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Influence of Macrostructural Parameters on the Physico-Mechanical Properties and Post-Recovery of Densified Scots Pine Wood

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This study investigates the effects of macrostructural parameters, particularly latewood proportion, on the physico-mechanical properties and post-recovery behaviour of Scots pine (*Pinus sylvestris* L.) wood subjected to partial delignification and radial densification. The latewood content ranged from 22.56% to 48.25%. Delignification resulted in a 27.1% reduction in lignin content, which decreased from 28.4% to 20.7%. Densification effectively doubled the wood's density, which reached 1056–1168 kg·m⁻³ in the delignified-densified (DD) group and 1008–1117 kg·m⁻³ in the delignified-furfurylated-densified (DFD) group. Brinell hardness increased from 16.06–24.99 N·mm⁻² in the controls to 87.85–145.27 N·mm⁻² (DD) and 68.65–103.54 N·mm⁻² (DFD). Specimens with higher earlywood content exhibited greater compressibility and densification. While furfurylation slightly decreased maximum density and hardness, it significantly enhanced dimensional stability, reducing post-recovery dimensional variation after 48 hours to 2.54–4.84% in DFD compared with 4.50–9.23% in DD. Statistically significant correlations were observed among density, compression ratio, and hardness. The results demonstrate that anatomical structure substantially influences densification efficiency, and furfurylation improves short-term moisture stability.

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Introduction

The growing interest in timber frame structures can be attributed to their usability, lightweight nature, aesthetic appeal, low construction costs, and high durability (Wang et al., 2022). Despite these advantages, wood has limitations, including low dimensional stability due to its affinity for moisture, susceptibility to decay, and relatively low strength properties. To expand the applications of wood and build confidence among architects, engineers, and consumers, it is essential to enhance these properties (Sandak & Sandak, 2021).

Various wood modification methods have been explored to enhance the properties of wood and mitigate its limitations. These methods include thermal, chemical, mechanical, and biological modifications (Sandberg et al., 2017). Thermal modification involves subjecting wood to high temperatures to improve its dimensional stability and resistance to biological attacks, although it can reduce its mechanical strength and toughness (Barboutsis & Kamperidou, 2019). Chemical modification, which involves using chemicals to alter the properties of wood, aims to enhance durability and resistance to decay; however, it can also involve toxic

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chemicals and high production costs (Hill, 2007; Repič et al., 2022; Wang et al., 2022).

Delignification and densification are crucial wood modification techniques. Delignification removes lignin from wood fibres to increase plasticity, while densification compacts wood structure under pressure, enhancing density and mechanical properties. Radial densification is the most commonly used process (Cabral et al., 2022). These techniques can transform low-density wood into high-performance materials, making previously less commercially viable materials suitable for various applications (Mania et al., 2020, 2022, 2024; Yunianti et al., 2019).

Scots pine (*Pinus sylvestris* L.) is particularly well-suited for delignification and densification due to its favourable properties. Scots pine is widely available in Europe and parts of Asia, ensuring a reliable supply for industrial applications (Mederski et al., 2015). Its moderate to high density and chemical composition, including high cellulose content and low resin, make it ideal for modification processes (Kilpeläinen et al., 2005). The low resin content is advantageous for delignification, as it reduces the amount of chemicals required and improves the effectiveness of lignin removal. The chemical composition of Scots pine wood consists of approximately 40–45% cellulose, 24–28% hemicelluloses, and 24–30% lignin, with minor constituents including organic acids, fatty acids, proteins, and minerals (Kilpeläinen et al., 2005; Rowell et al., 2005). This composition contributes to its dimensional stability, making it less prone to warping or swelling due to changes in moisture, which is beneficial for densification processes.

Thermo-mechanical densification encounters challenges such as the springback effect and set recovery. The springback effect occurs when wood partially regains its original dimensions after compression during densification (Báder et al., 2018). Set recovery refers to the gradual reversion of densified wood to its original shape over time due to environmental factors, such as moisture and temperature (Kutnar & Šernek, 2007). Addressing these issues involves optimising modification processes and using hydrophobic chemicals to enhance stability (Blanchet & Pepin, 2021).

Several post-delignification treatments have been investigated to improve the properties of wood, including impregnating it with chemicals such as sodium silicate, acetic anhydride, phenol-formaldehyde, and furfuryl alcohol. These treatments aim to enhance mechanical properties and dimensional stability (Jones & Sandberg, 2020; Kuai et al., 2022; Kumar et al., 2021). Each method has specific advantages and limitations. Acetylation, for instance, introduces acetyl groups into the wood, improving dimensional stability and reducing susceptibility to biodeterioration,

though it may not significantly enhance mechanical properties (Chung et al., 2018). Phenol-formaldehyde treatment enhances stability and resistance to swelling, but may be less effective in delignified wood due to the reduced lignin content. Furfuryl alcohol, derived from agricultural waste, enhances hardness, stability, and mechanical properties but can increase brittleness (Jones & Sandberg, 2020). Recent research shows that the success of impregnation treatments before densification heavily relies on species-specific anatomical and chemical traits. In Scots pine, the lower resin content and higher cellulose levels allow better penetration of agents like furfuryl alcohol, leading to enhanced dimensional stability and mechanical properties after densification. Additionally, densification conditions – especially radial compression at high temperatures – are more effective in collapsing earlywood zones, while latewood regions resist deformation due to thicker cell walls. However, the combined effects of macrostructural features and sequential treatments, such as delignification followed by impregnation and densification, are still not fully understood.

Understanding and addressing the challenges associated with wood modification techniques is crucial for selecting the most suitable method for specific applications, thereby ensuring the improved performance and sustainability of wood products. Future research should focus on optimising these methods to minimise drawbacks and maximise the benefits of modified wood, paving the way for broader applications in construction, manufacturing, and other industries. This research aimed to evaluate the impact of variable macrostructure properties, specifically growth ring width and latewood proportions, on the densification degree and density values of Scots pine (*Pinus sylvestris* L.) wood after undergoing partial delignification and radial densification. Additionally, the study aimed to evaluate the impact of furfuryl alcohol treatment on the density, hardness, and set recovery of partially delignified, furfurylated, and densified Scots pine wood. Comparisons were made with Scots pine wood that had undergone only partial delignification and densification, to isolate the effects of furfuryl alcohol treatment.

Materials and methods

1. Wood specimens

This study utilised wood samples from Scots pine (*Pinus sylvestris* L.). The samples were carefully selected from six different Scots pine trees. All logs were free of defects and displayed distinct macrostructural characteristics, such as variations in growth ring widths and

proportion of latewood. Scots pine was selected due to its wide availability and favourable anatomical and chemical characteristics, making it suitable for delignification and densification processes. The samples were prepared from mature sapwood of trees aged 85–95 years, providing a representative and well-developed wood structure for the applied modifications. Only sapwood near the bark was used, and the wood was repeatedly machined and cut. Each sample had dimensions of 25 x 25 x 35 mm along the radial, longitudinal, and tangential directions, respectively. A total of 90 samples were prepared for densification in this study. These were divided into six groups, with each group differing in growth ring width and the proportion of latewood. To ensure accurate measurements, a line was drawn perpendicularly across the growth rings on the smooth surface of each sample. Using a Biotronik BEPD-19 dendrometer (Warsaw, Poland), the earlywood and latewood sections of the growth rings were measured, and the percentages of each were calculated. Prior to the delignification process, the samples were oven-dried at 103 ± 2 °C. The density of each specimen was measured using the stereometric method according to the standard (ISO 13061-2 2014).

To examine the chemical composition of the wood samples, the specimens were first milled using a laboratory mill (Fritsch Pulverisette 15, Fritsch GmbH, Germany). The resulting wood powder, with a particle size range of 0.5–1.0 mm, was subsequently used for chemical analysis. Cellulose content was measured using the Kürschner–Hoffer method (Kürschner & Hoffer, 1929), while pentosan content was determined according to the TAPPI T 223 cm-01 standard (TAPPI, 2001). The content of acid-insoluble lignin was analysed according to the TAPPI T 222 om-06 standard (TAPPI, n.d.), which involved the hydrolysis and dissolution of carbohydrates using 72% sulfuric acid. Each analysis was performed at least three times for each specimen. The chemical composition was assessed both before and after the delignification process.

2. Preparation of densified wood: delignification and furfurylation

Delignification was performed using the laboratory method described by Song et al. (2018) and Mania et al. (2020). Scots pine samples were placed in a round-bottom flask containing 2.5 M NaOH and 0.4 M Na₂SO₃ aqueous solution. The flask was placed on a heating mantle and fitted with a reflux condenser. The solution was brought to boil at 110 °C and kept at that temperature for nine hours. Subsequently, the samples were transferred to boiling

distilled water and thoroughly rinsed several times to remove residual delignification chemicals.

For furfuryl alcohol impregnation, the procedures described by Bartkowiak et al. (2013) and Hadi et al. (2022) were followed. Directly after delignification and rinsing to remove residual chemicals, six samples from each group were selected and allowed to cool. These samples then underwent a two-stage furfuryl alcohol modification process: impregnation and curing. During the impregnation phase, the wood samples were immersed in a solution of 99% furfuryl alcohol under a vacuum of 8 bar. This vacuum was maintained for 45 minutes, and atmospheric pressure was restored for an additional 2 hours. The samples were then tightly wrapped in aluminium foil for the curing stage, where they were heated to 120 °C for the first 4 hours, and then held at 105 °C for an additional 9 hours.

The wooden specimens were then subjected to a pressure of approximately 5 N/mm² at a temperature of 100 °C for approximately 24 hours, resulting in densified wood with an initial thickness of around 8 mm.

Radial densification was chosen due to the anisotropic nature of wood and its more consistent deformation behavior along this axis. Compressive treatment perpendicular to the growth rings facilitates the more effective collapse of earlywood, leading to a more homogeneous density distribution. Conversely, tangential densification tends to induce non-uniform deformation, internal stresses, and defects attributable to the curvature of growth rings. Consequently, radial densification offers superior process control and results in more uniform enhancements of wood properties.

Three groups of samples were collected: control; delignified and densified (DD); and delignified, furfurylated, and densified (DFD).

The compression ratio (*CR*) was calculated according to equation (1) below, representing the degree to which the size of the wood sample decreased after radial densification, relative to its initial dimensions before the delignification process:

$$CR = \frac{IT - CT}{IT} \times 100(\%) \quad (1)$$

where *IT* is the initial thickness and *CT* is the compressed thickness.

3. Post-recovery of specimens

A total of 24 wood samples were selected, with four (4) samples taken from each group after the densification process. These consisted of two (2) samples that were partially delignified and densified, and two (2) that were partially delignified, furfurylated, and densified. Following this, the samples were oven-dried

at 103 ± 2 °C for 12 hours. After drying, the dimensions of each sample were measured in the radial, tangential, and longitudinal directions, and their masses were recorded. The samples were placed inside a desiccator with a relative humidity of over 97% to evaluate how the densified wood responded to humid conditions. Thickness measurements were repeated at specific time intervals: 24, 48, 72, and 144 hours. The maximum swelling degree was calculated using the collected data, reflecting the extent of expansion of a sample relative to its original size. This value was determined based on the recorded measurements of the dimensional changes.

4. Hardness test using the Brinell method

In accordance with the EN 1534 standard (EN 1534, n.d.), the Brinell method was applied to determine hardness in the radial direction. A 10 mm diameter steel ball and a 1 kN load were used for the measurements. The maximum force of 1 kN was reached over 15 seconds, held for 30 seconds, and then gradually reduced to zero within 15 seconds. The Brinell hardness was calculated using the following formula:

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

where HB is the Brinell hardness [$\text{N} \cdot \text{mm}^{-2}$], F is the nominal force [N], D is the diameter of the steel ball (10 mm), and d is the mean diameter of the residual indentation [mm]. Brinell hardness was determined in the radial direction of control and densified specimens.

5. Scanning electron microscopy (SEM)

Control and densified wood samples were prepared using different procedures for SEM observations. Control specimens were soaked in water for 24 h

to soften the tissue, after which cross-sections were smoothed using a rotary microtome with disposable blades to obtain cuts of approximately 10 μm thickness. Densified samples were sectioned in an air-dry state to prevent set-recovery during soaking. After smoothing, control samples were oven-dried overnight at 103 ± 2 °C. Prior to sectioning, densified specimens were shaped into a cone-like form to minimise the contact area with the blade (approx. 1 mm^2), and subsequently cut into slices of approximately 1 μm thickness. All samples were mounted on carbon tape and examined using a scanning electron microscope (HITACHI TM4000, Tokyo, Japan) at an accelerating voltage of 15 kV with a backscattered electron detector.

6. Statistics

The experimental data were analysed using STATISTICA 13.3 software (TIBCO Software Inc., Palo Alto, CA, USA) with analysis of variance (ANOVA). Tukey's HSD test was used to identify significant differences between the mean values of the groups. All comparisons were performed at a significance level of 0.05.

Results and discussion

1. Macrostructural parameters

The annual growth ring of a tree is composed of two distinct parts: the earlywood and the latewood. In coniferous trees, earlywood is differentiated from the latewood. According to Fernandes et al. (2017) the latewood percentage in Scots pine varies from 24% to 41%, based on an analysis of 100 adult samples and data from the literature. These findings offer valuable insights into the variation in latewood content within species, suggesting the potential for diverse wood characteristics and properties among individual trees. Figure 1

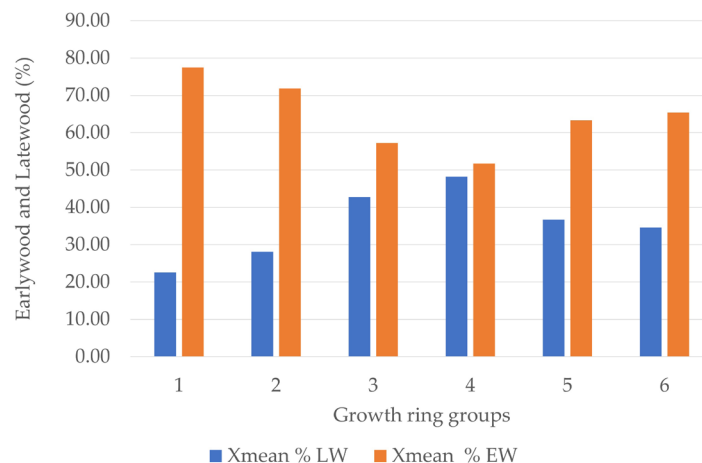


Fig. 1. Mean percentage of earlywood (EW) and latewood (LW) values

illustrates that the selected growth ring samples for this study had very small to very large average latewood percentages (22.56–48.25%). Latewood regions are typically distinguished from earlywood regions by their superior mechanical properties, higher density, and darker colours. It was also noted that growth ring group 1 had the highest percentage of earlywood, with an average value of 77.44%. The lowest mean earlywood percentage was 51.75%, which was observed in growth ring group 4. Conversely, growth ring group 4 had the highest mean latewood percentage among all the growth ring groups used in this study.

2. Chemical composition

Table 1 presents the lignin content in the pine samples before and after delignification, as well as the lignin loss that occurred during the process ($\pm$ SD). The reported value represents the average lignin content calculated for the six groups of samples characterised by variable macroscopic parameters. In the control samples, pine exhibited a typical lignin content consistent with values reported in the literature (Irbe et al., 2011; Liszka et al., 2023; Pandey & Pitman, 2004).

Following delignification treatment, the lignin content in Scots pine decreased from 28.4% to 20.7%, representing a 27.1% reduction (Table 1). This decrease was statistically significant, as shown by single-factor analysis of variance (ANOVA), indicating that the delignification process effectively removed lignin from the wood structure. The reduction highlights that the chemical treatment selectively targeted lignin without causing excessive loss of the polysaccharide fraction. The initial lignin content of the untreated Scots pine samples (28.4%) was in line with typical values reported in the literature. Softwoods, including Scots pine, generally have a lignin content of 27–33%, primarily composed of guaiacyl-type lignin, which is more condensed and chemically resistant than the guaiacyl/syringyl lignin found in hardwoods. Several authors have reported similar lignin values: Fengel et al. (1989) described softwood lignin contents of approximately 28–30%, while Kollmann & Côté (1968) reported an average lignin content of 29% in Scots pine wood. These findings support the classification of pine as a lignin-rich species and confirm that the control samples in this study fall within the expected range of natural

variability. The 27% reduction in lignin achieved by our delignification treatment allows it to be categorised as moderately efficient, rather than extremely aggressive. In more intensive pretreatment or pulping processes, the lignin removal rates can be significantly higher. For instance, studies on ionic liquids or deep-eutectic solvent (DES) systems applied to pine have reported lignin removal rates of up to ~80% under optimised conditions, with Sirviö et al. (2023) noting a removal rate of ~87% from pine using a DES system. Conversely, milder or less penetrating treatments on softwoods often result in lower delignification extents. For example, Imlauer-Vedoya et al. (2019) documented delignification ranges of approximately 5.2% to 24% for specific steam explosion pretreatments of *Pinus radiata*. Compared with these reports, the present results fall on the lower-to-moderate end of the removal range, indicating that the delignification conditions used in this study were efficient enough to significantly decrease the lignin content while avoiding excessive degradation of carbohydrates. Overall, the results demonstrate that the applied delignification method successfully modified the chemical composition of pine samples by reducing lignin levels. The achieved reduction aligns well with literature values for softwood delignification efficiency, confirming that Scots pine can undergo effective chemical modification despite the known resistance of softwood lignin to chemical degradation.

3. Densification

The density values of Scots pine (*Pinus sylvestris* L.) before and after densification are summarised in Table 2. The initial density range of the untreated specimens (395–586 kg·m⁻³) confirms that the proportion of cell-wall material relative to the volume of cell lumina and voids is the primary determinant of wood density. These values fall within the range reported for Scots pine in earlier studies. For instance, Fundova et al. (2018) reported densities from 365 to 566 kg·m⁻³ (SilviScan measurements). In comparison, Konofalska et al. (2021) found mean air-dry densities of approximately 514 kg·m⁻³ for Scots pine from Polish stands, with substantial natural variation between individual samples. The variability among the groups in the present study can be attributed mainly to differences in the proportion of earlywood and latewood. This observation is consistent with the

Table 1. Lignin content in pine wood before and after the delignification process

Specimens	Percentage of lignin [%]	Reduction of lignin [%]
Scots pine (control)	28.4 $\pm$ 1.19	27.1 $\pm$ 0.66
Scots pine after chemical treatment	20.7 $\pm$ 0.97	

Table 2. Wood density (ρ) before and after the densification process

Specimens	Wood Density [$\text{kg}\cdot\text{m}^{-3}$]					
	1	2	3	4	5	6
Control	457 $\pm$ 16.1	404 $\pm$ 6.1	574 $\pm$ 6.3	555 $\pm$ 12.6	469 $\pm$ 8.5	513 $\pm$ 9.9
DD	1168 $\pm$ 25.2	1056 $\pm$ 10.8	1130 $\pm$ 20.6	1079 $\pm$ 8.5	1100 $\pm$ 25.8	1118 $\pm$ 11.4
DFD	1031 $\pm$ 21.8	1008 $\pm$ 10.8	1069 $\pm$ 30.1	1058 $\pm$ 67.1	1117 $\pm$ 12.0	1104 $\pm$ 28.6

literature, which demonstrates that the latewood fraction is one of the strongest anatomical predictors of wood density due to its thick cell walls and reduced lumen volume (Pritzkow et al., 2014). In our material, the highest densities before densification were recorded for groups exhibiting elevated latewood percentages (groups 3 and 4: 573.98 and 554.96 $\text{kg}\cdot\text{m}^{-3}$), whereas groups characterised by a predominance of earlywood had markedly lower densities (groups 1 and 2: 457.17 and 404.02 $\text{kg}\cdot\text{m}^{-3}$). This pattern reflects a well-established relationship reported for Scots pine and other conifers: higher proportions of latewood and narrower growth rings are strongly associated with increased density. It should be noted that wood density is influenced by multiple factors, including species, site conditions, tree age, moisture content, and the applied measurement method; thus, broad natural variation is commonly observed across studies.

Following partial delignification and subsequent radial densification (DD), the highest density was observed in growth ring group 1, with an average value of 1168.11 $\text{kg}\cdot\text{m}^{-3}$. This group had the most significant amount of earlywood, resulting in the greatest porosity before the treatment. According to Mania et al. (2020), the density that can be achieved in densified wood is significantly influenced by the proportion of voids in the cell structure; areas with more pores experience greater deformation during compression. Because radial densification compresses wood across the grain, earlywood, characterised by thin cell walls and wide lumina, is more easily compressed than denser latewood bands. In contrast, growth ring group 2 recorded the lowest average density among the DD samples, at 1056.11 $\text{kg}\cdot\text{m}^{-3}$. The relatively high variation in latewood proportion within this group likely affected its compressibility, thereby reducing the densification effect. DD treatment notably decreased the density variation between the sample groups. Prior to delignification and densification, the difference between the lowest and highest average densities was 170 $\text{kg}\cdot\text{m}^{-3}$; after DD treatment, this difference was reduced to 112 $\text{kg}\cdot\text{m}^{-3}$. This suggests that applying the same delignification method and identical radial compression force to samples with varying earlywood/latewood ratios results

in densified wood with similar final densities, despite significant differences in the untreated material. Practically, Scots pine samples subjected to the DD process achieved final densities approximately twice their initial densities, with the main dimensional changes occurring in thickness and width rather than mass. The average density of the delignified + furfurylated + densified samples (DFD) was generally lower than that of the DD samples (Table 2). Only growth ring group 5 showed a slight increase of 17 $\text{kg}\cdot\text{m}^{-3}$ relative to the DD counterpart. Similarly as in the case of DD treatment, the density values of the DFD samples were more consistent across the growth ring groups. Despite having a lower density than DD, the combination of furfurylation and densification resulted in final densities approximately twice that of the untreated material. This confirms that both densification methods effectively increase density; however, furfurylation introduces an additional polymer network within the cell wall, which may affect the extent of cell wall collapse and the final compaction behaviour.

The calculated compression ratio (CR) values for all Scots pine sample groups ranged from 59% to 74%. The highest CR values were observed in growth ring groups 1, 2, and 5 (72.2%, 72.9%, and 73.7%, respectively), which had the lowest proportions of latewood. In contrast, the lowest CR values were found in groups 3 and 4 (60.8% and 59.2%, respectively), which had the highest latewood content. This suggests that samples with a higher proportion of latewood exhibit higher resistance to compression. This pattern aligns with previous findings indicating that latewood, due to its thicker cell walls and smaller lumina, shows significantly greater resistance to deformation during densification compared to earlywood (Mania et al., 2024). After furfurylation (DFD treatment), the CR values became more variable, ranging from 52% to 76%. The lowest CR values (52.3% and 53.3%) were recorded in groups 3 and 4, while the highest (76.4%) was observed in group 2. This variability suggests that furfurylation alters the compressive behaviour of the wood structure by modifying the distribution of polymerised furfuryl alcohol within the lumen or cell walls, thereby influencing the capacity for cell-wall collapse and volumetric reduction

(Mubarok et al., 2023). In summary, anatomical composition – specifically the earlywood-to-latewood ratio – remains the primary determinant of densification potential in Scots pine. At the same time, chemical modification via furfurylation introduces additional variability, which is dependent on polymer uptake and curing dynamics.

4. Brinell hardness

The Brinell hardness values (HB) of the Scots pine samples increased significantly after undergoing partial delignification and densification (DD) compared with the untreated controls (Table 3).

In the control group, HB values ranged from 16.06 to 24.99 N·mm⁻². After post-DD treatment, the mean hardness values increased significantly to 87.85–145.27 N·mm⁻², representing approximately a 5- to 8-fold increase. The highest hardness was recorded in group 1 (145.27 ± 19.63 N·mm⁻²), which also exhibited the highest compression ratio (CR) and the most significant reduction in porosity. The observed correlation between hardness and density profile aligns with previous research on thermomechanical and viscoelastic thermal compression (VTC) processes, which have demonstrated that surface hardness is strongly influenced by local density increases and the shape of the vertical density profile (Rautkari et al., 2011, 2013). In the partially delignified, furfurylated, and densified (DFD) samples, mean HB values were generally lower than in DD samples, ranging from 68.65 to 103.54 N·mm⁻², yet still substantially (approximately 4 to 6 times) higher than those of the untreated controls. The smaller hardness gains in DFD samples can be attributed to the effects of furfurylation on deformation mechanics. The polymerisation of furfuryl alcohol within lumina and partially within cell walls increases local stiffness. However, it reduces the ability of cell walls to collapse completely during compression, thereby lowering volumetric densification efficiency and limiting the maximum hardness increase relative to DD treatment (Esteves et al., 2011; Liu et al., 2021). Moreover, heterogeneity in earlywood-to-latewood distribution and variation in weight percent

gain (WPG) likely contributed to the higher variability in hardness among the DFD groups. Overall, the results confirm that the main factor determining hardness improvement after densification is the increase in the proportion of solid cell wall material per unit volume (local density rise). Chemical modification by furfurylation, however, alters compression behaviour and may both enhance and limit hardness, depending on polymer distribution and uptake within the wood structure. This conclusion is consistent with findings from recent reviews and experimental studies on the densification and chemical modification of softwoods (Cabral et al., 2022; Mania et al., 2024).

To assess the relationship between densification, compression ratio (CR), and Brinell hardness (HB), one-way analysis of variance (ANOVA) and Pearson correlation analysis were performed on all sample groups. ANOVA revealed statistically significant differences in HB among the control, DD, and DFD groups ($p < 0.001$), confirming that both densification and chemical modification significantly enhanced wood hardness. Post hoc Tukey tests showed that DD samples generally exhibited significantly higher hardness than DFD samples ($p < 0.05$), while all modified groups differed significantly from the controls. Pearson correlation analysis revealed a strong positive correlation between radial compression ratio and hardness ($r = 0.89$, $p < 0.01$) across all densified samples, indicating that greater densification resulted in higher hardness values. Similarly, a strong positive correlation was found between final density and HB ($r = 0.92$, $p < 0.01$), aligning with previous studies showing that Brinell hardness scales with the proportion of cell-wall material per unit volume (Mania et al., 2024; Rautkari et al., 2013). Notably, the correlation between CR and HB was slightly weaker in DFD samples ($r = 0.78$), likely due to the uneven distribution of polymerised furfuryl alcohol in earlywood and latewood, which partially limited lumen collapse and reduced the macro-scale hardness gain (Esteves et al., 2011). These statistical analyses confirm that mechanical densification is the primary driver of hardness improvements, while chemical modification through furfurylation introduces additional variability depending on polymer uptake, localisation, and curing conditions.

Table 3. Wood Brinell hardness (HB) before and after the densification process

Specimens		Brinell hardness [N·mm ⁻²]				
Group	1	2	3	4	5	6
Control	20.6±0.58	17.64±0.75	24.99±1.77	22.78±2.08	16.06±2.59	17.33±0.65
DD	145.27±19.63	114.10±7.89	113.08±9.93	95.16±12.06	87.85±6.86	104.87±5.67
DFD	103.54±20.42	85.18±12.06	68.65±10.12	77.26±7.02	79.48±8.87	68.69±7.16

5. Post-recovery

The dimensional recovery of Scots pine samples following partial delignification and densification (DD) and combined delignification, furfurylation, and densification (DFD) was evaluated under near-saturation humidity conditions (~97% RH) to investigate the effect of these treatments on moisture-induced dimensional changes (Table 4). Overall, the results demonstrate that furfurylation prior to densification significantly enhances dimensional stability, particularly during the initial period of moisture exposure.

During the first 24 to 48 hours, DFD samples exhibited substantially lower post-recovery percentages than DD samples across all growth ring groups. For instance, at 48 hours post-recovery, the dimensional change in DFD samples ranged from 2.54% to 4.84%. In contrast, the dimensional change in DD samples ranged from 4.50% to 9.23%, representing a reduction of approximately 40–50% in dimensional change due to furfurylation. This difference highlights the ability of furfuryl alcohol to limit swelling and shrinkage, as the resulting polymer penetrates the cell lumina and, to a limited extent, the cell walls, thereby reinforcing the wood microstructure and reducing water accessibility to hydrophilic sites within the cell wall. The earlywood zones, which are more prone to collapse during densification and more susceptible to water-induced expansion, benefit most from this chemical stabilisation.

Over longer exposure times (72–144 hours), the recovery differences between DD and DFD samples decreased. By 144 hours, all treated samples displayed similar post-recovery values, suggesting that hygroscopic equilibrium gradually mitigates the initial benefits of chemical modification. Nevertheless, DFD samples consistently maintained lower early recovery, demonstrating superior short-term dimensional stability, which is critical for applications where wood may be exposed to

sudden humidity changes. The post-recovery behaviour correlates with previously reported mechanisms of furfurylated wood: polymerisation of furfuryl alcohol within lumina and cell walls reduces the availability of hydroxyl groups for moisture sorption, increases cell-wall rigidity, and restricts volumetric expansion (Esteves et al., 2011; Liu et al., 2021; Mubarak et al., 2023).

Comparatively, DD samples, while densified and significantly more dimensionally stable than untreated controls, exhibited higher early recovery due to the absence of chemical stabilisation. In these samples, the elastic springback of the compressed earlywood cells is not restrained by a polymer, leading to higher initial swelling. Therefore, while densification alone can improve dimensional stability, furfurylation in combination with densification (DFD) provides superior control over moisture-induced recovery, particularly in the critical first 48–72 hours of exposure. This result demonstrates the synergistic effect of combining chemical modification with mechanical densification for producing wood products with enhanced moisture resistance.

6. Scanning electron microscopy (SEM)

Figure 2 shows SEM micrographs of Scots pine (*Pinus sylvestris* L.) wood before and after densification. In the untreated sample (Figure 2a), the cellular structure remains well preserved, with earlywood and latewood tracheids arranged in a regular radial pattern. Ray parenchyma cells are clearly seen as radially arranged structures. A close-up of the latewood-dominant area (Figure 2b) displays typical features of this zone, such as thicker cell walls and smaller lumen sizes. While differences in macrostructure and latewood content were evident among the samples before densification, SEM analysis of the densified specimens showed no notable intergroup differences after compression. All densified samples, regardless of their initial latewood amount, had

Table 4. Post recovery before and after the densification process

Specimens		Post recovery [%]					
Group	Time [h]	1	2	3	4	5	6
DD	24	5.49	4.46	4.63	2.30	3.10	2.22
	48	8.24	9.23	7.43	4.50	8.06	5.78
	72	12.57	13.54	8.87	5.75	13.80	7.79
	144	17.92	15.29	12.05	10.92	16.88	11.33
DFD	24	2.72	2.67	1.89	2.09	1.78	1.79
	48	3.72	4.84	3.31	2.54	2.92	3.03
	72	4.61	6.02	5.48	6.05	6.58	7.67
	144	8.45	9.69	10.02	10.34	9.42	10.14

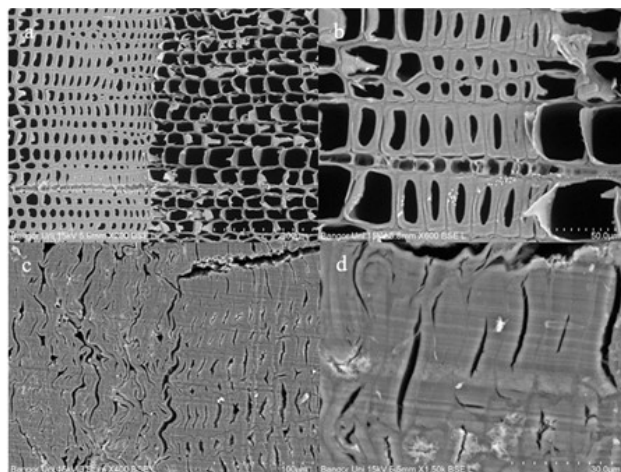


Fig. 2. SEM images of Scots pine wood before and after densification: (a) untreated structure, (b) latewood detail, (c) densified wood with reduced porosity and cracks, (d) densified latewood with collapsed lumina

very similar microstructures, with no visible features to distinguish between groups post-compression.

After densification (Figures 2c and 2d), a significant decrease in porosity is observed. At the scale of the entire growth ring (Figure 2c), tracheid outlines are still partially visible, though many lumina are collapsed or indistinct. Microcracks are common and tend to spread along ray regions, reflecting the wood's anatomical variability. These features relate to the different mechanical responses of tracheids and ray parenchyma cells under compression. Ray parenchyma cells, oriented perpendicular to the grain and with a distinct shape compared with tracheids, show less radial compressibility during densification. This mechanical mismatch causes stress concentration and encourages crack formation along ray interfaces. A close-up of latewood after densification (Figure 2d) reveals severe cell-wall deformation, lumen collapse, and widespread microcracking.

Conclusions

This research demonstrates that combining partial delignification with densification (DD) and with furfurylation followed by densification (DFD) significantly enhances the mechanical and dimensional properties of Scots pine wood. The findings indicate that wood density and compression ratio are heavily influenced by the earlywood-to-latewood ratio, with groups rich in earlywood exhibiting higher compressibility and, consequently, greater densification. Partial delignification and densification increased wood density up to twice that of untreated samples, while furfurylation adjusted densification by

strengthening cell walls and preventing luminal collapse. The Brinell hardness increased significantly after DD treatment, reaching up to $145.3 \text{ N}\cdot\text{mm}^{-2}$. It remained considerably higher than in the controls after DFD treatment, although the latter group exhibited slightly lower maximum hardness due to the furfuryl polymer's limiting effect on cell wall collapse. Statistical analysis confirmed strong correlations between density, compression ratio, and hardness, emphasising that mechanical densification is the primary factor in improving hardness. At the same time, furfurylation introduces variability based on polymer distribution and uptake. Post-recovery tests conducted under near-saturation humidity conditions revealed that furfurylation significantly reduces early moisture-induced recovery, thereby enhancing short-term dimensional stability, particularly during the first 48 to 72 hours of exposure. Although long-term equilibration lessens the differences between DD and DFD samples, the combination of chemical modification and densification consistently offers superior control over swelling and shrinkage, confirming the synergistic effect of these treatments. In conclusion, the results suggest that integrated delignification, densification, and furfurylation treatments can effectively improve the density, hardness, and moisture stability of Scots pine wood. These findings provide a solid framework for creating high-performance, durable softwood materials suitable for structural applications, flooring, and other load-bearing or moisture-sensitive uses. The study also highlights the significance of anatomical features, such as the earlywood-to-latewood ratio, in determining the effectiveness of treatment and the final performance of the wood.

Conflict of interest

The author(s) declare(s) that there is no conflict of interest concerning the publication of this article.

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