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How thermal treatment afects the chemical composition and the physical, mechanical and swelling properties of Scots pine juvenile and mature wood

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Abstract

High variations in juvenile wood properties in the radial direction and its worse performance than mature wood make it less suitable for some applications and often treated as waste material. This study aimed to assess how thermal modifcation afects the chemical composition and the physical, mechanical and swelling properties of Scots pine juvenile and mature wood. An additional goal was to evaluate if the modifcation can equalise the diferences in selected properties of juvenile wood to those of mature wood so that from waste material, juvenile wood can become a fully-fedged raw material for various industrial applications. Thermal treatment at 220 °C infuenced wood chemical composition, degrading mainly hemicelluloses but also afecting cellulose and lignin, which resulted in a reduction of hydroxyls and carbonyl/carboxyl groups. These changes were more pronounced for mature than juvenile wood. It reduced mass loss and swelling rate, and increased swelling pressure in the tangential and radial directions to a higher degree for juvenile than mature wood. Changes in mechanical properties in compression were statistically signifcant only for mature wood, while wood hardness remained unafected. Although the applied heat treatment improved the performance of juvenile wood by reducing its swelling rate, it did not equalise the examined properties between juvenile and mature wood. Since higher juvenile wood proportion is expected in the wood supply from the future intensively managed forests, there is still a need to fnd suitable modifcation methods or better processing techniques so that instead of being thrown away as waste, it could be used broadly in various industrial applications.

Extended author information available on the last page of the article

Introduction

Juvenile wood refers to wood formed usually in the frst 10–20 annual rings adjoining the pith. The term comes from the cambial age when this part of the wood was produced. The wood located further from the pith towards the bark is called mature wood (Zobel and Sprague [2012](#page-27-0); Moore and Cown [2017](#page-25-0)). Juvenile and mature wood distribution in a tree trunk is presented in Fig. [1.](#page-1-0)

Juvenile wood is characterised by high variations in wood properties in the radial direction (in the following annual rings). These include anatomical features like cell dimensions, cell wall thickness, microfbril angle in the S2 cell wall layer (MFA), and spiral grain angle, as well as other properties such as chemical composition, density, mechanical properties, susceptibility to internal checking, shrinkage and distortions during drying, latewood percentage content, resin content, and higher incidence of reaction wood. Some properties like MFA, longitudinal shrinkage, resin content and spiral grain angle typically decrease exponentially within the frst 20 annual rings, while others, including cell wall thickness, density, latewood percentage content, modulus of elasticity and modulus of rupture, cell length, mechanical strength, moisture content, and transverse shrinkage increase. Then, in the wood of older cambial age (mature wood), most of these radial alterations between annual rings are more subtle and mainly result from changeable growing conditions (Kretschmann and Cramer [2007](#page-24-0); Moore and Cown [2017\)](#page-25-0).

Several researchers have investigated the variability of wood properties in the radial direction across a broad range of wood species (Bao et al. [2001](#page-23-0); Alteyrac

Fig. 1 Schematic distribution of juvenile and mature wood in a tree trunk and its cross-section (from pith to bark)

et al. [2006](#page-22-0); Jordan et al. [2007](#page-24-1); Mansfeld et al. [2009](#page-25-1); Bal and Bektaş [2013](#page-23-1); Auty et al. [2014;](#page-22-1) Moore et al. [2015a](#page-25-2); Kimberley et al. [2015;](#page-24-2) Sedlar et al. [2019;](#page-26-0) Mania and Nowicki [2020;](#page-24-3) Roszyk et al. [2020](#page-26-1); Arslan et al. [2021](#page-22-2); Sarkhad et al. [2021](#page-26-2); Tumenjargal et al. [2022\)](#page-27-1). Based on that research, the main diferences between juvenile and mature wood properties have been recognised (Table [1\)](#page-2-0).

In general, the properties of juvenile wood difer from those of mature wood, and they are less desirable from a processing and practical application perspective. They strongly impact the performance of end products since it depends on the anatomical, chemical, physical and mechanical properties of wood (Moore and Cown [2017;](#page-25-0) Huang et al. [2021](#page-24-4); Zanuttini and Negro [2021](#page-27-2)). As a result of the radial variability of the wood properties, the end product of juvenile wood is expected to have diferent characteristics from that of mature wood.

Juvenile wood is considered a lower-quality raw material, particularly for solidwood products that require good dimensional stability, high strength and stifness (structural applications), and absence of defects, including resin features, knots and checks (appearance uses) (Moore and Cown [2015;](#page-25-3) Ruano et al. [2022](#page-26-3)). Due to low density and high microfbril angle, juvenile wood has higher longitudinal shrinkage and lower bending strength and modulus of elasticity compared to mature wood (Bhat et al. [2001;](#page-23-2) Adamopoulos et al. [2007](#page-22-3); Ivković et al. [2009;](#page-24-5) Moore et al. [2012;](#page-25-4) Roszyk et al. [2020](#page-26-1)). The resulting poor dimensional stability manifests in inequivalent swelling or shrinkage in diferent anatomical directions under unstable humidity conditions. Moreover, due to the strong gradient in microfbril angle within juvenile wood, the timber is prone to twisting and warping during drying (Danborg [1994;](#page-23-3)

Wood qualities	The characteristics of juvenile wood in relation to mature wood			
Anatomical features	Shorter cells (tracheids) with smaller diameters and thinner cell walls (where a lower contribution of the S2 layer in relation to the double cell wall thickness characterises softwoods)			
Microfibril angle	Higher MFA, with values typically higher for softwoods $(40-50^{\circ})$ than hard- woods $(25-35^{\circ})$			
Spiral grain angle	Higher spiral grain angle typically reaches a maximum in the first few annual rings from the pith and then drops to lower values in mature wood			
Annual rings	Wider growth rings with lower latewood percentage			
Reaction wood	Increased incidence of reaction wood—compression wood in softwoods and ten- sion wood in hardwoods			
Density	Density is typically lower by 10–20%. In some pine species, mature wood can be more than twice as dense as juvenile wood			
	Mechanical properties Lower mechanical strength and modulus of elasticity resulted from lower density and higher microfibril angle			
Shrinkage, distortions	Higher longitudinal shrinkage, which, together with high microfibril angle, spiral grain angle and incidence of reaction wood, increases the tendency for distor- tion			
Chemical properties	Higher lignin content in the cell wall at lower cellulose content. Higher resin content in wood			
Other features	Increased susceptibility to internal checking			

Table 1 Selected properties of juvenile versus mature wood (Moore and Cown [2017\)](#page-25-0)

Wagner et al. [2002;](#page-27-3) Soares et al. [2019](#page-26-4)). All these can cause severe problems for wooden structures, even if they impact only a certain number of timber elements (Barnett and Bonham [2004](#page-23-4); Moore et al. [2009;](#page-25-5) Lachenbruch et al. [2011](#page-24-6)). The only areas for juvenile solid-wood utilisation are interior fnishing and decorative elements, as well as some furniture designs where unique grain patterns are of higher importance than the mechanical strength of wood. Larger cells and lower density can be advantageous by making this part of wood easier to work with and providing an end product with unique aesthetic properties (Paes et al. [2015](#page-25-6); Blackburn et al. [2021](#page-23-5)).

The infuence of juvenile wood on the characteristics of reconstituted panel products (i.e., fakeboards, particleboards and fbreboards) is much smaller than that of solid-wood products. Although the dimensional stability of panels made of juvenile wood was lower compared to those made of mature wood, the mechanical prop-erties were usually similar (Pugel et al. [1990](#page-26-5); Shi et al. [2005](#page-26-6); Pecho et al. [2005;](#page-25-7) Cloutier et al. [2007;](#page-23-6) Moore and Cown [2017\)](#page-25-0). Nevertheless, the proportions of juvenile to mature wood should be limited to ensure the satisfactory performance of end products.

In the case of laminated wood products, such as glulam, cross-laminated timber (CLT), laminated veneer lumber (LVL), and plywood, utilisation of juvenile wood can be possible only as inner layers, while outer zones should be made of a material of higher strength and stifness to mitigate the adverse impact of juvenile wood on the mechanical properties of an end product (Lee et al. [2005;](#page-24-7) Nazerian et al. [2011;](#page-25-8) Hughes [2015](#page-24-8); Brandner et al. [2016;](#page-23-7) Moore and Cown [2017](#page-25-0)).

On the contrary, the pulp and paper industry is an application where juvenile wood can be preferentially used, particularly in the production of fne writing and magazine papers. Lower density and a higher proportion of larger, thin-walled cells make the pulping process easier and thus more economically viable, and the paper has sufficient tensile strength and optical behaviour (Corson and Richardson [2007](#page-23-8); Severo et al. [2013;](#page-26-7) Moore and Cown [2017](#page-25-0)). However, due to generally lower mechanical strength and stability, juvenile wood is unsuitable for manufacturing Kraft pulps that require high tear strength, boards for packaging grades or other paper products such as high-quality printing paper (Corson [1984,](#page-23-9) [1991;](#page-23-10) Myers [2002](#page-25-9); Moore and Cown [2017\)](#page-25-0).

Juvenile wood formation is a natural process that occurs in all tree species. The size of the juvenile wood zone and its properties are afected by environmental, genetic and silvicultural factors (Zobel and Sprague [2012\)](#page-27-0).

The presence of juvenile wood typically did not affect wood processing because the primary raw material resource was old-growth trees from natural forests where the early growth of trees was suppressed by surrounding plants so that the proportion of juvenile wood was small (Larson [2001](#page-24-9); Zobel and Sprague [2012\)](#page-27-0). Over the past few decades, however, genetic manipulations focused mainly on improving tree growth and stem form (Hannrup et al. [2004](#page-24-10); Gapare et al. [2007](#page-24-11); Wu et al. [2008](#page-27-4)), the intensifcation of forestry and the pursuit of more cost-efficient production of raw materials inevitably lead to a substantial reduction in rotation length thus an increase in the proportion of juvenile wood in managed planted forest stands. As a result, although the general productivity gains increase, the general quality of raw material available for the wood industry declines signifcantly (Zobel [1984;](#page-27-5) Zobel and Sprague [2012;](#page-27-0) Antony et al. [2012](#page-22-4); Moore et al. [2015b;](#page-25-10) Zhang et al. [2021](#page-27-6)).

Understanding the impact of juvenile wood on the performance of end products is necessary to devise efective processing strategies to fully utilise forest resources with increasing content of this material for industrial purposes. In order to address the issues caused by juvenile wood, wood processors may need to modify their systems to account for the growing amount of juvenile wood in future resources or modify the performance requirements for their products. These may be achieved by developing new types of wood products that are less adversely afected by juvenile wood drawbacks or using specifc modifcation methods to improve juvenile wood performance (Moore and Cown [2017](#page-25-0)).

One of the common methods for improving wood performance is a thermal modifcation developed to enhance its durability and dimensional stability. Several production plants worldwide utilise this well-established commercial technology in open or closed systems that mainly difer in the medium used to exclude oxygen and the treatment temperature (Sandberg and Kutnar [2016;](#page-26-8) Gérardin [2016](#page-24-12); Hill et al. [2021](#page-24-13)). The thermal modifcation afects wood chemical composition, resulting in the degradation of hemicelluloses, an increase in cellulose crystallinity and changes in amorphous cellulose structure. It also leads to lignin depolymerisation in the frst stage and further auto-condensation by forming new methylene bridges between aromatic rings. The rearrangement of cell wall polymers caused by thermal treatment reduces wood hygroscopicity, which translates into enhanced dimensional stability and biological durability. It increases wood surface hardness but usually reduces its other mechanical properties, such as stifness, bending, shear and compression strength (Tjeerdsma et al. [1998;](#page-26-9) Yildiz and Gümüşkaya [2007;](#page-27-7) Hill [2007](#page-24-14); Hannouz et al. [2015](#page-24-15); Candelier et al. [2016\)](#page-23-11).

In this research, Scots pine wood, one of the most common species grown in managed forests and used for industrial purposes in central Europe, was thermally modifed to assess how thermal treatment afects the selected physical and mechanical properties of juvenile wood compared to mature wood. To better comprehend these changes, the efect of thermal modifcation on the chemical wood composition was investigated using the Fourier Transform Infrared Spectroscopy (FTIR) technique. The results will enable a better understanding of the relations between anatomical features and chemical composition of juvenile wood and its susceptibility to thermal treatment compared to mature wood. It will also help to evaluate if the modifcation can equalise or at least signifcantly limit the diferences in selected properties of juvenile wood compared to those of unmodifed or modifed mature wood so that from waste material, in the past usually discarded in the production process, juvenile wood can become a fully-fedged raw material for various industrial applications.

Materials and methods

Materials

The research material was Scots pine (*Pinus sylvestris* L.) from a typical forest purchased from commercial timber merchants in the Wielkopolska Region, Poland.

Methods

Sample preparation

Wood samples for individual experiments were cut from pine plank according to the scheme presented in Fig. [2.](#page-5-0)

Determining the transition from juvenile to mature wood

Selection of juvenile and mature wood zones for the study was based on the length of tracheids, the width of annual rings and the percentage content of latewood (Clark et al. [2006;](#page-23-12) Mansfeld et al. [2016;](#page-25-11) Chen et al. [2016](#page-23-13)).

The tracheid length was measured in wood macerates of individual annual rings using a digital microscope 4 K Keyence VHX-7000N (KEYENCE, Osaka, Japan) with an accuracy of 0.01 mm. To prepare wood macerates, early- and latewood zones from selected annual rings were collected in separate weighing bottles and poured with a maceration mixture consisting of equal parts of glacial acetic acid and 30% hydrogen peroxide. Maceration was conducted at 60 °C for 48 h. Then, the

Fig. 2 A scheme for the preparation of juvenile (J) and mature (M) wood samples used for the measurements of wood swelling pressure and kinetics and FTIR analysis (**a**), and mechanical properties (1, 2, 3 –points for wood hardness measurements in the longitudinal, tangential and radial direction, respectively) (**b**); C–control wood (control), T–thermally-treated wood; all dimensions given in mm

macerated wood anatomical elements were washed with water and measured (Fabisiak and Fabisiak [2021\)](#page-23-14). The measurements were taken for every three annual rings from the 3rd to the 51st and every fve rings from the 51st to the 86th. Thirty tracheids were measured in both early- and latewood from each annual ring, and then average tracheid length values were calculated for each.

The width of individual annual rings and latewood zones were measured using a BIOtronik® BEPD-19 (BIOtronik, Warsaw, Poland).

Thermal modifcation

Juvenile and mature wood samples were subjected to thermal treatment according to the industrial method described in the ThermoWood® Handbook (Lahtela [2021\)](#page-24-16). The process consisted of an initial phase in moist air followed by heating in superheated steam after the temperature reached 130 °C, a maximum heating phase (temperature of 220 \degree C and duration time of 3 h), and a cooling phase with superheated steam followed by cooling in moist air only. Before modifcation, wood samples were dried to an oven-dry state to avoid hydrolysis of the material during the thermal treatment (Fengel and Wegener [2011](#page-23-15); Rautkari and Hill [2014](#page-26-10)). The mass loss of thermally modifed wood was determined in relation to the oven-dry mass of wood before the modifcation process.

Infrared spectroscopy

Fourier Transform Infrared Spectroscopy was used to analyse the wood chemical composition and investigate the changes caused by thermal treatment in both juvenile and mature wood. The samples were powdered and sieved, and the powder fraction particles with a diameter of $\langle 0.2 \text{ mm}$ were retained for the analysis. Infrared spectra were recorded in the 4000–400 cm−1 region with a resolution of 4 cm−1, using a Bruker ALPHA FT-IR spectrometer (Bruker, Billerica, MA, USA) in KBr pellets. Both sample powder and KBr were carefully weighted, using 3 mg wood powder and 200 mg KBr for each pellet. For each sample, five spectra were recorded, averaged and processed using OPUS 7.5 software. The second derivatives were calculated using Savitsky-Golay method with 21 points. For principal component analysis (PCA) and hierarchical cluster analysis (HCA), the 1550–840 cm⁻¹ region from the FTIR spectra was used. The processing was done in the Origin 2023b program (OriginLab Corporation, Northampton, MA, USA).

Physical properties

Wood density and moisture content Wood samples were conditioned for two weeks at a temperature of 23 ± 2 °C and air relative humidity of $35 \pm 3\%$ before all measurements. The density of seasoned samples before and after treatment was determined by employing a stereometric method according to ISO 13061-2 (ISO 13061-2 [2014\)](#page-24-17). Wood mass and dimensions were measured using an analytical balance with an accuracy of 0.001 g (Sartorius GmbH, Göttingen, Germany) and a digital calliper with an accuracy of 0.01 mm, respectively.

Wood moisture content after conditioning was determined using a standard ovendrying method at 103 ± 2 °C and calculated as a ratio of the mass of water (a difference between the mass of wood after conditioning and at a completely dry state) to the mass of a dry sample.

Additionally, part of the samples intended for swelling measurements were ovendried at a temperature of 103 ± 2 °C to a constant weight, and their density at a completely dry state was calculated based on dry masses and dimensions.

Kinetics of radial and tangential swelling and wood swelling pressure Wood swelling is an increase in dimensions caused by increased bound water content. The maximum degree of linear swelling is an increase in wood dimensions caused by an increase in moisture from a completely dry state to the fbre saturation point and is expressed as a percentage of wood dimensions in a completely dry state. Since wood is an anisotropic material, it swells diferentially in diferent anatomical directions. Generally, from dry to wet state (fbre saturation point), wood swells about 10% in the tangential direction, 5% in the radial directions, and only 0.1% in the longitudinal direction (Mantanis et al. [1994](#page-25-12); Arzola-Villegas et al. [2019](#page-22-5)).

Wood swelling in the tangential and radial direction was measured for control and treated juvenile and mature wood samples with dimensions of $30 \times 30 \times 10$ mm in the radial (R), tangential (T) and longitudinal (L) directions, respectively. Six replicates with similar density and the pattern of annual rings per variant were used for the measurement. Before the experiment, wood samples were oven-dried at a temperature of 104 °C to a completely dry state. Each specimen was placed in a support stand equipped with a fatbed micrometre so that the measurement direction was tangential or radial in relation to the wood anatomical directions. Then, the stands with samples were placed in water, and the measurements started. The micrometres indications were recorded at specifed intervals. For each interval, the degree of swelling was calculated using the following equation:

$$
\alpha = \frac{\Delta l}{l_0} \times 100
$$

where α is the degree of swelling, Δl is the dimension increase in the measured direction, and l_0 is the dimension of a completely dry sample in the direction of measured swelling.

Swelling pressure is the maximum stress in wood during restrained swelling, or, in other words, the stress required to prevent the wood from swelling completely (Perkitny and Kingston [1972](#page-25-13); Mazzanti et al. [2014](#page-25-14)).

The experiment was performed on the prototype device specially designed for this purpose (Molinski and Raczkowski [1980](#page-25-15)), allowing for the measurement of the total force needed to retract the permitted linear swelling. Swelling pressure of completely dry control and treated juvenile and mature wood was measured in the radial and tangential directions following the procedure described before (Roszyk et al. [2024](#page-26-11)), using three replicates of each variant per direction (sample dimensions were $30\times30\times10$ ($R\times T\times L$, like in the experiment described above). Directly before the measurement, each sample was preloaded to eliminate contact deformations. Swelling pressure was calculated as the quotient of the swelling force and the crosssectional area of a completely dry specimen according to the following equation:

$$
\sigma cp = \frac{P}{A}
$$

where $\sigma c p$ is the swelling pressure (in MPa), *P* is the value of swelling force (N), and *A* is the cross-sectional area of the sample in a completely dry state.

Mechanical properties

Compression tests and the Brinell hardness method were used to determine the selected mechanical properties of unmodifed and modifed juvenile and mature pine wood. Methods selection was based on the fact that thermally modifed wood is often used as a fooring material; therefore, its hardness (on which the abrasion resistance depends) and compression strength are the most crucial mechanical parameters. Moreover, since juvenile and mature wood difering in density are the research materials, the compression tests were chosen because of the highest relation between wood density and strength from all strength testing methods. Also, due to material restrictions related to the use of juvenile wood and a thermal modifcation performed at a laboratory scale, the mechanical tests that require as small samples as possible were selected to ensure enough replicates of similar wood specimens to obtain reliable results.

The data obtained were statistically analysed using STATISTICA 13.3 software (TIBCO Software Inc., Palo Alto, CA, USA). One-way analysis of variance (ANOVA) followed by a post-hoc Tukey's honest signifcance test was applied to fnd means that are signifcantly diferent from each other (signifcance was established at $p < 0.05$).

Compression tests Compression tests in the longitudinal direction (along the grain) were performed using a numerically controlled test machine, Zwick Z050TH (Zwick/ Roell, Ulm/Germany). Wood specimens with dimensions $20 \times 20 \times 30$ mm ($R \times T \times L$) were used in the study, with eight replicates of each variant. The modulus of elasticity (MOE), stress at proportionality limit (σ_{pl}), and stress to failure (so-called compressive strength—R c_L) were determined. Additionally, specific modulus of elasticity (sMOE), specific stress at proportionality limit ($s\sigma_{pl}$) and specific stress to failure (sRc_L) were calculated as the MOE, σ_{pl} , and Rc_L to wood density ratios, respectively.

Brinell hardness The hardness of unmodifed and modifed juvenile and mature wood in all three anatomical directions was determined using the Brinell method (EN 1534 [2000](#page-23-16)). Eight replicates were used per variant. The measurements were performed on an Amsler universal testing machine (Amsler UTM). A spherical steel indenter with a diameter of 10 mm was pressed against the sample surfaces (Fig. [2b](#page-5-0)) with an increasing force to 500 N for 15 s, then the maximum load of 500 N was kept for the next 30 s, and fnally, the indenter load was reduced to its initial value during the next 15 s.

The minimum and maximum indentation diameters were measured with a Brinell magnifer with an accuracy of 0.1 mm. Then, the averaged values were used to calculate hardness (HB) following the equation:

$$
HB = \frac{2F}{D\pi(D - \sqrt{D^2 - d^2})}
$$

where F is the loaded force (N) , D is the diameter of the pressed indenter (mm), and *d* is the average measured indentation (mm).

Results

Anatomical characteristics of juvenile and mature pine wood

Figure [3](#page-10-0) presents a set of anatomical parameters measured for studied pine wood. An apparent decrease in the width of annual rings (Fig. [3C](#page-10-0)) and an increase in the length of tracheids (Fig. [3](#page-10-0)A and B) and percentage content of latewood (Fig. [3](#page-10-0)D) in successive annual rings can be seen to about ffteenth increment, and then the values stabilise. Based on the graphs in Fig. [3](#page-10-0) and the fact that juvenile wood difers from mature wood by the values of the presented parameters and their gradient concerning the ring number and distance from the pith (Moore and Cown [2017](#page-25-0)), the transition zone between juvenile and mature wood was defned as 15th annual ring.

FTIR

Thermal modifcation resulted in wood mass loss of 6.6% for juvenile (J) and 5.1% for mature (M) wood. Alterations in the wood chemical composition were characterised using the FTIR technique. Infrared spectra and their corresponding second derivatives for the control and thermally modifed juvenile and mature wood are presented in Figs. [4](#page-10-1) and [5](#page-11-0). Figure [4](#page-10-1) shows the $3750-2730$ cm⁻¹ region, assigned to diferent stretching vibrations of hydroxyl groups and hydrogen (H) bonds, as well as to methyl and methylene groups from the wood structure (Popescu et al. [2011;](#page-25-16) Torniainen et al. [2021\)](#page-26-12). Due to the thermal treatment of mature wood, the large band from 3430 cm−1 shifted to 3416 cm−1. It was caused by modifcations appearing in the component bands, namely: the band from 3576 cm−1 (in MC spectrum), assigned to absorbed water weakly bound and intramolecular H bond in a phenolic group (in lignin), is shifted to 3569 cm⁻¹ in MT spectrum and decreased in intensity; the band from 3443 cm^{-1} , assigned to O2–H2⋯O6 intramolecular H bonds stretching modes, presents a slight decrease in intensity; the bands from 3336 and 3263 cm^{-1} (in MC spectrum), assigned to O5–H5⋯O3 intramolecular H bonds in cellulose and to O6–H6⋯O3 intermolecular H bonds in cellulose I_β , are shifted to 3342 and 3274 cm⁻¹ in MT spectrum and increased in intensity; while the band from 3211 cm^{-1} (in MC spectrum) assigned to O6–H6…O3 intermolecular in cellulose I_{α} is shifted to 3219 cm⁻¹ in

Fig. 3 Average values of tracheid length in earlywood (**A**) and latewood (**B**), ring width (**C**), and latewood content (**D**) in the following annual rings counted from pith to bark; green dots represent juvenile wood, brown dots—mature wood (color figure online)

Fig. 4 Infrared spectra and their second derivatives for the control (C) and thermally treated (T) mature (M) and juvenile (J) pine wood in the 3750–2700 cm−1 region

MT spectrum (Kondo [2005](#page-24-18); Popescu et al. [2011\)](#page-25-16). These modifications are wellvisible from the second derivative of the infrared spectra (see Fig. [4A](#page-10-1)).

Figure [4](#page-10-1)B shows that the modifcations appearing in the juvenile wood spectra following thermal treatment are less pronounced than for mature wood. In this case, the large band from 3426 cm⁻¹ in the JC spectrum is shifted to 3421 cm⁻¹ in the JT spectrum. From the second derivative spectra of juvenile wood, the following modifications were identified: the band from 3567 cm⁻¹ (in JC spectrum) is shifted to 3558 cm^{-1} (in JT spectrum) and shows a slight increase in intensity after thermal treatment; the band from 3342 cm^{-1} (in JC spectrum) is shifted

Fig. 5 Infrared spectra and their second derivatives for the control (C) and thermally treated (T) mature (M) and juvenile (J) pine wood in the 1850–800 cm−1 region

to 3345 cm−1 and increases in intensity (in JT spectrum); while the band from 3262 cm^{-1} (in JC spectrum) is shifted to 3273 cm^{-1} (in JT spectrum).

Table [2](#page-12-0) shows hydrogen bond energies for each O–H stretching band calculated using Struszczyk's formula (Struszczyk [1986](#page-26-13)) and hydrogen bonding distances calculated based on the equation given by Pimentel and Sederholm ([1956\)](#page-25-17).

The calculated energies vary between the control mature and juvenile wood, as well as between control and thermally treated wood. The hydrogen bonding energy for the band assigned to absorbed weakly bound water and an intramolecular hydrogen bond in a phenolic group (in lignin) is higher in JC compared to MC and increases after thermal treatment for both types of wood compared to control wood. A similar trend is observed for the energies of the O2–H2⋯O6 intramolecular and O6–H6…O3 intermolecular hydrogen bonds in cellulose $I_β$ hydrogen bonds (which give bands at about 3440 and 3270 cm⁻¹). All other types of hydrogen bonds present higher energies for the mature wood than the juvenile one. Analysing the thermally treated mature (MT) and juvenile (JT) wood, the energies are increasing for the

Samples	Hydrogen bonding energy (E_H) (kJ/mol)							
	3570 cm^{-1}	3440 cm^{-1}	3380 cm^{-1}	3340 cm^{-1}	3270 cm^{-1}	3215 cm ⁻¹		
МC	5.32	14.89	19.34	22.58	27.83	31.57		
MT	5.82	14.89	19.42	22.15	27.04	30.99		
JC	5.97	15.03	19.20	22.15	27.90	31.35		
JT	6.62	15.32	19.49	21.93	27.11	31.14		
Samples	Hydrogen bonding distance (R) (\dot{A})							
	3570 cm^{-1}	3440 cm^{-1}	3380 cm ⁻¹	3340 cm ⁻¹	3270 cm^{-1}	3215 cm^{-1}		
MC	2.8346	2.8045	2.7905	2.7804	2.7639	2.7522		
MT	2.8330	2.8045	2.7903	2.7817	2.7664	2.7540		
JC	2.8325	2.8041	2.7910	2.7817	2.7637	2.7528		
JT	2.8305	2.8032	2.7901	2.7824	2.7662	2.7535		

Table 2 The energy of the hydrogen bonds and the hydrogen bonding distance calculated for the studied samples

hydrogen bonds, which give bands at about 3570, 3440, and 3380 cm⁻¹ and decrease for the other three. Variations in the energy values indicate diferent positions of the specifed bands in juvenile wood compared to mature one, both before and after the thermal treatment. A decrease or increase of the hydrogen bonding energy is due to a shifting of the corresponding vibration bands to higher or lower wavenumber. The values of the hydrogen bonding distance vary similarly to the energies, although the variation is very low. An increase in the hydrogen bonding energy may mean a more compact structure, higher bonding energy induces higher activation energy for those bonds to break.

Further modifcations were also observed in the fngerprint region between 1850 and 830 cm⁻¹ (see Fig. [5\)](#page-11-0). Figure [5A](#page-11-0) presents the spectra and their second derivatives of the control (MC) and thermally treated (MT) mature wood. In this region, the modifcations observed are mainly assigned to C=O, C–O and C–O–C groups. The band from 1738 cm⁻¹ (in MC spectrum), assigned to C=O stretching vibration of carboxyl and acetyl groups in hemicelluloses (Popescu et al. [2011;](#page-25-16) Torniainen et al. 2021), decreases in intensity and is shifted to 1734 cm⁻¹ (in MT spectrum). The band from 1659 cm⁻¹, assigned mainly to absorbed O–H from water molecules (Popescu et al. [2011;](#page-25-16) Torniainen et al. [2021](#page-26-12)), decreases in intensity in the MT spectrum. Further, the bands from 1424, 1373, 1323, 1163, 1113, and 1066 cm^{-1} assigned to C–H deformation and stretching vibration in lignin and carbohydrates, C_l –O stretching vibration in syringyl derivatives, and C–O–C stretching vibration in cellulose and hemicelluloses show slight increase in intensity, while the bands from 1267, 1226 and 949 cm−1 assigned to C–O stretching vibration in lignin, C–O–C stretching vibration mode of the pyranose ring and C–O and C–C stretching vibration of the ring in cellulose and hemicelluloses decreases in intensity in thermally treated sample spectrum (Faix and Böttcher [1992;](#page-23-17) Pandey and Pitman [2003](#page-25-18); Popescu et al. 2011). At the same time, the bands from 1226, 1066, 992 and 949 cm⁻¹ (in

MC spectrum) assigned to C–O–C stretching vibration mode of the pyranose ring, C–O stretching vibration mainly from C(3)–O(3)H in cellulose I and C–O and C–C stretching vibration of the ring in cellulose and hemicelluloses are shifted to 1221, 1060, 989 and 943 cm−1 (in MT spectrum) (Popescu et al. [2011;](#page-25-16) Torniainen et al. [2021](#page-26-12)).

Figure [5](#page-11-0)B presents the spectra and their second derivatives of the control and modifed juvenile wood in the 1850–830 cm−1 region. In this case, a decrease in intensity of the band from 1736 cm−1 with slight shifting to 1734 cm−1 was observed for the JT sample. As in the previous case, the band from 1656 cm⁻¹ (in JC spectrum) decreases in intensity and is shifted to 1649 cm^{-1} (in JT spectrum), as well as the bands from 1270, 1221 and 949 cm⁻¹ (in JC spectrum) decrease in intensity following the thermal treatment. At the same time, the bands from 1373, 1114 and 1065 cm−1 increase in intensity in the JT spectrum compared to the JC one. No real shift towards a lower or higher wavenumber of the bands' maxima was identifed in this case.

In order to evidence the variations between the lignin and carbohydrates in the studied samples, the ratios of the integral area of the band from 1510 cm⁻¹, characteristic to lignin (as belongs purely from C=C aromatic skeletal stretching vibration), against diferent carbohydrate characteristic bands from 1730, 1373, 1155 and 895 cm⁻¹ (assigned to C=O stretching vibration of carbonyl, carboxyl and acetyl groups, C–H deformation in carbohydrates and C–O–C stretching vibration in carbohydrates) were calculated—see Fig. [6](#page-14-0).

The intensity ratio of the lignin characteristic band from 1510 cm⁻¹ against different carbohydrate characteristic bands indicates variations between the mature and juvenile wood as well as between control and thermally modifed wood. An increase in the intensity ratio means increased lignin values compared to carbohydrate-specific groups. For example, the value of the I_{1510}/I_{1730} from mature wood, both control and thermally treated, is smaller when compared to the juvenile wood ratio value, but at the same time, it decreases for JT compared to JC. A similar trend is also observed for the I_{1510}/I_{1373} ratio, while the I_{1510}/I_{895} ratio presents different trends the value for MT is much higher than for the MC, the value for JC is higher than that for MC, but the values of JT is lower than that for MT. This shows that the variation of the C–O–C bond in juvenile wood due to the treatment is lower than in mature wood.

Principal component analysis (PCA) gives detailed information about the difer-ences which may appear between series of similar wood samples (Chen et al. [2010;](#page-23-18) Popescu et al. [2013,](#page-25-19) [2020](#page-26-14)); thus, the chemical changes or chemical diferences in the wood structure of mature and juvenile wood and their physical properties as a result of structural diferences and thermal treatment are refected in both PC scores and loadings. In Fig. [7A](#page-14-1), the plot of PC1 (principal component factor (1) versus PC2 (principal component factor (2) is presented. PC1 describes 98.4%, and PC2 describes 1.1% of data variance; therefore, the variances existing in the wood spectra can be captured using these two dimensions instead of the initial spectra.

The JC samples presented positive values on PC1 and PC2, while MC presented negative values for PC1 and close to zero for PC2. Thermally treated mature wood (TM) presents negative values for both PC1 and PC2, while JT presents PC2 values **Fig. 6** Variation of the intensity ratio of lignin characteristic band from 1510 cm^{-1} against diferent carbohydrate characteristic bands from 1730, 1373, 1155 and 895 cm^{-1} for control (C) and treated (T) juvenile (J) and mature (M) pine wood

close to 0 and slightly positive values of the PC1 score. From here, we can conclude that PC1 is the most informative latent variable for the description of chemical differences between mature and juvenile wood, while the PC2 score separates mainly the modifed samples from the control ones. The loading plots (Fig. [7](#page-14-1)B) indicate the chemical features responsible for the grouping of the samples along the PC1 and PC2. They can be used to understand how much each wavenumber contributes to the meaningful variation in the data and to interpret variable relationships. Thus, PC1 indicates variations in the whole evaluated region between the mature and juvenile wood, while PC2 presents positive values mainly for lignin bands and negative ones for carbohydrates. Positive bands show an increase in lignin content, while the negative ones indicate a decrease in hemicelluloses content after the thermal treatment.

In addition to PCA, a hierarchical cluster analysis (HCA) was performed, which allowed us to emphasize the natural grouping in the data set and made it possible to visualise the relationships among diferent groups (Chen et al. [2010](#page-23-18); Popescu et al. [2013](#page-25-19), [2020](#page-26-14)).

Fig. 7 PC1 versus PC2 scores plot (**A**) and loading plots (**B**) of the control (C) and treated (T) juvenile (J) and mature (M) pine wood

The dendrogram of the analysed wood samples is presented in Fig. [8](#page-15-0), showing two main clusters: the MC is in the frst one, and the other three samples are in the second one. The second cluster is divided into two other sub-clusters, separating the MT and JC samples from the JT samples. A third sub-cluster separates the MT samples from the JC samples. As observed from the PC scores plot, it was a higher diference between the MC and the other three samples, and further, JT is separated from the other two samples as well.

During thermal modifcation of wood in moist conditions (depending on water quantity in the surrounding environment), the degradation reactions take place faster than in dry conditions. At the same time, the hydrolysis reactions occur before the oxidation ones. The water molecules act as a catalyst for the activation of the reactions—hydronium-catalysed reactions, with the further generation of acetic acid due to the hydrolysis of acetyl groups from hemicelluloses (Garrote et al. [2001](#page-24-19); Popescu et al. [2021\)](#page-26-15). These induce modifcations mainly in hemicelluloses but also in lignin and cellulose to some extent and lead to the formation of saturated and unsaturated low-molecular compounds. Moreover, the presence of water molecules can prevent oxidation reactions in favour of hydrolysis. A certain amount of newly formed low-molecular compounds, which vary in quantity and composition, remains trapped in the wood structure.

The modifcations observed in the spectra of the mature and juvenile wood indicate the reduction of hydroxyl, as well as the amount of carbonyl/carboxyl groups and an apparent increase of C–O and C–O–C groups from lignin and carbohydrates. The latter can also be due to the presence of these groups in the newly formed low-molecular compounds trapped in the wood structure.

Swelling properties

Kinetics of radial and tangential swelling

The rate and extent of radial and tangential wood swelling are presented in Fig. [9.](#page-16-0) Generally, for all wood variants, the values of linear swelling were higher in the tangential direction than in the radial direction, which is consistent with the current state of knowledge (Patera et al. [2013](#page-25-20); Arzola-Villegas et al. [2019\)](#page-22-5). The tangential swelling of mature wood was higher than that of juvenile wood, but their radial swelling was similar. However, mature wood swelled faster. Thermal treatment considerably reduced the extent of wood swelling and its rate for both wood types measured in both anatomical directions.

Swelling pressure

The maximum swelling pressure recorded for studied pine wood is presented in Fig. [10](#page-17-0). Generally, the stress needed to restrain swelling was higher in the T than in the R direction. However, it cannot be explained by unrestricted wood swelling of similar order, as described above (see Sect. "[Kinetics of radial and tangential](#page-16-1) [swelling"](#page-16-1).), because it is known from literature that wood swelling pressure has no direct correlation with the rate of its moisture-induced deformations (Rybarczyk and Ganowicz [1974](#page-26-16))—the smallest wood deformations are observed along the grains while swelling pressure in this direction is much higher than across the grains, for which moisture-induced deformations are much more pronounced. For JC and MC samples, swelling pressure was similar in the R direction, but in the T direction, it was higher for mature wood. Thermal treatment increased wood swelling pressure in

Fig. 9 Kinetics of linear swelling in tangential and radial directions measured for control (C) and thermally modifed (T) juvenile (J) and mature (M) pine wood in tangential (T) and radial (R) directions

Fig. 10 Maximum swelling pressure in tangential and radial directions measured for control (C) and thermally modifed (T) juvenile (J) and mature (M) pine wood; error bars represent the standard deviation

all cases. For juvenile wood, however, the increase was higher than for mature wood, and it was 40% in T and 70% in R compared to 5% in T and 24% in R for mature wood. It is important to note that juvenile wood swells much slower than mature wood (about 7.5 times), and thermally treated wood swells slower than control one (about 3 times for juvenile and 5 times for mature wood). Maximum swelling pressure was recorded after 13 min (R) and 17 min (T) for JC, 45 min (R) and 48 min (T) for JT, 3 min (R) and 2 min (T) for MC, and 16 min (R) and 14 min (T) for MT.

Mechanical properties

Table [3](#page-18-0) shows the mechanical parameters of pine wood measured in the compression test. Although there are diferences in moisture content between control and thermally treated samples, they do not alter the mechanical parameters much since they all remain in the range of low values (Côté and Kollmann [1984](#page-23-19)); therefore, they can be neglected in the discussion. The modulus of elasticity (MOE) for JC was 27% lower than for MC. Thermal modifcation reduced their values for both wood types, but the decrease was less pronounced for juvenile wood (about 10%) than mature wood (27%). The reduced MOE of MT was similar to the initial MOE of JC. No statistically signifcant diferences were observed between JC, JT and MT MOE values. Considering the specifc MOE (independent of wood density) of analysed samples, thermal treatment reduced the elasticity of mature wood, but that of juvenile wood was left unaltered.

Stress at the proportionality limit (σ_{pl}) was slightly higher for JC compared to MC. The thermal modifcation increased its value for both wood types, and the change was again more pronounced for mature wood (an increase of 31% for MT vs 20% for JT). The specific σ_{nl} was also higher for JC than MC, and thermal treatment changed its value by 32% for JT and 38% for MT.

Compressive strength (RcL) for JC was lower than for MC. Thermal modifcation increased its values by 2% for juvenile and 7% for mature wood, but that increase was not statistically signifcant. Interestingly, the treatment increased the specifc RcL for both wood types by 13%.

Wood hardness along the grains (Table [4](#page-19-0)) was higher than in the tangential and radial directions; hardness values for individual directions were similar for juvenile and mature wood. Thermal treatment slightly reduced wood hardness in both directions, but diferences were not statistically signifcant.

Discussion

Thermal modifcation of juvenile and mature pine wood afected all the measured parameters, from chemical composition through swelling behaviour to mechanical properties, showing correlations between them.

In our research on pine wood, we observed a slightly higher mass loss (by 1.5 percentage points) and a higher reduction in wood swelling in the tangential and radial directions (by about 44% for J vs 37–39% for M) for juvenile than mature wood, despite less pronounced changes in its chemical composition. It is known that the degradation of cell wall polymers caused by heat treatment mainly involves the least thermally stable hemicelluloses, but cellulose and lignin can also be afected, depending on the applied temperature and treatment conditions (Hill et al. [2021;](#page-24-13) Popescu et al. [2021](#page-26-15)). It results in wood mass loss, a decrease in density, and also a reduced number of hydroxyl groups accessible to water, which are present in wood polymers, most abundantly on hemicelluloses (Repellin and Guyonnet [2005](#page-26-17); Hill et al. [2021](#page-24-13)). Heat treatment also causes a reduction in wood swelling. It is usually attributed to the degradation of hemicelluloses and includes a decrease in hemicellulose absorption sites and also structural modifcations in the cell wall due to hemicellulose destruction. However, it is suggested that structural alterations of the cell wall due to hemicellulose degradation, along with chemical changes of lignin, also play an important role in swelling reduction (Repellin and Guyonnet [2005](#page-26-17)). Summarising, the results obtained show that the correlation between wood chemical composition, mass loss, and swelling is complex and also afected by other factors, e.g., structural features. They also suggest that in the case of juvenile and mature pine wood, the changes in wood swelling behaviour caused by thermal treatment may be

a,b,cDifferent superscripts denote a statistically significant $(p < 0.05)$ diference between mean values according to Tukey's honest signifcant diference (HSD) test

less attributable to alterations in wood chemical composition than to other factors, including mass loss and structural changes in the cell wall.

Thermal treatment also afected the measured mechanical parameters of juvenile and mature pine wood, such as MOE in compression, compressive strength, stress at proportionality limit (in the longitudinal direction), and hardness along and across the grains (Tables [3](#page-18-0) and [4](#page-19-0)). However, the observed diferences between control and treated wood were statistically important only in the case of MOE, σ_{pl} and RcL values for mature wood. It may suggest that the observed changes result mainly from the alterations in the wood chemical composition, which was much more pronounced for mature than juvenile wood. It is known that wood mechanical properties depend on several factors, including chemistry and the arrangement of individual polymers in the cell wall (Salmén et al. [2016;](#page-26-18) Gaff et al. [2019](#page-24-20)). Although the arrangement of the main structural wood polymers in the cell wall has still not been fully established, the results obtained using various techniques suggest that glucomannan and xylan from hemicelluloses chains are associated to the cellulose microfbrils while lignin flls the remaining spaces between the cellulose/hemicellulose aggregates and acts as an independent entity (Salmén [2022\)](#page-26-19). Cellulose and the arrangement of cellulose microfbrils are considered responsible for the strength of wood fbre, particularly in the longitudinal direction, and cellulose degradation leads to a reduction in its macro-strength properties (Sweet and Winandy [1999;](#page-26-20) Salmén et al. [2016\)](#page-26-18), while hemicelluloses contribute to wood elasticity (Berglund et al. [2020](#page-23-20)). Lignin is thought to afect wood compression stifness and take part in stress transfer in the cell wall (Salmén et al. [2016;](#page-26-18) Özparpucu et al. [2019;](#page-25-21) Serra-Parareda et al. [2020\)](#page-26-21). The recent results of the research where wood polymers were selectively removed from the cell wall suggest that the complementary action of fexible cellulose and rigid lignin is responsible for compressive response of wood; hemicelluloses seem to enhance the interactions between cellulose and lignin and stabilise wood structure without a signifcant impact on wood compression behaviour (Kurei et al. [2024](#page-24-21)). In the case of thermally treated mature pine wood, where chemical changes due to the modifcation were more pronounced than in juvenile wood, the signifcantly reduced hemicellulose content and structure, along with increased relative lignin content and structure alterations, contributed to the signifcant decrease in its elasticity, which was refected in much lower MOE value for MT than MC. The efect of increased lignin content on lowering MOE value has already been observed by other researchers (Xi [2018\)](#page-27-8). Slightly increased values of compressive strength and stress at proportionality limit along the grain after heat treatment for mature wood may result from an increase in the relative amount of lignin and crystalline cellulose due to the degradation of hemicelluloses and amorphous cellulose and the increased crosslinking of lignin acting as a stifener for cellulose microfbrils (Sweet and Winandy [1999](#page-26-20); Boonstra et al. [2007\)](#page-23-21). Since chemical changes in thermally-treated juvenile wood were much smaller, their efect on wood mechanical parameters was not that distinct.

Wood hardness across and along the grains remained unafected by the applied treatment (Table [4](#page-19-0)). It is known that wood hardness is determined by various factors, including grain direction, lignin content, wood density and moisture content (Hansson and Antti [2006;](#page-24-22) Xi [2018\)](#page-27-8). In our research, the combination of various changes caused by thermal treatment in wood chemistry and structure apparently led to maintaining the hardness values recorded for control wood.

It may seem surprising that thermal modifcation caused the growth of maximum swelling pressure in wood, particularly for juvenile samples (Fig. [10](#page-17-0)). Wood swelling pressure is considered a positive function of MOE (Keylwerth [1962](#page-24-23)) but is not directly linked with the rate of moisture-induced deformations (Rybarczyk and Ganowicz [1974](#page-26-16)). In our case, we cannot relate the obtained values of swelling pressure to recorded MOE values because they were measured in diferent anatomical directions. However, considering the fact that water interactions with wood polymers during absorption depend on the elastic energy needed to distort polymers in the cell wall (Bertinetti et al. [2013](#page-23-22); Arzola-Villegas et al. [2019\)](#page-22-5) and that "...the potential work of swelling originates from the elastic energy stored in the rigid structure…" of polymers (Nishiyama [2023\)](#page-25-22), it can be stated that the composition, structure and thermodynamics of wood polymers building the cell wall play an important role in creating swelling pressure in wood (Nishiyama [2023](#page-25-22)). In juvenile wood, the changes in chemical composition caused by thermal treatment were less pronounced than for mature wood. Despite this, its swelling pressure increased more than that of mature wood. Since we do not know the details about the quantitative and qualitative chemical composition of control and treated juvenile and mature pine wood and the efect of the chemical changes on the nanostructure of the cell wall, further more detailed studies in those areas are required to gain more knowledge and explain the phenomenon of the increased swelling pressure of heat-treated wood.

Conclusion

In this research, Scots pine wood of diferent cambial ages was thermally modifed to evaluate if the modifcation could improve the properties of juvenile wood and make its selected properties equal to those of mature wood so the modifed juvenile wood could be used as a fully-fedged raw material for industrial applications.

The results show that thermal treatment caused multiple changes in juvenile and mature pine wood, but the extent of these alterations difered between juvenile and mature wood. Mass loss and the decrease in the swelling rate in the tangential and radial directions were higher for juvenile wood. Swelling pressure increased for both wood types but was more distinct for juvenile wood. Changes in chemical composition due to the heat treatment included reducing carbohydrate content (particularly hemicelluloses) and decreasing the number of hydroxyls. The alterations were more pronounced for mature wood, making it more similar to juvenile both control and treated wood than to control mature wood. Also, more distinct alterations in the mechanical parameters measured in compression were observed.

Based on the above, although the applied thermal modifcation did not afect much the mechanical performance of juvenile samples while efectively reducing the swelling rate of both studied wood types, making them better suited for use under changeable moisture conditions, it is an inappropriate method to equalise or limit the diferences in examined properties between juvenile and mature wood. The results point to the conclusion that structural features may play an important role in

the changes in parameters measured; therefore, to gain more detailed knowledge in this area, we plan to extend research on other wood species, including deciduous species, both ring- and difuse-porous. Since the results also suggest that changes in wood chemical composition afect wood mechanical properties to a greater extent than its swelling behaviour, these may provide valuable guidance for fnding more accurate modifcation methods to improve juvenile wood performance so it could be used along with mature wood of the same species for a broader range of industrial applications.

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Declarations

Confict of interest On behalf of all authors, the corresponding author states that there is no confict of interest.

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