

# Changes in the Physical and Mechanical Properties of *Pinus taeda* and *Eucalyptus bosistoana* Wood Modified by Contact Charring

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Physical and mechanical properties were evaluated for all-sided charred *Pinus taeda* and *Eucalyptus bosistoana* wood by hot plate contact heating system followed by treatment with linseed oil. The water absorption, volumetric swelling, wettability, hardness, modulus of rupture, and modulus of elasticity in bending strength and compression strength parallel to grain were determined. The water absorption and volumetric swell were determined after immersion in water, as measured at various intervals of water immersion up to 120 h. The results suggested that the contact charring process with the addition of a linseed oil application improved water absorption and volumetric swell properties of charred specimens compared to un-charred controls. Hardness of the charred wood decreased by 38% and 43% in *P. taeda* and *E. bosistoana* specimens, respectively, compared with their respective controls. The highest reductions were seen in modulus of elasticity and compression strength values in charred *P. taeda* specimens, while modulus of rupture (MOR) values decreased more in charred *E. bosistoana* specimens than in charred *P. taeda* specimens. These results suggested that charring of *P. taeda* and *E. bosistoana* wood does improve the moisture-related characteristics; however, their mechanical behavior and hardness decreased.

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## INTRODUCTION

Thermal treatments are well-known modification methods that change the structure of wood, increase durability against wood degrading organisms, decrease water absorption, and improve dimensional stability. In general, temperatures below 300 °C are employed in the thermal treatment process to prevent issues related to mechanical strength loss, which may limit use of thermally modified wood particularly in load-bearing applications (Boonstra and Tjeerdsma 2006; Yan and Morrell 2014; Candelier *et al.* 2016; Čermák *et*

al. 2016; Sandberg and Kutnar 2016; Kymäläinen *et al.* 2018; Morozovs *et al.* 2021). Candelier *et al.* (2016) found that mechanical resistance, particularly modulus of elasticity (MOE) and modulus of rupture (MOR) in bending of heat-treated wood, decreases based on the intensity of the treatment (Kim 1998; Jimenez Jr *et al.* 2011; Bal 2014). However, the adverse effects of heat treatments on the mechanical properties of wood is one of the most important disadvantages of thermal modifications (Chotikhun *et al.* 2020).

Unlike conventional thermal modification methods, surface charring of wood has been used for centuries to increase wood durability. One-sided surface charring methods are an alternative to overcome reductions in mechanical strength properties of thermally modified wood (Morozovs *et al.* 2021). Surface charring methods modify only the exposed surface while maintaining the mechanical properties of underlying wood (Čermák *et al.* 2019; Buksans *et al.* 2021). One-sided surface charred wood is suitable for use as wood cladding and wooden shingles in structures, while all-side charred wood is used in more severe conditions such as wet environments (Lunguleasa and Spirchez 2017; Kymäläinen *et al.* 2018). Charring decreases water sorption due to increased hydrophobicity, which is caused by the degradation of more reactive components, the hemicelluloses and amorphous cellulose (Kymäläinen *et al.* 2017). As a result of reduced hygroscopicity, both the mechanical and biological durability is expected to be higher. However, more detailed studies are needed to find suitable temperatures and times for the charring process because elevated temperatures and/or extended treatment times can result in surface cracks, which increase water sorption and reduce mechanical properties.

Although many studies have examined moisture uptake and weathering properties of one-sided charred wood, research on all-side charred wood and its mechanical and water-sorption characteristics are lacking. Soytürk *et al.* (2023) examined the biological performance of all-sided charred wood with oil application and found correlations between total carbohydrate content and durability against decay and mold fungi, and subterranean termites in laboratory tests. To build on this work, the objective of this research was to evaluate the effect of an all-side wood surface charring method with protective linseed oil application on mechanical strength, hardness, and hydrophobic, and volumetric swell properties. Linseed (flax) oil was added, as it has already been incorporated as an ingredient in alkyd or polyurethane systems; in addition, it is the most used natural oil for surface treatments of wood in Europe (Kymäläinen *et al.* 2022a).

## EXPERIMENTAL

### Wood Specimens

Three 50-year-old *Eucalyptus bosistoana* F. Muell. trees belonging to a shelter-belt multispecies located in Bernardo Rosengurtt Experimental Station of the Faculty of Agronomy of Universidad de la República (32°21'26"S, 54°26'34"W, Cerro Largo, Uruguay), and two 22-year-old *Pinus taeda* L. trees from the same plantation were used in this study. Sapwood only portions of the selected trees were cut into 2-meter-long boards and air dried for four months. The sapwood boards were then cut in a sawmill into specimens of suitable dimensions based on the individual tests. For each, half the specimens were charred while the other half was used as controls. For modulus of rupture (MOR) and modulus of elasticity (MOE) tests, the specimens (4.5 x 4.5 x 71 mm) were prepared from charred wood blocks (12 x 20 x 71 mm), whilst compression tests and density evaluations were done by using charred and uncharred specimens (25 x 15 x 25 mm). Then water absorption, thickness swelling, hardness, and wettability tests were

carried out by using charred and uncharred specimens (25 x 15 x 50 mm).

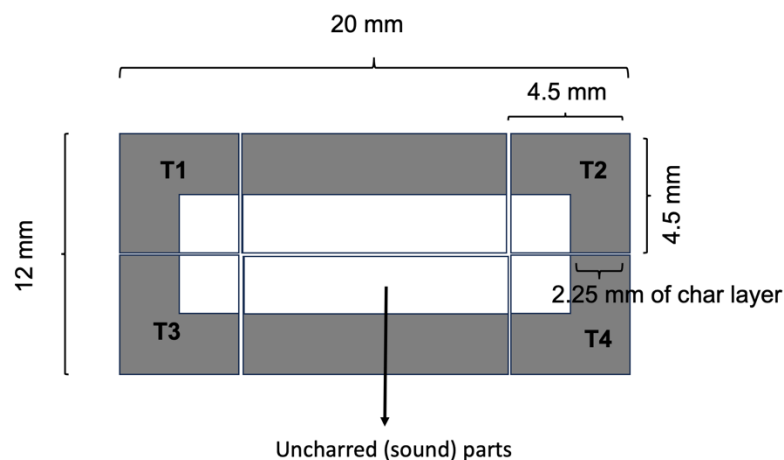
### Charring Process

Wood specimens were oven dried at 80 °C for 48 h and then charred on a metallic hot surface at an initial temperature of 120 °C. The metallic surface was heated with an electric resistance controlling the temperature every 15 min with an HI 99550-00 infrared thermometer (Hanna Inst. Mexico City, Mexico) (range -10 / 300 °C). To prevent cracking, temperature was always kept below 150 °C. For each test, wood samples were comprised of ten specimens of equal dimensions. Specimens were set on the hot plate and then weighed down by a 5 kg weight to ensure even contact throughout the entire surface and to avoid deformations. The conditions chosen were based on preliminary testing that focused on minimizing surface cracking while maintaining a high enough temperature to form a thick and uniform charred layer beneath the wood surface.

Contact time with the hot surface was determined by controlling the char size, which was measured using a caliper, with the goal of obtaining homogeneous specimens with a coal layer. Each surface of a given specimen was exposed to heat for 10 minutes and all surfaces of each specimen were exposed to the same charring conditions. For full surface charring, specimens were rotated to expose the other five faces to the same treatment conditions. Average thickness of the char layer in the charred specimens was measured as 2.25 mm ( $\pm 0.1$  mm) (N: 30). Once charring was completed, the specimens were immediately dipped in linseed oil for five seconds to homogenize the charred layer and prevent further cracking. Excess oil and loose charred particles were removed with a brush, and specimens were air dried for four days at room temperature.

### Mechanical Tests

*P. taeda* and *E. bosistoana* test specimens with 4.5 x 4.5 x 71 mm evaluated in a three-point bending test setup based on the ISO 13061-3 (2014) and ISO 13061-4 (2014) standard test methods with a 63 mm span length. Test specimens were prepared from four corners of previously charred wood blocks (12 x 20 x 71 mm) of which char thickness is approximately 2.25 mm (Fig. 1).



**Fig. 1.** Cutting pattern of specimens (T1, T2, T3 and T4) (4.5 x 4.5 x 71 mm) for MOR and MOE tests from previously charred wood (10 x 20 x 71 mm) (cross section)

Modulus of elasticity (MOE) and modulus of rupture (MOR) were determined by the below equations. Compression strength tests were performed on test specimens with

25 x 15 x 25 mm parallel to grain ( $\sigma_c$ ) based on the ISO 13061-17 standard (2017), calculated according to the equations below,

$$MOE = \frac{(F_2 - F_1) \cdot l^3}{4 \cdot b \cdot h^3 \cdot (w_2 - w_1)} \quad (1)$$

$$MOR = \frac{3 \cdot F_{max} \cdot a}{b \cdot h^2} \quad (2)$$

$$\sigma_c = \frac{F_{max}}{b \cdot h} \quad (3)$$

where MOE is the modulus of elasticity (MPa), MOR is the modulus of rupture (MPa),  $l$  is the span distance (mm),  $a$  is the distance between a loading position and the nearest support in a bending test (mm),  $F_2 - F_1$  is the load difference at 40% and 10% of maximum load (N),  $F_{max}$  is the maximum load for bending and compression test respectively (N),  $w_2 - w_1$  is the deflection difference at 40% and 10% of maximum load (mm),  $b$  is the width (mm) and  $h$  is the height of specimens (mm).

Wood specimens (25 x 15 x 25 mm) prepared for density ( $\rho$ ) measurement were weighed, and their dimensions were measured with an accuracy of 0.01 g and 0.01 mm, respectively, according to the ISO 13061-2 standard (2014). In addition, moisture content (MC) values were obtained from these specimens after mechanical testing from a piece of the test specimens following the procedure defined in the ISO 13061-1 standard (2014). The experimental values for the MOR, MOE,  $\sigma_c$  and the density ( $\rho$ ) of specimens were adjusted to 12% according to the following equations,

$$MOR_{12} = MOR_m \cdot [1 + \alpha \cdot (M - 12)] \quad (4)$$

$$MOE_{12} = \frac{MOE_m}{1 - \alpha \cdot (M - 12)} \quad (5)$$

$$\sigma_{12} = \sigma_m \cdot [1 + \alpha \cdot (M - 12)] \quad (6)$$

$$\rho_{12} = D_m \cdot \left(1 - \frac{(1-K) \cdot (m-12)}{100}\right) \quad (7)$$

where  $M$  is the moisture content of the specimen determined according to the ISO 13061-1 standard (2014) (%),  $\alpha$  is the correction factor for the moisture content; equal to 0.04 for MOR, 0.02 for MOE and 0.05 for  $\sigma_c$  and  $K$  is the coefficient of volumetric shrinkage for a change in moisture content of 1%.

The numerical value of  $K$  can be taken as equal to  $(0.85 \cdot 10^{-3} \cdot \rho_m)$  when the density ( $\rho$ ) is expressed in  $\text{kg/m}^3$ . Differences among groups averages were evaluated for statistical significance using an independent-specimen t-test at a 95% confidence level ( $p \leq 0.05$ ) (IBM SPSS 21.0 software).

### Hardness Tests

Hardness of test specimens (15 x 25 x 50 mm) was determined following the ISO 1522-2022 standard method (ISO 2022), which utilizes a pendulum (*The König pendulum apparatus*) damping method to evaluate coating hardness. Differences among groups averages were evaluated for statistical significance using a Mann-Whitney U Test at a 95% confidence level ( $p \leq 0.05$ ).

### Water Absorption and Volumetric Swell Tests

From each treatment group, 10 test specimens (15 x 25 x 50 mm) were oven-dried at 103 °C for 3 days and weighed. They were then placed in water and re-weighed after

120 h. Any excess water on the specimen surface was blotted with paper tissue before weighing.

Water absorption (WA) was evaluated as follows,

$$WA (\%) = \frac{(W_2 - W_1)}{W_1} \times 100 \quad (8)$$

where  $W_2$  is the weight of wood specimen after immersion in water for 120 h (g), and  $W_1$  is the oven-dried weight of wood specimen before immersion in water (g).

Volumetric swelling (VS) was determined by measuring the oven-dried and wet dimensions of the specimens as follows,

$$VS (\%) = \frac{(V_1 - V_0)}{V_0} \times 100 \quad (9)$$

where  $V_1$  is the wet volume of wood specimen after immersion in water for 120 h ( $\text{mm}^3$ ), and  $V_0$  is the oven-dried volume of wood specimen ( $\text{mm}^3$ ).

### Wettability

Contact angle measurements were taken with a Teta Lite optical tensiometer (Biolin Scientific) to determine the effect of charring on the wetting of wood. The test specimens (charred and uncharred) (15 x 25 x 50 mm) were conditioned at  $65 \pm 5\%$  relative humidity (RH),  $20 \pm 2$  °C for two weeks. The MC of the specimens was 10 to 12% (*P. taeda*) and 12 to 14% (*E. bosistoana*). Three test droplets were measured from a total of three specimens per treatment. The contact angle was measured by placing a droplet of 5  $\mu\text{L}$  of water onto the earlywood surface regions of the specimens, with a camera pointed parallel to grain. The reported values are averages of measurements from at least 3 different positions on each specimen.

## RESULTS AND DISCUSSION

Moisture content and density ( $\rho$ ) values of test specimens are shown in Table 1. As expected, charred wood specimens had lower moisture content due to less water absorption and linseed oil application. Despite the charring process, the density of charred specimens was higher than un-charred specimens, which was likely as a result of the linseed oil applications. Leitch (2009) found the amount of cell wall material to be a common metric for wood density, calculated by specimen weight and volume. It is apparent that linseed oil penetrated wood pores and lumens, increasing the weight of the specimens without any increasing wood volume, thus causing increases in density values of charred specimens. Kymäläinen *et al.* (2022a) states that linseed oil application in charred wood results in increases in hydrophobicity due to its penetration into wood pores. Wang and Cooper (2005) also state that wax and wax-based products can remain in cell lumens and impede moisture uptake.

A number of studies on thermal and charring treatments of wood showed decreases in wood densities due to chemical modifications in the wood (Metsä-Kortelainen *et al.* 2006; Boonstra *et al.* 2007; Gündüz *et al.* 2008; Korkut *et al.* 2008; Akyildiz *et al.* 2009; Gunduz *et al.* 2009; Won *et al.* 2012; Percin *et al.* 2016; Antons *et al.* 2018; Kapidani *et al.* 2019; Chotikhun *et al.* 2020; Wang *et al.* 2020; Esteves *et al.* 2021; Gennari *et al.* 2021; Ninane *et al.* 2021; Šeda *et al.* 2021; Taraborelli *et al.* 2022).

Hill *et al.* (2021) showed that thermal treatments for heat-modified wood generally resulted in increased mass loss and decreased wood density due to chemical degradation and that weight loss after heat treatment was generally linked to a reduction in density associated with treatment conditions and wood species (Pfriem *et al.* 2009). Leitch (2009) states that higher density woods typically demonstrate greater mechanical properties. However, there was a negative correlation between density and mechanical strength values since the changes in the density were the result of linseed oil accumulation in the lumen.

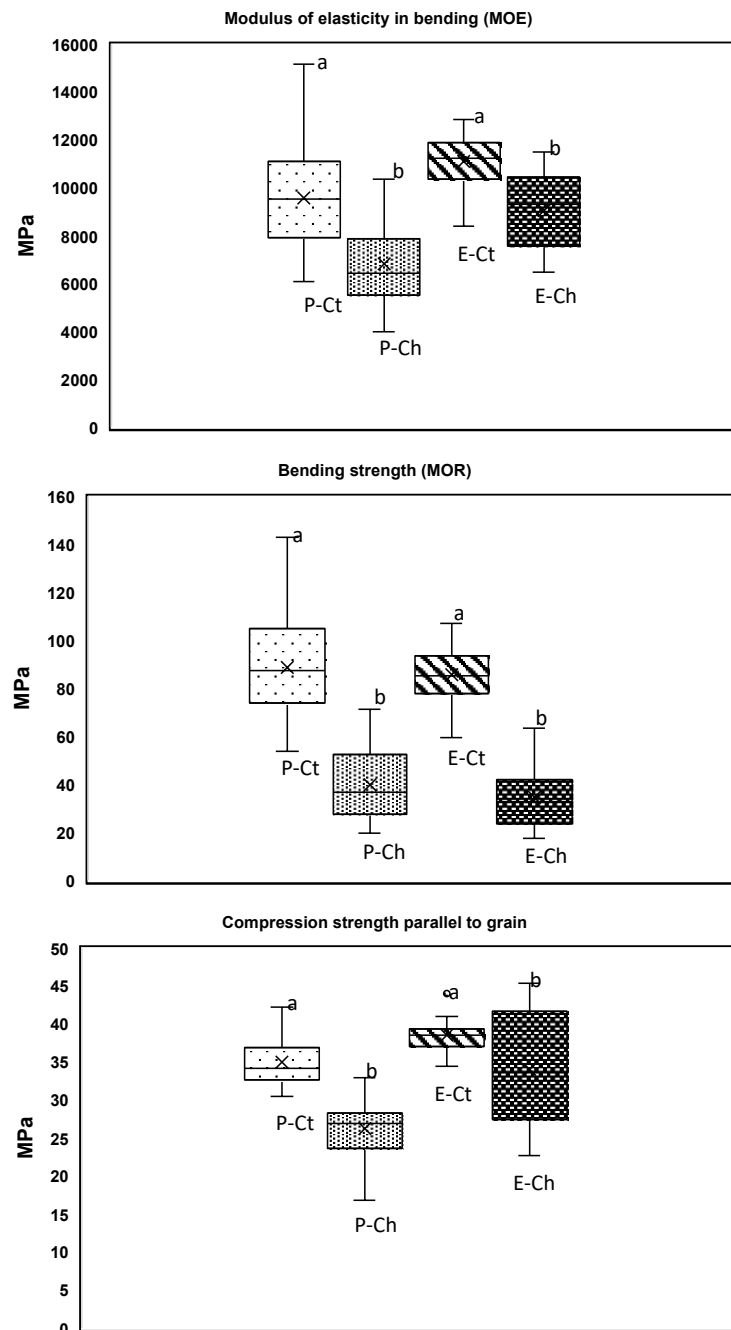
**Table 1.** Density and Mechanical Strength Values of the Test Specimens Adjusted to 12% MC Level

Specimens	MC (%)	$\rho_{12}$ (g/cm <sup>3</sup> )	MOE <sub>12</sub> (MPa)	MOR <sub>12</sub> (MPa)	$\sigma_{c12}$ (MPa)
P-Ct	10.1 (0.23)	0.503a (0.03)	9582a (2245)	89.1a (20.9)	35.1a (3.4)
P-Ch	6.1 (1.63)	0.724b (0.08)	6832b (1764)	39.8b (14.9)	26.3b (3.5)
E-Ct	9.1 (0.43)	0.516a (0.02)	11131a (1030)	85.9a (12.6)	38.6a (2.1)
E-Ch	6.8 (0.97)	0.590b (0.05)	9077b (1512)	34.6b (12.3)	34.0b (7.0)

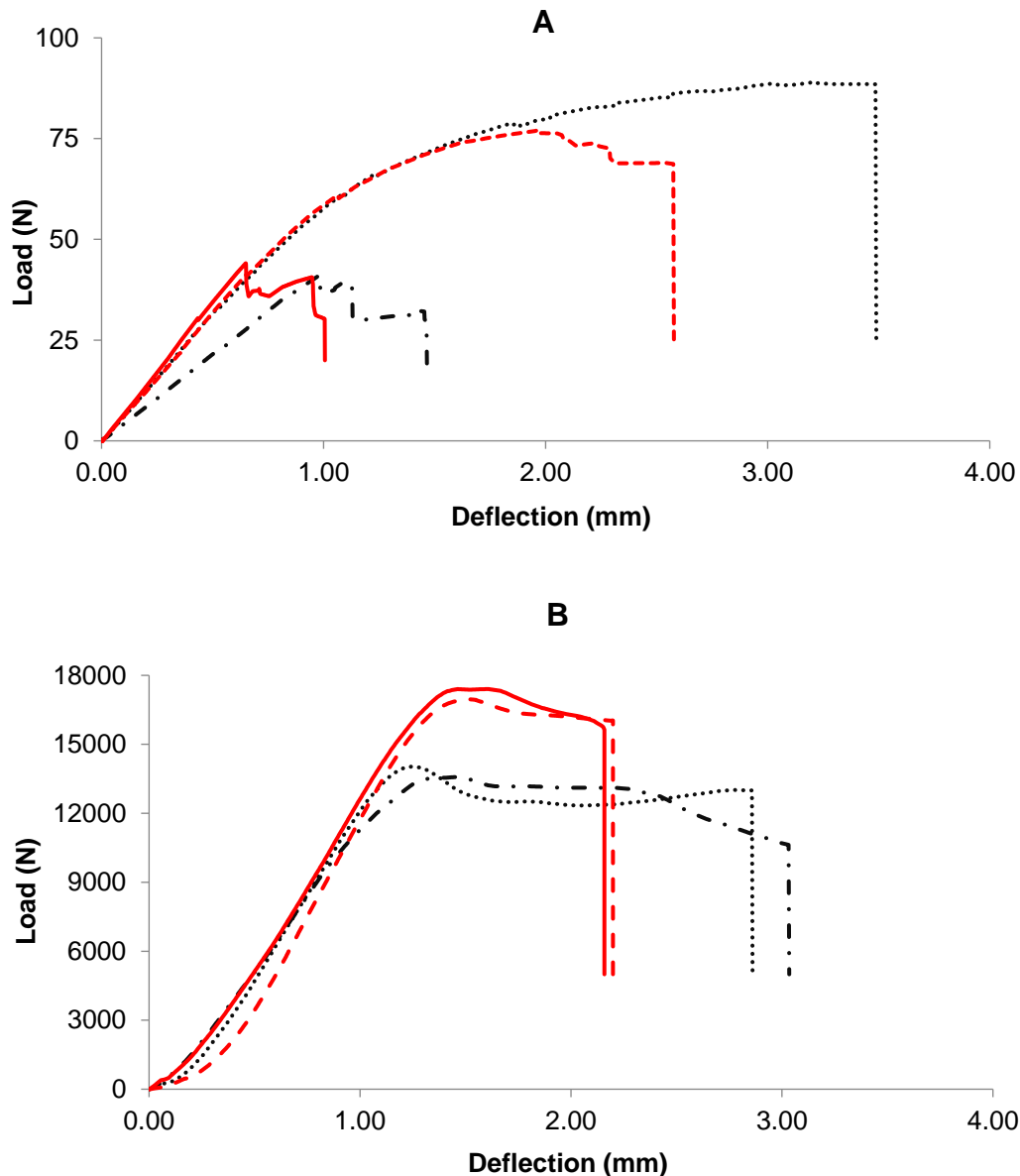
P-Ct: *P. taeda* control; P-Ch: *P. taeda* charred; E-Ct: *E. bosistoana* control; E-Ch: *E. bosistoana* charred. MC: Moisture content;  $\rho_{12}$ : Density at MC of 12%; MOE<sub>12</sub>: Modulus of elasticity at MC of 12%; MOR<sub>12</sub>: Bending strength at MC of 12%;  $\sigma_{c12}$ : Compression strength parallel to grain at MC of 12%. Values in parenthesis show standard deviation. Columns with the same letters indicate no statistical difference between the specimens according to the independent-specimen t-test ( $p \leq 0.05$ ). N for MC and D<sub>12</sub>: 20 and 30 for control and charred specimens, respectively. N for MOE<sub>12</sub> and MOR<sub>12</sub>: 33 for both control and charred specimens. N for  $\sigma_{c12}$ : 20 and 28 for control and charred specimens, respectively.

The values for MOE, MOR, and compression strength parallel to grain are shown in Fig. 2. These results indicate that charring has a negative effect on all strength properties evaluated in both wood species. Of the properties tested, the greatest decrease was in bending strength for both wood species, with reductions in MOR values reaching 55% and 60% in charred *P. taeda* and *E. bosistoana* specimens, respectively. The reductions in MOE (29%) and compression strength (25%) were higher in charred *P. taeda* specimens in comparison with *E. bosistoana* specimens (19% for MOE and 12% for compression strength).

Load-deformation curves from the bending and compression strength tests and load-deflection data obtained from a 3-point bending test are shown in Fig. 3. The load-deflection graph suggests that the semi-plastic regions of the charred *P. taeda* and *E. bosistoana* specimens were shortened compared to un-charred specimens. As a result, the maximum amount of deflection was reduced by 59% and 55% for *P. taeda* and *E. bosistoana* specimens, respectively. In addition, weakening of the tensile zone due to carbonization in the charred specimens resulted in a decrease in maximum load values of 54% for *P. taeda* and 42% for *E. bosistoana*. Compression strength measurements parallel to grain showed significant difference in charred or control specimens for either species. The differences between the compression strength of charred and control specimens were minor before the actual moisture content of the specimens was adjusted to 12% MC; after they were adjusted, the difference in compression strength differed significantly, decreasing by about 25% for *P. taeda* and 12% for *E. bosistoana* charred specimens.



**Fig. 2.** Mechanical properties of charred and control specimens (Box-and-whiskers plots encode five characteristics of a distribution by position and length. The box ranges from the first (Q1) to the third quartile (Q3) of the distribution and represents the interquartile range (IQR). A line across the box indicates the median. The whiskers are lines extending from Q1 and Q3 to end points that are typically defined as the most extreme data points within  $Q1 - 1.5 \times IQR$  and  $Q3 + 1.5 \times IQR$ , respectively. Each outlier outside the whiskers is represented by an individual mark. Alternatively, the minimum and maximum values in the data set are used as end points for the whiskers. P-Ct: *P. taeda* control; P-Ch: *P. taeda* charred; E-Ct: *E. bosistoana* control; E-Ch: *E. bosistoana* charred; the same letters in the bars indicate that there was no statistical difference between the specimens according to the independent-specimen t-test ( $p \leq 0.05$ ); N for  $MOE_{12}$  and  $MOR_{12}$ : 33 for both control and charred specimens. N for  $\sigma_{C12}$ : 20 and 28 for control and charred specimens, respectively). The box plot was used to plot the distribution of the data set. Independent-specimen t-test and IBM SPSS 21.0 software for statistical analyzes were used to verify differences of all among groups averages with 95% confidence level ( $p \leq 0.05$ ).

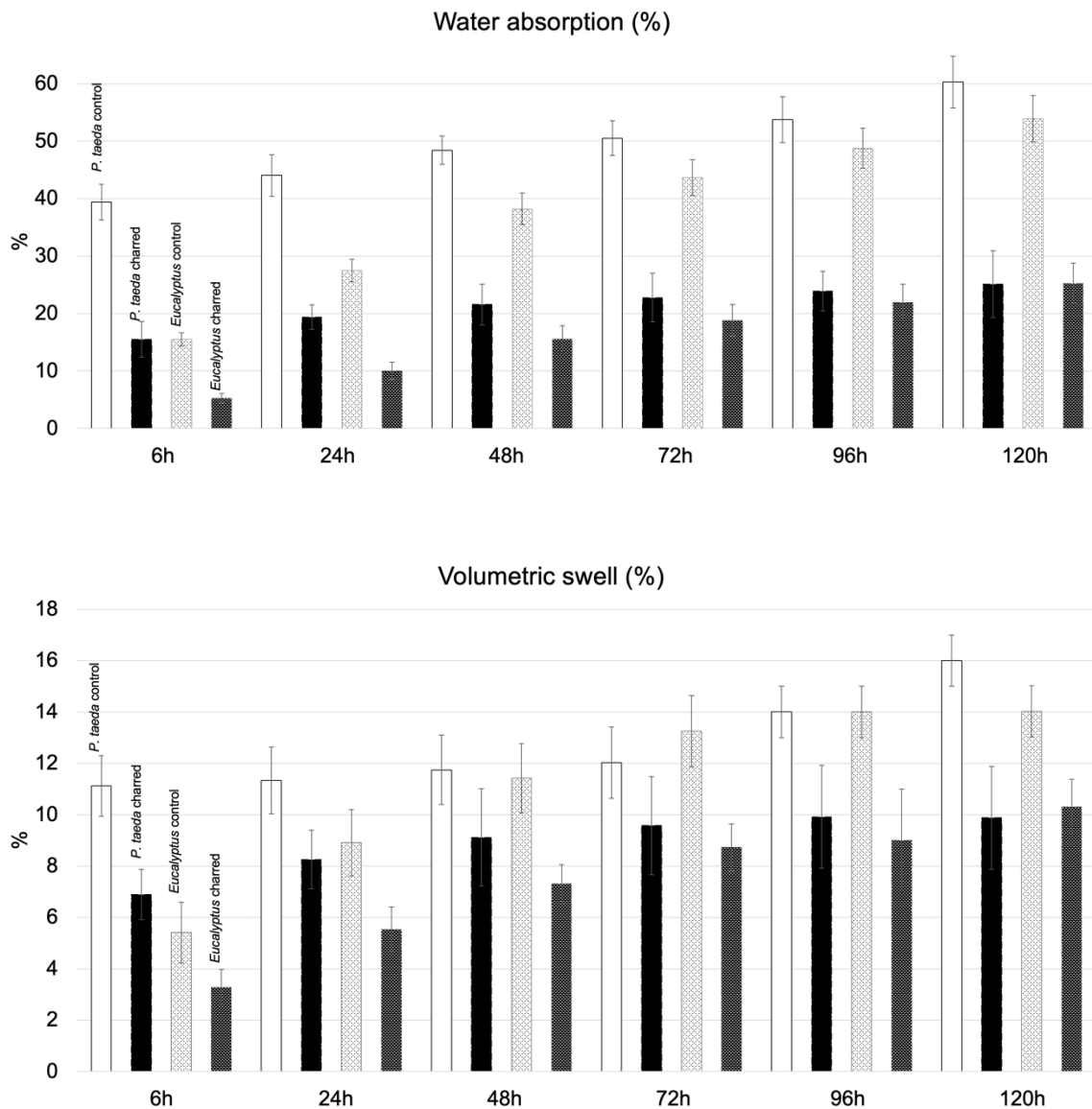


**Fig. 3.** Mean load-deformation curves for 3-point bending test (A); and compression strength parallel to grain (B) (red bold line: *E. bosistoana* charred; red dash line: *E. bosistoana* control; black round dot line: *P. taeda* control; black dash dot line: *P. taeda* charred)

Yilgor *et al.* (2001) showed that modifications in wood cell components play an important role in the strength of wood exposed to elevated temperatures. Other studies also found decreases in strength values in wood to be related to the removal of the hemicelluloses in modified wood (LeVan *et al.* 1990; Sweet 1995; Winandy 1995; Sweet and Winandy 1999). In a previous work by Soytürk *et al.* (2023), it was found that carbohydrate contents of charred *P. taeda* and *E. bosistoana* wood decreased by 31% and 30%, respectively, compared to un-charred specimens. This supports the idea that strength loss and carbohydrate content in charred wood material are connected. A one-sided charring procedure using the Yakisugi Method has been suggested as a possible alternative to thermal modification as a means for maintaining mechanical strength values of charred wood, (Kymäläinen *et al.* 2018; Morozovs *et al.* 2021).



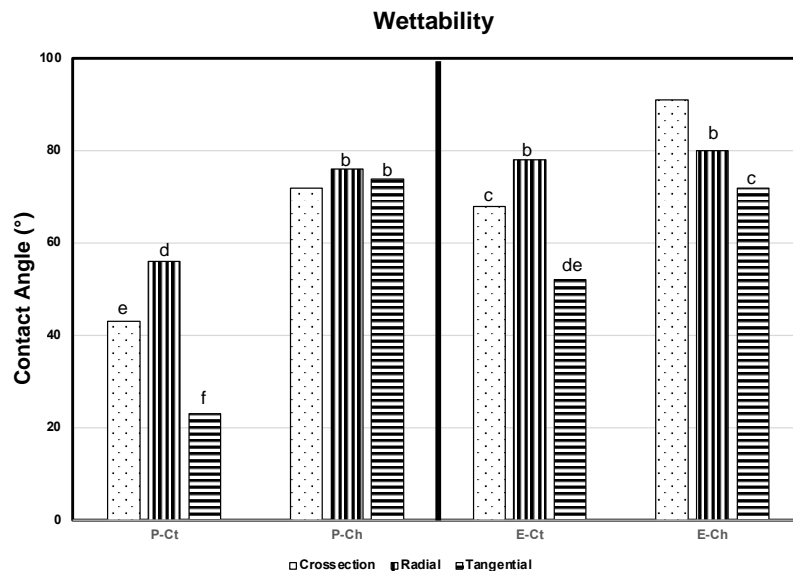
Average water absorption and volumetric swell curves of charred and un-charred specimens during 120 h of water immersion are given in Fig. 4. The water absorption of charred wood specimens was considerably reduced compared to un-charred wood specimens for both wood species. In the first five immersion durations (6, 24, 48, 72 and 96 h), both water absorption and volumetric swell rates for charred *E. bosistoana* specimens were much lower than those for charred *P. taeda* specimens; however, at the end of tests the values were almost equal in both wood species. Water absorption values in charred specimens showed a 58% and 53% decrease for *P. taeda* and *E. bosistoana*, respectively compared to un-charred specimens at the end of the test. The reductions in volumetric swell were lower in comparison to water absorption with volumetric swell decreased by 38% and 27% in charred *P. taeda* and *E. bosistoana* specimens, respectively.



**Fig. 4.** Water absorption and volumetric swell of charred and control specimens (n: 10)

Wettability test results (Fig. 5) were consistent with water absorption tests, showing much higher contact angle values in charred specimens. This indicated lower water absorption capability and improved hydrophobicity after charring and linseed oil

application. In a previous study by Soytürk *et al.* (2023), char formation followed by linseed oil application also appeared to contribute to decreased water absorption and wettability. This type of linseed oil application following wood charring method was used by Kymäläinen *et al.* (2022a) specifically as a means to increase hydrophobicity of the modified surfaces as well as to help prevent the carbon layer from cracking. Their results indicated that the decrease in water uptake was comparable to addition of oil on spruce reference specimens, measuring a reduction of up to 79%. A reduction in OH- groups on charred wood surfaces was found to be correlated with decreased absorption capacity of charred wood in a study by (Kymäläinen *et al.* 2018). Crosslinking in the carbohydrate-lignin matrix during thermal degradation was suggested by Tjeerdsma *et al.* (1998) and Wannapeera *et al.* (2011), whereas the migration and enrichment of extractives on the wood surface was considered another factor contributing to decreased wettability (Rautkari 2012). On the other hand, softening of lignin polymer in thermally modified wood might be effective in forming hydrophobic surfaces (Nuopponen *et al.* 2003; Kymäläinen *et al.* 2018). Results from a previous study by Soytürk *et al.* (2023) showed that total carbohydrates in charred *P. taeda* and *E. bosistoana* wood specimens decreased by approximately 30%. In addition, use of oil in our studies during charring processes might have also contributed to decreased wettability and better water absorption and volumetric swell properties along with chemical modifications and decreases in OH- content by thermal degradation in the hemicelluloses. The effect of charring processes on wood carbohydrates was also examined by Kymäläinen *et al.* (2022b); with results showing increased water resistance in charred spruce sapwood after one-sided charring. However, crack formation increased porosity in charred wood specimens at elevated temperatures of 400 °C which resulted in increased wettability (Kymäläinen *et al.* 2017).

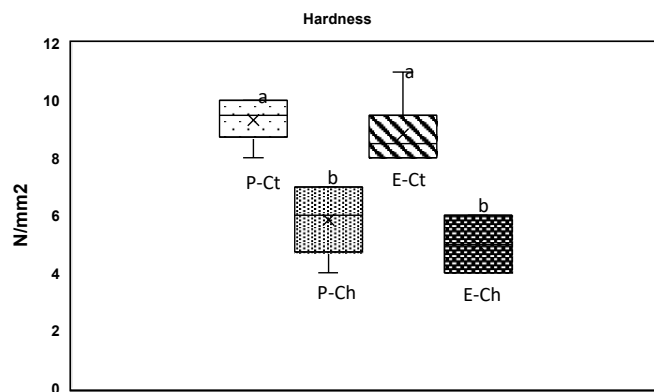


**Fig. 5.** Contact angle values of test specimens (P-Ct: *P. taeda* control; P-Ch: *P. taeda* charred; E-Ct: *E. bosistoana* control; E-Ch: *E. bosistoana* charred; the same letters in the bars indicate that there was no statistical difference between specimens according to Duncan's multiple range test ( $p \leq 0.05$ ); N: 3).

Čermák *et al.* (2019) found that considerable increases in soluble wood carbohydrates and phenolic compounds in one-side surface charred wood resulted in reduced water absorption due to decreased hydroxyl groups and cross-linking of lignin. Machová *et al.* (2021) also noted substantial changes in total carbohydrates after one-sided

charring of wood. Water absorption characteristics of charred wood is discussed by Kymäläinen *et al.* (2022a,b) and several mechanisms were proposed including, removal and conversion of carbohydrates (Gosselink *et al.* 2004), increased hydrophobicity, and alterations in the functional groups of wood surface (Gomez-Serrano *et al.* 1996; Labbe *et al.* 2006; MacBeath *et al.* 2011; Kymäläinen *et al.* 2015). Extractives condensing on wood surfaces due to evaporation during the charring process was also mentioned as contributing to the hydrophobicity of charred wood. Šeda *et al.* (2021) also found decreased water absorption tied to decreases in OH- groups in the celluloses and hemicelluloses and increases in cellulose crystallinity and cross-linking in lignin in one-sided charred beech wood.

Hardness tests results are given in Fig. 6. Charring resulted in decreased hardness in both wood species with reductions of nearly 38% and 43% for charred *P. taeda* and *E. bosistoana* specimens, respectively, compared to un-charred controls. Recent studies by Kymäläinen *et al.* (2022a,b) found that charring by contact or flame methods generally had adverse effects on surface properties because of degradation of the wood cell wall. On the other hand, the effects of heat-treatments rather than surface charring have been found to positively affect hardness of modified wood in various studies (Kocaefe *et al.* 2008; Korkut and Guller 2008; Dilik and Hiziroglu 2012; Won *et al.* 2012; Bakar *et al.* 2013; Salca and Hiziroglu 2014; Chotikhun *et al.* 2020). Other studies have shown no or slight changes in hardness of thermally-treated wood when compared to untreated controls (Boonstra *et al.* 2007; Leitch 2009). Akyildiz (2009) showed that increased temperatures and durations (230 °C, 2 and 8 h) decreased hardness values of wood when compared to the treatments of 130 and 180 °C for 2 h and 8 h. Hardness values have also been found to differ based on wood species. In one study, heat-treated cumaru wood had increased hardness, while hardness decreased in heat-treated peroba wood and bracinga wood was less affected by heat treatments (de Oliveira Araújo *et al.* 2016). The decreased hardness values in heat-treated wood have been correlated with deterioration of the cell wall structure (Salca and Hiziroglu 2014). Won *et al.* (2012) and Chotikhun *et al.* (2020) discussed that in addition to mechanical properties of heat-treated wood, hardness is also adversely affected by the degradation of hemicelluloses. Chotikhun *et al.* (2020) additionally state that changes in lignin structure may impact hardness of modified wood by heat (Kocaefe *et al.* 2010; Bakar *et al.* 2013).



**Fig. 6.** Hardness values of charred and control specimens (For explanation for box-and-whiskers plots, refer Fig. 1. P-Ct: *P. taeda* control; P-Ch: *P. taeda* charred; E-Ct: *E. bosistoana* control; E-Ch: *E. bosistoana* charred; the same letters in the bars indicate that there was no statistical difference between the specimens according to the Mann-Whitney U Test ( $p \leq 0.05$ ;  $N: 6$ ).

## CONCLUSIONS

1. The linseed oil application resulted in significant improvements in wood in terms of decreased water absorption and volumetric swelling properties compared to un-charred specimens.
2. The results showed significant differences based on wood species with higher decreases in charred *P. taeda* compared to charred *E. bosistoana*. In terms of strength properties, greater reductions were observed in MOR and compression strength in charred *P. taeda* specimens when compared to charred *E. bosistoana* specimens.
3. Decreases in hardness of charred specimens were smaller in *P. taeda* specimens.
4. Further studies are in progress to more comprehensively evaluate chemical and anatomical properties of charred wood after the charring process followed by linseed oil application as well as accelerated and natural weathering characteristics of charred wood material.

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## Author Contributions

All authors contributed to the conceptualization and methodology of the study. Material preparation was performed by CMI, data collection and analysis were performed by SNK, EES, CMI, FK, MSÖ, NÇ, SŞ, and ABT. The first draft of the manuscript was written by SNK and all authors commented on subsequent versions of the manuscript. All authors read and approved the final manuscript.

## Availability of Data and Material

Data used for this work are available from the corresponding author upon reasonable request.

## REFERENCES CITED

- Akyildiz, M. H., Ates, S., and Özdemir, H. (2009). "Technological and chemical properties of heat-treated Anatolian black pine wood," *African Journal of Biotechnology* 8 (11), 2565-2572. DOI:10.5897/AJB09.306
- Antons, A., Cirule, D., Verovkins, A., and Kuka, E. (2018). "Effect of thermal treatment on physical and mechanical properties of birch and pine wood," *Research For Rural Development*, 1, 78-85. DOI: 10.22616/rrd.24.2018.012
- Bakar, B. F. A., Hiziroglu, S., and Tahir, P. M. (2013). "Properties of some thermally modified wood species," *Materials & Design*, 43, 348-355. DOI: 10.1016/j.matdes.2012.06.054
- Bal, B. C. (2014). "Some physical and mechanical properties of thermally modified juvenile and mature black pine wood," *European Journal of Wood and Wood Products*, 72, 61-66. DOI: 10.1007/s00107-013-0753-9

- Boonstra, M. J., and Tjeerdsma, B. (2006). "Chemical analysis of heat treated softwoods", *Holz als Roh-und Werkstoff* 64, 204-211. DOI: 10.1007/s00107-005-0078-4
- Boonstra, M. J., Van Acker, J., Tjeerdsma, B. F., and Kegel, E. V. (2007). "Strength properties of thermally modified softwoods and its relation to polymeric structural wood constituents," *Annals of Forest Science* 64, 679-690. DOI: 10.1051/forest:2007048
- Buksans, E., Laiveniece, L., and Lubinskis, V. (2021). "Solid wood surface modification by charring and its impact on reaction to fire performance," in: *Proceedings of 20<sup>th</sup> International Scientific Conference - Engineering for Rural Development*. Jelgava, Latvia Vol. 20, pp. 1899-1905. DOI:10.5897/AJB09.306
- Candelier, K., Thevenon, M.-F., Petrissans, A., Dumarcay, S., Gerardin, P., and Petrissans, M. (2016). "Control of wood thermal treatment and its effects on decay resistance: a review," *Annals of Forest Science* 73, 571-583. DOI: 10.1007/s13595-016-0541-x
- 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, 9497-9506. DOI: 10.1007/s10853-019-03589-3
- Čermák, P., Vahtikari, K., Rautkari, L., Laine, K., Horáček, P., and Baar, J. (2016). "The effect of wetting cycles on moisture behaviour of thermally modified Scots pine (*Pinus sylvestris* L.) wood," *Journal of Materials Science* 51, 1504-1511. DOI: 10.1007/s10853-015-9471-5
- Chotikhun, A., Kittijaruwattana, J., Salca, E.-A., and Hiziroglu, S. (2020). "Selected physical and mechanical properties of microwave heat treated rubberwood (*Hevea brasiliensis*)," *Applied Sciences* 10(18), 6273. DOI: 10.3390/app10186273
- de Oliveira Araújo, S., Rocha Vital, B., Oliveira, B., Oliveira Carneiro, A. d. C., Lourenço, A., and Pereira, H. (2016). "Physical and mechanical properties of heat treated wood from *Aspidosperma populifolium*, *Dipteryx odorata* and *Mimosa scabrella*," *Maderas. Ciencia y Tecnología* 18(1), 143-156. DOI: 10.4067/S0718-221X2016005000015
- Dilik, T., and Hiziroglu, S. (2012). "Bonding strength of heat treated compressed Eastern redcedar wood," *Materials & Design*, 42, 317-320. DOI: 10.1016/j.matdes.2012.05.050
- Esteves, B., Şahin, S., Ayata, U., Domingos, I., Ferreira, J., and Gürleyen, L. (2021). "Effect of heat treatment on shore-D hardness of some wood species," *BioResources* 16(1), 1482-1495. DOI: 10.15376/biores.16.1.1482-1495
- Gennari, E., Picchio, R., and Lo Monaco, A. (2021). "Industrial heat treatment of wood: Study of induced effects on Ayous wood (*Triplochiton scleroxylon* K. Schum)," *Forests* 12(6), 730. DOI: 10.3390/f12060730
- Gomez-Serrano, V., Pastor-Villegas, J., Perez-Florindo, A., Duran-Valle, C., and Valenzuela-Calahorra, C. (1996). "FT-IR study of rockrose and of char and activated carbon," *J. Anal. Appl. Pyrol.* 36(1), 71-80. DOI: 10.1016/0165-2370(95)00921-3
- Gosselink, R., Krosse, A., Van der Putten, J., Van der Kolk, J., de Klerk-Engels, B., and Van Dam, J. (2004). "Wood preservation by low-temperature carbonisation," *Industrial Crops and Products* 19(1), 3-12. DOI: 10.1016/S0926-6690(03)00037-2
- Gunduz, G., Korkut, S., Aydemir, D., and Bekar, İ. (2009). "The density, compression strength and surface hardness of heat treated hornbeam (*Carpinus betulus* L.) wood," *Maderas. Ciencia y Tecnología* 11(1), 61-70. DOI: 10.4067/S0718-221X2009000100005

- Gündüz, G., Korkut, S., and Korkut, D. S. (2008). "The effects of heat treatment on physical and technological properties and surface roughness of Camiyanı Black Pine (*Pinus nigra* Arn. subsp. *pallasiana* var. *pallasiana*) wood," *Bioresource Technology* 99(7), 2275-2280. DOI: 10.1016/j.biortech.2007.05.015
- Hill, C., Altgen, M., and Rautkari, L. (2021). "Thermal modification of wood—A review: Chemical changes and hygroscopicity," *Journal of Materials Science* 56(11), 6581-6614. DOI: 10.1007/s10853-020-05722-z
- ISO 13061-3 (2014). "Physical and mechanical properties of wood -Test methods for small clear wood specimens - Part 3: Determination of ultimate strength in static bending," International Organization for Standardization (ISO) Geneva, Switzerland.
- ISO 13061-4 (2014). "Physical and mechanical properties of wood - Test methods for small clear wood specimens - Part 4: Determination of modulus of elasticity in static bending," International Organization for Standardization (ISO) Geneva, Switzerland.
- ISO 13061-2 (2014). "Physical and mechanical properties of wood — Test methods for small clear wood specimens — Part 2: Determination of density for physical and mechanical tests," International Organization for Standardization (ISO) Geneva, Switzerland.
- ISO 13061-1 (2014). "Physical and mechanical properties of wood — Test methods for small clear wood specimens — Part 1: Determination of moisture content for physical and mechanical tests," International Organization for Standardization (ISO) Geneva, Switzerland.
- ISO 13061-17 (2017). "Physical and mechanical properties of wood -Test methods for small clear wood specimens - Part 17: Determination of ultimate stress in compression parallel to grain," International Organization for Standardization (ISO) Geneva, Switzerland.
- ISO 1522 (2022). "Paints and varnishes - Pendulum damping test," International Organization for Standardization (ISO) Geneva, Switzerland.
- Jimenez, Jr, J. P., Acda, M. N., Razal, R. A., and Madamba, P. S. (2011). "Physico-Mechanical properties and durability of thermally modified malapapaya (*Polyscias nodosa* (Blume) Seem.) wood," *Philippine Journal of Science* 140(1), 13-23.
- Kapidani, A., Hajdini, D., and Beqo, S. (2019). "The effect of heating temperature on static hardness of beech (*Fagus sylvatica* L.) wood," *Int. J. Curr. Eng. Technol.* 9(3), 363-367. DOI: 10.14741/ijcet/v.9.3.3
- Kim, G.-H. (1998). "Effect of heat treatment on the decay resistance and the bending properties of radiata pine sapwood," *Material und Organismen*, 32(2), 101-108.
- Kocaefe, D., Poncsak, S., and Boluk, Y. (2008). "Effect of thermal treatment on the chemical composition and mechanical properties of birch and aspen," *BioResources* 3(2), 517-537. DOI: 10.15376/biores.3.2.517-537
- Kocaefe, D., Poncsak, S., Tang, J., and Bouazara, M. (2010). "Effect of heat treatment on the mechanical properties of North American jack pine: Thermogravimetric study," *J. of Materials Science* 45, 681-687. DOI: 10.1007/s10853-009-3985-7
- Korkut, D. S., and Guller, B. (2008). "The effects of heat treatment on physical properties and surface roughness of red-bud maple (*Acer trautvetteri* Medw.) wood," *Bioresource Technology* 99(8), 2846-2851. DOI: 10.1016/j.biortech.2007.06.043
- Korkut, S., Akgül, M., and Dündar, T. (2008). "The effects of heat treatment on some technological properties of Scots pine (*Pinus sylvestris* L.) wood," *Bioresource Technology* 99(6), 1861-1868. DOI: 10.1016/j.biortech.2007.03.038
- Kymäläinen, M., Rautkari, L., and Hill, C.A.S. (2015). "Sorption behaviour of torrefied wood and charcoal determined by dynamic vapour sorption," *J. Mater. Sci.* 50, 7673-

7680. DOI: 10.1007/s10853-015-9332-2
- 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., Turunen H., Čermák P., Hautamäki S., and Rautkari L. (2018). "Sorption-related characteristics of surface charred spruce wood," *Materials* 11(11), 2083-2098. DOI: 10.3390/ma11112083
- Kymäläinen, M., Dömény, J., and Rautkari, L. (2022a). "Moisture sorption of wood surfaces modified by one-sided carbonization as an alternative to traditional façade coatings," *Coatings* 12(9), article 1273. DOI: 10.3390/coatings12091273
- Kymäläinen, M., Belt, T., Seppäläinen, H., and Rautkari, L. (2022b). "Decay resistance of surface carbonized wood," *Materials* 15, 8410. DOI: 10.3390/ma15238410
- Labbé, N., Harper, D., Rials, T., and Elder, T. (2006). "Chemical structure of wood charcoal by infrared spectroscopy and multivariate analysis," *J. Agric. Food Chem.* 54(10), 3492-3497. DOI: 10.1021/jf053062n
- Leitch, M. A. (2009). "Hardness values for thermally treated black ash," *Wood and Fiber Science* 41(4), 440-446.
- LeVan, S., Ross, R. J., and Winandy, J. E. (1990). *Effects of Fire Retardant Chemicals on the Bending Properties of Wood at Elevated Temperatures* (Research Paper FPL-RP-498), U.S. Department of Agriculture, Forest Service, Forest Products Laboratory, Madison, WI, USA.
- Lunguleasa, A., and Spirchez, C. (2017). "Torrefaction of lamellar panels made of oak and spruce wood species," *Wood Research* 62(2), 319-328.
- MacBeath, A.V., Smernik, R.J., Schneider, M.P.W., Schmidt, M.W.I., and Plant, E.L. (2011). "Determination of the aromaticity and the degree of aromatic condensation of a thermosequence of wood charcoal using NMR," *Org. Geochem.* 42(10), 1194-1202. DOI: 10.1016/j.orggeochem.2011.08.008
- Machová, D., Oberle, A., Zářybnická, L., Dohnal, J., Šeda, V., Dömény, J., Vacenovská, V., Kloiber, M., Pěňčík, J., and Tippner, J. (2021). "Surface characteristics of one-sided charred beech wood," *Polymers* 13(10), 1551. DOI: 10.3390/polym13101551
- Metsä-Kortelainen, S., Antikainen, T., and Viitaniemi, P. (2006). "The water absorption of sapwood and heartwood of Scots pine and Norway spruce heat-treated at 170 °C, 190 °C, 210 °C and 230 °C," *Holz als Roh-und Werkstoff* 64(3), 192-197. DOI: 10.1007/s00107-005-0063-y
- Morozovs, A., Laiveniece, L., and Lubinskis, V. (2021). "Wood one-side surface charring of timber for claddings or recycled wood," in: *Proceedings of the 20th International Scientific Conference - Engineering for Rural Development Latvia*, Latvia University of Life Sciences and Technologies, 26-28 May, 2021, Jelgava, Latvia, 994-1002. DOI: 10.22616/ERDev.2021.20.TF221.
- Ninane, M., Pollet, C., Hébert, J., and Jourez, B. (2021). "Physical, mechanical, and decay resistance properties of heat-treated wood by Besson® process of three European hardwood species," *Biotechnologie, Agronomie, Société et Environnement* 25(2), 129-139. DOI: 10.25518/1780-4507.19050
- Nuopponen, M., Vuorinen, T., Jämsä, S., and Viitaniemi, P. (2003). "The effects of a heat treatment on the behaviour of extractives in softwood studied by FTIR spectroscopic methods," *Wood Science and Technology* 37, 109-115. DOI: 10.1007/s00226-003-0178-4

- Percin, O., Peker, H., and Atilgan, A. (2016). "The effect of heat treatment on the some physical and mechanical properties of beech (*Fagus orientalis* Lipsky) wood," *Wood Research* 61(3), 443-456.
- Pfriem, A., Zauer, M., and Wagenführ, A. (2009). "Alteration of the pore structure of spruce (*Picea abies* (L.) Karst.) and maple (*Acer pseudoplatanus* L.) due to thermal treatment as determined by helium pycnometry and mercury intrusion porosimetry," *Holzforschung*, 63(1), 94-98. DOI: 10.1515/HF.2009.027
- Rautkari, L. (2012). *Surface Modification of Solid Wood using Different Techniques*, Ph.D. Dissertation, Aalto University, Finland.
- Salca, E.-A., and Hiziroglu, S. (2014). "Evaluation of hardness and surface quality of different wood species as function of heat treatment," *Materials & Design*, 62, 416-423. DOI: 10.1016/j.matdes.2014.05.029
- Sandberg, D., and Kutnar, A. (2016). "Thermally modified timber: recent developments in Europe and North America," *Wood and Fiber Science* 48, 28-39.
- Šeda, V., Machová, D., Dohnal, J., Dömény, J., Zárýbnická, L., Oberle, A., Vacenovská, V., and Čermák, P. (2021). "Effect of one-sided surface charring of beech wood on density profile and surface wettability," *Applied Sciences* 11(9), article 4086. DOI: 10.3390/app11094086
- 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 and Eucalyptus woods modified by contact charring method," *Journal of Wood Materials Science and Engineering*. DOI: 10.1080/17480272.2023.2198993
- Sweet, M. S. (1995). *Determination of the Chemical Mechanism of Strength Loss in Fire-Retardant-Treated Wood*, Master's Thesis, University of Wisconsin-Madison, WI, USA.
- Sweet, M. S., and Winandy, J. E. (1999). "Influence of degree of polymerization of cellulose and hemicellulose on strength loss in fire-retardant-treated southern pine," *Holzforschung* 53(3), 311-317. DOI: 10.1515/HF.1999.051
- Taraborelli, C., Monteoliva, S., Keil, G., and Spavento, E. (2022). "Effect of heat treatment on hardness, density and color of *Populus × canadensis* 'I-214' wood," *Forest Systems* 31(3), article e023. DOI: 10.5424/fs/2022313-19558
- Tjeerdsma, B., Boonstra, M., Pizzi, A., Tekely, P., and Militz, H. (1998). "Characterisation of thermally modified wood: Molecular reasons for wood performance improvement," *Holz als Roh-und Werkstoff* 56, 149-153.
- Wang, J., and Cooper, P. (2005). "Effect of oil type, temperature and time on moisture properties of hot oil-treated wood," *Holz als Roh-und Werkstoff* 63, 417-422. DOI: 10.1007/s00107-005-0033-4
- Wang, X., Cheng, D., Huang, X., Song, L., Gu, W., Liang, X., Li, Y., and Xu, B. (2020). "Effect of high-temperature saturated steam treatment on the physical, chemical, and mechanical properties of moso bamboo," *Journal of Wood Science* 66, 52. DOI: 10.1186/s10086-020-01899-8
- Wannapeera, J., Fungtammasan, B., and Worasuwanarak, N. (2011). "Effects of temperature and holding time during torrefaction on the pyrolysis behaviors of woody biomass," *Journal of Analytical and Applied Pyrolysis* 92(1), 99-105. DOI: 10.1016/j.jaap.2011.04.010
- Winandy, J. E. (1995). *Effects of Fire Retardant Treatments after 18 Months of Exposure at 150°F (66°C)* (Research Note FPL-RN-0264), U.S. Department of Agriculture, Forest Service, Forest Products Laboratory, Madison, WI, USA.



Won, K.-R., Kim, T.-H., Hwang, K.-K., Chong, S.-H., Hong, N.-E., and Byeon, H.-S. (2012). "Effect of heat treatment on the bending strength and hardness of wood," *Journal of the Korean Wood Science and Technology* 40(5), 303-310. DOI: 10.5658/WOOD.2012.40.5.303

Yan, L., and Morrell, J. J. (2014). "Effects of thermal modification on physical and mechanical properties of Douglas-fir heartwood," *BioResources* 9(4), 7152-7161. DOI: 10.15376/biores.9.4.7152-7161

Yilgor, N., Unsal, O., and Kartal, S. N. (2001). "Physical, mechanical, and chemical properties of steamed beech wood," *Forest Products Journal* 51(11), 89-93.

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