulic laboratory press at room temperature and at a pressure of 0.8 N/mm^2 for 90 minutes. After one week of conditioning in standard climate (65 % relative air humidity, 20 °C), the bonded assemblies were cut into pre-treated shear specimens prior to testing (EN 204). The shear strength of the adhesive bonds was tested (EN 205) using a Zwick Z005 universal testing machine (Zwick Roell, Ulm, Germany).
In the second part, three different laminated wood composites (Figure 1) with laminations of *Robinia pseudoacacia* (RP), *Acer negundo* (AN), *Ailanthus altissima* (AA), *Picea abies* (PA), and *Fagus sylvatica* (FS) were bonded with Mitopur E45 polyurethane adhesive and tested in a four-point bending test (EN 480).

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<u>Findings</u>

The shear strength of the bond depends on the wood properties, especially density, and the type of adhesive (Table 1). The highest shear strength of specimens after Preparation 1 (conditioning in standard climate) was shown by *Ailanthus altissima* bonded with PVAc adhesive (15.1 N/mm^2), and the lowest strength was measured for specimens of *Aesculus hippocastanum* (7.2 N/mm^2). The minimum shear strength of the bond prescribed for Preparation 1 is 10 N/mm^2. PVAc bonds had higher strengths than PU for all wood species except *Aesculus hippocastanum*. The percentage of wood failure was generally high, so the shear strength of the bond was mainly related to the shear strength of the wood species, which was higher for wood species with higher density.

Table 1. Shear strength and percentage of wood failure of tested wood species and adhesives for different pre-treatments of shear specimens

Treatment according to EN 204	Preparation 1		Preparation 3		Preparation 4	
Wood species and adhesive	Shear strength (N/mm^2)	Wood failure (%)	Shear strength (N/mm^2)	Wood failure (%)	Shear strength (N/mm^2)	Wood failure (%)
Robinia pseudoacacia PU	9.6	42	2.1	0	3.7	0
Robinia pseudoacacia PVAc	**11.1**	**100**	**2.9**	**0**	**12.7**	**87**
Acer negundo PU	9.2	83	3.5	1	6.8	23
Acer negundo PVAc	9.8	100	2.3	0	8.8	50
Aesculus hippocastanum PU	7.5	100	5.8	22	7.8	100
Aesculus hippocastanum PVAc	7.2	100	2.4	0	6.9	77
Gleditsia triacanthos PU	8.1	42	5.6	0	8.2	1
Gleditsia triacanthos PVAc	**12.0**	**88**	**2.1**	**0**	**8.8**	**14**
Ailanthus altissima PU	**11.0**	**53**	**3.0**	**0**	**9.4**	**37**
Ailanthus altissima PVAc	**15.1**	**99**	**2.2**	**0**	**11.3**	**76**
Fagus sylvatica PU	12.4	100	2.1	0	6.8	0
Fagus sylvatica PVAc	12.5	100	1.4	0	9.1	85

Preparation 3 (soaking in cold water for 4 days) resulted in a significant decrease in bond strength. The highest shear strength of the wet specimens was exhibited by *Aesculus hippocastanum* (5.8 N/mm^2) and *Gleditsia triacanthos* (5.6 N/mm^2), both of which were bonded with PU, which chemically cures with OH groups and is more water resistant. The minimum shear strength of the bond prescribed for Preparation 3 is 2 N/mm^2. The percentage of wood failure was 0, except for two groups of specimens, but the values were still low.

Samples pre-treated according to Preparation 4 (soaking in water for 4 days and subsequent conditioning in standard climate for 7 days) showed higher bond strength than samples pre-treated according to Preparation 3. *Robinia pseudoacacia* bonded with PVAc had the highest shear strength (12.7 N/mm^2), and *Robinia pseudoacacia* bonded with PU had the lowest (3.7 N/mm^2). The minimum shear bond strength prescribed for Preparation 4 is 8 N/mm^2.

Four groups of specimens (shown in bold in Table 1) achieved the requirements of durability class D3 according to EN 205. It was shown that lesser-used invasive wood species can be used for the

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production of wood composites (Figure 1). From an engineering point of view, it is advantageous to use stronger wood species with higher density in the outer layers of the composite to be loaded in bending.

E_m	7,0 GPa	12,5 GPa	15,0 GPa
f_m	80 MPa	90 MPa	100 MPa
ρ	550 kg/m^3	600 kg/m^3	630 kg/m^3

Figure 1. Laminated wood composites made of different wood lamellae and their properties (E_m = modulus of elasticity, f_m = bending strength, ρ = density)

It can be concluded that all investigated lesser-used wood species showed quite good bonding and bond strength for dry specimens. The PVAc adhesive showed better performance than PU for dry specimens, while the PU adhesive was better than PVAc for bonded specimens exposed to water. The shear strength as well as the bending properties strongly depend on the wood density.

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Application of renewable impregnation agents and extracts in lesser-used tree species by P. Rademacher (MENDELU)

Keywords: wood modification; renewable impregnation agents; outdoor wood applications; dimensional stability; durability

Introduction

The production and application of the sustainable and renewable material WOOD under local conditions and its higher value use under outdoor conditions have to be evaluated under the following background conditions:

- Climate change, global warming, CO_2 re-emission from fossil sources
- Forest conversion, limited high-value wood assortments, non-sustainable produced tropical wood assortments or biocide treatment as well as plastic, metal, or concrete materials
- Renewable, sustainable and locally produced wood, environmentally friendly chemical modification treatments (CMT) & treatment agents (impregnation, finishing, adhesive)
- Research interactions in EU-Networks (COST FP1407, DE-CZ-HU-SE-SI-NET, DANUBE-Network, CZ-SE-DE-AT-SI - Hor2020-ASFORCLIC)

The objective of the present research was to evaluate the possibilities of both, the utilization of non-durable local wood species as well as their property improvement for outdoor applications by wood-modification agents from native, renewable origin.

State-of-the-art: During the last decades the production and utilization of renewable biomass increased and are important for the future biomass supply with options for different usages. Reasons for this are 1) overuse of traditionally managed forests (Mantau and Bilitewski 2005); 2) decreasing storage of fossil raw materials and energy sources (FAO 2005); 3) increasing amount of carbon dioxide in the atmosphere and global warming due to the burning of fossil carbon sources (Cox et al. 2000); 4)

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need of higher application of more and climatically better-adapted tree species under changing climate conditions (Blohm et al. 2014).
During the development of wood cell walls until the maturation and functionality of the cells, wall architecture and chemical composition become distinctly changed (Zimmermann and Brown 1980; Fengel and Wegener 1989; Wagenführ 1999; Faix 2008). Secondary changes, especially in the case of durable heartwood, occur at the transition from sapwood to heartwood by the incorporation of phenolic compounds in the carbohydrate-lignin complex (Hillis 1962; Klumpers et al. 1994; Faix 2004). During subsequent ageing of cell walls, additional organic components (Gierlinger et al. 2003; Haupt et al. 2003; Grabner et al. 2005) or mineral elements can be bonded on or included in the fibril-structure of wood cell walls (Rademacher et al. 1986; Faix, 2004), leading to both increased durability and dimensional stability of wood. Also wounding (Rademacher et al. 1984; Shigo 1986; Frankenstein and Schmitt 2006), emissions (Hapla 1992) or climate stress (Schweingruber 1993) can initiate tertiary structural or chemical changes in cell walls.
The knowledge about the basic mechanisms of wood formation and successive changes in the wood structure and chemical composition has been strengthened due to many investigations in the past (see overviews of Schwager and Lange 1998, Faix 2008, Wagenführ and Wagenführ 2008, Schmitt and Koch 2009), mainly dealing with most utilized tree species like spruce and pine or beech and oak. However, the knowledge about the structure and chemistry of lesser-used wood species or even the mechanism and process of technical or biotechnical changes in cell walls due to wood modification – leading to an improvement of properties – is still not fully understood (Zimmermann and Brown 1980; Ermeydan et al. 2012).
The improvement of wood properties by wood modification (Hill 2006; Militz and Mai 2008) became a more and more important goal and suitable methods have been developed for innovative and additional use of wood as a renewable raw material (Klemm et al. 2005). The transformation of our knowledge about mechanisms and processes of modification from technical (Militz et al. 1997) to small-scaled cellular (Mahnert et al. 2013) or molecular level (Grabner et al. 2005) could help to deepen our understanding and enlarge our instruments to establish new materials (Meier et al. 2001; Heiduschke and Haller 2010).
A lot of efforts have been done in the past to improve the properties of wood by chemical or mechanical treatments (Rowell 1983; Wepner and Militz 2005; Hill 2006), but the cellular or even sub-cellular mechanisms (mode of action) behind this are more or less unclear (Zimmermann and Brown 1980; Ermeydan et al. 2012). The following results deal with the exposure of basic mechanisms which lead to higher density, higher strength, higher dimensional stability and higher durability of wooden material. Collecting information on the microscopic structures as well as the chemical composition of cell walls before and after modification treatments is essential to understand the mechanisms behind changed 1) cell wall stability, 2) cell wall penetrability and 3) cell wall impregnation.

Methodology
A set of renewable agents for wood impregnation was developed for the following material resources and processes (see also Table 1):

- Production of liquid residues from thermal treatment (TT), Hydro-Thermal Carbonisation (HTC), pyrolysis processes (Pyrol), Liquid Wood (LW) and Robinia extracts (RobExtr); see Table 1.
- Impregnation of beech, poplar and pine sapwood samples with these liquids (using vacuum 20 kPa/1 hour) under calculation of the weight-percent-gain (WPG) and leaching tests, using 10 cycles to yield the weight-percent-remain (WPR) of the non-leached fraction.
- Concentration (1 agent: X H_2O): Pyrol 1:10; 1:2, original (1:1=100%); TT and HTC conc. 10:1; LW 1:3.
- Conditioning, drying, volume/weight, leaching, and bulking measurement following standards.
- Durability tests: Bravery Test (1978); fungi: *Trametes versicolor,* time of exposure 6 weeks.
- Durability samples: 9 samples of 5 x 10 x 30 mm^3 (Bravery 1978) for each treatment.

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- Swelling measurement: 10 samples of 14 x 14 x 28 mm^3 for each treatment.
- UMSP: UV-light absorption at 278 nm, using Zeiss-UMSP 80 (Koch and Grünwald 2004). - Light and UV-microscopic investigations.
-

Table 1: Chemical sources and process for impregnation of wood with renewable solutions

No.	Used Raw-Materials	Abbreviation	Synonym	Process Conditions
1	Miscanthus sp.	HTC_Misc	HTC-AG, HTC- D. Tschok,Misc.3/2013	Hydro-Thermal-Carbonisation = HTC, pressure, temp.
2	Spruce sawdust	HTC_Saw	HTC/ S. Vondran	HTC, pressure, temp.
2.2	Brewery residuest	HTC_Brew	HTC Schlamm	HTC, pressure, temp.
3	Mixed spruce, beech, oak, ash, poplar	TT_180	Thermal Treatment, Heat treatment, HT	TT, 180°C
4	Mixed spruce, beech, oak, ash, poplar	TT_200	Thermal Treatment, Heat treatment, HT	TT, 200°C
5	Canadian beech	Pyrol_Can	Bio-Oil, BO, Old Canadian	Fast pyrolysis, Canadian process (Dynamotive)
6	European beech	Pyrol_ProF1	German 1	Slow pyrolysis, D-ProFagus process
7	European beech	Pyrol_ProF2	German 2	Slow pyrolysis, D-ProFagus process
8	European beech	Pyrol_ProF3orig	Hamburg bio oil, crude Pro Fagus	Slow pyrolysis, D-ProFagus process
9	European beech	Pyrol_ProF3low	Hamburg low fraction,CHNSCCO2	Slow pyrolysis, D-ProFagus proc., supercritical CO_2-extr. low molecular weight
10	European beech	Pyrol_ProF3big	Hamburg PH 200, residue	Slow pyrolysis, D-ProFagus proc., supercritical CO_2-extr. high molecular weight
11	European beech	Pyrol_NLfre	Btg wood oil fresh, NL fresh	Fast pyrolysis, NL- BTG process, fresh production
12	European beech	Pyrol_NLold	Btg wood oil from storage, NL old	Fast pyrolysis, NL-BTG proc., stored/ old production
13	Robinia-heartwood, milled wood	Rob_Extr		Methanol-water 1:1 extract of heartwood, 3 concentrations
14	Forest poplar	LW_Pop_for1:1	LW poplar 1:1	Liquified wood
15	Forest poplar	LW_Pop_for1:3	LW poplar 1:3	Liquified wood
16	Plantation poplar	LW_Pop_plant	LW poplar, fast growing	Liquified wood
17	Forest spruce	LW_Spruce	LW spruce	Liquified wood

<u>Findings</u>

Chemical Analysis of Robinia Extracts and thermally produced liquid Agents; The extraction process of Robinia wood with methanol-water mixture 1:1 resulted in a wide range of different concentration levels of extractives, reaching or exceeding 10000 µg g^{-1} dry-wood. Most substances showed maximal concentration in older Robinia heartwood compared to a median or young plantation wood; Robinia sapwood and especially bark have the lowest concentration of single extracts (<5 µg g^{-1}dw), mainly ranked from robinetinidin > dihydrorobinetin > robinetin > gallic acid > fisetin >> catechine, whereas barks shows higher catechine amount (Sablík and Rademacher 2013; Sablík et al. 2016; Rademacher et al. 2017).

Chemical Analysis of Process Residues after Pyrolysis Treatment of Bio-Materials; The dominating molecules in most pyrolysis process products are gallic acid, furfural, catechol and phenol, reaching or exceeding 1000 µg per gram of pyrolysis liquid. Also 5-methylfurfural, syringaldehyde and eugenol reach in some processes a range of 200-400 µg g^{-1}, whereas all other detected molecules show concentrations <<100 µg g^{-1}. Some molecules, like gallic acid, have a widely similar range of concentrations (880-1150 µg g^{-1}), while others (div. ProFagus products; Bodenfelde/ DE; e.g. ProF3) show a 2-3 times higher amount, especially compared to NL- or CAN- products (Rademacher et al. 2017).

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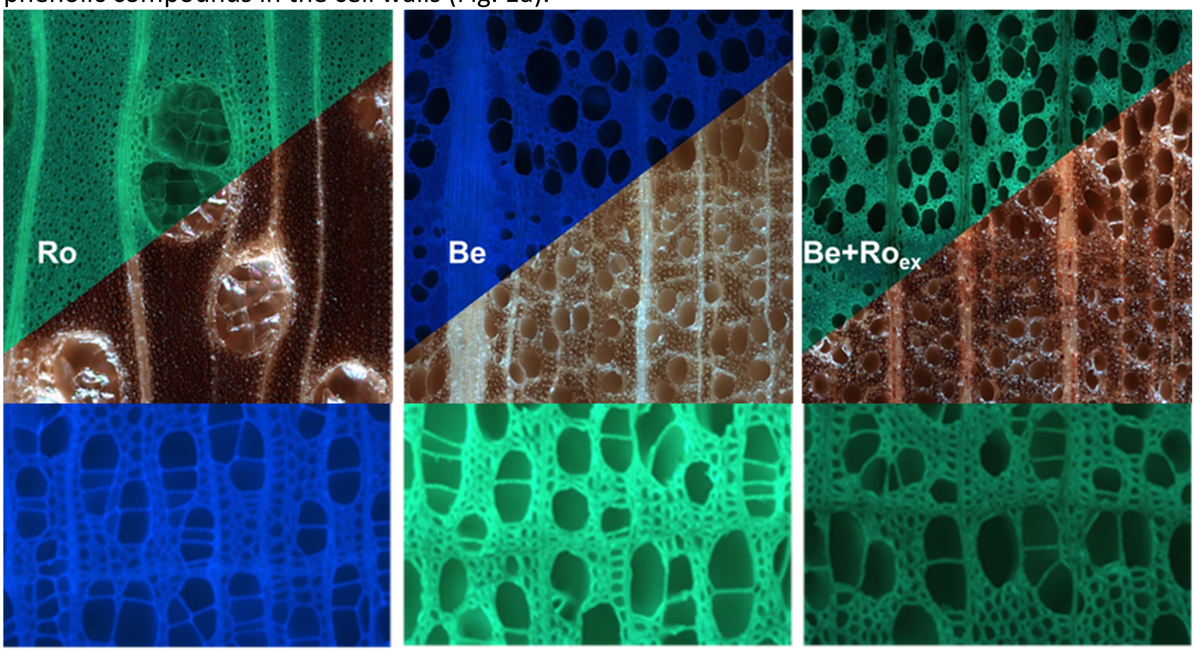
Chemical Analysis of Process Residues after Thermal Treatment (TT) or Hydro-Thermal-Carbonisation (HTC) of Biomass Materials (add. Graphics see: Rademacher et al. 2017); In the liquid process-residues after TT of wood (using 180 or 200°C) or HTC of organic waste assortments (Miscanthus grass, or spruce sawdust) the robinetin-derivatives as well as fisetin, catechin and gallic acid are present in higher concentrations in the HTC-process residues (about 0.5-10 µg ml^{-1}) compared to the TT process (mainly 0.1-0.8 µg ml^{-1}, gallic acid also 1-5 µg ml^{-1}). Furfural even appears only in the HTC-Sawdust), and phenol only in the TT process.

Microscopic Observations and UV Detection of impregnated Modification Agents; Microscopy of pyrolysis-impregnated (Pyrol_Can) beech and poplar wood show – compared to untreated native wood - a distinct deepening of the colour in the cell walls and a strong colour deepening in the cell lumina due to the more or less achieved full-impregnation of the entire micro- and macro-porous space of the wood tissue. The maximal WPG, realized by full impregnation of the cell walls as well as the filling of all cell-lumina – has no further positive effect on the property improvement, realised mainly by the cell wall impregnation and the interaction of the impregnation agent and the cell wall hydroxyl groups, resulting in chemical and micro-structural changes (Rademacher et al. 2017).

To counteract an overloading of the cell lumina a post-vacuum or leaching treatment of the impregnated wood just following the impregnation process helps to minimize these effects by eliminating the free liquid from the macro-pores.
The microscopic view of UV-light illuminated cross sections of older Robinia heartwood as well as impregnated beech and poplar samples showed a stronger detection of phenolic compound response in kind of yellow-green fluorescence shining compared to younger Robinia or non-treated beech or poplar samples (blue colour). There was a ranking of shining, starting with native beech and poplar wood <<< young Robinia < leached Robinia-extract impregnated beech and poplar < unleached Robinia-extract impregnated beech and poplar wood < old Robinia heartwood, indicating increasing phenolic compounds in the cell walls (Fig. 1a).



Po Po+Ro_{ex} Po+Ro_{ex}+$_{leach}$

Fig. 1a: Above: Increase of UV-light induced green fluorescence shining, detecting an increasing amount of phenolic compounds in cell walls of Robinia heartwood (left; upper part of pictures: green = detection of phenolic compounds); native beech (middle; upper part of the picture: blue = no phenolic compounds); Robinia - extract impregnated beech wood (right: upper part green = phenolic

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extract detection) compared with normal light (lower parts of upper 3 pictures: brown). Each image has a dimension of 0.9 x 0.9 mm^2.
Below: UV-light induced fluorescence shining in poplar wood: left: Reference, no impregnation (→ no green, but blue); mid: after Robinia extract impregnation (intensive green shining); right: after leaching (lower green coloured). Photos: R. Rousek

Cellular UV-Microspectrophotometry (UMSP); UMSP images of untreated as well as pyrolysis-liquid and Robinia-extract treated poplar wood showed high differences in UV-light (278 nm) absorption, indicating a higher amount of aromatic, condensed components in impregnated cell walls compared to untreated wood of native poplar (Fig. 1b). The presence of these components also in leached samples confirms their strong cell wall fixation.

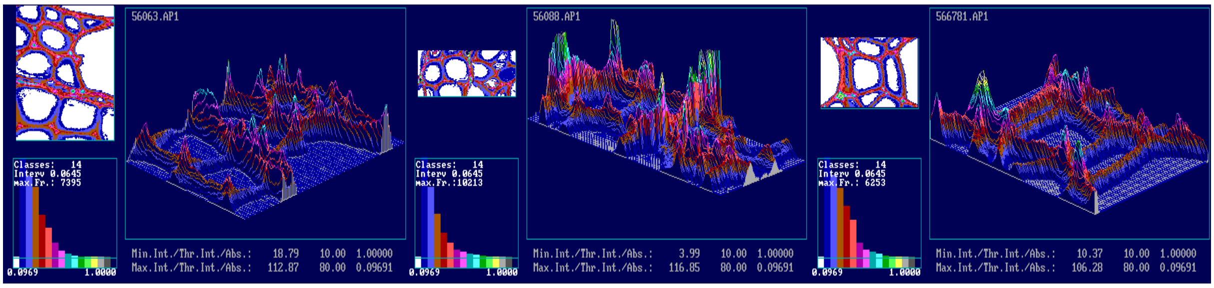

Fig. 1b: UV-absorption UMSP-field scan of reference (left) and pyrolysis-treated poplar (Pyrol_Can, mid), as well as Robinia extract, impregnated poplar wood (right); wave length-setting of absorption 278 nm, absorption scale 1.0

Weight Percentage-Gain (WPG), Weight Percentage–Remain (WPR) and Bulking Effect; The maximal WPG, possible to be reached after vacuum-pressure impregnation with thermal liquid residues is due to the lower density and higher porous system in pine sapwood about 2-times higher (up to 120% of wood dry mass) than in beech (30-70%). In poplar wood, which has a similar or even lower density than pine, this difference as compared to beech is much lower, explained by the fact, that the facultative heartwood of poplar is classified as 'difficult to impregnate', whereas the sapwood is easy to treat like pine sapwood (Kumar and Donriyal 1991; Hoffmann 2008; EN 350:2016). Due to the washout of the non-fixed agent after the leaching test, only 25-40% of the impregnated pyrolysis liquids remained in the wood (Weight-Percent-Remain = WPR), in case of low-molecular fraction after supercritical CO_2-fractionation of pyrolysis liquids and a case of liquid wood from poplar and especially from spruce the washout was even higher: only 25% of liquefied poplar wood and 15% of liquefied spruce remained in the wood, in case of low-molecular pyrolysis fraction even only 10-15% of the impregnated amount remained. The corresponding WPR was 12-45% of the wood mass in the case of pyrolysis liquids, 10-30% of the liquefied wood and 5% of the low molecular fraction of pyrolysis liquid (Rademacher et al. 2017).

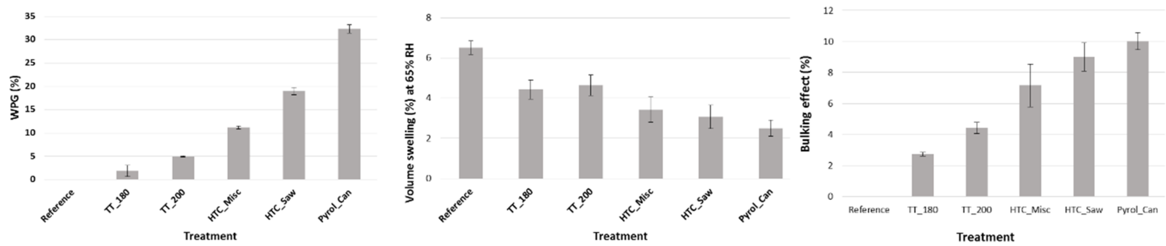

Fig. 2: Weight percentage-gain (%; left) of impregnated poplar using wood-processing residues (thermal treatment (TT: 180 and 200°C), hydro-thermal-carbonisation (HTC: Miscanthus, spruce-sawdust) and pyrolysis-liquid from Canadian producer (Pyrol_Can), reduced volume-swelling (%; mid) and bulking effect (%; right). Residues and process compare Table 1

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The determined bulking effect (permanent swelling of the wood due to structural modification (e.g. cross-linking of hydroxyl groups in the cell wall) of the remaining agents after the leaching test was surprising: Despite the highest washout rate in the case of the low-molecular pyrolysis fraction and the high washout of the liquefied wood the bulking effect is still in average (low pyrolysis fraction) or even maximal (compare Fig. 2, right). This indicates that not only the amount of impregnated or remaining impregnation agent is responsible for the wood-property improvement, but also the selection of reactive components with special functional groups (Ermeydan et al. 2012), especially in the case of higher active slow-pyrolysed fractions after fractioning or wood liquefaction.

Moisture related Behaviour of the modified Wood; Compared to this, liquid thermal residues from thermal treatment, hydro-thermal carbonisation or Canadian fast pyrolysis process show a related behaviour between the amount of impregnated agent (WPG) and the corresponding bulking effect as well as the decreasing volume swelling response under moisture conditions (Fig. 2).
The water uptake (WU) of pyrolysis and liquid wood-impregnated beech wood shows a strong relationship (r^2 = 0.91) between the amount of impregnated and remaining (after leaching) agent (WPR) and the amount of included water, covering a range of 90% WU in case of WPR after leaching of < 5% until < 70% WU in case of WPR > 20% (Fig. 3, left).

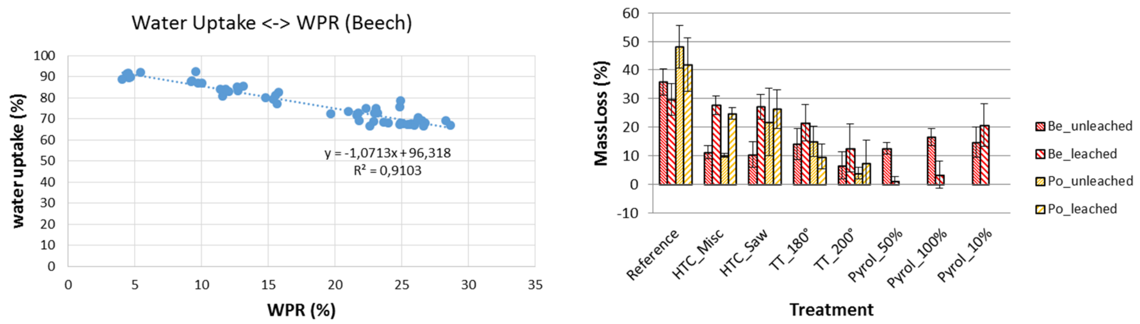

Fig. 3: Reduced water uptake (%; left) in relation to weight-percent-remain (WPR [%]) after leaching of impregnated beech using wood-processing residues (pyrolysis-liquid, liquid wood). Right: Mass loss of beech and poplar wood (%, right) impregnated with native residues from technical processes. Pyrolysis-Liquids (Pyrol) of different dilution degrees with water, TT = Thermal-Treatment residues, 180 rsp. 200°C, HTC solution = Hydrothermal Carbonisation of Miscanthus and Spruce sawdust residues. Residues and process compare Table 1

Fungal Decay and Durability; The mass loss due to fungal decay (*Trametes versicolor*) of native beech (30-35%) and poplar wood (40-50%) was reduced to 2% in the case of pyrolysis treatment and to 4% in the case of TT, depending on the wood species, impregnation method, concentration and leaching process (Fig. 3 right, n = 9). HTC treatment and also TT showed acceptable results only in the case of un-leached samples, whereas leaching resulted in 10-30% decay due to fungal activity. In contrast, the pyrolysis treatment of beech in the concentration of 100% and 50% showed good durability - less than 3% of decay - also in leached samples, resulting in durability classes 2 or even 1 against basidiomycetes, whereas a concentration of 10% was too low. All un-leached pyrolysis samples showed mass losses due to the leaching of exceeding amount of pyrolysis liquids out of cell lumina, which was leached under culture conditions on agar and resulted in a mass loss due to lower weight after the tests.

Conclusions

The results show that increasing demand for additional high-quality wood assortments – following rising environmentally friendly standards – can be delivered by new bio-based processes, producing and applying renewable and sustainable produced wood impregnation agents for various wood modification processes. These innovations allow not only the material wood but also the entire wood

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modification process, can operate as a renewable and sustainable process, excluding more and more fossil sources from the material production and application processes.
Nevertheless, the industrial implementation of natural and renewable produced wood modification or impregnation agents depends on the technical and constitutionally frame conditions. Under the viewpoint of sustainability in nature and production it has to be discussed, compared, balanced, and decided, if the application of defined and tested single modification agents from fossil origin, or the use of an undefined mixture of plant components and their process residues are more confirming with the requirements of sustainability, close to nature approach, environmental and health protection as well as renewability.

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