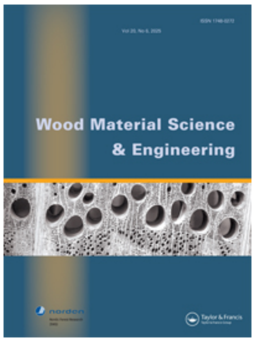

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Preservative retention and penetration in Norway spruce wood as influenced by different microwave treatment energy levels

Sauradipta Ganguly [a,b], Marko Petrič [c], Angela Balzano [c], Sanjeet Kumar Hom [a], Varun Sharma [a] and Davor Kržišnik [c,d]

[a]Wood Preservation Discipline, Forest Products Division, Forest Research Institute, Dehradun, India; [b]Natural Resources Institute Finland (LUKE), Production Systems, Joensuu, Finland; [c]Biotechnical Faculty, Department of Wood Science and Technology, University of Ljubljana, Ljubljana, Slovenia; [d]NOVA School of Science and Technology, MARE - Marine and Environmental Sciences Centre, NOVA University Lisbon, Caparica, Portugal

ABSTRACT

Norway spruce (*Picea abies*) is often used in construction but is difficult to impregnate with wood preservatives due to pit aspiration, limiting its treatability. The low natural durability of spruce necessitates preservation treatments. Microwave treatment improves the permeability, increasing preservative retention and penetration. However, treatability assessment has often been conducted using visual methods, which can over - or underestimate success. This study used image processing to evaluate MW-treated spruce's treatability with two water-based preservatives. The effect of MW treatment at five energy levels was analysed for sapwood and heartwood. The results showed improved retention values and penetration percentages for the MW-treated sapwood and the heartwood. The retention values of both preservatives increased between 50% and 124% for different treatment combinations. Spruce heartwood MW-treated with 1260 MJ/m^3 improved treatability by 24% when analysed with ImageJ. The anatomical analysis showed that the effect of MW on heartwood was more pronounced than on sapwood, leading to the destruction of rays and parenchyma cells that facilitated the absorption and penetration of the preservatives. The study demonstrates the potential of ImageJ for more accurate treatability assessment and suggests that MW treatment if optimised, could improve the natural durability of spruce wood.

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Wood preservation; treatability; ImageJ analysis; anatomical analysis; pit aspiration

1. Introduction

Wood is gaining worldwide attention for various applications in the construction sector, mainly because of its positive impact on the environment, its ability to sequester carbon during use, good workability, easy availability, aesthetics, and sustainability. However, its use is limited by inherent properties such as lower dimensional stability, flammability, and susceptibility to decay and degradation (Jiang *et al.* 2015, Sandberg *et al.* 2017). Harsh climatic conditions further impair its performance (Scheffer and Morrell 1998, Råberg *et al.* 2005). The susceptibility of wood to various biotic and abiotic decay factors outdoors is significant to stakeholders and must be regularly monitored and controlled (Tripathi *et al.* 2022). As the biodegradation of wood is a natural process, it needs to be controlled for efficient utilisation of resources (Kutnik *et al.* 2014). Furthermore, various wood species with low permeability and high refractoriness lead to an obstruction of the impregnation process (Ramezanpour *et al.* 2015, Hess *et al.* 2021) and in return have a shorter service life, so they need to be replaced frequently. The impregnation of wood with various treatment chemicals is used to preserve the wood for long service life. However, the extent of retention and penetration of these chemicals in the wood depends on the anatomical structure of the wood, the treatability, and the penetration index (Tarmian *et al.* 2020). The poor treatability and refractory nature of some commercially essential wood species results in very low retention and little to no penetration, which does not fulfil the purpose of ensuring a substantial lifespan of wood (Mascarenhas *et al.* 2024a).

India is a timber deficit country, as evidenced by the fact that as a major importer of round timber and other related products, India has had imports worth an estimated USD 2 billion in the last decade (Sood 2019), with round timber accounting for an estimated 74% of total imported forest products (Montiel 2016, AHEC 2016). However, the durability and performance of such imported non-native species under India's tropical climate require special attention as most of the imported species are reported to perform poorly when exposed outdoors (Sundararaj *et al.* 2015, Tripathi *et al.* 2022). This may result in the need for early and frequent replacement of the wood used, which may not be economically and environmentally viable (Samani *et al.* 2019). In recent decades, several wood modification processes have proven to be effective solutions to counteract the undesirable properties of wood (Sandberg *et al.* 2017). Several non-durable wood species, both of domestic and imported origin, can be made suitable for

CONTACT Davor Kržišnik davor.krzisnik@bf.uni-lj.si Biotechnical Faculty, Department of Wood Science and Technology, University of Ljubljana, Jamnikarjeva 101, Ljubljana 1000, Slovenia

specific purposes through appropriate modification processes, ensuring sustainable utilisation of available resources. The treatment of refractory wood and its modification for particular end uses with microwaves (MWs) has gained importance in the field of wood research in recent years (Torgovnikov and Vinden 2010, Samani *et al.* 2019, Ganguly *et al.* 2021a, Kol and Çayır 2021, Mascarenhas *et al.* 2024a), mainly due to its diverse applications in wood processing (Oloyede and Groombridge 2000, Torgovnikov and Vinden 2010, Balboni *et al.* 2018, Weng *et al.* 2021) and also because MW wood modification is relatively fast and less energy-intensive compared to the other available modification methods (Brodie 2010, Sethy *et al.* 2016).

Advanced research in the field of MW modification has already proven that it improves the permeability of wood, allowing more effective impregnation of preservatives and leading to more accessible treatment of refractory wood species of commercial importance (Treu and Gjølsjø 2008, Balboni *et al.* 2018, Kol and Çayır 2021, Weng *et al.* 2021, Ganguly *et al.* 2021a). This increased permeability is primarily due to the rupture of weak anatomical elements leading to the formation of micropores and microcracks (Mekhtiev and Torgovnikov 2004, Terziev *et al.* 2023), as well as an increase in pore diameters along with the destruction of pit membranes and damage to cell walls (Weng *et al.* 2020, Ganguly *et al.* 2021b). The microcracks in the wood during treatment are mainly due to the high-pressure gradient of the vapour generated during MW heating. Greenwood performs better than wood with low moisture content (MC). A low MC leads to rapid destruction of the cell wall components (Wang *et al.* 2022). MWs were researched by Gašparik and Gaff (2013) for wood plasticisation, and they also reported the importance of higher MC in wood (e.g. above the fibre saturation point) for this intensive process, which was later confirmed by Gašparik and Barsik (2014). When very high microwave (MW) power is applied to green wood, the water molecules in the microstructure rapidly heat up to the boiling point and generate a vapour pressure in the wood cells, which intensifies with increasing MW energy and can reach up to 600 kPa (Terziev *et al.* 2023), breaking the weak anatomical elements and cell wall structures and creating free pathways for liquid and gas transport. When the MC of wood is well above its Fibre Saturation Point (FSP), more MW energy is used to evaporate free water in the form of steam, resulting in damage to the microstructure (Ganguly *et al.* 2024). MC below the FSP leads to a rapid temperature rise in the wood, resulting in localised heating and rapid destruction of the cell wall structure and anatomical elements (Brodie 2005, Wang *et al*. 2022), leading to a loss of mechanical strength (Mascarenhas *et al.* 2023) compared green wood when exposed to MW. Therefore, optimising MC is crucial before applying MW energy to wood destined for specific end-use. Apart from the initial MC, other factors such as the applied MW energy, the power, the wood species, part of the stem (sapwood or heartwood), and the refractoriness of the wood species in question can significantly influence the effectiveness of the MW treatment (Wang *et al.* 2022, Mascarenhas *et al.* 2023).

The penetration of wood preservatives into the microstructure of the wood is often an accurate indicator of the sustainable use of wood indoors and outdoors. It is, therefore, of significant importance (Tarmian *et al.* 2020). Retention may be a surface phenomenon, but if the treatment chemical is not properly and evenly distributed, even higher retention and absorption over an extended period may not provide much protection. Research to estimate the penetration of wood preservatives has typically been conducted using visual methods, i.e. spot tests in which treated wood develops a distinct colouration after being sprayed with specific chemical solutions (Ramezanpour *et al.* 2015). However, the visual estimation method may have limitations and often results in over- or underestimation of the treated area in the wood. Therefore, the accuracy of the data obtained with this method is usually questionable, calling for better and more precise estimation techniques (Craciun *et al.* 2011). In this study, thus, a more accurate estimation of the penetration percentage of treatment chemicals into the treated wood was attempted using the ImageJ software, which is unique and has hardly been explored (Ramezanpour *et al.* 2015) by other researchers in this field. ImageJ, an open-source image processing software developed in Java, is ideal for fast analysis of greyscale and binary images. It efficiently extracts basic image information, including resolution, colour mode, channels, units, and pixel distribution. In wood science, ImageJ has been used for various analyses, such as measuring wood formation and morphological parameters (Camarero *et al.* 2024, Mpapa *et al.* 2024, Niu *et al.* 2024, Sun *et al.* 2024), evaluating the size and shape of wood particles (Mazzoli and Favoni 2012, Dobrzanski *et al.* 2024), studying the behaviour of glue lines in glued joints (Alade *et al.* 2022, Maffioletti *et al.* 2024), the study of the distribution and properties of pores in wood (Thilagashanthi *et al.* 2021, Mastantuoni *et al.* 2024), the estimation of the depth and extent of carbonisation in wood samples (Aguiar *et al.* 2024). In addition, the use of ImageJ to assess the depth of impregnation in wood has been reported in the literature (Zhang *et al.* 2024), emphasising its versatility and usefulness in wood science research. However, studies focusing on the analysis of the impregnation depth with image analysis software remain limited to date. Therefore, an attempt was made through the present study to elucidate the potential of the image analysis system ImageJ to estimate the impregnated area in treated wood accurately. The preservative formulations of boron-based agent (BBA) and Silvanolin were selected based on their commercial importance, availability, and ease of use in India and Slovenia,. Norway spruce was selected considering its high import potential to the Indian subcontinent and the known fact that this species, when dried below 30% moisture content, is refractory to impregnation due to the aspirated pits in the tracheid (Kurjatko and Reinprecht 1993, Hansmann *et al.* 2002, Usta 2005) which could be reduced or eliminated through MW. Five different energies comprising medium to high intensities of MW were investigated to understand the effects of MW on ameliorating the retention and penetration in heartwood and sapwood.

This study aimed to investigate the effect of microwave (MW) treatments on the impregnation and retention potential of Norway spruce sapwood and heartwood. In particular, we investigated whether MW treatments improve, reduce, or have no significant effect on these properties depending on the MW energy applied. By evaluating the variations in impregnation and retention performance at different treatment intensities, we aimed to determine the extent to which MW modification affects the permeability of Norway spruce wood.

2. Materials and methods

2.1. Materials

The study used 35-year-old Norway spruce (*Picea abies* (L.) H. Karst.) from the northeastern part of Slovenia. The round timber was processed into planks in a local sawmill. Further processing, such as resawing and planing started about 3 weeks after felling the tree. The dimensions of the samples used for the entire length of the study were l × w × h = 5 × 2.5 × 1.5 cm (EN 113: 2020). The samples were considered if they had no visual anomalies or defects such as knots, resin pockets, cracks or similar and were predominantly semi-radial in orientation; a grain orientation that is between the radial and tangential directions. Sapwood and heartwood samples were stored separately for each test. The outermost part of the board was selected for the preparation of the sapwood samples, as it is difficult to distinguish between sapwood and heartwood in Norway spruce. Since there is no visual difference between the two, we assumed the outermost part of the board to be sapwood. Initial moisture content (IMC) was determined for twenty randomly selected samples for heartwood and sapwood using the gravimetric method. The sapwood samples were in the range of 50–65%, while the IMC of the heartwood samples was 28–33%. The dimensions of the samples prescribed by EN 113 (2020) were used so that they would fit into the microwave chamber due to their small size. A total of ten replicates were considered for each test series (Table 1).

2.2. Preservative formulation

Two preservative solutions were used for the present study. A 6% solution of borax:boric acid (BBA) was prepared by dissolving 30 g of borax and 30 g of boric acid in one litre of deionised water. Silvanolin is a commercially available preservative manufactured by Silvaprodukt. The ingredients of Silvanolin include copper hydroxide carbonate, ethanolamine, quaternary ammonium compounds, boric acid, and octanoic acid; the detailed composition of the product can be found in the patent by Humar and Pohleven (2008). Silvanolin had a total concentration of 4%, with an active copper concentration of 2.5%.

2.3. Microwave treatment

The treatment of wood with microwaves (MW) was carried out using a MW oven (Model: M020MW, Gorenje, Velenje, Slovenia) with a frequency of 2.45 GHz and a maximum power of 700 W. Five MW energies in the range of 700 and 1260 MJ/m^3 were applied to the wood. The energy range was selected based on our preliminary tests and considering the highly refractory nature of Norway spruce wood, especially the heartwood. The duration of MW irradiation and details of the energies are given in Table 1. The parameters of the MW treatments were determined based on the MW power and volume of the samples, as suggested by Kol and Çayir (2021) and as performed by Ganguly *et al.* (2021b). Only one sample was exposed at a time per treatment cycle. The samples were allowed to cool in a desiccator and weighed before being subjected to further treatments. Samples were weighed to the nearest 0.01 g before and after MW treatment to determine weight percentage loss (WPL).

2.4. Calculation of weight percentage loss (WPL)

The WPL for the replicates after MW modification was calculated for each sample as described by Kol and Çayir (2021).

$$WPL = \frac{M_1 - M_2}{M_1} \quad (1)$$

where M_1 is the initial weight of the sample before the MW treatment, and M_2 is the final weight at the end of the MW treatment.

2.5. Dipping and pressure treatment

Two impregnation methods were used: immersion at atmospheric pressure (dipping) and high-pressure treatment with an initial and final vacuum step. These methods were used to evaluate the effect of microwave treatment and to compare different commonly used impregnation techniques. Dipping was carried out for 20 h in a plastic vessel under laboratory conditions. The samples were fully immersed in the preservative solutions and weighed to prevent floating. The samples were then wiped off and weighed to the nearest 0.01 g.

Table 1. Specification for microwave (MW) treatment and sample distribution. S and B denote the preservatives Silvanolin 4% and BBA 6% used in the study.

			No. of samples (Set 1: dipping)		No. of samples (Set 2: dipping + pressure)		
Time (s)	Supplied MW energy (MJ/m^3)	Treatments	Sapwood	Heartwood	Sapwood	Heartwood	Total No. of samples
Control	No MW	CP	20 (10S + 10B)	20 (10S + 10B)	20 (10S + 10B)	20 (10S + 10B)	80
20	700	MW 20	20 (10S + 10B)	20 (10S + 10B)	20 (10S + 10B)	20 (10S + 10B)	80
24	840	MW 24	20 (10S + 10B)	20 (10S + 10B)	20 (10S + 10B)	20 (10S + 10B)	80
28	980	MW 28	20 (10S + 10B)	20 (10S + 10B)	20 (10S + 10B)	20 (10S + 10B)	80
32	1120	MW 32	20 (10S + 10B)	20 (10S + 10B)	20 (10S + 10B)	20 (10S + 10B)	80
36	1260	MW 36	20 (10S + 10B)	20 (10S + 10B)	20 (10S + 10B)	20 (10S + 10B)	80
Total samples for the study							480

The pressure treatments were carried out in a laboratory-scale impregnation chamber (Kambič, Semič, Slovenia) and were conducted at the Biotechnical faculty of the University of Ljubljana at 20 °C. The samples were subjected to pressure impregnation using the full-cell treatment. The samples were first kept under vacuum (−0.085 MPa) for 20 min, then under high pressure (1 MPa) for 3 h, and finally under vacuum (−0.085 MPa) for 20 min. The samples were immersed for a further 20 h at atmospheric pressure to determine retention as accurately as possible, as specified in the leaching standard (ENV 1994). At the end of each treatment, the samples were carefully wiped to remove any dripping preservatives and weighed to the nearest 0.01 g.

2.6. Calculation of preservative retention

Retention of preservatives (active substances) was calculated using:

$$R = \frac{GC}{V} \quad (2)$$

where:

R – retention of preservative,
G – mass of the treating solution absorbed by block,
C – concentration of the treating chemical, and
V – volume of the test block.

2.7. Treatability evaluation

For the evaluation of treatability, only the samples of Set 2, i.e. pressure treatment (Table 1) was considered, as they had higher retention values. The samples were conditioned for two weeks at 60% relative humidity (RH) and 20 °C to achieve better mobilisation and fixation of the preservative solution. Afterwards, the samples were cut into two halves with approximate dimensions (l × w × h = (2.5 × 2.5 × 1.5) cm^3). The BBA-treated samples were then sprayed with curcumin extract and salicylic acid, which developed a reddish colour on the treated parts. The Silvanolin-treated samples were sprayed with a 2% solution of potassium ferrocyanide, which gave the treated parts the characteristic brown colour. For detailed analysis using ImageJ software, four samples with a cumulative cross-sectional area of 30 cm^2 were considered for treatability evaluation and discussed in this article. Eight halves of these four samples were then scanned together to obtain a high-quality image, which was then processed in ImageJ software for binary colour correction. ImageJ can count the number of black (0) and white (255) pixels and calculate their ratio. Black pixels indicated treated areas, and the untreated areas were white (Figure 1). Using ImageJ software, there is a possibility that the dark-coloured parts of the latewood are classified as treated, especially for samples treated with Silvanolin. This could be a possible limitation of the study, and the methodology used, but understandably, this case is rare, and even if it occurs, it is a slight overestimation of the actual data. The percentage of treatability was calculated by measuring the area covered by black pixels and expressing it as a percentage of the total area (30 cm^2).

2.8. Scanning electron microscopy of microwave-treated samples

Scanning Electron Microscopy was performed according to the methodology proposed by Merela *et al.* (2020) to obtain an optimal surface for observation, as described in detail in Ganguly *et al.* (2021b). The purpose of the SEM study was to assess the extent of delamination in the wood microstructure after MW modification that can facilitate the uptake of preservatives. The SEM examination was only conducted for the following samples: Untreated controls, MW 20, and MW 36 for both sapwood and heartwood, respectively. The samples used for the SEM examination were not treated with preservatives.

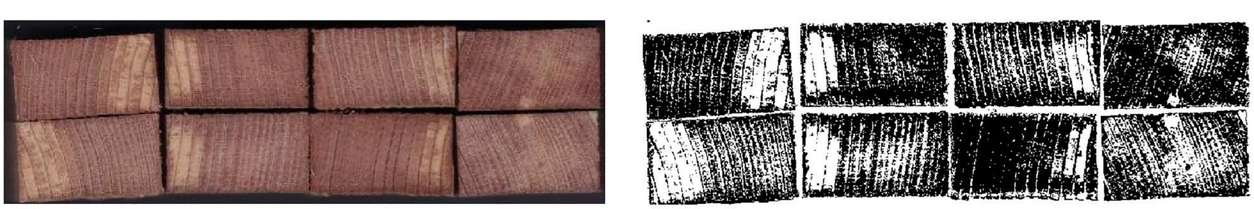
2.9. Statistical analysis

IBM® SPSS® Version 28 software (IBM 2021) was used for the statistical analysis. The mean values of the different treatments were evaluated to analyse the data. A one-way ANOVA was performed for the mean values of the treatment. In addition, a Duncan post hoc analysis was performed at $\alpha = 0.05$ to test for significant difference between treatment means. The samples from Set 1 and Set 2 were analysed separately, and the mean values of the MW-treated heartwood and sapwood samples were compared for statistical significance.

3. Results and discussion

3.1 Weight percentage loss (WPL)

The mean WPL value obtained after the different treatments is shown in Table 2. ANOVA for each wood type showed a significant difference ($p < 0.05$) between the treatment sets. A further post-hoc data analysis was performed using Duncan's homogeneity test. The statistical differences between treatments were represented by different alphabets (a, b, c: for sapwood and m, n, o for heartwood) and the data are presented in Table 2.



a) b)

Figure 1. Treatability evaluation using ImageJ: (a) original scanned photo, and (b) ImageJ representation after binary correction.

Table 2. Mean WPL % exhibited by different sets of MW treatment by sapwood and heartwood samples (Values in parenthesis indicate standard error).

Treatments	Sapwood WPL (%)	Heartwood WPL (%)
MW 20	7.31^{a} (± 1.05)	9.83^{m} (± 0.35)
MW 24	8.58^{a} (± 0.60)	11.98^{n} (± 0.24)
MW 28	12.96^{b} (± 0.99)	12.74^{n} (± 0.19)
MW 32	13.31^{b} (± 1.06)	15.85^{o} (± 0.74)
MW 36	16.95^{c} (± 1.19)	16.62^{o} (± 0.44)

The same letters in the columns indicate no significant difference between treatments, and different letters indicate significant differences between treatments according to Duncan's post hoc analysis ($\alpha = 0.05$).

Table 2 shows that the percentage of WPL in both sapwood and heartwood of Norway spruce increased with increasing MW energy. On average, a weight reduction of 7% to 17% was observed after applying MW energy to wood, and the WPL percentage for the different treatment groups was statistically significant ($p < 0.05$). The trend of WPL observed in this study conformed with the findings of previous studies (Kol and Çayır 2021, Mascarenhas *et al.* 2024b) where higher applied energy led to higher WPL. It is essential to mention here that the extent of mass loss of wood during microwave treatment is a synergistic effect of several factors, which will be explained below. When subjected to microwave treatment, wood is initially partially dried by removing most of the free water. If the treatment is continued over a longer period, the bound water also begins to escape from the matrix in the form of vapour (Oloyede and Groombridge 2000, Xing *et al.* 2023, Mascarenhas *et al.* 2024b). The evaporation and vaporisation of this free and bound water present in the wood can cause the wood to become lighter after MW treatment. Understanding the temperature development in wood during the MW treatment is particularly important (Xiao *et al.* 2018) as prolonged MW exposure can lead to the development of substantially high temperatures., which can also contribute to the thermal decomposition of specific wood components. Xing *et al.* (2023) reported cellulose and lignin decomposition after high-intensity MW. In addition, the high temperatures achieved during higher intensity MW treatment (Terziev *et al.* 2023) can decompose complex organic compounds in the wood, releasing volatile by-products such as gases and small molecules, which can also contribute to weight loss. At higher MW intensity, extractives such as resins, oils and tannins present in the wood matrix often leak out of the vessels, channels, and other cell structures, making the wood lighter. However, WPL can depend on various other factors, including the specific conditions of the treatment (e.g. output, frequency, and duration), the initial moisture content of the wood and the anatomical features (like porosity and pore volume) of the species itself. The process often needs to be carefully controlled to achieve the desired changes without excessive weight loss that could compromise the structural integrity of the wood.

3.2. Preservative retention

Table 3 shows the mean retention obtained in both the sapwood and heartwood samples and in the control samples after treatment with 4% Silvanolin and 6% BBA for different exposure levels. The data set obtained shows that the retention increase is proportional to the increasing exposure to MW radiation, regardless of the preservatives used and their concentrations.

The MW-modified samples lost an average of 7%–17% moisture in all treatment combinations after MW treatment. This partial drying of the samples facilitated the retention of the preservative during the dipping and impregnation process. From Table 3, it can be deduced that the MW modification led to a significant increase in the retention of the preservative solutions for both preservatives in the samples of Set 1 and Set 2. This finding is in line with the results of previous studies (Hermoso and Vega 2016, Samani *et al.* 2019). When analysing the data for Set 1: the immersion set, it was found that the retention values of the untreated controls were significantly ($p < 0.05$) lower than those of the MW-treated samples for all treatment combinations, regardless of the preservatives used or the wood part (sapwood and heartwood). MW 36, i.e. the samples treated with the highest MW energy, showed about 2–2.3 times higher retention in both sapwood and heartwood than CP, the controls. Interestingly, the MW 24 and MW 28 treatments showed statistically similar immersion results to a greater extent. This is especially true for the heartwood samples compared to the sapwood samples, where some statistical significance was found. When comparing the retention values of sapwood and heartwood for the sample from Silvanolin Set 1, a statistical significance of the retention values was found for all treatment sets apart from CP and MW 32. However, a similar comparison for BBA showed that only the control sets for sapwood and heartwood showed statistical similarity, while all other treatment sets differed significantly. In this comparison between sapwood and heartwood, the data sets showed identical increasing trends in preservative retention, apart from the set for MW 32 for Silvanolin-treated sapwood. The decrease in retention values could be due to the internal charring of the samples that occurred after the application of very high MW energy. This led to a thermal runaway phase in which MW absorption increases but heat conduction decreases with increasing temperature, leading to intense localised heating of the wood and production of char, resulting in rapid destruction (Brodie 2005). In addition, the partial charring of the wood delays the absorption of Silvanolin.

The BBA-treated samples showed slightly higher retention values for both sets across all the treatment combinations, including the controls. The higher concentration of BBA used for the study is hypothesised to be the reason for this.

Samples used for Set 2 gave comparable results to those of Set 1 for MW-modified samples. Statistically similar retention values were observed for the control sets of both preservatives regardless of the wood type. The retention values obtained for the MW-modified samples were significantly different from the CP, for all treatment types and in accordance with the results of Set 1. For the Silvanolin-treated heartwood samples, statistically similar values were obtained for MW 24 and MW 28, as well as for the MW 32 and MW 36 treatments. For the sapwood samples, the data were more consistent, and statistical similarities were observed between the MW 20 and MW 24, MW 24 and MW 28, MW 28 and MW 32, and MW 32 and MW 36 treatment sets. The statistical comparison for BBA-treated sapwood showed the same trend, although for BBA sapwood,

Table 3. Mean retention (kg/m^3) values for sapwood and heartwood for different sets of MW-modified samples.

	Set 1: Dipping				Set 2: Dipping + Pressure			
	Heartwood		Sapwood		Heartwood		Sapwood	
Treatments	Silvanolin	BBA	Silvanolin	BBA	Silvanolin	BBA	Silvanolin	BBA
CP	2.53^{a} (± 0.06)	3.04^{ab} (±0.09)	3.16^{abc} (± 0.36)	3.56^{bcd} (± 0.21)	9.60^{p} (± 0.26)	11.30^{q} (± 0.24)	10.05^{p} (± 0.14)	11.71^{q} (± 0.39)
MW 20	3.94^{cde} (± 0.11)	4.67^{efg} (± 0.06)	4.95^{fgh} (± 0.22)	5.65^{hij} (± 0.24)	17.50^{r} (± 0.40)	19.82^{tu} (± 0.73)	17.80^{rs} (± 0.28)	20.04^{tuv} (± 0.32)
MW 24	4.18^{def} (± 0.09)	4.92^{fgh} (±0.09)	5.13^{ghi} (± 0.31)	6.31^{jkl} (± 0.46)	18.19^{rs} (± 0.40)	20.65^{tu} (± 0.43)	18.78st (± 0.36)	21.13^{vwx} (± 0.52)
MW 28	4.65^{efg} (± 0.14)	5.33^{ghi} (± 0.08)	5.93^{ijk} (± 0.29)	7.05^{lm} (± 0.50)	19.00st (± 0.30)	21.10^{vwx} (±0.43)	19.49^{tu} (± 0.42)	21.84^{wxy} (± 0.61)
MW 32	5.02^{fgh} (± 0.14)	5.71^{hij} (± 0.10)	5.56^{hij} (± 0.27)	7.31^{mn} (± 0.57)	19.60^{tu} (± 0.30)	21.87^{wxy} (± 0.29)	20.00^{tuv} (± 0.49)	22.18^{xy} (± 0.45)
MW 36	5.66^{hij} (± 0.20)	6.61^{klm} (±0.10)	6.71^{klm} (± 0.19)	7.99^{n} (± 0.52)	20.10^{tuv} (± 0.32)	22.80^{yz} (± 0.38)	20.98^{vwx} (±0.33)	23.50^{z} (± 0.63)

Note: Standard errors are presented within parentheses. The body of the table shows the average values (at ± 95% confidence interval) of 20 replicates per treatment. Equal alphabets in columns indicate no significant difference between treatments, and different alphabets indicate a significant difference between treatments according to Duncan's post hoc analysis ($\alpha = 0.05$). Alphabets "a" to "n" denote the statistical significance of Set 1 samples. Alphabets "p" to "z" denote the statistical significance of Set 2 samples.

the retention values of MW 36 differed significantly from all other MW-treated sets. The sapwood of Norway spruce is more permeable and, therefore, allows better impregnation. The results obtained with both treatment sets are consistent with the findings of several previous researchers (Torgovnikov and Vinden 2000, Hong-Hai *et al.* 2005, Treu and Gjølsjø 2008), where uptake and retention were increased 2.5–5.3 times and by factors of 8–14. When MW is applied to wood, the water molecules in wood are set in motion and generate heat through movement, friction, and collision, which in return generates internal vapour (Torgovnikov and Vinden 2010, Xiao *et al.* 2018). The increasing vapour pressure can damage the cell tissue of the wood to varying degrees. For example, structures such as tyloses in the vessels, pit membranes in the cell walls (Figure 2) and ray cells can crack. These changes in microstructure can lead to shifts in porosity and pore diameter distribution (He *et al.* 2023), creating new pathways for the passage of fluids. Consequently, they influence critical aspects of the properties of the wood, such as permeability. The heat generated in the wood can also melt, boil, and vaporise resin and other extractives contained in the wood, creating open spaces in the matrix that further aid the retention of preservative solutions. The overall results of this study demonstrate the effectiveness of MW in increasing preservative retention many times over. After pressure treatment, the highly refractory spruce heartwood showed almost comparable retention values to the sapwood samples for BBA and Silvanolin (Figure 3), which is remarkable.

After further analysing the data, to determine the increase in retention compared to the untreated controls, it was found that an increase of 54% to 117% was obtained in the BBA-treated heartwood samples in Set 1. In comparison, in the sapwood the increase varied between 58% and 124% at different applied MW energy intensities (Figure 3). The same trend continued in the Silvanolin samples from Set 1. They showed increases of 60% to 113% for the sapwood and 54% to 123% for the heartwood. It can be concluded that regardless of the wood type, both waterborne preservatives showed similar trends and significant improvements in retention values were recorded with increasing MW energy.

Pressure-treated samples belonging to Set 2 showed a very high retention increase for Silvanolin, especially in heartwood, compared to the untreated controls, which showed an increase in retention values of 82% to 109%. For the sapwood set, the range was from 77% to 108%. The rise for BBA for both sapwood and heartwood samples in Set 2 ranged from 71% to 100% at different treatment energies. It is interesting to note that the MW modification at the highest energy level (MW 36) resulted in an increase in retention values for both preservatives of more than 100% for all treatment combinations. Although, MW modification is not a very energy-intensive process, this is an achievement and a good step towards commercialisation.

Similar trends in retention improvement were observed between the two sets, demonstrating the effectiveness of MW treatment in both submersion and pressure impregnation. The results regarding the increase in retention are consistent with our previous findings, where the effect of MW on heartwood was found to be more pronounced and destructive than on sapwood (Ganguly *et al.* 2021b). This could be since the heartwood samples had a lower moisture content, which resulted in the absorbed MW energy causing rapid vapour formation and higher levels of delamination and destruction (Figure 2) during the thermal runaway phase (Brodie 2005). In sapwood, however, the higher moisture content meant that more energy was used to heat and vaporise the water present in the wood, resulting in less damage.

3.2. Anatomical changes facilitating preservative retention

To further investigate the study's objectives and clarify the effectiveness of MW modification in refractory Norway spruce wood, it is essential to analyse the microstructural changes that occur after MW irradiation. A detailed study on this topic has already been published (Ganguly *et al.* 2021b), in which the effect of MW irradiation on spruce wood at the same applied energy levels were explained. The results of the present study were analysed anatomically with SEM micrographs to detect similar structural changes that facilitate the

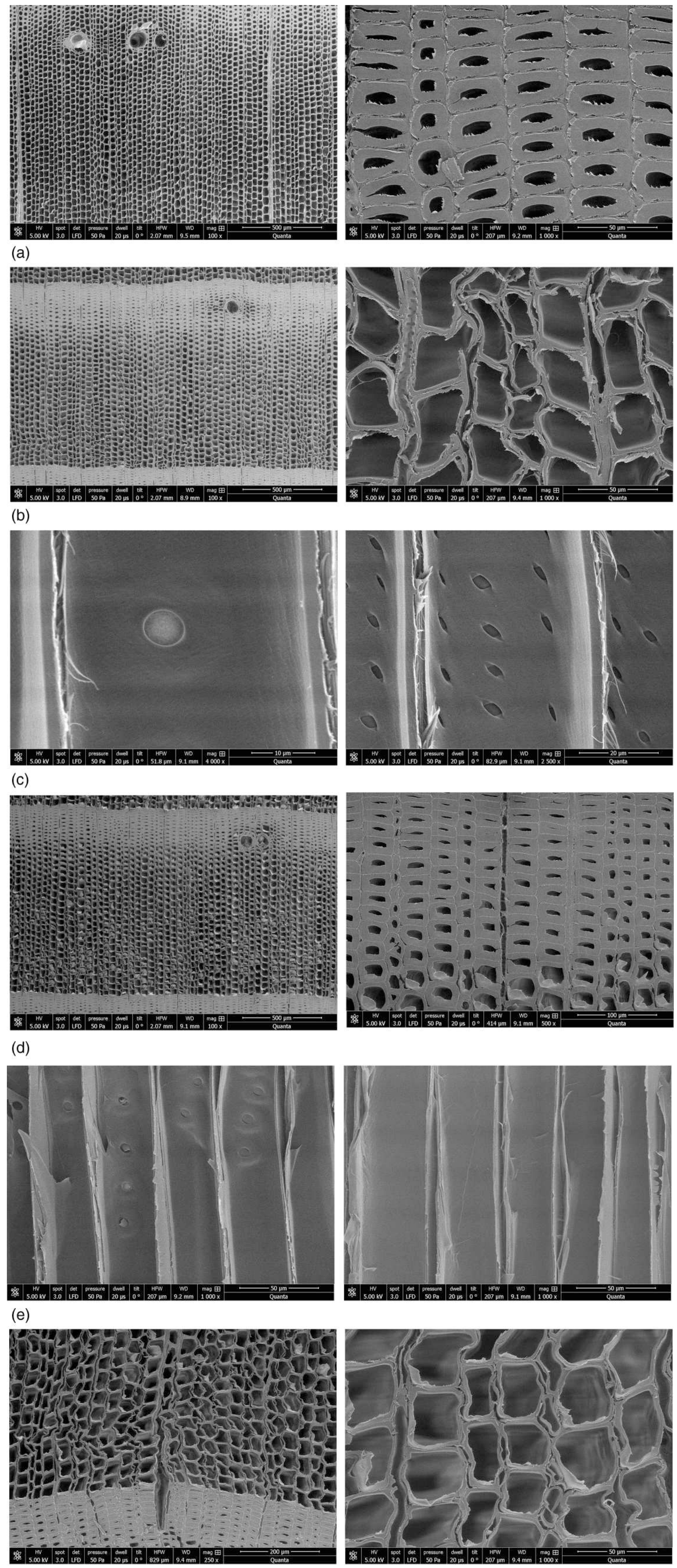


Figure 2. Anatomical structure of control and MW-modified Norway spruce: (a) control samples showing sound anatomical structure, (b) MW 20 heartwood samples (cross section) exhibiting structural delamination, (c) MW 20 heartwood samples (radial section) exhibiting structural delamination, (d) MW 20 sapwood samples (cross section) exhibiting structural delamination, (e) MW 20 sapwood samples (radial section) exhibiting structural delamination, (f) MW 36 heartwood samples (cross section) exhibiting structural delamination, (g) MW 36 heartwood samples (radial section) exhibiting structural delamination, (h) MW 36 sapwood samples (cross section) exhibiting structural delamination, and (i) MW 36 sapwood samples (radial section) exhibiting structural delamination.

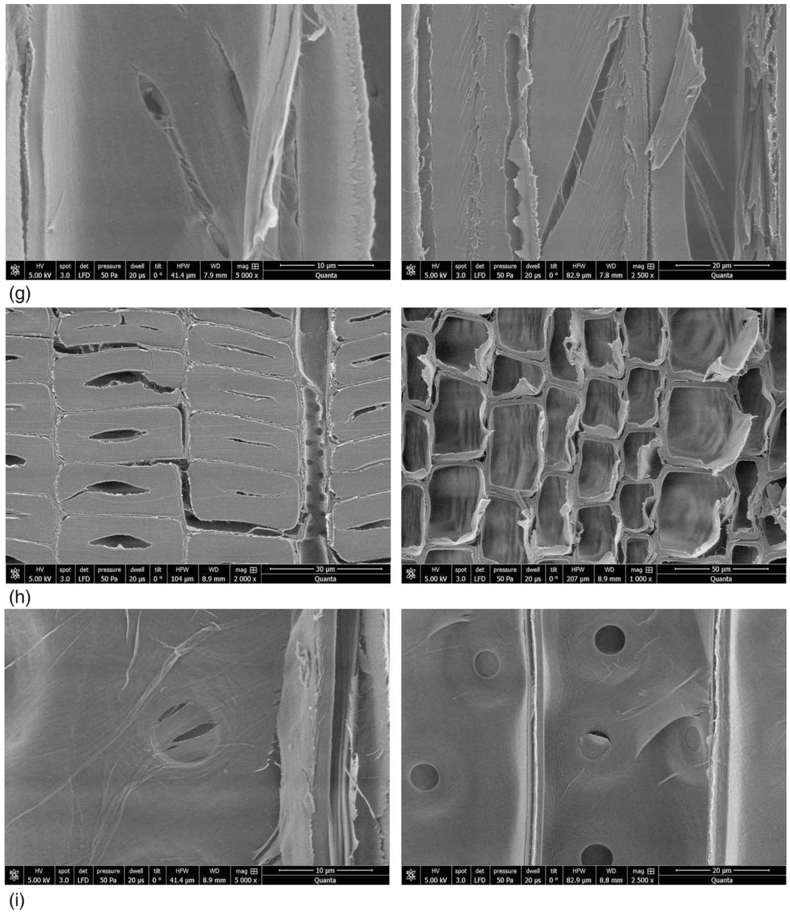


Figure 2. *Continued.*

retention and penetration of treatment chemicals. The SEM analyses revealed distortions in the structure of the earlywood, delamination of the cell walls in the earlywood and latewood of the MW-treated heartwood samples. Microcracks occurred along the radial and tangential walls of the tracheid between the middle lamella and the primary wall. Similar anatomical delamination in MW modified Chinese Fir was also reported by Weng *et al.* (2020), who also correlated the anatomical changes to increase in pore diameter and number, which was also endorsed by Samani *et al.* (2019). No deformation of the earlywood was observed in the sapwood, but in addition to the delamination of the tracheid cell wall and the microcracks, the bordered pit membranes and cross-field pits were also damaged (Figure 2).

The treatment of the heartwood with MW 20 thus improved the impregnation of the wood by allowing the wood preservatives to flow more quickly through the cell wall, improving its permeability and retention capacity for the preservatives, as shown in Table 2. On the other hand, the treatment of the heartwood with MW 36 was extreme and led to rapid delamination of the microstructure, which could have a negative effect on the strength properties. The impact of MW 36 on the sapwood were less pronounced, which is consistent with our previous results (Ganguly *et al.* 2021b). A likely reason for this could be the difference in IMC of heartwood and sapwood samples. Liao *et al.* (2024) also reported a similar impact of high intensity MW on the anatomy of Chinese Fir heartwood. The higher IMC of the sapwood (50–65%), which was significantly higher than its FSP, resulted in more MW energy being used for free water evaporation, leading to less changes in the anatomical matrix. The anatomical study therefore demonstrates the importance of IMC in the wood before it is subjected to MW irradiation. Due to the more pronounced effect on the heartwood, which has less IMC, the MW modification reduced the refractoriness of the spruce heartwood very effectively and caused it to retain the preservative solution to an extent statistically comparable to that of the sapwood when subjected to pressure impregnation (Set 2; Table 3).

3.3. Treatability improvement

The retention of wood preservatives into the wood alone often does not provide sufficient protection unless they continuously penetrate the microstructure of the wood. Previous studies have shown that Norway spruce wood is not resistant to fungi and insects, which necessitates the use of preservatives (Beikircher *et al.* 2012). However, due to the low permeability of the wood species, the retention of preservatives is often limited, a challenge that can be effectively solved by microwave treatment (MW) (Harris *et al.* 2008). In this study, MW treatment of Norway spruce wood improved its treatability with preservatives (Table 4 and Figure 4).

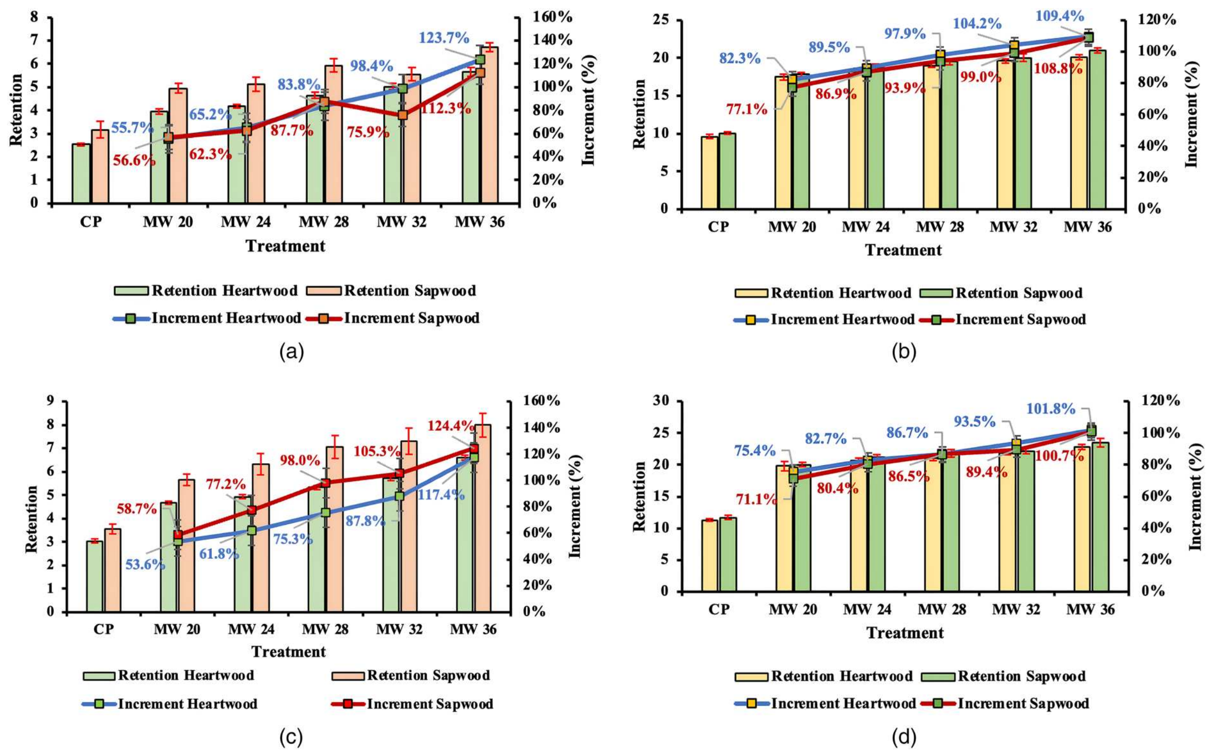


Figure 3. Increment in retention values obtained by different treatments: (a) Silvanolin dipping (Set 1), (b) Silvanolin pressure (Set 2), (c) BBA dipping (Set 1), and (d) BBA pressure (Set 2).

The highest treatability of Norway spruce heartwood was observed with the preservative BBA at MW 36 (Figure 4), with a maximum treatability of 92.49%, and a minimum value was observed for CP at 68.75%. The treatability of samples irradiated with MW energy was between 77% and 93%, 8%–14% higher than the control.

In contrast, Norway spruce sapwood showed a higher overall treatability (Figure 5), with a maximum treatability of 96.45% and a minimum of 85.89% for intensities MW 36 and MW 20, respectively. The increase in treatability due to microwave treatment was 7%–17% for sapwood, indicating that the preservatives can easily pass through the wood.

The samples treated with Silvanolin also showed a significant increase in treatability. However, in the case of Silvanolin, the heartwood samples showed lower treatability compared to their BBA counterparts (Figure 6), which could be due to the high diffusivity of boron-containing preservatives in the wood.

The high diffusivity of boron in wood was also supported by the creation of new pathways after MW modification, which led to a significantly higher penetration of the preservative. Diffusion continues if there are concentration differences between the solution on the wood surface and the chemical concentration in the free water in the wood (Ra *et al.* 2001). Overall, treatability increased with increasing microwave intensity and ranged from 43% to 54% (Table 4), with MW 24 and MW 28 giving almost identical percentages for the heartwood. The treatability of sapwood was higher than that of heartwood, ranging from 62% to 76% for microwave-modified samples compared to 57% for the control. Both the sapwood and heartwood samples showed an average increase of 20% between the control set and MW 36. The results of the current study are consistent with those of Samani *et al.* (2019), in which the authors reported a significant increase in treatability of *Melia composita* wood modified with MW and treated with various preservative formulations after 24 h of immersion compared to untreated samples. The researchers found that a MW intensity of 840 MJ/m^3 was most effective in improving the retention of these preservatives.

Table 4. Treatability percentage of samples modified with different MW energies.

Set 2: Dipping + Pressure	Treatability(%) heartwood	Treatability (%) sapwood	Treatability (%) heartwood	Treatability (%) sapwood
Preservative used	BBA		Silvanolin	
CP	68.75%	79.27%	33.02%	57.36%
MW 20	76.96%	85.89%	42.87%	62.21%
MW 24	81.15%	86.17%	44.12%	69.85%
MW 28	87.44%	91.34%	44.54%	70.84%
MW 32	90.77%	93.58%	49.47%	71.85%
MW 36	92.49%	96.45%	53.65%	76.12%

The study by Xing *et al.* (2023) investigated the effects of microwave (MW) treatment on the chemical composition, chemical structure, and cellulose crystallinity of wood. The results showed that MW treatment changed the chemical composition of wood by increasing the relative concentration of lignin and decreasing the relative content of cellulose and hemicellulose. In addition, the O/C ratio decreased with increasing microwave energy density. The researchers concluded that MW treatment leads to a degradation of holocellulose and an increase in the relative lignin content. Lignin and hemicelluloses, in their various forms are the main elements responsible for the performance of the wood when treated with preservatives. In addition, at very high energy intensities, the anatomical changes at the micro level and the micro

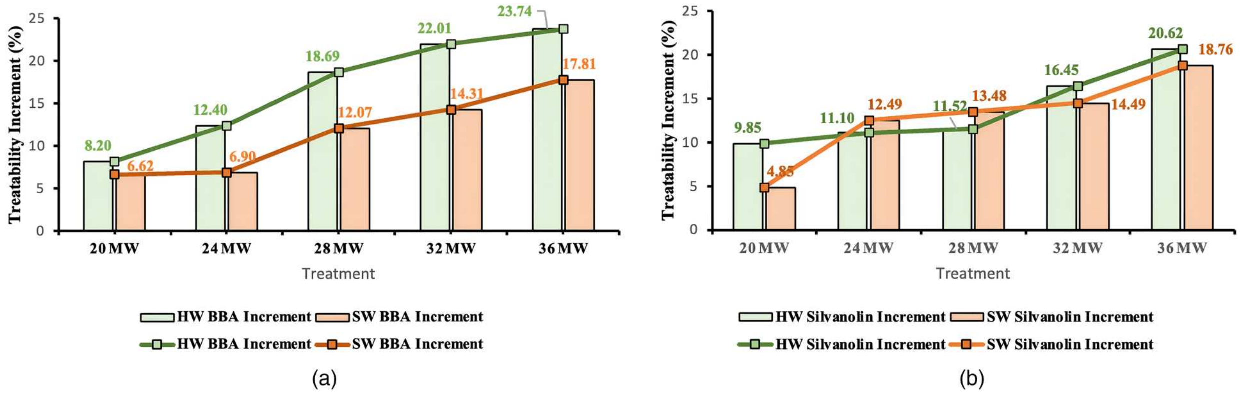


Figure 4. Percentage increase in treatability: (a) BBA (SW: sapwood; HW: Heartwood) and (b) Silvanolin (SW: sapwood; HW: Heartwood).

cracks and fissures formed on the wood surface increase in extent and size, transforming the wood into a highly porous material, which also leads to a significant improvement in permeability (Weng *et al.* 2020, Terziev *et al.* 2023). These findings of anatomical changes were also observed in the present study (Figure 2), which significantly impacted the treatability and subsequent penetration. Possible alteration in the chief polymeric constituents after MW, as analysed by Xing *et al.* (2023), could also be one of the reasons behind the improved performance in terms of treatability. The sapwood showed less improvement in treatability by microwave modification than the heartwood, which could be due to the higher moisture content leading to less anatomical and chemical changes. This was also emphasised by previous researchers for radiata pine wood (Xing *et al.* 2023), where the changes in the chemical structure of the heartwood were more pronounced. In addition, the lower permeability of the heartwood may have resulted in a significantly higher vapour pressure and temperature causing

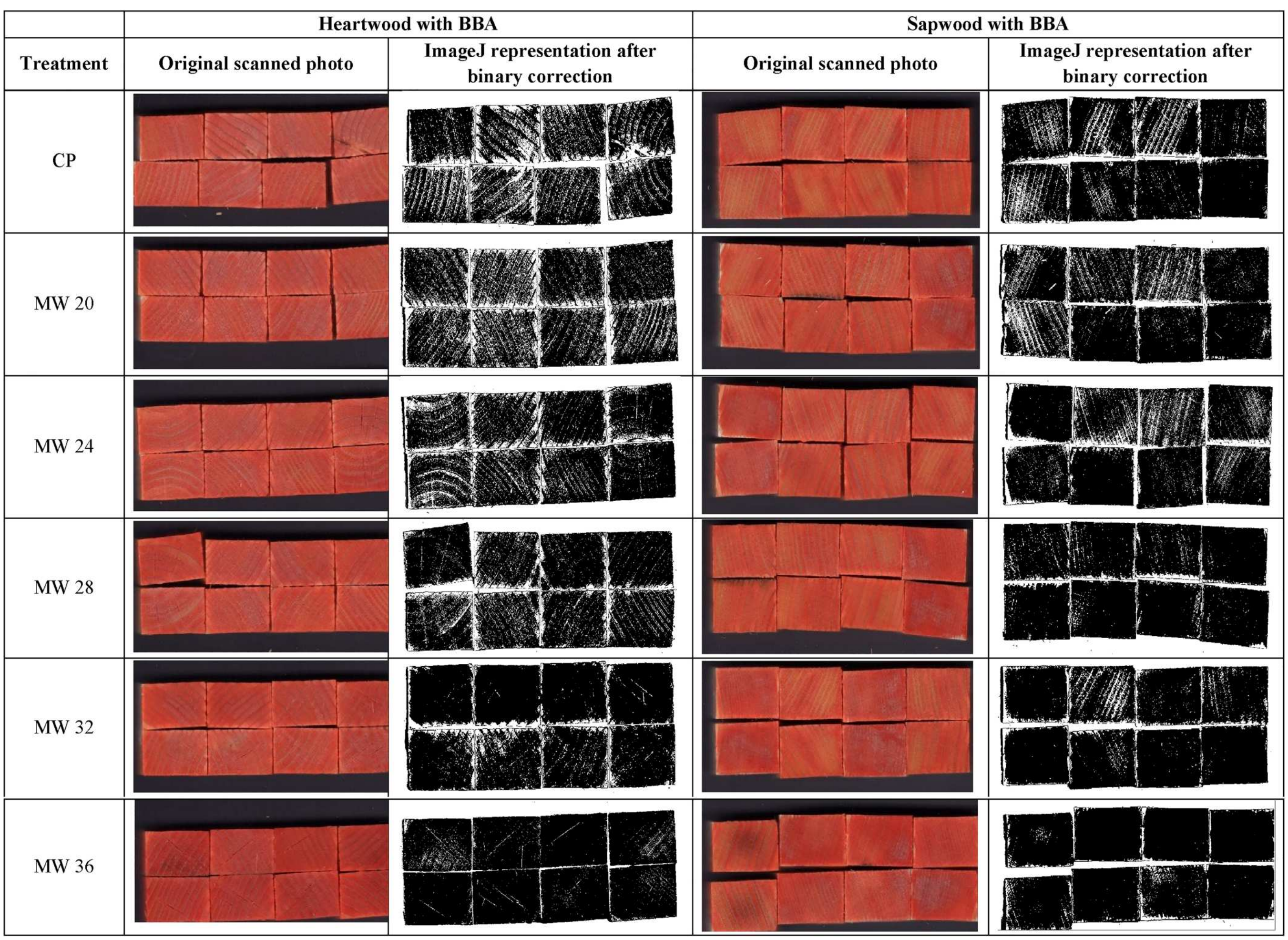


Figure 5. Treatability of samples treated with BBA.

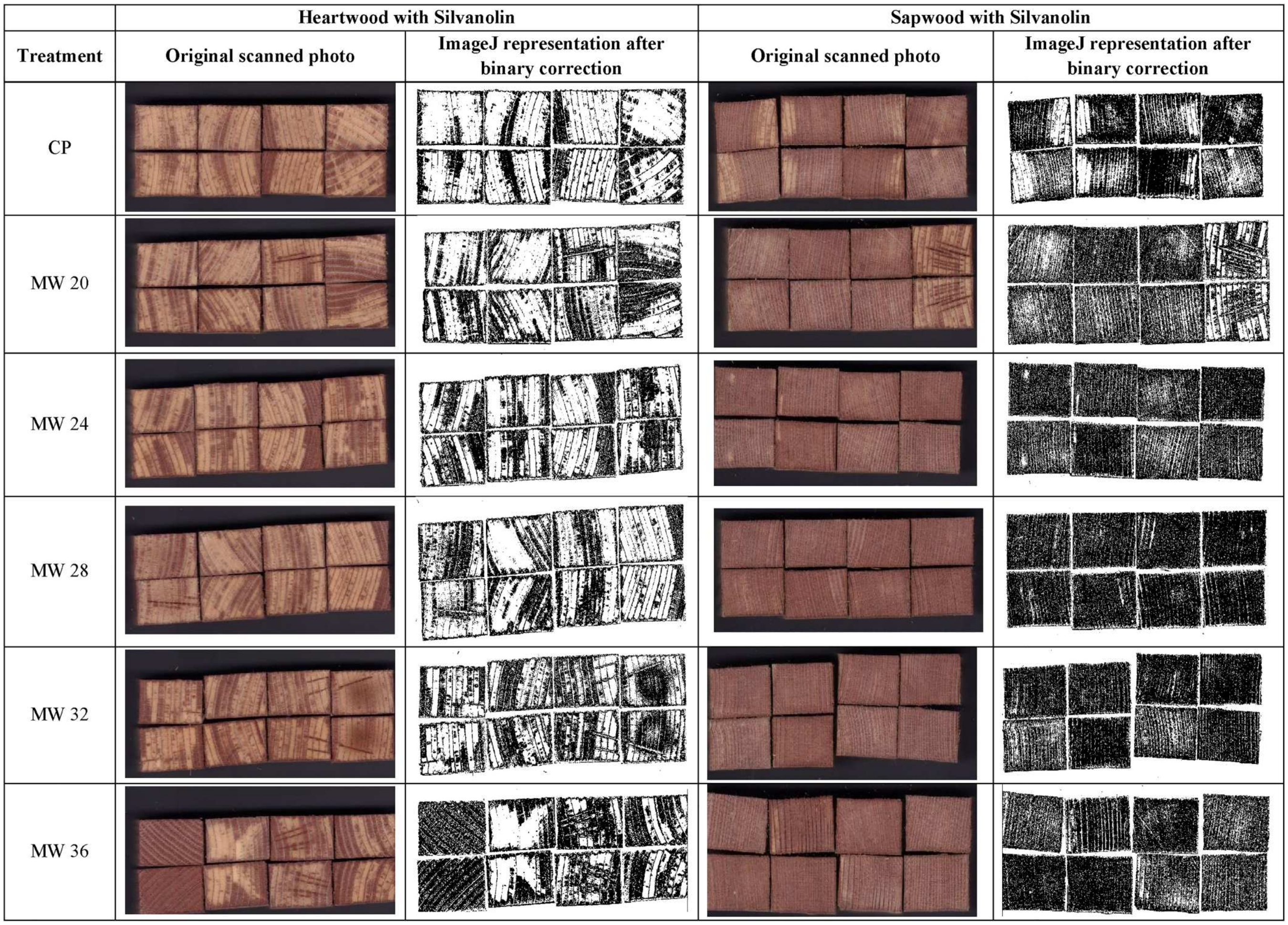


Figure 6. Treatability of samples treated with Silvanolin.

hydrothermal alteration, so the effects of MW on the heartwood were more pronounced (Ganguly *et al.* 2021b, Xing *et al.* 2023). In addition, MW can reduce the free and bound water in the cell lumens and cavities, which can lead to better absorption and distribution of preservatives.

4. Conclusions

The aim of the study was to investigate the effect of microwave treatments (MW) on the impregnation and retention potential of Norway spruce sapwood and heartwood to improve the service life and preservation of the wood, especially in the tropical climate of India where many imported wood species perform poorly. The focus was on the application of microwave treatment abundant, which has attracted attention due to its efficiency and effectiveness in modifying wood for better preservation.

MW treatment was primarily aimed at reducing the refractoriness of Norway spruce wood. The study has shown that microwave treatment (MW) is an effective method for improving preservative retention and permeability of Norway spruce wood, especially in heartwood, which is typically refractory. Compared to other thermal and chemical modification methods, MW treatment proved to be faster and more energy efficient. The results showed that MW treatment increased the retention of the two preservatives BBA and Silvanolin, with the highest retention observed at the highest MW energy levels. In addition, the MW treatment improved the treatability of the wood, especially for water-based preservatives, by disrupting anatomical structures and forming pathways that facilitated the movement of the preservatives. Analysis with ImageJ software ensured accurate measurements and minimised the risk of overestimating the treated area. In particular, the MW treatment significantly improved the treatability of Norway spruce heartwood, which is normally refractory.

To summarise, MW treatment has proven to be effective in improving the retention and penetration of wood preservatives in Norway spruce wood. It is an energy-efficient and time-saving pre-treatment technique that improves the treatability of low permeability wood species, thus offering the potential for improved wood protection and contributing to the development of more sustainable and durable wood products.

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ORCID

Sauradipta Ganguly http://orcid.org/0000-0001-9190-2621
Marko Petrič http://orcid.org/0000-0002-0283-7985
Angela Balzano http://orcid.org/0000-0002-6494-5850
Sanjeet Kumar Hom http://orcid.org/0000-0003-1289-7161
Varun Sharma http://orcid.org/0009-0004-1717-6931
Davor Kržišnik http://orcid.org/0000-0003-0324-4740

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