

Article

Improving the Physical Characteristics and Durability of Wood Through a Combined Modification Process Using Thermal Treatment and Wax Impregnation in One Step

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Abstract

In this paper, a combined modification method using thermal modification and wax impregnation was investigated. The advantage of this method is that the two modification steps are completed in one step. Two different wood species, beech (*Fagus sylvatica*) and Scots pine (*Pinus sylvestris*), were investigated. The effects of the treatments were tested regarding the wax uptake, mass loss, density, equilibrium moisture content, swelling, water contact angle, strength properties, and durability. Through the synergistic effect of the combined modification, it was possible to significantly improve the dimensional stability and decrease the hygroscopicity and equilibrium moisture content, while swelling anisotropy was not affected. It was proven that the wax uptake during this method is highly dependent on the treatment temperature, resulting in a large density increase. The treatment resulted in an obvious color change as well. Bending strength was not affected by the combined treatment, while impact bending, compression strength, and Brinell hardness were improved. High durability was observed after the combined modification method, indicating that lower treatment temperatures are enough to efficiently protect the wood.

Keywords: thermal modification; wax impregnation; paraffin; wood durability; physical properties; color change; dimensional stability; wettability



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1. Introduction

Wood thermal modification and wax impregnation are well known methods to enhance the performance and durability of wood materials and make them more suitable for various applications, especially outdoors. These processes have gained significant attention in recent years due to their ability to enhance wood properties without using biocides.

Thermal modification involves the application of controlled heat to wood, changing its physical and chemical structure. Processes are typically carried out at temperatures between 160 and 220 °C and can be performed under various conditions, including inert gas (most commonly nitrogen), vacuum, steam, or oil medium [1,2]. The primary effects of thermal modification include increased dimensional stability, improved decay resistance, and enhanced aesthetic qualities, such as uniform color [3,4]. These improvements make thermally modified wood suitable for both indoor and outdoor applications, extending its usability and lifespan. However, it has to be mentioned that thermal modification processes also have some negative effects, like reduced mechanical properties, brittleness, poor wettability, and color changes, which might be considered unwanted effects [5–7]. Thus, it is necessary to optimize the parameters of thermal modifications to improve their outcome; one common way is a combination with other modification processes [8–11].

Wax impregnation significantly enhances the durability and stability of wood by providing a protective barrier that reduces water absorption and increases resistance to biological decay. The mode of action of this process involves filling the wood micro- and macropores with waxes, such as paraffin or beeswax, and forming a hydrophobic layer. This layer effectively reduces the wood's hygroscopicity, thereby improving its dimensional stability and minimizing warping or swelling when exposed to moisture [12,13]. Enhanced stability makes wax-impregnated wood particularly suitable for outdoor applications, where exposure to varying environmental conditions is common [14].

Thermal modification alters the color and texture of wood, enhancing its aesthetic appeal. Wax impregnation not only enhances the protective qualities of wood but also improves its aesthetic appeal. The process can enrich the wood's natural color and pattern, providing a smooth, glossy finish. This aesthetic enhancement is particularly valuable in applications where the appearance of the wood is critical, such as in high-end furniture and interior design. The wax layer can also highlight the natural grain patterns of the wood, which adds to its visual appeal [2,15].

The synergy between thermal modification and wax impregnation offers a comprehensive approach to improving wood properties. Combined treatments have been shown to optimize both the physical and mechanical properties of wood, making it more versatile and suitable for a wider range of applications [8,13,14,16]. These processes also provide solutions to improve wood's performance while minimizing its environmental impact. As the demand for sustainable building materials grows, the importance of these techniques is likely to increase, driving further research and development in the field. These processes offer eco-friendly alternatives to traditional chemical-based preservative treatments. Wax impregnation is considered an environmentally friendly wood treatment method, as it utilizes natural or synthetic waxes that are less harmful to the environment compared to traditional chemical preservatives during utilization. The process does not release toxic substances, making it safer for both the environment and human health [17]. By extending the lifespan of wood products, thermal modification and wax impregnation also contribute to sustainability by reducing the need for frequent replacements and conserving forest resources [18]. By improving physical and mechanical properties, these treatments add economic value to lower-grade wood species, making them suitable for a wider range of applications [12,13]. Both processes have been successfully applied to a variety of wood species, demonstrating versatility and adaptability in different contexts and environments [14,19]. The advancements in wood thermal modification and wax impregnation highlight their potential as sustainable alternatives to traditional wood preservation methods [4,20]. These factors underscore the significance of wood thermal modification and wax impregnation in advancing the wood industry toward more sustainable and efficient practices.

Current research often lacks optimization studies that consider the full range of variables in thermal modification and wax impregnation processes. These include temperature, duration, type of wax, and wood pre-treatment conditions. Optimizing these parameters could significantly enhance the efficiency and effectiveness of the treatments, leading to better performance outcomes. However, recent studies are usually based on separate impregnation and thermal modification steps, which make the process rather complicated and long. On the other hand, oils are used as a treatment medium instead of waxes, which are less efficient regarding dimensional stabilization or decay resistance compared to wax impregnations [8,16,19,21–24]. The aim of this study was to use a simplified one-step method for the combination of thermal modification and paraffin wax impregnation. Most important physical (density, color, and weight percent gain), mechanical (bending, impact bending, and compression strengths and Brinell hardness) properties, wood–water rela-

tions (equilibrium moisture content, swelling anisotropy, swelling coefficient, anti-swelling efficiency, and surface contact angle) and decay resistance were tested to have an overview on the changes caused by the combined modification.

2. Materials and Methods

2.1. Materials

Defect-free European beech (*Fagus sylvatica* L.) and Scots pine (*Pinus sylvestris* L.) sapwood were used for the tests, with an initial moisture content of $10 \pm 2\%$. Paraffin wax was purchased from the company MOL Nyrt. (Budapest, Hungary). The type of paraffin was a commercially available product, with the trade name FR DWC 6264. The melting range of the wax is 62–64 °C, while its density is 0.84 g/cm³.

2.2. Modification Method

Thermal modification was conducted in an experimental autoclave produced at the University of Sopron (Sopron, Hungary). Dimensions of the autoclave vessel are 75 cm in diameter and 100 cm in length. The equipment has a PLC-regulated temperature control coupled with a pt-100 thermometer in the autoclave. The modification autoclave is sealed, providing a closed system during the thermal modification process. The heating medium in the vessel was air. Treatment temperatures used were 180 °C and 200 °C, while treatment duration was fixed at 5 h, based on some of our preliminary experiments. Beech and Scots pine laths with the dimensions of 25 mm × 50 mm × 350 mm were used for the treatments.

For the combined treatment, the paraffin wax was pre-melted in a smaller vessel placed inside the autoclave and heated up to 80 °C (over its melting range). The parallelly pre-heated (beside the wax in the treatment vessel) wooden laths were immersed in the wax and weighed down by inert, non-porous weights to keep them covered entirely by the wax throughout the treatment process. The impregnation was promoted by keeping the laths in the hot wax during the cooling phase. This way, the samples were impregnated with the wax in one step within the thermal modification process, without using an overpressure step. These treatments are referred to as TMW180 and TMW200 later on. Detailed treatment parameters are presented in Table 1.

Table 1. Process parameters of the thermal modification method. Where immersion is indicated, there is no significant paraffin uptake; paraffin is only a heat-transferring medium. In case impregnation is indicated, paraffin uptake occurs during the process.

Modification Step/Parameters	Temperature [°C]	Duration [h]	Heating Rate [min/°C]	Wax Application Steps in TMW
Heating up phase	up to 80	8	8	no
Immersion of laths into wax (only TMW)	80	-	0	immersion
Temperature increase-I	80 → 100	3	9	immersion
Drying phase	100	12	0	immersion
Temperature increase-II	100 → 180/200	10.5/12	8	immersion
Thermal modification phase	180 or 200	5	0	immersion
Cooling phase-I	down to 80	10/12	6	impregnation
Cooling phase-II	down to room temperature	2	not controlled	no

Another set of laths was thermally modified in an air atmosphere, without using paraffin wax. This material served as a control treatment for comparison, to determine the

additional effects of the combined treatment. Except for the treatment medium, the same parameters were used for this treatment as well. These treatments are referred to as TM180 and TM200 later on.

2.3. Test Methods

Wax uptake (WU), the pore filling ratio (PFR), and mass loss (ML) were calculated during the process. Laths were conditioned at 20 °C and 65% relative humidity (RH) before the treatment (Binder KBF 115). Additionally, their moisture content was determined after conditioning by cutting 20 mm long pieces from their ends (25 mm × 50 mm × 20 mm). These pieces were weighed, oven-dried, and weighed again to determine the initial moisture contents (MC_i) of the laths. Laths were weighed before treatment (m_u). The initial dry weight (m_0) of the laths was calculated according to Equation (1):

$$m_0 = \frac{m_u}{1 + \frac{MC_i}{100}} \quad (1)$$

Wax uptake was measured indirectly, by weighing the wax before (m_{w1}) and after (m_{w2}) the treatment. It was calculated according to Equation (2):

$$WU = \frac{m_{w1} - m_{w2}}{m_{w1}} \cdot 100 \quad (2)$$

Mass loss (ML) was measured indirectly by weighing the dry weights of the laths after the treatment (m_{0w}). Initial dry weight (m_0) was calculated according to Equation (1). To determine the dry weight of the laths without the wax (m_{0TM}), it was necessary to correct the weights by subtracting the weight of wax taken up ($m_{w1} - m_{w2}$) from the dry weight of the modified laths (m_{0M}). Thus, ML, as a result of thermal modification, was calculated according to Equation (3):

$$ML = \frac{m_0 - m_{0TM}}{m_0} \cdot 100 \quad (3)$$

The pore filling ratio (PFR) was calculated according to the method described by a previous study [25], where m_p is the weight of the paraffin taken up by the lath, m_0 is the oven-dry weight of the lath, ρ_p is the density of the paraffin (0.84 g/cm³), ρ_0 is the oven-dry density of the lath, and ρ_{CW} is the density of the cell wall (1.472 g/cm³ for beech and 1.489 g/cm³ for Scots pine) [26]. PFR is a theoretical value in this case, as the literature value is used for the cell wall density. It was calculated according to Equation (4):

$$PFR = \frac{\frac{m_p}{m_0} \cdot \frac{1}{\rho_p}}{\frac{1}{\rho_0} - \frac{1}{\rho_{CW}}} \cdot 100 \quad (4)$$

The color of the laths was measured before and after the modification processes according to the CIELab color system using Konica Minolta CM2600d equipment. After the treatments, the color was measured directly after cooling down to room temperature and after planing the surface (~1 mm depth), as there were visible differences detected between the surface and the middle of the laths. Changes in each color coordinate (ΔL^* , Δa^* , and Δb^*) and the total color change (ΔE^*) were calculated, as described previously [27].

Oven-dry density (ρ_0) was measured using samples with dimensions of 20 mm × 20 mm × 30 mm, according to ISO 13061-2 [28]. A total of 10 samples were tested for each modification, and 10 untreated samples served as controls.

Equilibrium moisture content (EMC) at 20 °C and 65% RH and after immersion under water was measured using samples with dimensions of 20 mm × 20 mm × 30 mm, according to ISO 13061-1 [29]. A total of 10 samples were tested for each modification, and

10 untreated samples served as controls. The same set of samples was used to determine volumetric swelling [30–33] after both climatization (S_V) and saturation by immersion under water ($S_{V_{max}}$) and, additionally, anti-swelling efficiency ($ASE_{20/65}$ and ASE_{sat}) [27]. Measurements started from oven-dry conditions, followed by climatization at 20 °C and 65% RH. The last step was to immerse the samples under water for 10 days. The swelling coefficient (SC) was calculated between the oven-dry and climatized conditions using S_V and $EMC_{20/65}$, according to Equation (5):

$$SC = \frac{S_V}{EMC_{20/65}} \quad (5)$$

Swelling anisotropy (A_{sw}) was calculated from the tangential and radial swelling (S_T and S_R) of the same set of samples between oven-dry and climatized conditions, according to Equation (6):

$$A_{sw} = \frac{S_T}{S_R} \quad (6)$$

The water contact angle (θ) was measured with a PGX+ Pocket Goniometer (Columbia, S.C., USA) using 4 μ L distilled water droplets. Ten measurements were completed for each modification and for the untreated control material on planed surfaces. Planing ensured the removal of excess paraffin wax from the surface in TMW samples. The same preparation was used for all samples (TM, TMW, and control) before measurement.

Before mechanical testing, all samples were climatized at 20 °C and 65% RH until reaching constant mass. Static bending was tested with Instron 4208 (Norwood, MA, USA) equipment using samples with dimensions of 20 mm $\times$ 20 mm $\times$ 300 mm, according to ISO 13061-3 [34]. A total of 10 samples were tested for each modification, and 10 untreated samples served as controls. Impact bending was tested with $\dot{U}T$ 1000-1 Charpy hammer (Budapest, Hungary) equipment using samples with dimensions of 20 mm $\times$ 20 mm $\times$ 300 mm, according to ISO 13061-10 [35]. A total of 10 samples were tested for each modification, and 10 untreated samples served as controls. Compression strength parallel to the grain was tested with Instron 4208 (Norwood, MA, USA) equipment using samples with dimensions of 20 mm $\times$ 20 mm $\times$ 30 mm, according to ISO 13061-5 [36]. A total of 10 samples were tested for each modification, and 10 untreated samples served as controls. Brinell hardness at the tangential surface was tested with Instron 4208 (Norwood, MA, USA) equipment using samples with dimensions of 50 mm $\times$ 50 mm $\times$ 20 mm (longitudinal $\times$ tangential $\times$ radial), according to EN 1534 [37]. A total of 10 samples were tested for each modification, and 10 untreated samples served as controls.

Durability was tested according to CEN/TS 15083-1 using samples with dimensions of 15 mm $\times$ 25 mm $\times$ 50 mm. The test fungus was *Coniophora puteana*. A total of 5 samples were tested for each modification, and 5 untreated samples served as controls. The duration of the test was 16 weeks. No leaching test was performed prior to inoculation.

Distribution normality of the data (WU, ML, ρ_0 , EMC, S_V , ASE, SC, A_{sw} , θ , color change, mechanical tests, and durability) was verified, and statistical significance tests (ANOVA, Fischer LSD-test, $p < 0.05$) were conducted for the effect of the treatment on the investigated material properties with Statistica 10.0 (Statsoft) software.

3. Results and Discussion

3.1. Wax Uptake and Mass Loss

Paraffin wax uptake and mass loss results are represented in Table 2. Pine sapwood represented significantly higher WU compared to beech at both treatment temperatures. This is considered a result of the different pore volumes of the species (~67% of pine and ~55% [38]) and differences in the anatomical structure, as the treatability of the materials is

equivalent according to EN 350 [39]. WU was remarkably higher in the 180 °C treatment for both wood species. It was approximately two times the value of the higher treatment temperature. Optical observation did not indicate inhomogeneity in the wax uptake after cutting the laths in half longitudinally (Figure 1). A similar phenomenon was reported previously for oil heat treatment of wood. Oil uptake is inversely proportional to the treatment temperature in oil heat treatment of wood [17,40–42]. The oil uptake occurs for two reasons during the process. On the one hand, there is the natural absorption of the liquid treatment medium into wood, as they are in contact during the process. However, if wood is immediately taken out from the medium at the end of the thermal modification process, the uptake is rather small, as the outflow of the degradation products hinders the deeper uptake. On the other hand, if there is a cooling step at the end, uptake is highly promoted, and cooling time has a high influence on the uptake ratio. The impregnation was driven by thermal contraction of the gaseous atmosphere of the cell lumens and cell wall pores during this process step. It caused a relative vacuum, resulting in the penetration of the liquid treatment media [17,23,43].

Table 2. Paraffin wax uptake (WU), mass loss (ML) caused by thermal modification as a result of thermal modification in air atmosphere (TM180 and TM200) and combined one-step thermal modification and wax impregnation (TMW180 and TMW200), and pore filling ratio (PFR). Values in brackets represent the standard deviation, while different superscript letters indicate a significant difference between the WU or ML of samples at the $p < 0.05$ level.

	WU [%]		ML [%]		PFR [%]	
	Beech	Scots Pine	Beech	Scots Pine	Beech	Scots Pine
TM180	-	-	3.77 ^a (0.78)	3.50 ^a (0.19)	-	-
TM200	-	-	7.14 ^b (0.87)	6.57 ^b (0.56)	-	-
TMW180	52.84 ^a (1.30)	133.15 ^b (2.64)	7.27 ^b (0.42)	7.82 ^b (1.27)	64.81 ^a (1.92)	85.54 ^b (3.07)
TMW200	28.22 ^c (1.29)	55.94 ^a (4.39)	12.37 ^c (2.05)	12.32 ^c (1.34)	41.04 ^c (1.83)	48.35 ^d (4.87)

Mass loss was significantly affected by the treatment medium (Table 2); as a result, ML was higher when using melted paraffin as a treatment medium (TMW) instead of a gaseous atmosphere (TM). Using a liquid medium for thermal modification is considered a more intense treatment method, as the specific heat of the liquid medium is remarkably higher; thus, the heat transfer is more intense as well. This causes additional thermal degradation in methods using liquid treatment medium, compared to the methods using gaseous atmospheres, with all the other treatment parameters being the same [17,42]. Wood species did not influence ML, as there was no significant difference between ML of the different wood species under the same treatment conditions. Oxidation and evaporation of the extractive have a minor role in mass loss during thermal modification as well; however, the main reason for this is the degradation of hemicellulose and lignin, as cellulose is considered to be less sensitive to thermal degradation because of its partly crystalline structure [44–48].

The pore filling ratio was calculated to show the efficiency of the impregnation step. In accordance with the WU results, there was a significant effect of the treatment temperature. The PFR was considerably higher in TMW180 treatment compared to TMW200, as the PFR was 64.81% and 41.04% for beech and 85.54% and 48.35% for Scots pine, respectively. The results show that the impregnation did not fill every void in the wood; however, it has to be taken into consideration that no overpressure was used during the process. Especially from this point of view, this can be considered a good result.

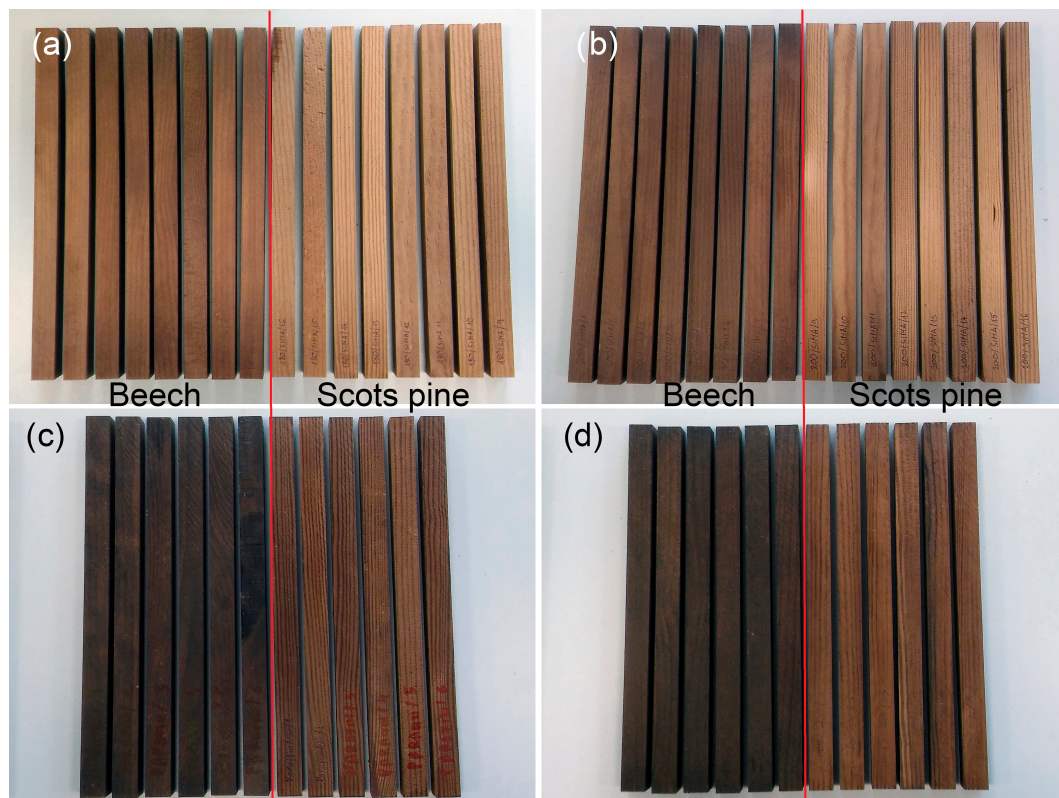


Figure 1. Images of beech and Scots pine laths cut in half after TM180 (a), TM200 (b), TMW180 (c), and TMW200 (d) treatments, optically representing the impregnation homogeneity and color.

3.2. Color Change

Thermal modification processes resulted in considerable color changes. Brightness (ΔL^*) decreased proportionally to the temperature in both treatment media, but wax treatment caused significantly more darkening (Figure 2a). The surface of the modified laths was affected more by the treatments, as results show less change in the brightness on planed surfaces. This difference in brightness after planing the surface was slight in air medium (5–10 units, depending on treatment parameter and wood species), but in wax medium, there was a remarkable difference (30–40 units, depending on treatment parameter and wood species). Differences between the untouched and the planed surface were larger in 180 °C treatments, which indicates the role of wax uptake in the color change. Brightness decreased similarly in both wood species. Red hue (Δa^*) increased as a result of all treatments in Scots pine; however, the red hue of beech showed no change after TM180 on the planed surface and decreased after TMW180 and TMW200 (Figure 2b). The increase in the red hue of the planed surface was lower in Scots pine compared to the outer surface; however, there was an increase for beech, except after the TM180 treatment. Treatment temperature did not significantly affect the red hue change in Scots pine, but there was a significant difference in beech. Results show the same trend comparing the TM and TMW treatments as well. Yellow hue (Δb^*) showed an increase, except for the outer surface after the TMW180 and TMW200 treatments (Figure 2c). However, after planing, there was an increase observed as well. This result points out again that, during thermal modification, the surface is more affected compared to the inner layers. This is especially valid using a liquid treatment medium, like paraffin wax. In this case, the wax is deposited in a higher ratio on the surface, while the intensity of heat transfer is higher and causes larger color changes. This is supported by the total color change as well, as there are no significant differences between the planed surfaces of the TM and TMW treatments (Figure 2d). On

the other hand, TMW treatments resulted in higher mass losses, which is considered to correlate with the total color change [49,50]. The result that significantly different mass losses result in equivalent color changes shows that this correlation is not directly usable in comparison of different methods [50]. According to the literature, the discoloration of wood due to thermal modification is caused by the degradation of hemicelluloses and the subsequent formation of low molecular weight sugars, as found for *Betula papyrifera* and *Pinus pinaster* [51,52]. In addition, the formation of oxidation products such as quinones was investigated in *Pinus sylvestris* and was found to also contribute to this [53]. The relative increase in the ratio of extractives and lignin also contributes to the darkening of wood as a result of heat treatment. Changes in the red color are caused by the degradation, condensation, and oxidation of various cell wall components, as in *Robinia pseudoacacia* [54]. The increase in the amount of polyphenols was found to contribute in different hardwood and softwood species (*Larix decidua*, *Eucalyptus grandis*, and *Eucalyptus saligna*) to the intensification of the red color [55,56].

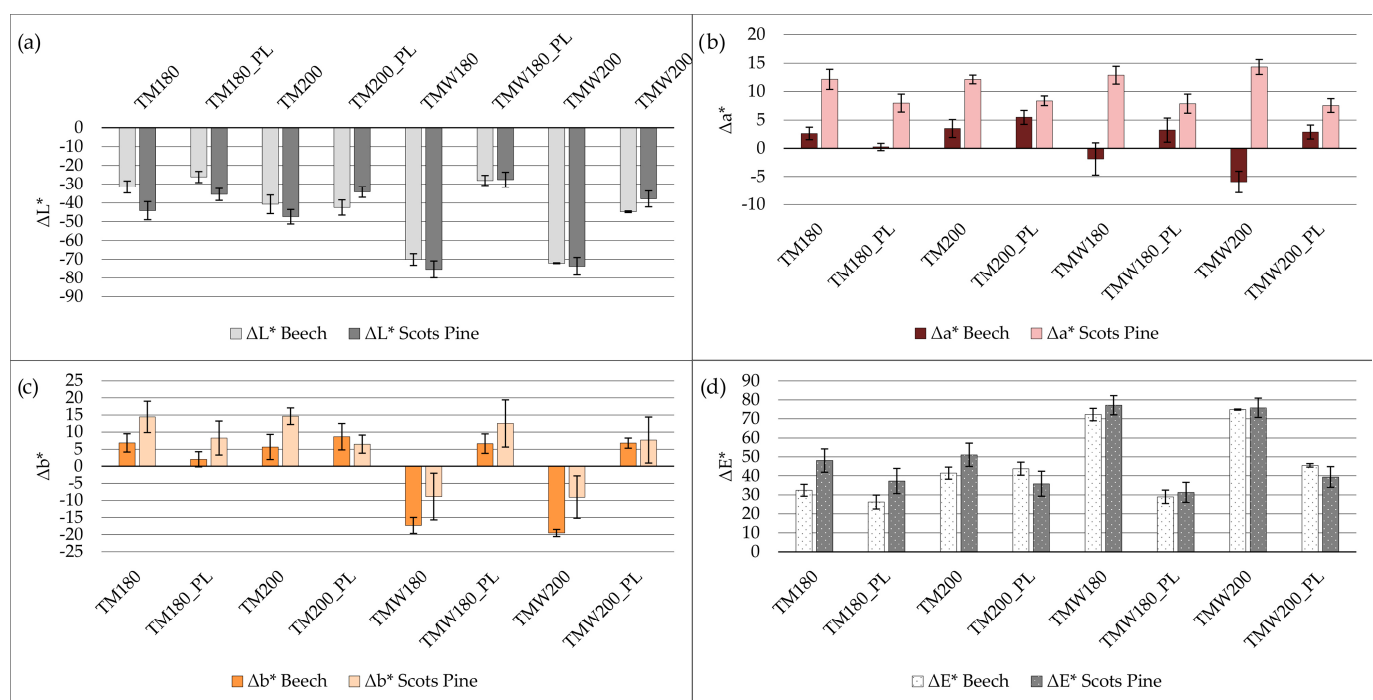


Figure 2. Values of brightness change (a), red hue change (b), yellow hue change (c), and total color change (d) of beech and Scots pine as a result of different thermal modification processes. Scale bars indicating standard deviation.

3.3. Ovendry Density

Thermal modification processes resulted in significant changes in the ovendry density. The density increased significantly as a result of both TM and TMW compared to the untreated material. Density increase in the TM-treated materials indicates that the volume decrease was not proportional to the ML caused by thermal degradation (Table 2). In TM treatment, the treatment temperature resulted in slight differences (Figure 3). In correlation with WU (Table 2), the TMW treatments resulted in a large increase in density. In this case, the 180 °C treatments resulted in the highest density (0.94 and 0.82 g/cm³ for beech and pine, respectively). The large differences between the 180 and 200 °C TMW treatments indicate the higher WU of 180 °C-treated wood. Density is usually reported to decrease as treatment intensity (temperature and time) increases [57–59]. However, some studies reported increased density as a result of thermal modification as well [60]. The decrease in density is attributed to the mass loss caused by thermal degradation of the cell wall

components. However, the controversial reports on density change show that the treatment process is an influencing factor on density. Besides mass loss, the volume of the wood is changing during thermal modification. If volume change surpasses mass change, the density is increasing. Impregnation with waxes causes a significant increase in density by filling the pores of wood with a solid substance [8]. The result that the density of TMW-treated wood increased considerably is in correlation with the WU results.

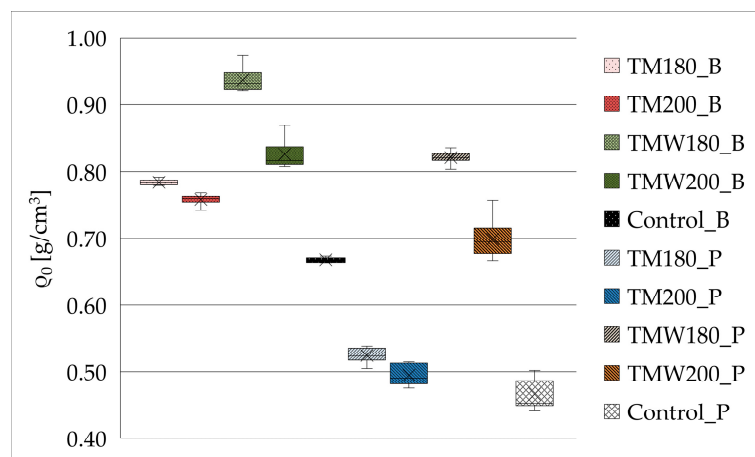


Figure 3. Oven-dry density of beech (_B) and Scots pine (_P) before and after TM and TMW treatments. Scale bars indicating standard deviation.

3.4. Water Contact Angle

The contact angle (θ) of distilled water measured on the planed surfaces of samples was not influenced by the TM treatments. θ of untreated beech was 75.78, in addition to 76.86 and 73.68, as a result of TM180 and TM200, respectively. The values for the equivalent Scots pine specimens were 80.75, 79.71, and 80.00 for untreated, TM180, and TM200 treatments, respectively. θ increased to 112.11 and 107.91 for beech and 114.04 and 109.52 for Scots pine as a result of TMW180 and TMW200 treatments, respectively (Figure 4). The wax impregnation significantly turned the wood hydrophobic. The higher WU of TMW180 treatment resulted in significantly higher water contact angles compared to TMW200 treatment. However, the large difference between WU of TMW180 and TMW200 (Table 2) was not represented in a large further increase in the hydrophobic character. These results are in correlation with the finding that liquid water uptake (EMC_{sat}) of TMW180 is lower compared to TMW200 (Figure 5b). The water contact angle is usually reported to increase as a result of thermal modification; however, some studies report unchanged values as well for spruce and hornbeam [61,62]. Additionally, the time elapsed since the surface was formed influences the water contact angle, as it shows increasing values over time for both untreated and thermally modified wood. The wettability changes rapidly after the preparation of the surface during the interaction with the surrounding atmosphere. The reason for this is reported to be the decrease in the polar character of the surface, indicated by the decrease in the O/C ratio. This process is similar in untreated and thermally modified wood [61]. During the preparation for water contact angle measurements, the control samples were produced prior to the modification processes; thus, the control sample surfaces were prepared 2–3 days earlier than the TM and TMW surfaces. According to a previous study [63], it is enough to alter the WCA of the wood, diminishing the differences between control and TM wood. This information must be considered during the interpretation of these results.

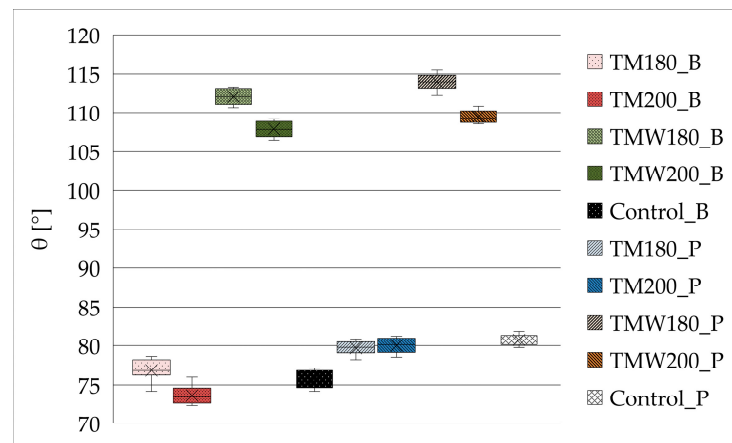


Figure 4. Water contact angle (θ) of beech (_B) and Scots pine (_P) before and after TM and TMW treatments. Scale bars indicating standard deviation.

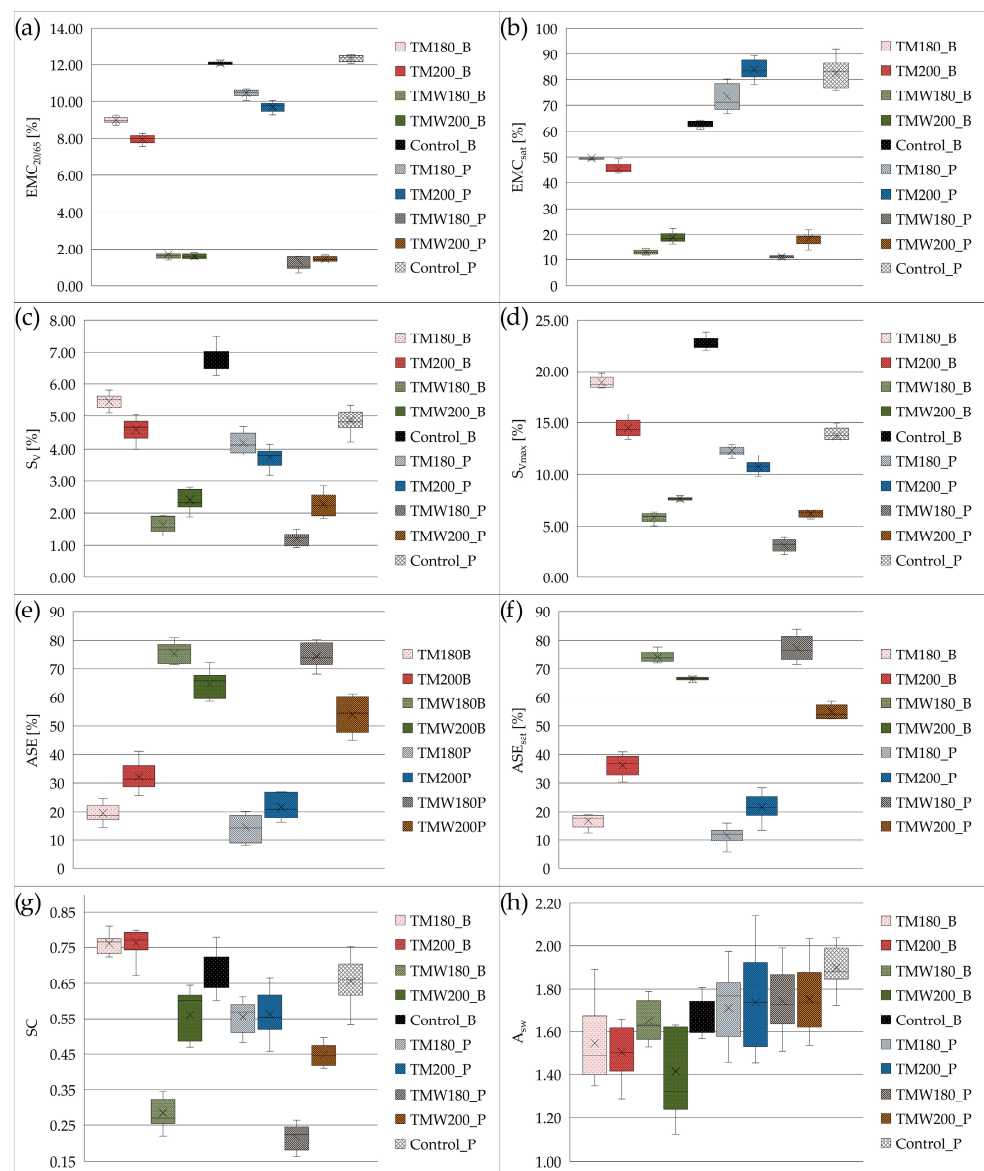


Figure 5. Moisture related properties of beech (_B) and Scots pine (_P) before and after TM and TMW treatments: (a) equilibrium moisture content after conditioning ($EMC_{20/65}$); (b) equilibrium moisture content after immersion under water (EMC_{sat}); (c) volumetric swelling after conditioning (S_v);

(d) volumetric swelling after immersion under water (S_{Vmax}); (e) anti-swelling efficiency after conditioning (ASE); (f) anti-swelling efficiency after immersion under water (ASE_{sat}); (g) swelling coefficient (SC); (h) swelling anisotropy (A_{sw}). Scale bars indicating standard deviation.

Water contact angle of paraffin wax is 105–120° according to previous studies, depending on paraffin type, surface roughness, and other factors [64]. According to the results, the water contact angle of TMW-treated wood is in this range, indicating the efficient hydrophobization of wood. These results are in line with other studies dealing with wax impregnation of wood; however, water contact angles up to 140° were reported using oxidized paraffin tested on the cross section of wood or bamboo as well [24,65,66].

3.5. Wood–Water Relations

EMC was measured at two different conditions, after conditioning at 20 °C/65% ($EMC_{20/65}$) and after immersion under water (EMC_{sat}). $EMC_{20/65}$ decreased significantly after the TM treatments. $EMC_{20/65}$ of untreated beech was 12.08%, which decreased to 9.02% and 7.95% as a result of TM180 and TM200, respectively. The values for the equivalent Scots pine specimens were 12.34%, 10.45%, and 9.40% for untreated, TM180, and TM200 treatments, respectively (Figure 5a). Thus, as expected, the higher temperature resulted in a significantly lower moisture content in both beech and pine. By changing the treatment medium to paraffin wax, $EMC_{20/65}$ further decreased to a high extent to 1.73% and 1.59% for beech and 1.33% and 1.44% for Scots pine as a result of TMW180 and TMW200 treatments, respectively. EMC_{sat} showed slightly different tendencies in TM treatments of Scots pine (73.36% and 83.96%), as there were no significant differences in moisture content in saturated conditions compared to the control (82.52%). In spite of that, TM treatments of beech resulted in significantly lower moisture content (49.67% and 45.53%) compared to the control (62.72%) (Figure 5b). However, the treatment temperature did not affect the EMC_{sat} . Similar to the results of $EMC_{20/65}$, paraffin wax impregnation further decreased the EMC_{sat} to 13.12% and 18.96% for beech and 11.15% and 18.35% for Scots pine as a result of TMW180 and TMW200 treatments, respectively. Moisture content of TMW wood remains under 20% even during underwater conditions, which makes it a promising possibility when used in ground or freshwater contact (use class 4) [67]. Besides the decreased amount of hydroxyl groups through removal of hydroxyl groups, increased crosslinking within the cell wall polymers, and a stiffer cell wall matrix as a result of thermal degradation processes [68], the wax impregnation excludes water through the hydrophobic character of paraffin wax and by clogging the pores of the wood, hindering the penetration of both vapor and liquid phase water [69]. Mass losses correlated with treatment temperature, as higher temperatures resulted in higher mass loss (Table 2). Nevertheless, EMC of TMW180 was lower compared to that of TMW200. This result indicates the important role of hydrophobic paraffin wax impregnation regarding sorption properties of wood [69].

Volumetric swelling was measured at two different conditions, after conditioning at 20 °C/65% (S_V) and after immersion under water (S_{Vmax}). S_V decreased similarly to the EMC. The 200 °C TM temperature resulted in significantly lower swelling after conditioning compared to 180 °C. S_V of untreated beech was 6.79%, which decreased to 5.46% and 4.59% as a result of TM180 and TM200, respectively. The values for the equivalent Scots pine specimens were 4.87%, 4.19%, and 3.72% for untreated, TM180, and TM200 treatments, respectively (Figure 5c). S_V was further decreased in TMW treatments, but in this case, the 180 °C treatment resulted in the lowest volumetric swelling. S_V decreased to 1.66% and 2.40% for beech and 1.21% and 2.27% for Scots pine as a result of TMW180 and TMW200 treatments, respectively. This again points out the importance of wax uptake in this combined modification method. The same tendencies were detected by calculating the S_{Vmax} as well, TMW180 treatments showing the lowest maximum volumetric swelling

(5.84% for beech and 3.10% for Scots pine), followed by TMW200 (7.59% for beech and 6.19% for Scots pine), TM200 (14.47% for beech and 10.76% for Scots pine), and TM180 (18.90% for beech and 12.38% for Scots pine), all significantly lower compared to the control (22.71% for beech and 13.73% for Scots pine) (Figure 5d). Treatment temperature affected the results less, compared to the additional wax impregnation, but its effect was also significant. ASE was calculated from the volumetric swelling; thus, the tendencies were equivalent. After conditioning, the ASE of beech was 19.59% and 32.38% as a result of TM180 and TM200, respectively. The values for the equivalent Scots pine specimens were 14.46% and 21.69% for TM180 and TM200 treatments, respectively. ASE increased to 75.58% and 64.63% for beech and 74.59% and 53.91% for Scots pine as a result of TMW180 and TMW200 treatments, respectively (Figure 5e). After immersion under water, ASE_{sat} of beech was 16.75% and 36.27% as a result of TM180 and TM200, respectively. The values for the equivalent Scots pine specimens were 11.58% and 21.66% for TM180 and TM200 treatments, respectively. ASE_{sat} increased to 74.30% and 66.56% for beech and 77.44% and 54.93% for Scots pine as a result of TMW180 and TMW200 treatments, respectively (Figure 5f). TMW treatment resulted in the highest ASE, reaching the range of over 70%, which makes wood highly dimensionally stable under varying conditions, by keeping the moisture content of wood very low (Figure 5a,b). Higher WU further improved dimensional stability, showing the importance of WU in this regard. A higher ratio of WU in Scots pine did not result in higher ASE (Table 2). This indicates that the WU is not a universal measure of impregnation efficiency. A pore filling ratio could be used as a better indicator for the efficiency of the impregnation treatments. However, this was not possible during these measurements. EMC, S_V, and ASE results show good correlation, as the reason behind the changes is the same. The dimensional stabilization effect of thermal modification could be improved by combining it with an additional wax impregnation. This was possible because thermal modification and wax impregnation have different modes of action (crosslinking, removal of hydroxyl groups, stiffer cell wall, and cell wall/cell lumen filling) [44,68–70]. Swelling of Scots pine is reported to be lower compared to that of beech. This phenomenon is generally explained by the lower solid content in Scots pine [48].

The swelling coefficient was calculated as a result of conditioning the samples at 20 °C/65%. It is a specific value, expressing the swelling caused by a 1% increase in the moisture content of wood. Thus, it makes comparable the swelling of different materials (treatments, wood species). There was no significant effect of the TM treatment compared to the control, as SC of untreated beech was 0.68, in addition to 0.76 and 0.76, as a result of TM180 and TM200, respectively. The values for the equivalent Scots pine specimens were 0.66, 0.56, and 0.56 for untreated, TM180, and TM200 treatments, respectively. SC was decreased in TMW treatments, where the 180 °C treatment resulted in the lowest swelling coefficient. SC decreased to 0.28 and 0.56 for beech and 0.21 and 0.45 for Scots pine as a result of TMW180 and TMW200 treatments, respectively (Figure 5g). SC was reported unchanged after thermal modification, which is explained by the constant water sorption capability in addition to the decreased sorption capacity as a result of a decreased amount of hydroxyl groups [46,47,71]. The decreased SC in correlation with WU indicates further changes in addition to the thermal degradation processes. This shows that, in addition to the same moisture uptake, the wood swells less, which indicates the effective penetration of paraffin wax into the cell wall. The wax present in the cell wall acts as a bulking agent (fixation) that hinders the dimensional changes by increasing the dry dimensions of wood [72–74].

Swelling anisotropy was not affected by the modification processes, as no significant differences could be detected between the modified and untreated samples. A_{SW} of untreated beech was 1.66, in addition to 1.55 and 1.51, as a result of TM180 and TM200,

respectively. The values for the equivalent Scots pine specimens were 1.90, 1.71, and 1.74 for untreated, TM180, and TM200 treatments, respectively. A_{SW} was 1.65 and 1.41 for beech and 1.74 and 1.75% for Scots pine as a result of TMW180 and TMW200 treatments, respectively (Figure 5h). This result indicates that the swelling in the radial and tangential directions changed to the same extent as a result of TM and TMW treatments. This shows that neither thermal treatment nor additional wax impregnation affected differently the radial and tangential swelling. From the utilization point of view, it is important that wood does not become more prone to deformation after modifications. Thermal modification usually reduces the swelling anisotropy by decreasing tangential swelling to a greater extent than radial swelling [75,76]. However, unchanged swelling anisotropy was reported in the case of different impregnation modifications because of the bulking effect [72,77,78]. This indicates the remarkable role of the bulking effect, reducing the effect of thermal modification on the swelling anisotropy.

3.6. Mechanical Properties

MOR did not change significantly as a result of the TM or TMW treatments. However, the standard deviation was remarkably increased after the modifications, especially in beech (Figure 6a). This indicates the degradation processes of the cell wall [79]. EMC decreased as a result of TM and TMW, which resulted in a relative increase in the MOR compared to the control. In the tested modifications (TM and TMW), the decreasing effect of thermal degradation is balanced by the increasing effect of lower moisture content and paraffin impregnation. The controversial results from the literature highlight the importance of treatment parameters (wood species, treatment medium, presence of moisture, etc.) during the treatment in terms of changes in the mechanical properties [5,22,68].

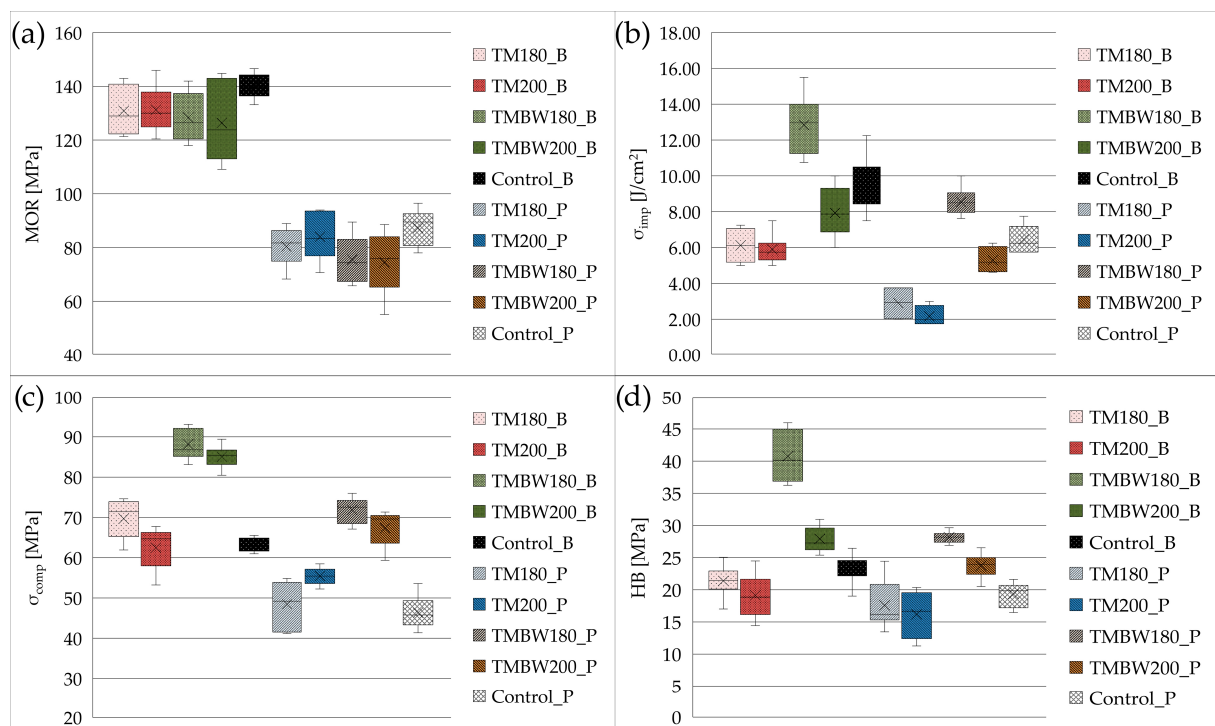


Figure 6. Mechanical properties of beech (B) and Scots pine (P) before and after TM and TMW treatments: (a) bending strength (MOR); (b) impact bending strength (σ_{imp}); (c) compression strength (σ_{comp}); (d) Brinell hardness (HB). Scale bars indicating standard deviation.

Impact bending strength (σ_{imp}) was influenced by the TM treatments. σ_{imp} of untreated beech was 9.57 J/cm², in addition to 6.05 J/cm² and 5.68 J/cm², as a result of

TM180 and TM200, respectively. The values for the equivalent Scots pine specimens were 6.46 J/cm², 3.17 J/cm², and 2.23 J/cm² for untreated, TM180, and TM200 treatments, respectively. σ_{imp} significantly increased to 12.46 J/cm² in TMW180 of beech and to 8.63 J/cm² for Scots pine. No significant changes were observed in TM200 treatment compared to control, as σ_{imp} was 7.93 J/cm² for beech and 5.27 J/cm² for Scots pine (Figure 6b). It was reported previously that thermal modification remarkably decreases the impact bending strength, while wax impregnation increases it [8,79]. The treatment temperature is a crucial factor in TMW modifications regarding WU, which seems to have a large influence on the impact bending strength as well. This is supported by the result that only TMW180 treatment improved σ_{imp} , which correlates with the WU results (Table 2). Impact bending strength changes are reported to correlate well with the thermal degradation-induced chemical changes. The highest correlation was found with the relative content of cellulose. The factors with the most significant effect on the impact bending strength are the modification temperature and the wood species [79,80].

Compression strength was not affected by the TM modifications, while TMW increased it remarkably. Compression strength of beech was 62.99 MPa, 70.36 MPa, and 62.06 MPa for the control, TM180, and TM200, respectively. The same for Scots pine was 46.25 MPa, 48.37 MPa, and 55.09 MPa for control, TM180, and TM200, respectively. Compression strength of beech increased to 88.40 MPa and 85.10 MPa as a result of TMW180 and TMW200, respectively, while that of Scots pine increased to 71.62 MPa and 67.02 MPa as a result of TMW180 and TMW200, respectively (Figure 6c). Treatment temperature did not affect the compression strength. Oil heat treatment results in more advantageous mechanical properties compared to methods using gaseous atmospheres [17]. The treatment medium fills the lumen and bulks the cell wall, providing better lateral stability to the wood. This way, the buckling of relatively thin cell walls is hindered [81]. Another explanation of the increasing compression strength is the increase in lignin condensation as a result of thermal modification [82]. Decreased moisture content, an increased ratio of crystalline cellulose, and improved crosslinking of lignin are also explanations behind this phenomenon [83].

Brinell hardness at the tangential surface was significantly influenced only by TMW. TM modifications did not affect the hardness. TMW200 slightly increased the surface hardness as a result of the wax impregnation, as the improvement was 18.89% in beech and 23.76% in Scots pine compared to the control. A remarkable improvement was observed in TMW180 treatment, showing 70.88% in beech and 48.16% in Scots pine compared to the control (Figure 6d). Comparing the results, TM and TMW at the same temperature make it visible that wax impregnation increased the surface hardness, especially in Scots pine, where higher WU was measured. Thermal modification in paraffin was reported to decrease the Brinell hardness [16]. The effect of thermal modification is reported to have varying effects on surface hardness depending on process parameters. Thermal modification mostly decreases the hardness with increasing treatment temperature. Especially at higher temperatures, a large decrease is observed [84]. However, a slight increase in the hardness is also reported [22,85]. Oil heat treatment has a cell wall thickening effect, which explains the increased mechanical properties, including compression strength and hardness [85,86]. The same effect prevails even more in wax impregnation, where the impregnation agent solidifies in the cell wall [81]. In addition, the increased ratio of crystalline cellulose, lower moisture content, and crosslinking of lignin contribute to increased hardness of wood thermally modified in liquid media (oils, waxes) [83,87].

Both thermal and impregnation modifications affect the mechanical properties of wood; however, these effects differ in their character. Thermal modifications usually decrease the mechanical properties because of the degradation processes of the cell wall

components, but several studies reported improved strength properties after thermal modification as well [5,22,68]. The increase in some mechanical properties is explained by several reasons. One is the decreased equilibrium moisture content of thermally modified wood, as it is in an inverse proportional relation with mechanical properties [5,22,68]. Additionally, the increased mechanical properties are also explained by hornification, which is a structural rearrangement of the cell wall polymers, resulting in irreversible hydrogen bonding between adjacent carbohydrate elements [47,70]. Another phenomenon that balances the negative effects of thermal degradation on mechanical properties is the glass transition and rearrangement of lignin during the process. This phenomenon occurs at temperatures up to 200 °C, while the bonds between hemicellulose and lignin are broken, and low molecular weight fragments of lignin are formed. The result of this process is the repolymerization of these lignin fragments, which serves as an internal support in the cell wall [44,83,88]. Oppositely, impregnation with resins, polymers, or waxes usually increases the mechanical properties through providing internal support to the structure by filling the pores with solid-state materials [8,24,69]. In general, strength losses due to thermal modification might be partly compensated by using an additional wax impregnation, especially in compression-type loading, as was observed in the compression strength and Brinell hardness.

3.7. Durability

Durability was significantly improved by TM and TMW as well. Mass loss in beech caused by *Coniophora puteana* (ML_D) was 62.59%, 14.40%, 4.04%, 1.75%, and 1.03% for control, TM180, TM200, TMW180, and TMW200, respectively. The same results in Scots pine were 62.01%, 18.95%, 5.24%, 3.35%, and 2.66% for control, TM180, TM200, TMW180, and TMW200, respectively (Figure 7). According to these results, TM180 cannot be evaluated as an effective modification, while TM200 reaches the minimum temperature that is considered to provide efficient protection against decay [89]. However, even in this case, it was not possible to go under the limit of ML_D, i.e., under 3% [90]. The most effective decay protection was provided by the TMW treatments, where the 180 °C temperature provided ML under 3%. This indicates the role of wax impregnation in decay protection, as a remarkably higher wax uptake was observed for TMW180 treatments compared to TMW200, which is in line with the fungal decay results. The wax is acting as a physical barrier to the hyphae of the fungi, blocking their penetration into the cell lumens [91]. On the other hand, the moisture content of the TMW wood is remarkably decreased as a result of the hydrophobization effect of the wax (Figures 4 and 5a,b). According to previous studies, 180 °C is not considered an effective treatment temperature to provide decay protection [89], but the advantage of the combined effects of thermal modification and wax impregnation is that, already, this temperature is enough for full protection. Fungal degradation of wax-impregnated wood is slower, as wax fills the cell lumens and possibly the cell wall pores, hindering the spreading of hyphae and the movement of fungal enzymes and fungal decomposition products through diffusion [92]. In addition, thermal modification induces changes in the chemical composition of the cell wall polymers, most importantly resulting in a decrease in the hydroxyl groups. This results in decreased moisture content, leading to improved decay resistance of wood [89]. Wax impregnation further decreased the moisture content (Figure 5a,b), which, besides the clogging effect, explains the improved decay resistance of TMW compared to TM treatments.

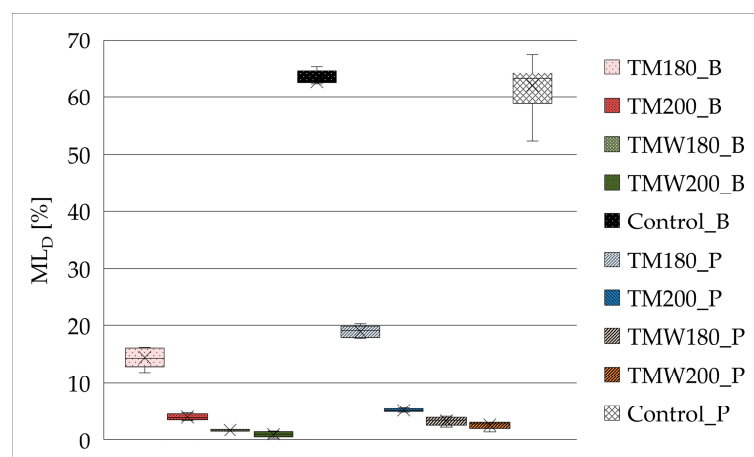


Figure 7. Durability of beech (_B) and Scots pine (_P) before and after TM and TMW treatments against *Coniophora puteana*. Scale bars indicating standard deviation.

4. Conclusions

With the use of the combination of thermal modification and paraffin wax impregnation in one step, it is possible to improve several properties of wood. Wax uptake depended on the treatment temperature, where a lower temperature resulted in remarkably larger wax uptake. Equilibrium moisture content, swelling, and dimensional stability were further improved by the combination of thermal modification and wax impregnation. Swelling anisotropy was not changed; thus, the material did not become more prone to deformations. The improved hydrophobicity of the wood through the wax impregnation makes these combined treatments more effective than the thermal modification or the wax impregnation separately. Mechanical properties were improved using the combined treatment compared to the air medium thermal modification, which is attributed to the effect of the additional paraffin impregnation. Especially the surface hardness could be improved in the combined modification method. The combined modification improved decay resistance as well. As a side effect of the treatments, an explicit color darkening occurred, in addition to a large difference between the surface and the inner part of the paraffin wax-impregnated wood. As a simple one-step method, it might be used as a commercial method in the future. As outdoor use is expected, improved durability and dimensional stability are the main advantages of this method.

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