Artificial Weathering Resistance and Biological Durability of Surface-Charred Beech Wood Combined with Linseed Oil Coating

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Specimens of European beech wood (*Fagus sylvatica* L.) charred at 250 °C (4 min), 300 °C (2 min), and 350 °C (1 min) in combination with linseed oil coating were studied. The influence of the surface charring process and artificial weathering on surface discoloration, water absorption, and decay resistance were analyzed. Discoloration analysis showed a decrease in all parameters *L**, *a**, and *b** due to the charring process. Coating with linseed oil caused a decrease in the *L** parameter. An influence on parameters *a** and *b** was also demonstrated. As a result of the artificial weathering, the *L** parameter increased in almost all groups. The group charred at 350 °C showed a decrease in *L**. The parameters *a** and *b** were also affected. The effect of oil coating on discoloration during artificial weathering was negligible. The average water absorption of uncoated charred specimens decreased more than 50%. Damage due to artificial weathering disrupted the effect of the charring process. The effect of oil coating decreased depending on the length of partial immersion. Surface charring and oil impregnation slightly reduced the loss of beech wood mass caused by *T. versicolor* and *P. placenta* but could not fully protect the wood.

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INTRODUCTION

Wood has been used by humans for thousands of years because of its significant benefits such as sustainability, availability, utility, and price. It is a suitable material for ecological constructions (Jones 2017), for which there is a developing trend. However, according to Dubois *et al.* (2020), there is a potential risk of a shortage of wood raw material for industrial purposes because of the crises of forest health and climate change. Therefore, there is pressure to develop new technologies that lead to more efficient use of resources, for example, by extending the service life of wood products such as wooden facades (Werner *et al.* 2006). During external use, wood is subject to degradation called weathering, which should not be confused with degradation caused by fungi or other microorganisms that can reduce structural strength (Teacă *et al.* 2013). Weathering mainly affects the appearance and properties of the surface layers (Evans *et al.* 2005). Photodegradation is one of the main causes of weathering, decreasing the lignin content sharply in the surface layers, which changes the wood's color. Changes in the surrounding

environment – for example, in temperature, rain, relative humidity, *etc.* – result in cyclical shrinkage and swelling of the exposed wooden elements, which leads to deterioration of the appearance and mechanical resistance of the wood (Popescu *et al.* 2011; Teacă *et al.* 2013). The discontinuous cracked surface and increased moisture content (20%) allow biological agents to attack the wood more easily. Surface coatings made of natural or synthetic chemicals are currently used to protect facade cladding from the adverse effects of the external environment. The main disadvantage of surface coatings is that they need to be renewed periodically as, due to the influence of the external environment, there is degradation, cracking, and peeling of the applied coatings (Grüll *et al.* 2013; Kymäläinen *et al.* 2022a). An alternative to surface coatings is thermal treatment. It is well known that exposure to high temperatures exceeding 160 °C causes decomposition of hemicelluloses, which reduces the free hydroxyl groups and the equilibrium moisture content (EMC) of the wood, and consequently increases its dimensional stability and biological durability. Temperatures exceeding 300 °C lead to significant weight loss, as a result of the extensive degradation of cell-wall compounds (Jämsä *et al.* 2000; Santos 2000; Kamdem *et al.* 2002; Repellin and Guyonnet 2005). Such treatment, using temperature exceeding 300 °C, is typically called wood charring. Traditional methods of charring the wood surface include the Japanese "*Yakisugi*" method, which employs an open flame. Modern methods include the use of a contact heating device or gas flame method. The charring produced by the contact heating device is more homogeneous and less brittle than that from "*Yakisugi*" or gas flame, because the contact heating device allows the conditions during the charring process to be controlled to create a uniformly charred surface. Unstable conditions are created during the open-flame method and the resulting charred layer can be heterogeneous, as well as surface made by gas flame method. The thickness of the charred layer depends on the temperature reached during the process and its duration (Ebner *et al.* 2021; Machová *et al.* 2021; Šeda *et al.* 2021; Kymäläinen *et al.* 2022b).

It has been shown that surface – charred wood has better hydrophobic properties, lower equilibrium moisture content and, in the short term, lower water absorption. Čermák *et al.* (2019) studied the moisture properties of beech wood charred at 220 °C for 40 min and showed an 11 to 18% reduction in water absorption. The increased hydrophobicity of wood is one of the reasons for the increased resistance of charred wood to fungal attack. Another reason is the presence of extractive substances with fungicidal potential arising during the charring process and the significant degradation of hemicelluloses in the charred layer (Militz 2002; Weiland and Guyonnet 2003). Soytürk *et al.* (2023) admit that a reduction in water absorption and a significant decrease in carbohydrate content due to surface carbonization can lead to an improvement in the biological resistance of the surface. The possibility of fungal attack can also be reduced by treating the surface with water – repellent substances, such as drying oils and wax emulsions, which fill cell lumina and pore surfaces and thereby slow down the penetration of water into the wood substance. A reduction in the average moisture content is then sufficient to increase resistance to fungal attack (Hyvönen *et al.* 2005; Schultz *et al.* 2007; Lesar and Humar 2011; Thybring 2013).

As a result of climate change, the coniferous forests of Central Europe are being converted to mixed forests (Dubois *et al.* 2020), in which European beech (*Fagus sylvatica* L.) could be significant. Beech wood is preferred because of its material properties, especially because it is easily workable and impregnable. However, it suffers from low resistance to fungi and insects, and high dimensional instability under changing weather conditions; therefore, a new way to modify its properties would be of great value. Based

on the information presented in the literature review, it is assumed that the use of a charring process with appropriate process parameters (temperature and time) in combination with a natural oil coating would increase the hydrophobicity of the surface, which should also favorably affect biological resistance. Therefore, an interesting alternative to traditional wood treatments could be provided. This study was set up to investigate the effect of surface charring and/or linseed oil coating on selected material properties.

EXPERIMENTAL

European beech (*Fagus sylvatica* L.) from a Czech enterprise forest with an average density of 762 kg/m^3 (standard deviation 24 kg/m^3) was studied. Reference and one – sided surface charred specimens with and without linseed oil coating before and after artificial weathering were used to evaluate surface discoloration and water absorption. Reference and four – sided surface charred specimens with and without linseed oil coating were used to evaluate decay resistance.

Specimen Preparation
Surface charring

The specimens with a deviation of the annual rings of 10 °– 25 ° from the exposed surface were used for the surface charring process. Prepared specimens were oven – dried to 0% moisture content, planed, and oven dried to 0% moisture content again to limit dimensional changes. The specimens were charred at temperatures of 250 °C for 4 min (250), 300 °C for 2 min (300), and 350 °C for 1 min (350), at atmospheric pressure, using a laboratory contact heating system with a hot plate (Ceran 500) and an electronic NiCrNi temperature sensor (regulation between 50 and 500 °C).

Surface coating

The uncharred parts of the specimens were sealed using Epolex S1300/Epolex S7300 epoxy sealant to prevent water absorption. Oil–coated specimens were brushed (three times; 24 h between brushing) with linseed oil on the charred sides. The specimens were kept at 65% RH and 20 °C to start the oil drying process, which was later accelerated by oven-drying at 103 °C. The oven-dry mass (m_0) of all specimens was recorded and specimens were conditioned at 65% RH and 20 °C until an equilibrium state was reached. The average oil intake of specimens for surface discoloration and water absorption was 168 g/m^2. The average oil intake of specimens for decay resistance was 150 g/m^2.

Artificial weathering

Prepared specimens were conditioned at 65% relative humidity (RH) and a temperature of 20 °C until a constant mass was achieved and exposed to a weathering process in a xenon test chamber Q – Sun Xe –1 (Q – Lab Corp.). A 2 h repeat cycle was set according to modified standard ASTM G155 (2013). Rain was simulated for 18 min in the first step of the cycle, and solar radiation of intensity 70 W/m^2 (TUV) was simulated for the next 102 min. The process of artificial weathering was paused for surface discoloration and gloss measurement.

Properties Evaluation

Surface discoloration

Surface discoloration was measured on 24 specimens of dimensions $65 \times 20 \times 80$ mm^3 (RT $\times$ RT $\times$ L) divided into two sets – with and without linseed oil coating. Each set included 3 reference specimens and 3 specimens for each temperature of the surface charring process. The surface discoloration was measured before and during artificial weathering process after 50, 200, 500, and 1000 h using a Konica Minolta CM-2500 (measurement area 8 mm, 10° standard observer, D65 illuminant) using the CIELab color system. CIELab resolves colour into 3 coordinates: L^* – lightness, with a range from 0 (black) to 100 (white); a^* – position between red and green, where negative values mean green and positive values mean red; and b^* – position between yellow and blue, where negative values mean blue and positive values mean yellow. Each specimen was measured at four points; the final values are the average of these measured values for all specimens in the group.

The gloss parameters were measured using a KSJ Glossmeter MG268 – F2 at an angle of incidence of 85° and measuring aperture 12×60 mm^2 with a deviation of +/- 1.5% GU at three points.

Water absorption

Measurement of water absorption was performed on 2 groups of specimens – fresh specimens and artificial weathered specimens. Each group contained two sets of 12 specimens of dimensions $65 \times 20 \times 80$ mm^3 (RT $\times$ RT $\times$ L). Each set includes 3 reference specimens and 3 specimens for each temperature of the surface charring process. One set from each group was surface coated with linseed oil. Water absorption during water floating was measured according to the EN – 927 – 5 (2006) standard as weight increase (g/m2) and was recorded after 1, 2, 4, 8, 24, 48, and 72 h.

Decay resistance

Reference and four-sided charred test specimens of dimensions $25 \times 15 \times 50$ mm^3 (RT $\times$ RT $\times$ L) were exposed to white – rot fungus *Trametes versicolor* L. Lloyd (BAM 116) and brown – rot fungus *Coniophora puteana* (Schumach.) P. Karst. (BAM 112). The testing groups included different charring temperatures (reference, 250, 300, and 350 °C) – with and without linseed oil coating; each group contained 6 test specimens. Sterile malt agar medium in Kolle flasks was inoculated with fungal mycelium and incubated at 22 °C and 70% RH until the mycelia completely covered the agar medium. All specimens were sterilized using steam (Tuttnauer 3150E) at 120 °C for 20 min, inserted into the flasks and stored at 22 °C and 70% RH. After 16 weeks, the mycelium on the specimen surface was carefully removed, the specimens were oven – dried (103 °C; m_1) and weighed in order to calculate the mass loss according to the following equation,

$$\text{ML (\%)} = m_0 - m_1/m_0 \times 100 \tag{1}$$

where ML is mass loss (%); m_0 is the dry mass before decay (g); m_1 is the dry mass after decay (g).

Data processing and statistical analysis

The data of surface discoloration and decay resistance were processed, evaluated using one – factor analysis of variance (ANOVA), completed with Tukey's honest significant difference (HSD) test, and graphed using OriginPro (OriginLab Corporation,

version 9.0, Northampton, MA, USA). The data for water absorption were processed, evaluated using the Kruskal – Wallis test and completed with Dunn's multiple comparison test. Statistically significant differences were considered at $p \leq 0.05$.

RESULTS AND DISCUSSION

Surface Discoloration

The measured values of color coordinates L^*, a^*, and b^* for beech specimens charred at different temperatures and for different times were compared. The specimens were divided into groups with oil coating and without oil coating. The surface appearance of the examined specimens is presented in Fig. 1.

Fig. 1. Surface discoloration of artificial weathered specimens after 1000 h of exposure; REF – Reference; 250, 300, 350 – charred at different temperature; O – coated with linseed oil; W – artificial weathered

According to visual analysis, the carbonized surface darkened due to the surface carbonization process. This statement is confirmed by the values shown in Table 1. According to the indicated values, the process of surface carbonization had a significant effect on all measured coordinates. The surface $L*$ of the carbonized specimens was significantly darker than the reference, almost identically for all measured groups. Lovrić *et al.* (2014), Mitani and Barboutis (2013), and Unsal *et al.* (2003) state that the decrease in the values of all color parameters, especially the lightness of wood $L*$, during the carbonization process depends mainly on the length of the process and the temperature. However, the observed values do not correspond to this trend. This discrepancy may be caused by the setting of the temperature – time regime of the surface carbonization process. A relatively short duration was set for this process, which simultaneously decreased with increasing temperature. The decrease in $L*$ is caused by the degradation of hemicelluloses with the formation of colored degradation products and extracts in the pyrolysis process (Sundqvist and Morén 2002; González-Peña and Hale 2009). The pyrolysis process is influenced by atmospheric pressure, temperature, time, and the chemical composition of the wood. This process leads to the formation of a charred layer, the composition of which depends mainly on temperature and time (Ermolenko *et al.* 1990; Byrne and Nagle 1997). As the length of pyrolysis increases, especially at temperatures above 300 °C, there is a significant change in the structure and composition of the charred layer. There is accelerated decomposition of cellulose and hemicellulose and the content of fixed carbon increases, which subsequently contributes to a decrease in lightness (Yoo *et al.* 2018).

Table 1. Color Coordinates for Reference (REF) and Charred Specimens with Oil and Without Oil Coating After 0 and 1,000 h of Artificial Weathering

	0 h			1,000 h			
	$L*$	$a*$	$b*$	$L*$	$a*$	$b*$	ΔE
REF	70.3 (1.4)	6.7 (0.6)	15.8 (1.4)	88.2 (0.8)	1.6 (0.2)	8.5 (1.2)	20.0 (2.1)
O REF	61.7 (2.3)	10.8 (0.5)	22.5 (2.4)	86.3 (1.4)	1.9 (0.4)	9.6 (1.3)	29.4 (2.1)
250	29.5 (1.2)	3.4 (1.0)	4.7 (1.5)	64.3 (5.7)	3.3 (0.7)	10.8 (2.0)	35.4 (5.1)
O 250	26.8 (0.9)	2.3 (0.9)	2.9 (1.2)	67.8 (7.0)	2.3 (0.6)	9.8 (1.1)	41.6 (6.1)
300	29.2 (0.9)	2.7 (0.8)	3.6 (1.2)	52.5 (8.9)	4.2 (0.9)	10.3 (1.3)	24.7 (8.2)
O 300	26.0 (0.6)	1.5 (0.4)	1.8 (0.5)	58.2 (9.3)	3.6 (1.0)	9.9 (1.6)	33.5 (8.9)
350	29.3 (0.5)	0.9 (0.4)	1.1 (0.4)	20.6 (2.2)	2.6 (1.3)	3.1 (2.1)	9.6 (1.4)
O 350	28.4 (0.8)	0.3 (0.1)	0.7 (0.3)	19.8 (1.0)	0.7 (0.2)	0.5 (0.3)	8.6 (1.0)
* Standard deviation in parentheses							

As a result of the surface carbonization process, the values of parameters $a*$ and $b*$ decreased. A sharp decrease in value was recorded for both parameters with increasing treatment temperature during the process. This greatest decrease was noted for the 350 group. According to these values, the decrease in parameter $b*$ was more pronounced than the decrease in parameter $a*$ in all groups of examined specimens compared to the reference. The same result was presented by Machová *et al.* (2021). They charred radial and tangential specimens of beech using a heating plate in the range 200 to 400 °C and recorded a significant decrease in the $L*$ parameter of all investigated groups. They also noted the same downward trend for parameters $a*$ and $b*$. As a result of carbonization, the monitored parameters approached zero, which means a neutral gray color. The specimens therefore tended towards gray in all parameters. The value of $a*$ is influenced by the content of thermal wood degradation products and due to the condensation and repolymerization of lignin (Chen *et al.* 2012). The combination of high temperature and

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wood moisture content leads to hydrolytic reactions and the formation of yellow phenolic substances. This leads to an increase in the value of parameter b^*. For 0% moisture content, these reactions are infrequent, and the spectrum shift trend is opposite (Wikberg and Maunu 2004; Chen *et al*. 2012; Machová *et al*. 2021).

As a result of the oil coating, the L^* parameter decreased in all investigated groups. The largest decrease was recorded in the reference specimens. The smallest decrease was recorded in the O 350 group. In the reference specimens, the values of parameter a^* and b^* increased. For the carbonized specimens, there was a decrease in the values of parameters a^* and b^*. The most significant decrease in the a^* parameter was recorded in the O 350 group, while the most significant decrease in the b^* parameter was recorded in the O 300 group.

According to visual analysis, it can be concluded that due to artificial weathering (W) there was an increase in the lightness of the surface of the reference specimens and the carbonized specimens of groups 250 and 300. In contrast, in groups 350 and O 350 the carbonized surface darkened only slightly. Visible cracks were noted on the surface of the carbonized specimens. The results of visual analysis are confirmed by the values shown in Table 1. The greatest increase in parameter L^* was recorded in group 250 W after 1,000 h of artificial weathering. In the groups 350 and O 350, there was a decrease in the parameter values after 1,000 h of artificial weathering.

According to Fig. 2, the values of ΔL^* followed a stable trend of growth (REF, 250 and 300) or decrease (350) during the process of artificial weathering compared to the values of Δa^* and Δb^*, for which the trend during artificial weathering was changing. In the reference and groups 250 and 300, there was an increase in the values of Δa and Δb in the initial stages of artificial weathering and then a subsequent decrease. The least significant change in trend was noted in the reference specimens (REF), where these changes were extreme. In the groups 250 and 300, the increase and decrease in values during the artificial weathering process were gradual. For the group 350, a slight increase in Δa and Δb values was recorded. The change in surface color during artificial weathering is determined by a set of chemical processes. Due to the carbonization of the surface, there was an increase in the proportion of lignin in the surface layers compared to hemicelluloses, because hemicelluloses are sensitive to heat treatment, but during artificial weathering they are more stable than lignin (Huang *et al*. 2012). According to Cogulet *et al.* (2016), lignin is highly susceptible to degradation by light radiation.

The initial increase in the values of Δa and Δb presented in Fig. 2 is probably caused by significant degradation of lignin and the formation of chromatic products in the initial stages of during artificial weathering. The subsequent decrease in values is caused by the leaching of chromatic products by water, which, in combination with leaching of extractive substances by water, also causes a lightening of the surface (Huang *et al*. 2012). The decrease in surface lightness in the group 350 is probably connected with the leaching of degradation products and the limitation of the chemical composition of the surface layers only on fixed carbon (Yoo *et al.* 2018).

According to the evaluation of the data presented in Table 1 in terms of discoloration during artificial weathering, the effect of surface coating with linseed oil was negligible. According to the values shown in Fig. 2, the values of changes in color parameters during artificial weathering reached a similar course for almost all groups of examined specimens. A different course of values was recorded for the Δa and Δb parameters of the group O 350. Compared to group 350, the increase in Δa values was more moderate, and there was even an opposite trend for Δb values. The small importance

of oil coating is explained by the properties of the oil used. Linseed oil is hydrophobic but has little resistance to photo-degradation. According to Mallégol *et al.* (2000), when linseed oil is exposed to light, it rapidly degrades due to photo-oxidation processes.
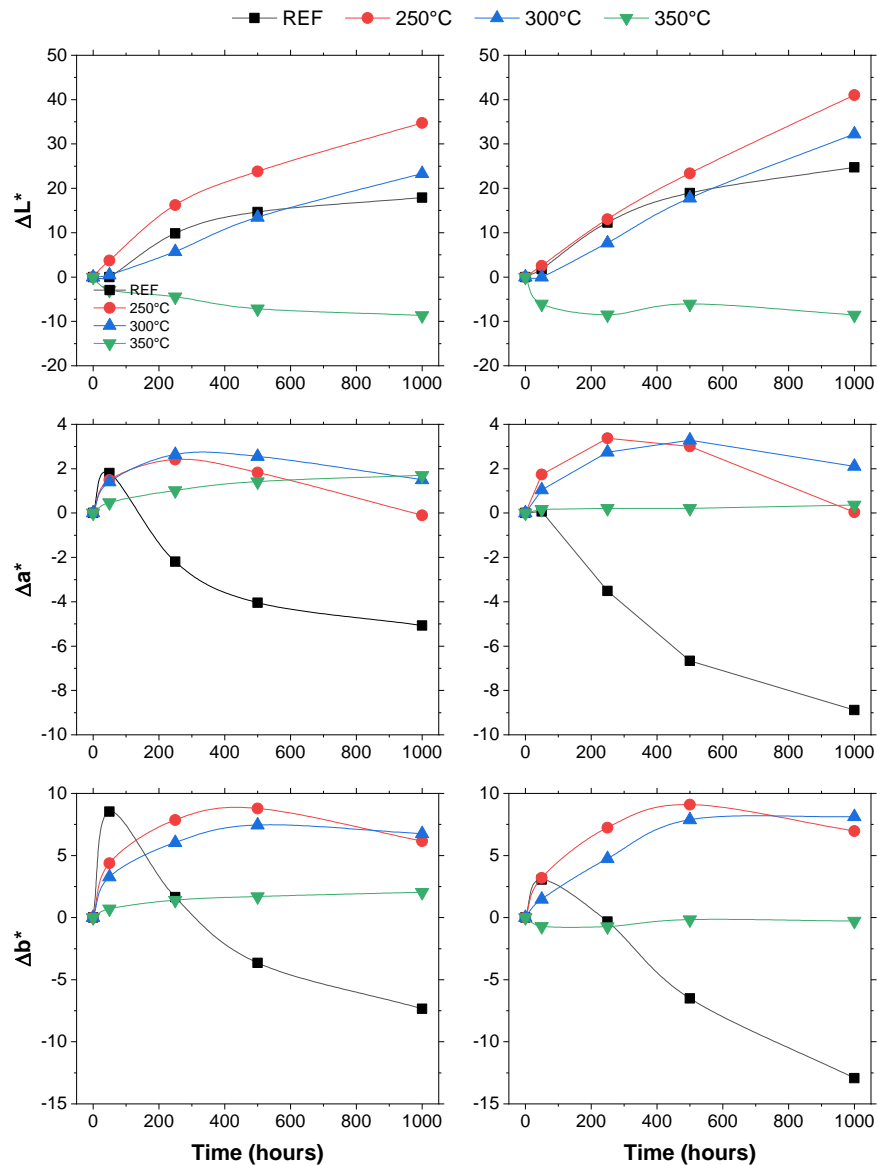
Fig. 2. Individual color parameter changes of charred specimens without oil coating (left) and with oil coating (right) during weathering; REF – Reference; 250, 300, 350 – charred at different temperature

According to the Δ*E* values in Table 1, all exposed specimens underwent significant changes due to the artificial weathering process. The smallest changes were noted for groups 350 and 350 O, which was probably due to the presence of a carbon structure in the surface layers, which is very resistant to photodegradation (Kymäläinen *et al.* 2022b).

The measured gloss values for all groups of examined specimens are shown in Table 2. It can be seen from the given values that the heat modification process did not have a significant effect on the gloss change, as did the oil surface coating. This result does not correspond with the result presented by Bekhta *et al.* (2014), who measured specimens

carbonized at 200 °C while pressing with a force of 12 MPa. The increase in gloss was probably due to a reduction in surface roughness during pressing. Surface gloss is therefore inversely proportional to surface roughness. The values shown in Table 4 present a sharp drop in gloss due to the artificial weathering process. The decrease in gloss could be due to the increase in surface roughness due to during artificial weathering.

Table 2. Gloss Values (expressed in gloss units) for Uncharred (REF) and Charred Specimens with Oil and Without Oil Coating After 0 and 1,000 h of Artificial Weathering

	0 h	1000 h
REF	7.7 (1.5)	2.4 (0.7)
REF O	6.1 (1.1)	1.3 (0.5)
250	7.1 (2.1)	1.5 (0.3)
250 O	6.5 (0.8)	0.5 (0.2)
300	8.7 (1.7)	1.4 (0.4)
300 O	8.0 (1.0)	0.6 (0.2)
350	6.3 (2.5)	1.1 (0.5)
350 O	9.4 (1.9)	0.3 (0.1)
* Standard deviation in parentheses		

Water Absorption

The measured values of water absorption by the beech specimens after partial immersion are presented in Fig. 3. Even though the specimens were soaked during various time intervals, for better clarity Fig. 3 shows the data after 2 and 72 h only. Presented data represent a trend continuing throughout the measurement period. However, the measured data were affected by the limited number of specimens and, at later times of partial immersion, by severe damage due to dimensional changes and cracks. Therefore, the data were burdened with a high variability and were evaluated as statistically inconclusive.
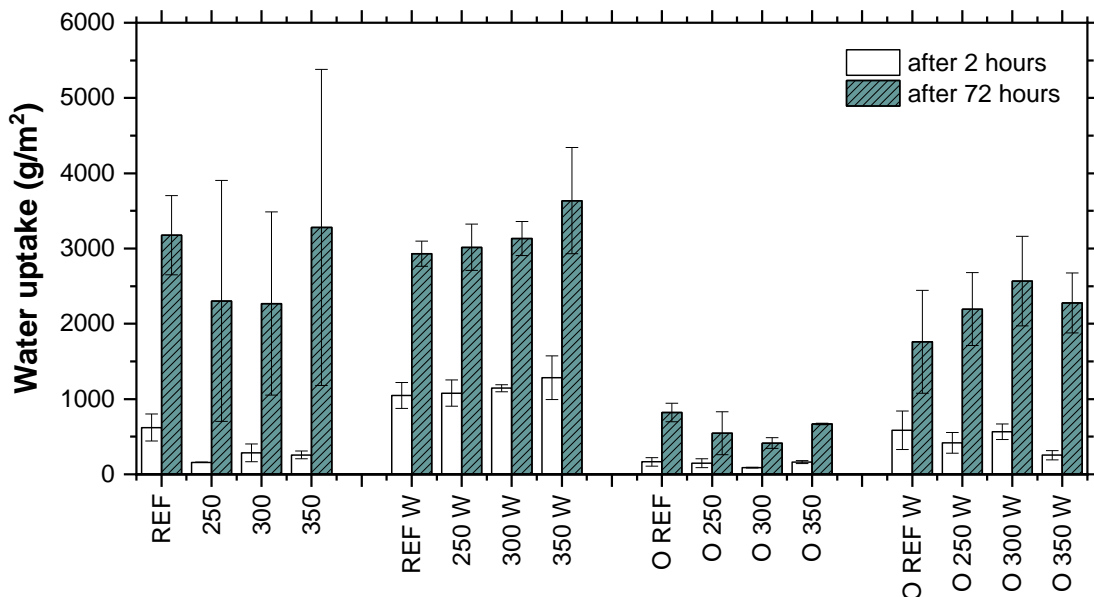
Fig. 3. Average water absorption of partially immersed groups of specimens after 2 and 72 h; REF – Reference; 250, 300, 350 – charred at different temperature; O – coated with linseed oil; W – artificially weathered

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The average water absorption of charred specimens without oil coating after 2 h of partial immersion decreased more than 50% compared to the references. Kymäläinen *et al.* (2017) charred specimens of Norway spruce (*Picea abies* L.) and Scots pine (*Pinus sylvestris* L.) at temperatures of 250 and 400 °C. The charred spruce showed a 24 to 51% reduction in water absorption compared to references. The presented results are supported by other studies describing the chemical changes of the basic components of wood in the surface layers during the application of high temperatures to the surface of charred specimens. A hydrophobic layer forms on the surface as a result of the reduction of hydroxyl groups, crystallization reactions of cellulose, and cross linking of lignin (Mitsui *et al.* 2008; Čermák *et al.* 2019).

As a result of the artificial weathering process, the water absorption of the reference specimens without oil coating was 41% compared to the reference specimens that did not undergo the artificial weathering process. The average water absorption of the charred weathered specimens was significantly higher than the water absorption of the charred non-weathered specimens. The obtained data indicate the disruption of the charring process effect due to significant damage during artificial weathering.

The damage to the charred layer is related to cyclical changes in moisture content during artificial weathering and the structure of unmodified beech wood. According to Šeda *et al.* (2021), the thickness of thermally affected layer of beech specimens charred at temperatures of 250 to 300 °C in short time regimes is over 2 mm. The charred layer is porous and brittle. As the distance from the heating plate increases, the effect of high temperature on the properties of wood decreases (Fortino *et al.* 2013). With increasing distance from the charred surface, the charred layer gradually goes into native wood. Beech is a species whose wood is subject to significant dimensional changes. The cyclic penetration of moisture through the charred layer leads to cracking of the unmodified wood, thereby disrupting the charred layer. The cracked surface leads to increased water absorption and makes the material unsuitable for outdoor use. Drying oils or wax emulsions can be used to slow down the penetration of water through the charred layer and to stabilize the surface (Thybring 2013).

The most significant decrease in water absorption due to surface coating with linseed oil was recorded for the reference specimens. The average water absorption after 2 h of partial immersion of reference specimens with linseed oil was 75% lower than the reference specimens without oil coating. For surface charred specimens, the decrease in water absorption due to the oil coating was not so significant and the water absorption values almost equalled the values of the oiled reference specimens. A more significant decrease (70%) was noted only in the group 300 O.

The average water absorption 2 h of partial immersion of the reference weathered specimens with oil coating was 46% lower than the weathered specimens without oil coating. For the charred specimens, the decrease in water absorption of weathered specimens with oil coating was from 52% to 81% compared to charred weathered specimens without oil coating. However, the effect of the oil coating decreased depending on the duration of the partial immersion due to the leaching out of the oil (Thybring 2013). According to the measured values, the combination of surface charring and oil coating is a suitable option for preparing beech wood for external use. However, the measured values were not statistically clear. Therefore, further measurements are needed to confirm the applicability of this modification.

Decay Resistance

The mass loss due to white-rot and brown-rot fungi of surface charred beech wood at different temperatures is shown in Figs. 4A (*T. versicolor*) and 4B (*P. placenta*). It is clear from the figures that surface charring and oil impregnation slightly decrease the beech wood mass loss caused by *T. versicolor*, but are not able to protect it fully. The effect of different charring temperatures became evident only when 350 °C was used. It is noteworthy that the mass loss was significantly higher than at lower temperatures. Generally, the subsequent oil impregnation did not significantly influence the resistance of charred wood; only at the highest temperature did the surface oiling cause a decrease in mass loss compared to an unoiled charred surface. Similar results were obtained with brown-rot fungus *P. placenta*. Only oiled charred specimens showed significantly lower mass losses than other groups, but this time no differences between temperatures were observed. The result of the charring effect on wood resistance is in line with other studies (Hasburgh *et al.* 2021; Machová *et al.* 2021), which, however, examined only one-sided charred specimens, where the substantial protective effect was also not demonstrated.
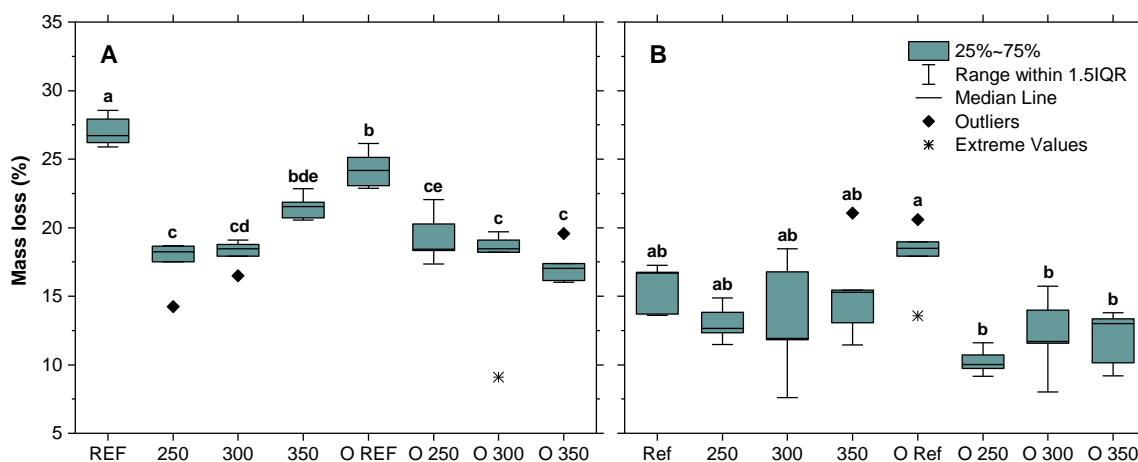

Fig. 4. Mass loss of test specimens after 16 weeks of exposure to fungal degradation; A – *Trametes versicolor*, B – *Poria placenta* (Ref – reference uncharred; 250, 300, 350 – charred at different temperature; O – coated by linseed oil)

It is supposed that a charred wood layer is able to protect the untreated wood below it. It was suggested by Ascough *et al.* (2010) that charcoal produced at 300 to 400 °C is a low – quality growth substrate for fungi *Pleurotus pulmonarius* and *T. versicolor*. Although neither species decomposed charred wood, they were able to colonize the surface, and to a lesser extent also the interior, in less than three months. Additionally, Singh *et al.* (2013) observed that torrefied chips (at 300 °C) were biologically inert, based on mass loss, when they were exposed to *Oligoporus placenta* and *T. versicolor*. Although the charred wood was not a suitable substrate with a readily available source of nutrients, hyphae were able to penetrate it through pre – existing cracks and fissures that extended from the surface to varying depths. However, fungal colonization was characterized by a sparse, exploratory growth form (Ascough *et al.* 2010), which was also observed in the current study. In both sets – oiled and non – oiled, the surface layer (0.5 to 1 mm in depth) was without visible fungal degradation, rather individual hyphae were present in vessel lumina (Fig. 5b, d; 6b, d). Below this layer typical degradation patterns for white rot (Fig. 5a, c) and brown rot

(Fig. 6a, c) with abundant mycelia were observed. Clearly, the charred layer can act as a physical barrier, which delayed the colonization and decomposition of untreated wood.

As Kymäläinen *et al.* (2017) stated, proper tuning of the temperature and time of charring is needed; too high a temperature or too long a time makes the surface crack, creating points of entry to the interior of the specimen, which should be the reason for faster wetting and also the fungal colonization of the inner part of test specimens charred at 350 °C (Figs. 5, 6 and 7). A similar effect was an increase in specimen moisture content during fungus exposure. The swelling of inner untreated beech wood led to the creation of distinct cracks in the charred surface layer due to the hygroscopicity difference and also to its brittleness. These cracks were tightly filled by mycelium at the end of the exposure. Generally, more dimensionally stable and naturally durable softwoods are more suitable for surface charring, because they are easier to burn, and intensive water absorption resulting in excessive swelling is limited.
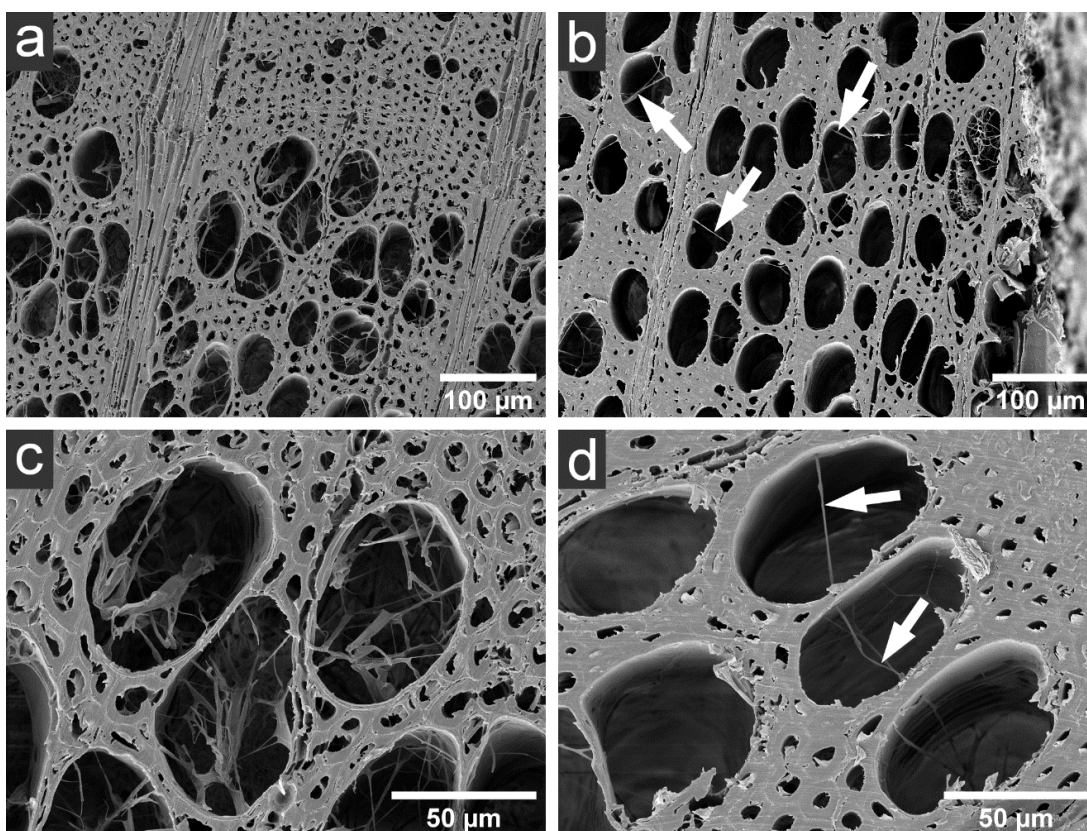
Fig. 5. SEM images of specimens (350) decayed by *T. versicolor* (transverse sections) showing different depth position: (a, c) inner part in depth >1 mm; (b, d) surface layer; sporadic hyphae in vessel lumina (arrows)

Charring of wood is primarily used in exterior applications above ground – *e.g.,* cladding and decking – where it is exposed to weathering and slowly erodes. It is crucial that a minimum charred layer of 2 to 3 mm is created; its weathering resistance is further improved by surface impregnation with drying oils (Sandberg *et al.* 2021). Unfortunately, in our study the real charred layer merely few tenths of millimetre thick was created on the specimen's surface due to selected treatment time and temperature (Fig. 7). The layers deeper under surface (up to 2.5 mm) were rather thermally treated than charred.
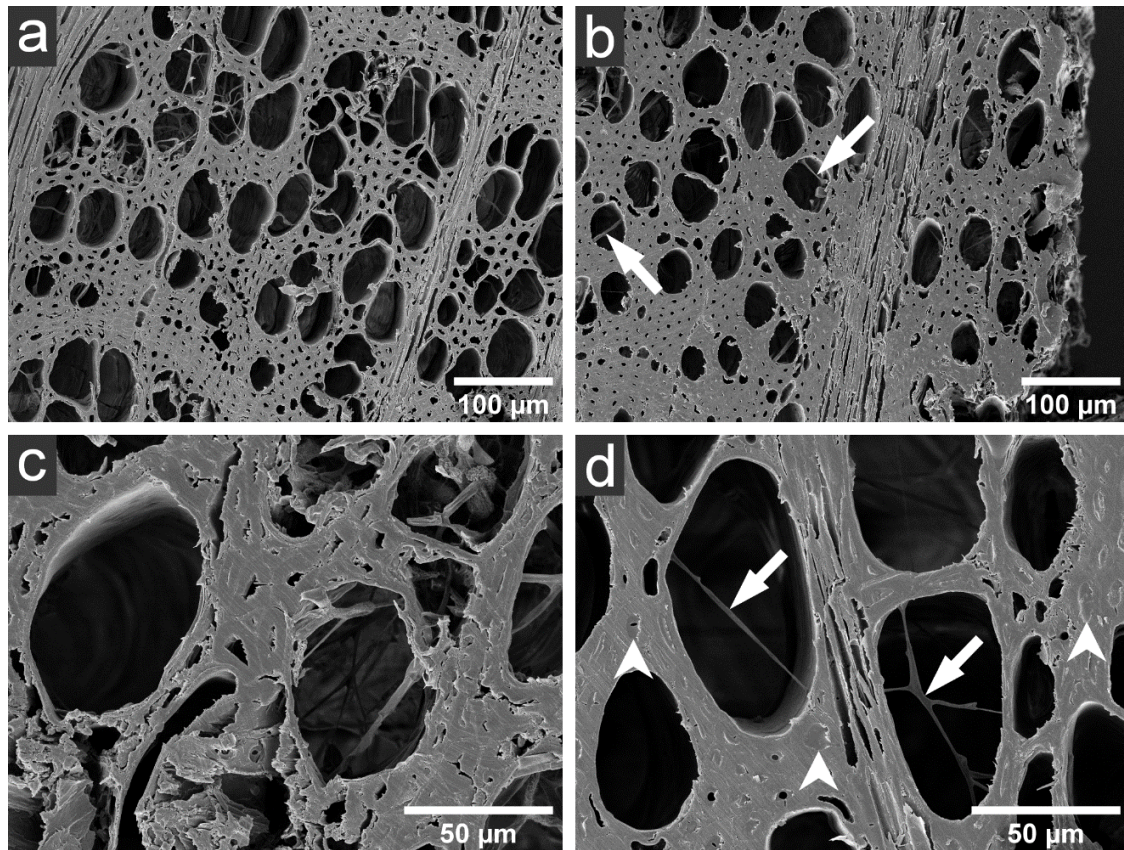
Fig. 6. SEM images of specimens (a, b, c – 350; d – O350) decayed by *P. placenta* (transverse sections) showing different depth position: (a, c) inner part in depth >1 mm; (b, d) surface layer; sporadic hyphae in vessel lumina (arrows), fiber lumina filled by linseed oil (arrowheads)

It was shown in the water absorption test (Fig. 3), that wood torrefaction changed its properties from hydrophilic to hydrophobic, which is caused by dehydration reactions where the destruction of many hydroxyl groups and the formation of unsaturated, non – polar structures take place (Adeleke *et al.* 2021). Linseed oil impregnation increased surface hydrophobicity even further (Fig. 3), whereas microscopic observation showed that the fiber lumina were primarily filled with oil and vessel lumina remained empty, allowing hyphal growth (Fig. 6d). However, there was no significant difference between the mass loss by decay in specimens charred at 250 and 300 °C that were or were not impregnated with linseed oil. However, the limited wetting of the test specimen surface can also be a reason for slower colonization by fungi in real exterior applications.

In contrast, too much charring leads to surface cracking and increased porosity, which enables increased capillary absorption of water and easier wetting of the inner untreated part (Kymäläinen *et al.* 2015a). Torrefied wood or charcoal used in exterior is intensively wetted and subsequently colonized by Ascomycetes or even Basidiomycetes, which both degrade wood to a low degree (Kymäläinen *et al.* 2015b). Additionally, Wengel *et al.* (2006) found that the fungus *Schizophyllum commune* decomposes charcoal at a low rate, so a too thin layer of charred surface does not have to necessarily work as protective barrier against fungi.

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Fig. 7. The decay specimen surface (below) and charring depth (on top) after individual treatments; specimen cross section dimensions 15 × 25 mm²

CONCLUSIONS

Surface-charred beech wood (*Fagus sylvatica* L.) with oil and without oil coating was studied.

1. Analysis of the color change showed that because of the charring process there was a significant decrease in all parameters L^*, a^*, and b^*.

2. As a result of the oil coating, the lightness (L^*) decreased in all groups as well as O REF, O 250, O 300 and O 350. There were different trends between groups for parameters a^* and b^*. There was an increase in these parameters in the group of reference (O REF) and a decrease in the groups of charred specimens (O 250, O 300 and O 350).

3. As a result of artificial weathering, the lightness (L^*) of the surface increased in all groups, except the ones charred at 350 °C, in which it was slightly decreased. The values of ΔL^* followed a stable trend of growth or decrease during the entire process of artificial weathering compared to the values of Δa^* and Δb^*, for which the trend during artificial weathering was irregular.

4. All exposed specimens underwent significant color change ΔE^* due to the artificial weathering process. The smallest changes were noted for groups 350 and 350 O.

5. The average water absorption of charred specimens without oil coating after 2 h of partial immersion decreased more than 50% compared to the references. The damage caused by artificial weathering cancelled the beneficial effect of the charring process. The oil coating benefits were decreased depending on the length of partial immersion.

6. Surface charring and oil impregnation slightly reduced the mass loss of beech wood caused by *T. versicolor* and *P. placenta* but could not protect the wood fully.

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ACKNOWLEDGMENTS

REFERENCES CITED

Adeleke, A. A., Odusote, J. K., Ikubanni, P. P., Lasode, O. A., Malathi, M., and Paswan, D. (2021). "Essential basics on biomass torrefaction, densification and utilization," *International Journal of Energy Research* 45(2), 1375-1395. DOI: 10.1002/er.5884

Ascough, P. L., Sturrock, C. J., and Bird, M. I. (2010). "Investigation of growth responses in saprophytic fungi to charred biomass," *Isotopes in Environmental and Health Studies* 46(1), 64-77. DOI: 10.1080/10256010903388436

ASTM G155 (2013). "Standard practice for operating xenon arc light apparatus for exposure of non-metallic materials," ASTM International, West Conshohocken, PA, USA.

Bekhta, P., Proszyk, S., Lis, B., and Krystofiak, T. (2014). "Gloss of thermally densified alder *(Alnus glutinosa* Goertn.), beech *(Fagus sylvatica* L.), birch *(Betula verrucosa* Ehrh.), and pine *(Pinus sylvestris* L.) wood veneers," *European Journal of Wood and Wood Products* 72, 799-808. DOI: 10.1007/s00107-014-0843-3

Byrne, Ch. E., and Nagle D. C. (1997). "Carbonization of wood for advanced materials applications," *Carbon* 35(2), 259-266. DOI: 10.1016/S0008-6223(96)00136-4

Čermák, P., Dejmal, A., Paschová, Z., Kymäläinen, M., Dömény, J., Brabec, M., Hess, D., and Rautkari, L. (2019). "One-sided surface charring of beech wood," *Journal of Materials Science* 54(13), 9497-9506. DOI: 10.1007/s10853-019-03589-3

Chen, Y., Fan, Y., Gao, J., and Stark, N. M. (2012). "The effect of heat treatment on the chemical and color change of black locust *(Robinia pseudoacacia)* wood flour," *BioResources* 7(1), 1157-1170. DOI: 10.15376/biores.7.1.1157-1170

Cogulet, A., Blanchet, P., and Landry, V. (2016). "Wood degradation under UV irradiation: A lignin characterization," *Journal of Photochemistry and Photobiology B: Biology* 158, 184-191. DOI: 10.1016/j.jphotobiol.2016.02.030

Dubois, H., Verkasalo, E., and Claessens, H. (2020). "Potential of birch *(Betula pendula* Roth and B. *pubescens* Ehrh.) for forestry and forest-based industry sector within the changing climatic and socio-economic context of Western Europe," *Forests* 11(3), article 336. DOI: 10.3390/f11030336

Ebner, D. H., Barbu, M. C., Klaushofer, J., and Čermák, P. (2021). "Surface modification of spruce and fir sawn-timber by charring in the traditional Japanese method—Yakisugi," *Polymers* 13(10), article 1662. DOI: 10.3390/polym13101662

EN 927-5 (2006). "Paint and varnishes – Coating materials and coating systems for exterior wood – Part 5: Assessment of the liquid water permeability," European Committee for Standardization, Brussels, Belgium.

Ermolenko, I. N., Lyubliner, I. P., and Gulko, N. V. (1990). *Chemically Modified Carbon Fibers and Their Applications*, VHC Publishers Inc., Weinheim, Germany.

Evans, P., Chowdhury, M. J., Mathews, B., Schmalzl, K., Ayer, S., Kiguchi, M., and Kataoka, Y. (2005). "Weathering and surface protection of wood," in: *Handbook of Environmental Degradation of Materials*, Myer Kutz Associates, Inc., Delmar, NY, USA, pp. 277-297. DOI: 10.1016/B978-081551500-5.50016-1

Fortino, S., Genoese, A., Genoese, A., and Rautkari, L. (2013). "FEM simulation of the hygro-thermal behaviour of wood under surface densification at high temperature," *Journal of Materials Science* 48, 7603-7612. DOI: 10.1007/s10853-013-7577-1

González-Peña, M. M., and Hale, M. D. C. (2009). "Colour in thermally modified wood of beech, Norway spruce and Scots pine. Part 1: Colour evolution and colour changes," *Holzforschung* 63(4), 385-393. DOI: 10.1515/hf.2009.078

Grüll, G., Truskaller, M., Podgorski, L., Bollmus, S., De Windt, I., and Suttie, E. (2013). "Moisture conditions in coated wood panels during 24 months natural weathering at five sites in Europe," *Wood Material Science and Engineering* 8(2), 95-110. DOI: 10.1080/17480272.2013.771212

Hasburgh, L. E., Zelinka, S. L., Bishell, A. B., and Kirker, G. T. (2021). "Durability and fire performance of charred wood siding (Shou Sugi Ban)," *Forests* 12(9), article 1262. DOI: 10.3390/f12091262

Huang, X., Kocaefe, D., Kocaefe, Y., Boluk, Y., and Pichette, A. (2012). "A spectrocolorimetric and chemical study on color modification of heat-treated wood during artificial weathering," *Applied Surface Science* 258(14), 5360-5369. DOI: 10.1016/j.apsusc.2012.02.005

Hyvönen, A., Piltonen, P., and Niinimäki, J. (2005). "Tall oil/water – emulsions as water repellents for Scots pine sapwood," *Holz Als Roh-und Werkstoff* 64(1), 68-73. DOI: 10.1007/s00107-005-0040-5

Jämsä, S., Ahola, P., and Viitaniemi, P. (2000). "Long-term natural weathering of coated ThermoWood," *Pigment & Resin Technology* 29(2), 68-74. DOI: 10.1108/03699420010317807

Jones, D. (2017). "Introduction to the performance of bio-based building materials," in: *Performance of Bio-Based Building Materials*, Woodhead Publishing, Duxford, United Kingdom, pp.1-19. DOI: 10.1016/b978-0-08-100982-6.00001-x

Kamdem, D. P., Pizzi, A., and Jermannaud, A. (2002). "Durability of heat-treated wood," *Holz Als Roh-und Werkstoff* 60(1), 1-6. DOI: 10.1007/s00107-001-0261-1

Kymäläinen, M., Rautkari, L., and Hill, C. A. S. (2015a). "Sorption behaviour of torrefied wood and charcoal determined by dynamic vapour sorption," *Journal of Materials Science* 50(23), 7673-7680. DOI: 10.1007/s10853-015-9332-2

Kymäläinen, M., Mäkelä, M. R., Hildén, K., and Kukkonen, J. (2015b). "Fungal colonisation and moisture uptake of torrefied wood, charcoal, and thermally treated pellets during storage," *European Journal of Wood and Wood Products* 73(6), 709-717. DOI: 10.1007/s00107-015-0950-9

Kymäläinen, M., Hautamäki, S., Lillqvist, K., Segerholm, K., and Rautkari, L. (2017). "Surface modification of solid wood by charring," *Journal of Materials Science* 52(10), 6111-6119. DOI: 10.1007/s10853-017-0850-y

Kymäläinen, M., Sjökvist, T., Dömény, J., and Rautkari, L. (2022a). "Artificial weathering of contact-charred wood – The effect of modification duration, wood species and material density," *Materials* 15(11), article 3951. DOI: 10.3390/ma15113951

Kymäläinen, M., Lourençon, T. V., and Lillqvist, K. (2022b). "Natural weathering of soft-and hardwoods modified by contact and flame charring methods," *European Journal of Wood and Wood Products* 80(6), 1309-1320. DOI: 10.1007/s00107-022-01864-w

Lesar, B., and Humar, M. (2010). "Use of wax emulsions for improvement of wood durability and sorption properties," *European Journal of Wood and Wood Products* 69(2), 231-238. DOI: 10.1007/s00107-010-0425-y

Lovrić, A., Zdravković, V., and Furtula, M. (2014). "Influence of thermal modification on colour of poplar (*Populus* x *euramericana*) rotary cut veneer," *Wood Research* 59(2), 661-670.

Machová, D., Oberle, A., Zárybnická, L., Dohnal, J., Šeda, V., Dömény, J., Vacenovská, V., Kloiber, M., Pěnčík, J., Tippner, J., *et al.* (2021). "Surface characteristics of one-sided charred beech wood," *Polymers* 13, article 1551. DOI: 10.3390/polym13101551

Mallégol, J., Gardette, J.-L., and Lemaire, J. (2000). "Long-term behavior of oil-based varnishes and paints. Photo- and thermooxidation of cured linseed oil," *Journal of the American Oil Chemists' Society* 77(3), 257-263. DOI: 10.1007/s11746-000-0042-4

Militz, H. (2002). "Thermal treatment of wood: European processes and their background," in: *The 33rd Annual Meeting on the International Research Group on Wood Preservation*, Cardiff, UK, IRG/ WP 02-40241.

Mitani, A., and Barboutis, I. (2013). "Heat treatment effect on colour changes of beech (*Fagus sylvatica*) wood," *Pro Ligno* 9(4), 140-145.

Mitsui, K., Inagaki, T., and Tsuchikawa, S. (2008). "Monitoring of hydroxyl groups in wood during heat treatment using NIR spectroscopy," *Biomacromolecules* 9(1), 286–288. DOI: 10.1021/bm7008069

Popescu, C.-M., Popescu, M.-C., and Vasile, C. (2011). "Structural analysis of photodegraded lime wood by means of FT-IR and 2D IR correlation spectroscopy," *International Journal of Biological Macromolecules* 48(4), 667-675. DOI: 10.1016/j.ijbiomac.2011.02.009

Repellin, V., and Guyonnet, R. (2005). "Evaluation of heat-treated wood swelling by differential scanning calorimetry in relation to chemical composition," *Holzforschung* 59(1). DOI: 10.1515/hf.2005.005

Sandberg, D., Kutnar, A., Karlsson, O., and Jones, D. (2021). *Wood Modification Technologies: Principles, Sustainability, and the Need for Innovation*, CRC Press, Boca Raton, FL, USA, 442 pp., eBook ISBN 9781351028226

Santos, J. A. (2000). "Mechanical behaviour of eucalyptus wood modified by heat," *Wood Science and Technology* 34(1), 39-43. DOI: 10.1007/s002260050006

Schultz, T. P., Nicholas, D. D., and Ingram, L. L. (2007). "Laboratory and outdoor water repellency and dimensional stability of southern pine sapwood treated with a waterborne water repellent made from resin acids," *Holzforschung* 61(3), 317-322. DOI: 10.1515/hf.2007.044

Šeda, V., Machová, D., Dohnal, J., Dömény, J., Zárybnická, L., Oberle, A., Vacenovská, J., and Čermák, P. (2021). "Effect of one-sided surface charring of beech wood on density profile and surface wettability," *Applied Sciences* 11(9), article ID 4086. DOI: 10.3390/app11094086

Singh, T., Singh, A. P., Hussain, I., and Hall, P. (2013). "Chemical characterisation and durability assessment of torrefied radiata pine (*Pinus radiata*) wood chips," *International Biodeterioration & Biodegradation* 85, 347-353. DOI: 10.1016/j.ibiod.2013.07.014

Soytürk, E. E., Kartal, S. N., Arango, R. A., Ohno, K. M., Solhan, E., Çağlayan, İ., and Ibanez, C. M. (2023). "Surface carbonization of wood: Comparison of the biological performance of *Pinus taeda* and *Eucalyptus bosistoana* woods modified by contact

charring method," *Wood Material Science & Engineering*, Online. DOI: 10.1080/17480272.2023.2198993

Sundqvist, B., and Morén, T. (2002). "The influence of wood polymers and extractives on wood colour induced by hydrothermal treatment," *Holz als Roh-und Werkstoff* 60(5), 375-376. DOI: 10.1007/s00107-002-0320-2

Teacă, C. A., Roşu, D., Bodîrlău, R., and Roşu, L. (2013). "Structural changes in wood under artificial UV light irradiation determined by FTIR spectroscopy and color measurements – A brief review," *BioResources* 8(1), 1478-1507. DOI: 10.15376/biores.8.1.1478-1507

Thybring, E. E. (2013). "The decay resistance of modified wood influenced by moisture exclusion and swelling reduction," *International Biodeterioration & Biodegradation* 82, 87-95. DOI: 10.1016/j.ibiod.2013.02.004

Unsal, O., Korkut, S., and Atik, C. (2003). "The effect of heat treatment on some properties and colour in eucalyptus (*Eucalyptus camaldulensis* Dehn.) wood," *Maderas. Ciencia y Tecnología* 5(2), 145-152.

Weiland, J. J., and Guyonnet, R. (2003). "Study of chemical modifications and fungi degradation of thermally modified wood using DRIFT spectroscopy," *Holz als Roh-und Werkstoff* 61(3), 216-220. DOI: 10.1007/s00107-003-0364-y

Wengel, M., Kothe, E., Schmidt, C. M., Heide, K., and Gleixner, G. (2006). "Degradation of organic matter from black shales and charcoal by the wood-rotting fungus *Schizophyllum commune* and release of DOC and heavy metals in the aqueous phase," *Science of The Total Environment* 367(1), 383-393. DOI: 10.1016/j.scitotenv.2005.12.012

Werner, F., Taverna, R., Hofer, P., and Richter, K. (2006). "Greenhouse gas dynamics of an increased use of wood in buildings in Switzerland," *Climatic Change* 74(1-3), 319-347. DOI: 10.1007/s10584-006-0427-2

Wikberg, H., and Maunu, S. L. (2004). "Characterisation of thermally modified hard- and softwoods by C CPMAS NMR," *Carbohydrate Polymers* 58(4), 461-466. DOI: 10.1016/j.carbpol.2004.08.008

Yoo, S., Kelley, S. S., Tilotta, D. C., and Park, S. (2018). "Structural characterization of loblolly pine derived biochar by X-ray diffraction and electron energy loss spectroscopy," *ACS Sustainable Chemistry & Engineering* 6(2), 2621-2629. DOI: 10.1021/acssuschemeng.7b04119