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RESEARCH ARTICLE

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Surface carbonization of wood: comparison of the biological performance of *Pinus taeda* and *Eucalyptus bosistoana* woods modified by contact charring method

Ekim Elçin Soytürk [a], S. Nami Kartal [a], Rachel A. Arango [b], Katie M. Ohno [b], Edanur Solhan[c], İclal Çağlayan[c] and Claudia Marcela Ibanez [d]

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ABSTRACT

This study evaluated biological resistance, water absorption, and total carbohydrate contents of carbonized woods by contact charring method followed by linseed oil submersion. Charred and control specimens were exposed in laboratory fungal resistance tests to two white – and two brown-rot fungi and in mold resistance tests by spraying the surface with a mold spore-suspension comprised of three different mold species. Charred and control specimens were also evaluated for resistance to the eastern subterranean termites. To help understand biological durability results, carbohydrate content and water absorption of test specimens were also determined. In all cases, durability against wood degrading fungi increased with the exception of charred *Eucalyptus bosistoana* wood exposing to the white-rot fungus, *Irpex lacteus*. Mold resistance of charred *Pinus taeda* specimens increased considerably when compared to controls however, no mold growth was observed on either control or charred specimens of *E. bosistoana*. In termite tests, significantly less weight losses were shown in charred specimens of both wood species tested compared to controls although, no complete protection was achieved against termite attack. Total carbohydrate content of charred wood specimens decreased after the carbonization process and as expected, increased hydrophobicity was observed with >50% less water uptake in charred specimens.

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Durability; charred wood; fungi; termites; mold

Introduction

Wood modification methods are generally applied to increase durability, enhance physical and mechanical properties, and/or improve esthetic characteristics of timber, veneer or particles (Sandberg *et al*. 2017). As a result of increased environmental concerns over broad spectrum chemicals used in certain wood preservatives, there is growing market interest in these wood modification technologies and the potential for novel modification methods that can be used as alternatives to chemical wood preservation strategies (Gérardin 2016). Currently, wood modification technologies fall under the following categories: chemical modification; thermal-based processes including thermo-only, thermo-hydro, thermo-mechanical and thermo-hydro-mechanical treatments; electromagnetic, plasma and laser treatments; and biological treatments, biomimetics and mineralization, supercritical and ionic liquid treatments, graft polymerization (Sandberg *et al*. 2021).

One type of thermal-based modification involves the charring of wood surfaces by controlled burning procedures. Charring as a surface treatment has been in use in Japan for centuries, which is a traditional process called yakisugi (translated to mean "burned cedar") (Sandberg *et al*. 2021). Although wood charring is an ancient practice used to protect wood, it has gained increased popularity in recent years (Kymäläinen *et al*. 2020; Hasburgh *et al*. 2021; Zelinka *et al*. 2022; Kymäläinen *et al*. 2022a, 2022b). Kymäläinen *et al*. (2022a) stated that charred wood products have recently been introduced to the market mainly due to its mainly esthetically pleasant surface properties. As a result of this increased interest in charred materials, researchers have begun to focus more on defining the various properties of charred wood (Kymäläinen *et al*. 2017; White 2017; Kymäläinen *et al*. 2018; Čermák *et al*. 2019; Kymäläinen *et al*. 2020; Buksans *et al*. 2021; Ebner *et al*. 2021; Hasburgh *et al*. 2021; Machová *et al*. 2021; Morozovs *et al*. 2021; Šeda *et al*. 2021; Ebner *et al*. 2022; Kymäläinen *et al*. 2022a, 2022b, 2022c).

Compared to thermally modified wood, which has shown evidence of improved stability and resistance to wood decaying fungi and wood attacking insects (Sandberg *et al*. 2021), much remains unknown regarding durability and service life of surface-charred products. Scientific knowledge is particularly lacking for understanding differences in charred wood properties associated with various application methods and wood species (Hasburgh *et al*. 2021). The charring process is believed to improve durability by way of several proposed mechanisms, such as removal of surface carbohydrates, production of hydrophobic surfaces, and infusion of wood with smoke which may be toxic to biodeterioration agents. Hasburgh *et al*. (2021) indicates that charring is a thermochemical process involving incomplete combustion of solid matter resulting in removal

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of water vapor and organic components and formation of a predominantly carbon layer on the outer surface. Since char formation results in a surface layer that is mainly devoid of surface carbohydrates, in principle, charred wood is predicted to be resistant to fungi. However, Kymäläinen *et al*. (2022a, 2022b) state that few studies are available on the biological durability of carbonized wood and that published findings seem to be rather conflicting. Okamura *et al*. (2017) found increased durability against a white-rot fungus (*Pleurotus*) in carbonized sugi wood by the yakisugi method on agar plate tests. Sandberg *et al*. (2021) also indicate that the charring process makes the wood more durable against fungi and protects wood against insect attack.

Čermák *et al*. (2019) emphasized the importance of carbohydrates in biological performance of charred wood against fungal degradation and suggested that degradation of these compounds was closely tied to mass losses in charred wood samples. Machová *et al*. (2021) found charred wood to be more resistant to fungal attack than un-charred wood due to changes in chemical composition and carbohydrates after surface charring. Hasburgh *et al*. (2021) studied the fungal resistance of commercially available wood products that had been charred using the yakisugi process and found that the charred wood samples were not systematically protected from fungal decay. They concluded that the yakisugi process does not provide complete wood protection and that the effect of charring on biological resistance is likely to be highly influenced by wood species tested based on results showing limited protection for cypress wood but little or no protection in other wood species.

A recent review paper by Zelinka *et al*. (2022) discussed that although use of surface charring has been known for a long time, further studies are needed to determine optimal methods for producing charred wood. Variations in charring parameters such as different charring practices, wood species, temperatures, durations, and wood moisture content make it difficult to define exact charring treatment process guidelines. Natural durability of the wood material itself is another factor likely to be contributing to conflicting results in published literature regarding biological resistance of the charred wood.

The aim of this study was to evaluate the effectiveness of a surface charring process followed by linseed oil emersion in protecting two species of wood, *Pinus taeda* (L.) and *Eucalyptus bosistoana* (F. Muell.) from wood decay fungi, mold fungi and termites in laboratory tests. We examined charred wood specimens of two species and tested them with standard laboratory test methods for resistance to biodeterioration. To understand some of the factors that may be contributing to durability, total carbohydrates and water absorption of charred wood specimens were also determined. To support growing interest for development of a timber construction industry in Uruguay, the materials examined in this study consisted of two commercially available wood species from this region, *E. bosistoana* and *P. taeda*. These species show ranges in natural durability and research is currently being conducted to find methods and processes for increasing the useful service-life of materials fabricated from both species. Therefore, these species were selected to evaluate the potential for increased usability and competitiveness of *P. taeda* and *E. bosistoana* woods by providing a sustainable material with increased durability properties. The novelty of the current study is the utilization of *P. taeda* and *E. bosistoana* woods as low and high-durability wood species, which are not widely tested in this manner and the use of contact heating and linseed oil application methods to produce smooth charred surfaces, offering an advantage over the traditional methods that tend to result in rather brittle surfaces.

Materials and methods

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 selected for this study. Heartwood of *E. bosistoana* is reported as class 1, a very high-density wood as well as a highly durable species (Scheffer and Morrell 1998; Bootle 2005). Meanwhile, heartwood of *P. taeda* is reported as class 3–4 (moderately resistant to non-resistant) according to Scheffer and Morrell (1998). In this study, only sapwood portions only of the harvested trees were used since it is considered to be susceptible to deterioration, for which reason it must be protected. 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 depending on the test. For each, half the specimens were charred while the other half was used as controls.

Charring process

The 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, Mx) (range – 10 / 300°C) every 15 min. To prevent cracking, temperature was always kept below 150°C. Each set was comprised of ten specimens to be used in the further tests (decay, termite and mold resistance and water absorption tests). A 5 kg weight was placed on top of the specimens to ensure even contact throughout the entire surface and to avoid deformations. The conditions were chosen 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 surface.

Contact time with the hot surface was determined by controlling the char depth with a caliper with the aim of obtaining homogeneous specimens with a char layer no thicker than three mm, depending on specimen size. The average range of exposure for one face of a specimen was ten minutes. For full surface treatment, specimens were rotated to expose all six faces to the same conditions. Once charring was completed, the specimens were immediately dipped in linseed oil for five seconds to homogenize the char layer and prevent cracking.

Figure 1. Appearance of wood specimens before and after the charring process (P-Ct: *P. taeda* control; P-Ch: *P. taeda* charred; E-Ct: *E. bosistoana* control; E-Ch: *E. bosistoana* charred).

Excess oil and loose char were removed with a wire brush and then specimens were air dried for four days at room temperature. Differences in the appearance of charred vs control specimens for each wood species are shown in Figure 1.

Fungal resistance tests

Wood decay tests were performed according to the EN 113–2 standard test method (CEN 2020) with slight modifications. Fungal cultures used were originally obtained from the Forest Products Laboratory (FPL) (Madison, WI, USA) and sub-cultured at the Wood Microbiology and Protection Laboratory, Istanbul University-Cerrahpaşa (Istanbul, Turkey). For each specimen group, twelve wood specimens ($15 \times 25 \times 50$ mm (tangential x radial x longitudinal)) were subjected to white-rot fungi, *Trametes versicolor* (L.) Lloyd (1920) (MAD 697) and *Irpex lacteus* (Fr.) Fr. (1828) (MAD 517), and brown-rot fungi, *Coniophora puteana* (Schum. ex Fr.) P. Karst. (1868) (MAD 515) and *Tyromyces palustris* (Berk. & M.A. Curtis) Murrill. (TYP-6137) in Kolle flasks. After 12 (for *C. puteana* and *T. palustris*) and 16 (for *T. versicolor* and *I. lacteus*) weeks of incubation in a conditioning chamber at 27 ± 2°C and 70 ± 2% relative humidity (RH), mycelium was removed from the specimens by brushing with a soft toothbrush prior to drying at 60 ± 2°C until reaching a constant weight. Specimen weight losses were then calculated based on the initial constant weights. Weight loss values were then used to determine durability class assignments for each specimen test group according to EN 113–2 (CEN 2020) as shown in Table 1.

Mold resistance tests

Test specimens ($7 \times 20 \times 70$ mm (tangential x radial x longitudinal)) were evaluated for resistance to mold fungi with a slight

Table 1. Durability class (DC) assignments based on resistance to fungal attack by basidiomycetes fungi according to EN 113–2 (CEN 2020).

Durability class	Description	Weight loss (%)
DC 1	Very durable	≤5
DC 2	Durable	>5 to ≤ 10
DC 3	Moderately durable	>10 to ≤ 15
DC 4	Slightly durable	>15 to ≤ 30
DC 5	Not durable	>30

modification of the ASTM D4445-10 standard test method (ASTM 2019). Three mold fungi, *Trichoderma harzianum*, Rifai (1969) FS-19, *Aspergillus niger*, Tiegh. (1867) Wilcox 117 and *Penicillium fellutanum*, Biourge, 1923 FS-66, were sub-cultured from original strains obtained from the Forest Products Laboratory (FPL) (Madison, WI, USA). Mold cultures were maintained on 2% malt agar at 27 ± 2°C, and 80 ± 5% RH prior to use. A mixed spore-suspension of the three test fungi was prepared by washing the surface of individual two-week-old Petri plate cultures with 10–15 mL of sterile DI water. Washings were combined in a spray bottle and diluted to approximately 100 mL with DI water to yield approximately 3×10^7 spores m/L. The spray bottle was adjusted to deliver ∼1-mL inoculum per spray. Specimens were sprayed with the mixed mold spore-suspension and then incubated at 27 ± 2°C and 80 ± 5% RH for four weeks. Following incubation, specimens were visually rated on a scale of 0–5, with 0 indicating the specimen was completely free of mold growth and 5 signifying complete coverage with mold growth (0: no growth, 1: 20%, 2: 40%, 3: 60%, 4: 80%, and 5: 100% coverage with mold fungi).

Termite resistance tests

Specimens for testing ($20 \times 20 \times 10$ mm (tangential x radial x longitudinal)) were conditioned at 27 ± 2°C and 30 ± 5% RH to a constant weight which was recorded prior to termite exposure. No-choice termite tests were conducted on replicate specimens ($n = 5$) for each specimen group following ASTM D3345 standard test methods (ASTM 2022). Briefly, specimens were tested in polyethylene jars containing 150 g sifted sand and 20 mL sterile distilled water. Test specimens were placed directly on the sand surface and then 300 individual termites (eastern subterranean termite – *Reticulitermes flavipes* (Kollar)) were added to each jar. An additional set of untreated southern yellow pine (SYP) specimens was also included as a reference species ($25.4 \times 25.4 \times 6.4$ mm). Jars were maintained at 27 ± 2°C and 60 ± 5% RH in the dark for a four-week testing period. At the end of the testing period, the remaining termites per jar were counted, and the wood specimens were removed from jars, brushed free of debris, and then dried in a hood for one week prior to re-conditioning at 27 ± 2°C and 30 ± 5% RH for an additional week. Specimens were then re-weighed to determine the amount of weight loss from termite feeding. Percent termite survival and daily feeding rate was also calculated at the end of the four-week testing period. Daily feeding rate was calculated according to Eq. (1):

$$DFR = (W/N)/d \quad (1)$$

where;

DFR: Daily feeding rate (mg/per termite)
W: Weight of wood eaten (mg)
N: Number of live termites at the end of the test.
d: Duration of testing period (e.g. 28 (days))

Determination of total carbohydrates

Chemical analysis was done on three replicate blocks from each of the specimen test groups. Samples from charred specimens were prepared differently than controls in that outer portions of the blocks were analyzed separately. These specimens were prepared by sectioning off the outer portion of the block using a blade so that analyzes could be performed to determine differences between the charred surface layer versus the inner core. All samples for chemical analysis were prepared by grinding in a Wiley mill to pass size 20 mesh and approximately 200 mg was collected from each sample. Chemical analysis was run in duplicate and included evaluation of total carbohydrates based on the NREL procedure, "Determination of Structural Carbohydrates and Lignin in Biomass", NREL/TP-510-42618 (NREL 2012). The analytical procedure included the quantification of lignin (soluble and insoluble) and carbohydrates. Briefly, the procedure consisted of a two-stage acid hydrolysis followed by a filtration step to remove the insoluble fraction. The hydrolysates were then diluted and injected into an ion chromatography system (DIONEX ICS 3000) to quantify the soluble sugars.

Water absorption

Ten specimens from each treatment group were cut into dimensions of 15 × 25 × 50 mm (tangential x radial x longitudinal), oven-dried at 103 ± 2°C for three days and then weighed. To determine water absorption, specimens were placed in water and then re-weighed after 120 h. Before weighing, any excess water on the surfaces was blotted with paper tissue.

Water absorption (WA) was evaluated according to Eq. (2):

$$WA\ (\%) = (W2 - W1)/W1 \qquad (2)$$

where;

W1: oven-dried weight of wood specimen before immersion in water (g)

W2: weight of wood specimen after immersion in water for 120 h (g)

Data analysis

The test results are presented here as box-and-whisker plots depicting five characteristics of data distribution by position and length (Figures 2, 4, 7, 8 and 9). The box ranges from the first (Q1) to the third quartile (Q3) of the distribution, representing the interquartile range (IQR). The 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 × IQR and Q3 + 1.5 × IQR, respectively. The minimum and maximum values in the data set are used as end points for the whiskers. Each outlier outside the whiskers is represented by an individual circle. For all tests, data analysis for statistical significance was determined by Duncan's multiple range test ($p \le 0.05$) using the InerSTAT-a V1.3 program (Vargas 1999).

Results and discussion

Fungal decay and mold resistance tests

Weight losses in test specimens exposed in decay resistance tests and representative images of test specimens post decay testing are given in Figure 2 and Figure 3, respectively. Results showed statistically significant differences in weight losses of both charred *Pinus taeda* and *Eucalyptus bosistoana* specimens compared to their corresponding un-charred

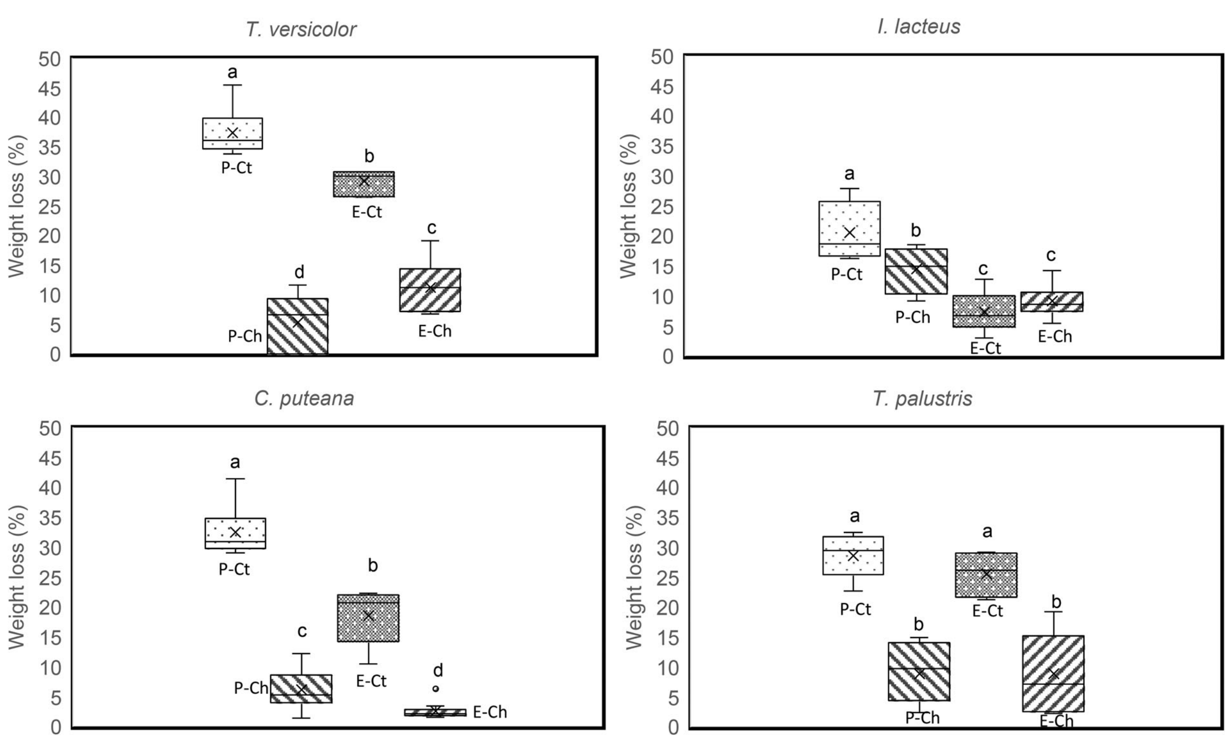

Figure 2. Weight losses in the test specimens in fungal decay resistance tests (P-Ct: *P. taeda* control; P-Ch: *P. taeda* charred; E-Ct: *E. bosistoana* control; E-Ch: *E. bosistoana* charred). Bars with the same letters indicate that there was no statistical difference between the specimens according to Duncan's multiple range test ($p \le 0.05$) (n: 12).

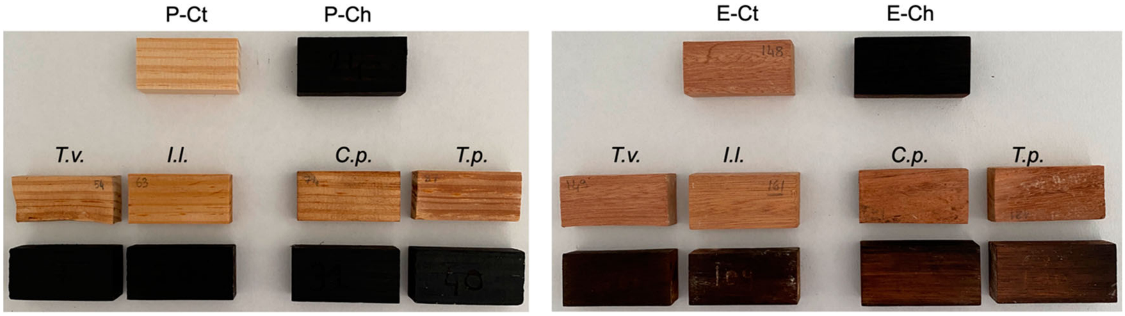

Figure 3. Representative test specimens post fungal decay resistance tests (P-Ct: *P. taeda* control; P-Ch: *P. taeda* charred; E-Ct: *E. bosistoana* control; E-Ch: *E. bosistoana* charred; *T.v.: T. versicolor, I.l.: I. lacteus, C.p.: C. puteana, T.p.: T. palustris*).

controls for all fungi except for *E. bosistoana* exposed to *I. lacteus*. Although, the weight loss caused by *I. lacteus* in *E. bosistoana* specimens was much lower overall compared to that caused by the other fungal species. For un-charred control specimens, all fungi caused significantly higher weight losses in *P. taeda* compared to *E. bosistoana* specimens except for *T. palustris* where weight loss of un-charred control *P. taeda* and *E. bosistoana* were similar. Overall, fungal decay resistance tests suggest that the charring process utilized in the present study did result in increased durability against fungal decay. However, the durability assignments based on the percent weight losses of charred specimens indicates that, in some instances, complete protection (i.e. classification of very durable) has not yet been reached. According to the definitions outlined in the EN113-2 (CEN 2020), all durability assignments improved with charring except for *E. bosistoana* exposed to *I. lacteus* (Table 2). The results of this study indicate charred *P. taeda* would be considered "durable" to *T. versicolor*, "durable" against both *C. puteana* and *T. palustris*, and only "moderately durable" against *I. lacteus*. Assignments for charred *E. bosistoana* would be "very durable" against *C. puteana*, "durable" against *I. lacteus* and *T. palustris*, and "moderately durable" against *T. versicolor*.

Visual ratings for specimens exposed in mold resistance tests are shown in Figure 4. For the duration of the test, no mold growth was seen on either un-charred controls or charred *E. bosistoana* specimens (Figure 4 and Figure 5). In *P. taeda* specimens, average mold growth rating was 0.7 for charred specimens while un-charred controls were completely covered by mold fungi at the end of the 4-week period (i.e. average rating of 5). In charred *P. taeda* specimens, mold growth was not observed until the beginning of the third week of the test, where mold growth on *P. taeda* controls was observed almost immediately on the second day after inoculation with complete coverage achieved by week three. These results suggest that the charring process considerably inhibited growth of mold fungi on *P. taeda* specimens. Conversely, mold resistance observed in *E. bosistoana* was most likely due to underlying natural durability properties of the wood material itself, regardless of treatment.

Thermal modifications are well-known methods that have the potential to improve wood durability by increasing biological resistance against decay fungi which may be useful for wood materials in above ground contact. The effect of thermal modifications on biological resistance of wood has been well studied and evaluations have included a wide range of wood species, process parameters, and varying test methods (Candelier *et al.* 2016; Presley *et al.* 2022). However,

Table 2. Durability class assignments of test specimens based on the EN 113–2 (CEN 2020).

Test specimens	*T. versicolor*	*I. lacteus*	*C. puteana*	*T. palustris*
P-Ct	Not durable	Slightly durable	Not durable	Slightly durable
P-Ch	Durable	Moderately durable	Durable	Durable
E-Ct	Slightly durable	Durable	Slightly durable	Slightly durable
E-Ch	Moderately durable	Durable	Very durable	Durable

P-Ct: *P. taeda* control; P-Ch: *P. taeda* charred; E-Ct: *E. bosistoana* control; E-Ch: *E. bosistoana* charred.

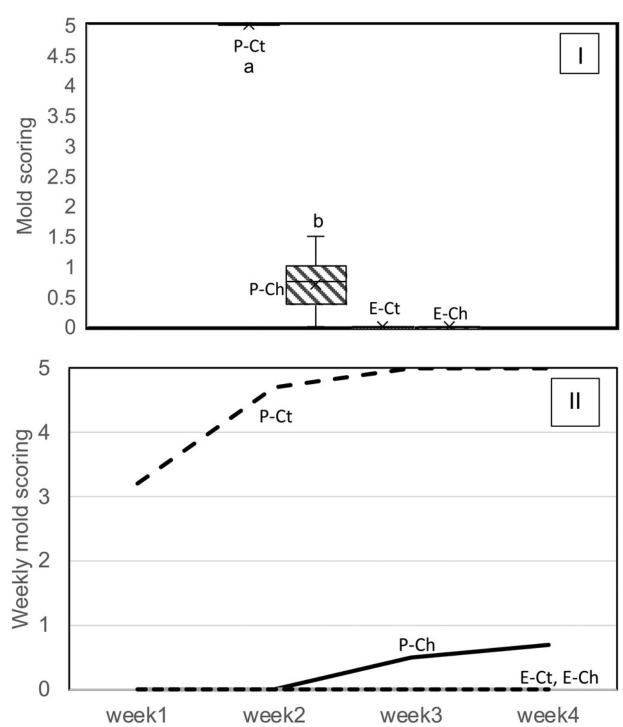

Figure 4. Visual rating score at week4 (I) and weekly rating scores (II) in mold resistance tests (P-Ct: *P. taeda* control; P-Ch: *P. taeda* charred; E-Ct: *E. bosistoana* control; E-Ch: *E. bosistoana* charred). Bars with the same letters indicate that there was no statistical difference between the specimens according to Duncan's multiple range test ($p \leq 0.05$) (*n*: 10).

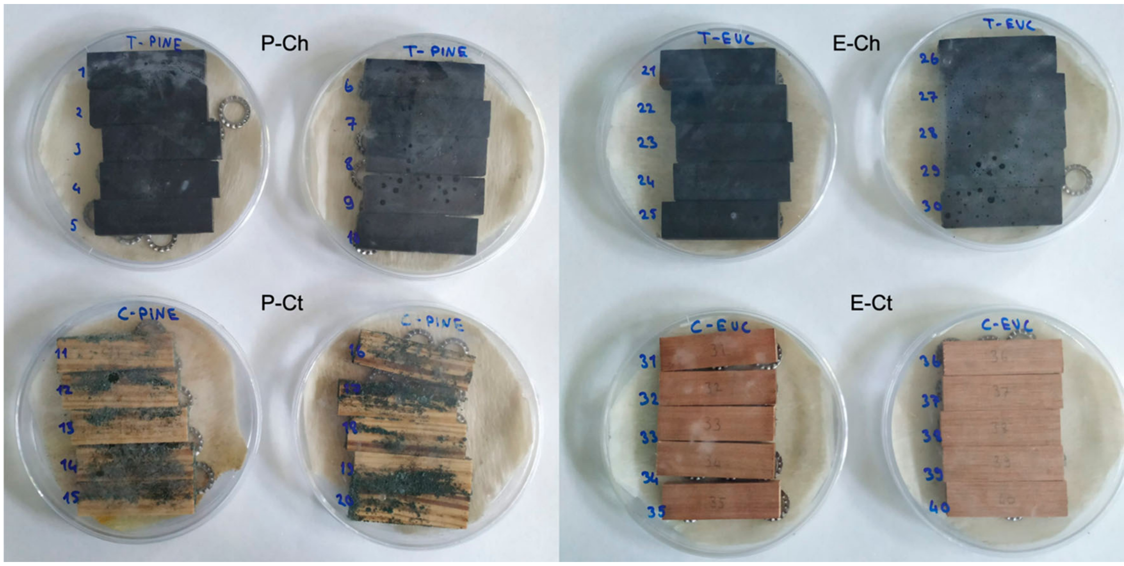

Figure 5. Test specimens post mold resistance tests (P-Ct: *P. taeda* control; P-Ch: *P. taeda* charred; E-Ct: *E. bosistoana* control; E-Ch: *E. bosistoana* charred).

Unsal *et al.* (2009) stated that despite improvements in fungal decay resistance of wood in above ground applications, thermally-modified wood is not recommended for outdoor exposure (Jämsä and Viitaniemi 1998; Tjeerdsma *et al.* 1998; Kamdem *et al.* 1999; Wienhaus 1999; Rapp *et al.* 2000; Kamdem *et al.* 2002; Tjeerdsma *et al.* 2002; Weiland and Guyonnet 2003; Doi *et al.* 2004; Welzbacher and Rapp 2005; Kartal 2006; Boonstra *et al.* 2007). Nevertheless, the possible reasons for increased durability in thermally-modified wood has be attributed to low water absorption properties (increased hydrophobicity), creation of harmful compounds to biological organisms, chemical modifications of wood cell components (i.e. degradation of carbohydrates, a main nutrition source for fungi) and the loss of hydroxyl groups affecting the ability of fungal enzymes to degrade the wood cells (Kamdem *et al.* 2000; Kamdem *et al.* 2002; Weiland and Guyonnet 2003; Hakkou *et al.* 2006; Hill 2006; Kartal 2006; Kartal *et al.* 2007; Del Menezzi *et al.* 2008; Kartal *et al.* 2008). While there is an increasing understanding of the effect of thermal treatment on wood properties, much less research has been conducted on biological resistance of charred wood and the factors that may be contributing towards durability. It has been suggested that the decay resistance of charred wood is increased more or less; however, the question as to whether the durability of wood after charring processes fulfills the requirements of various standards remains. Thus, more information is needed to understand the conditions under which charring processes results in protection against fungal decay (Hasburgh *et al.* 2021). Hill *et al.* (2022) hypothesized that charring leads to elevated durability by the removal of carbohydrates and formation of a hydrophobic surface layer. The release of volatile pyrolysis products resulting from the charring processes which move deeper into the charred wood is another proposed mechanism of protection against fungi. Regardless, it does seem clear that there is substantial variability in durability associated with wood species that still needs to be understood. Hasburgh *et al.* (2021) tested commercially available charred wood by the yakisugi methods from various wood species (i.e. black walnut, cypress, hemlock and white oak) and showed that the fungal resistance of charred wood materials did not systematically improve. They also stated that the charring process was just as likely to increase the amount of decay as it was to protect charred wood from fungal decay; however, some trends could be observed upon closer analysis of the weight loss data. In their study, no considerable changes in weight losses were observed associated with wood species except for cypress which did show some improvements with charring. Machová *et al.* (2021) studied decay resistance and total carbohydrate content of beech wood subjected to one-side surface charring method and their results revealed significant changes in total carbohydrate content and lower weight losses in fungal resistance tests compared to controls. Čermák *et al.* (2019) assert that the char layer is theoretically free of carbohydrates, thus causing resistance to wood decay fungi and that further wood protection may be achieved via pyrolysis under the char layer. The most recent study by Kymäläinen *et al.* (2022c) studied the biological resistance of one-sided charred wood by both contact and flame charring methods after sealing uncharred surfaces to evaluate the protective effect of the charred surface. They concluded that even though weight losses in charred wood were considerably lower than unmodified controls, no complete protection was achieved against decay and that, in general, the contact charring method was more effective in obtaining less weight loss (i.e. slowing the fungus down) compared to the flame charring method.

Termite resistance tests

Termites readily fed on all wood specimens regardless of treatment (i.e. control or charred) (Figure 6). Weight losses and termite daily feeding rates were higher in control specimens for both species, but termite survival was significantly higher in charred specimens compared to their associated controls for both *P. taeda* and *E. bosistoana* (Figure 7). This suggests that the termites preferred feeding on controls over charred

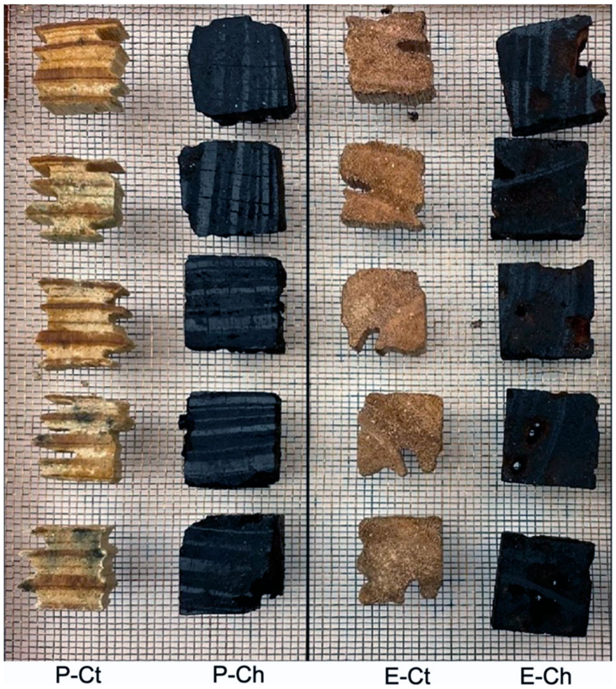

Figure 6. Test specimens post termite resistance tests (P-Ct: *P. taeda* control; P-Ch: *P. taeda* charred; E-Ct: *E. bosistoana* control; E-Ch: *E. bosistoana* charred).

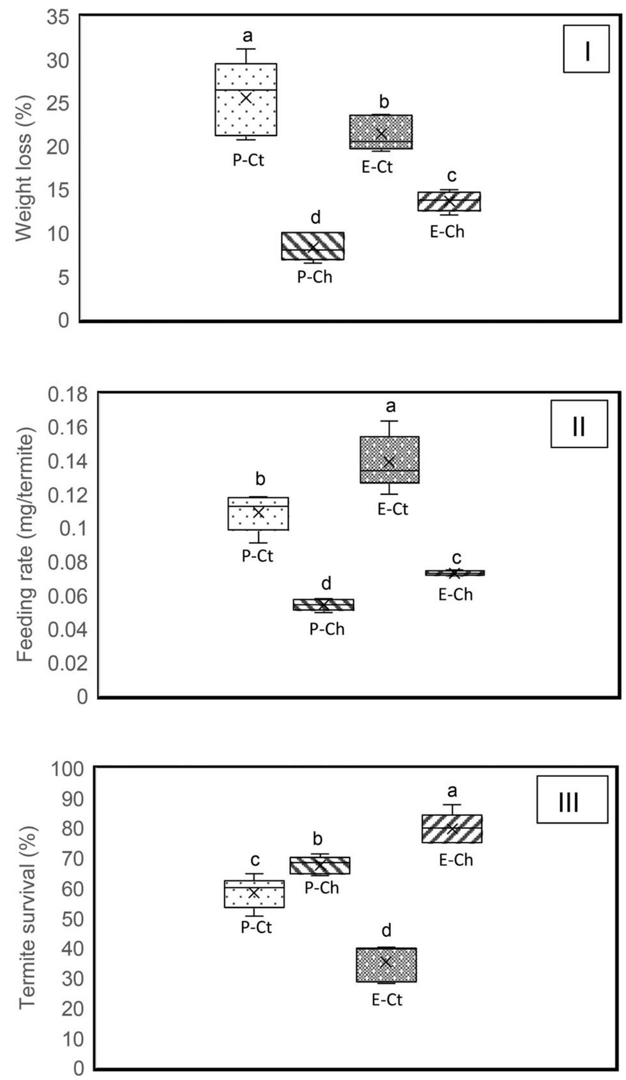

Figure 7. Weight losses of wood blocks (I), feeding rates (II) and termite survival rates (III) in termite resistance tests (P-Ct: *P. taeda* control; P-Ch: *P. taeda* charred; E-Ct: *E. bosistoana* control; E-Ch: *E. bosistoana* charred). Bars with the same letters indicate that there was no statistical difference between the specimens according to Duncan's multiple range test ($p \leq 0.05$) ($n = 5$). Southern yellow pine (SYP) reference controls: weight loss – 35.11% (± 2.27); feeding rate – 0.13 mg/termite (± 0.007); termite survival – 79.8% (± 5.16).

materials however, feeding on control specimens, particularly *E. bosistoana*, resulted in significantly higher mortality than their charred counterparts. In terms of overall termite resistance, the ASTM D3345 standard test method (ASTM 2022) does not specifically provide resistance classifications however, weight loss of test specimens may be used to determine the resistance class of wood (Hadi *et al*. 2014). The Indonesian standard, SNI 01-7207-2006 (BSN 2006), provides a reference for evaluating resistance class of wood exposed to subterranean termite attack with Class I corresponding to weight loss values less than 3.52% (very resistant), and class V corresponding to weight loss values greater than 18.94% (very poor resistance) (Table 3). The Japanese standard, JIS K 1571–2004 (JIS 2004), utilizes termite mortality for evaluating wood preference while the European standards, EN 117 (CEN 2012) and EN 118 (CEN 2013), require counting the number of live termite workers, soldiers, and nymphs at the end of the test as well as a visual examination of the test specimens. Termite feeding rates can also be used to determine how much wood each termite consumes per day, which are affected by the environmental conditions, wood availability, and other factors influencing termite survival (Hadi *et al*. 2014). Termite resistance class assignments based on SNI 01-7207-2006 (BSN 2006) for the different specimen groups evaluated are shown in Table 4. Based on these designations, charring did improve resistance class assignments however, none were determined as resistant and only charred *P. taeda* was moderately resistant. In general, termite feeding rates corresponded well with the weight losses of the test specimens, i.e. as the feeding rates decreased, the weight losses decreased accordingly. However, termite survival rates in the charred specimens from both species were significantly higher than their respective un-charred controls. In charred *E. bosistoana* specimens, termite survival rates were greater than two-fold higher when compared to controls.

Few published studies exist on the effect of the charring process on termite resistance. Peterson *et al*. (2008) evaluated pine sapwood boards charred by a propane torch and subjected to *R. flavipes* attack in both laboratory (47 days) and field tests. In their laboratory tests, termites attacked the charred wood boards more readily than the un-charred boards. Similar results were obtained by Bernklau *et al*. (2005)

Table 3. Termite resistance class assignments according to SNI 01-7207-2006 (BSN 2006).

Resistance class	Description	Weight loss of wood specimen (%)
1	Very resistant	<3.52
2	Resistant	3.52–7.50
3	Moderately resistant	7.30–10.96
4	Poorly resistant	10.96–18.94
5	Very poorly resistant	>18.94

Table 4. Termite resistance class assignments based on results from the current study.

Treatment group	Weight loss of wood specimen (%)	Resistance class assignment (following BSN 2006)
P-Ct	25.6 (4.3)	5 – Very poorly resistant
P-Ch	8.4 (1.6)	3 – Moderately resistant
E-Ct	21.4 (2.0)	5 – Very poorly resistant
E-Ch	13.7 (1.2)	4 – Poorly resistant
SYP	35.1 (2.3)	5 – Very poorly resistant

P-Ct: *P. taeda* control; *P*-Ch: *P. taeda* charred; E-Ct: *E. bosistoana* control; E-Ch: *E. bosistoana* charred.
Values in parentheses are standard deviations.

where charred stakes were more readily attacked than un-charred stakes. They propose thermal decomposition or volatile loss of extractive compounds in the charred wood as the possible reasons for increased attack by termites. Results presented here seem to differ from many of these previously described findings from the literature, which may be related to differences in the charring process itself. In this study, charring was performed as a two-step process in which specimens were first surface charred and then immediately submerged in linseed oil to homogenize the char layer. Other studies have already shown an effect of linseed oil treatment on feeding and mortality in several termite species (Temiz *et al*. 2013; Hassan *et al*. 2020). Hassan *et al*. (2020) demonstrated improved wood durability caused by a synergistic effect of linseed oil combined with wood extractives. Thus, the higher termite survival rates in charred specimen groups, paired with the lower weight loss values obtained from the current study, may suggest a repellent effect from either the charring process, oil treatment after charring, or a combination.

A study by Avitabile *et al*. (2015) discussed the factors that influence the distribution of termites and the effects of fire history on the occurrence of termite species. They stated that while forest fires may decrease food availability after woody material is incinerated, it may also elevate food availability if charred wood is as likely, or more readily, colonized than unburnt wood. Sufficient food is therefore likely to remain post-fire to support termite colonies that survive at a site, either above ground as burnt or charred wood or below ground as lignotubers, roots and stems. In a study by Peterson *et al*. (2008), they found that force-feeding char to termites significantly decreased protozoa populations in the termite gut which was distinctive to that of starvation. They suggest that termites may obtain some beneficial nutrients from char, as demonstrated by partial survival of gut protozoa in comparison with starved termites, but that charred wood cannot be considered as a food source alone.

Termite tests of heat-treated wood materials have generally shown increased susceptibility to termites, although many of these do not include charring. In a study of heat-treated sapwood (*Cryptomeria japonica* D. Don) (180°C for 2 and 4 h), Kartal (2006) found heat treatments increased the susceptibility of wood to termites showing more weight loss compared to controls. Another study by Unsal *et al*. (2009) subjected to thermally compressed wood panels (120 and 150°C, 60 min) to *R. flavipes* attack in laboratory conditions and showed lower weight losses in thermally compressed wood specimens when compared to control specimens. Doi *et al*. (1999) evaluated the effects of steam or dry-heat treatments of the feeding behavior of two subterranean termites, *Coptotermes formosanus* Shiraki and *Reticulitermes speratus* Kolbe. Their results showed that some steam-treated wood specimens were profoundly attacked by termites, while the weight losses in both dry-heated wood and control specimens were almost similar except for the Japanese larch-*R. speratus* combination where the weight loss in heat-treated specimens was considerably higher than that in controls. Doi *et al*. (2004; 2005) have also indicated that there is no increased resistance of heat-treated wood against termites. Similarly, in a more recent study by Surini *et al*. (2012) thermally modified wood at 160, 180, 230 and 260 °C did not show improved durability against termites. A general tendency towards greater mortality had been observed with elevated temperature likely due to the formation of toxic elements generated during heat treatment and thus, the mortality rates were considered as a positive effect of thermal treatment on wood protection.

Total carbohydrates and water absorption

Results from total carbohydrate analyses showed clear differences for both wood species associated with charring when compared to control specimens (Figure 8). Specifically, charring resulted in a significant decrease in carbohydrates for both *P. taeda* and *E. bosistoana* specimens compared to their un-charred counterparts. Results for water absorption rates showed values of charred *P. taeda* specimens decreased by 58% compared to controls after 120-h-immersion in water (Figure 9). In *E. bosistoana* specimens, charring resulted in 53% less water uptake than control specimens.

Kymäläinen *et al*. (2022d) showed that one-sided wood charring by contact and flame methods resulted in decreases in carbohydrate content and that the contact charred wood was nearly totally depleted of the hemicelluloses, while the flame charred wood surfaces were almost completely free of all carbohydrates. Kymäläinen *et al*. (2017) evaluated contact angle and water uptake properties of charred spruce and pine wood by one-sided method using a hot plate and their results showed elevated water resistance of charred spruce sapwood and increased water absorption of pine sapwood while the contact angles of both wood species increased compared to un-charred wood. However, they stated that the

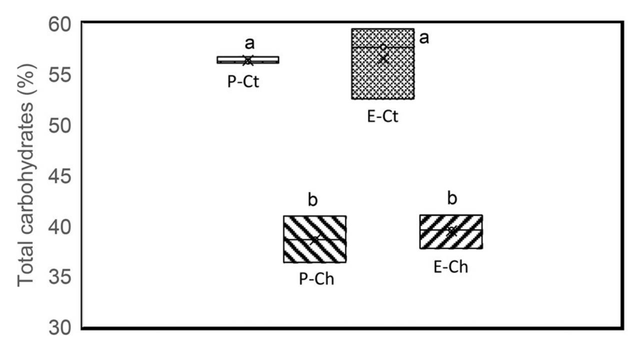

Figure 8. Total carbohydrate content in tests specimens (P-Ct: *P. taeda* control; P-Ch: *P. taeda* charred; E-Ct: *E. bosistoana* control; E-Ch: *E. bosistoana* charred). Bars with the same letters indicate that there was no statistical difference between the specimens according to Duncan's multiple range test ($p \leq 0.05$). (*n*: 3).

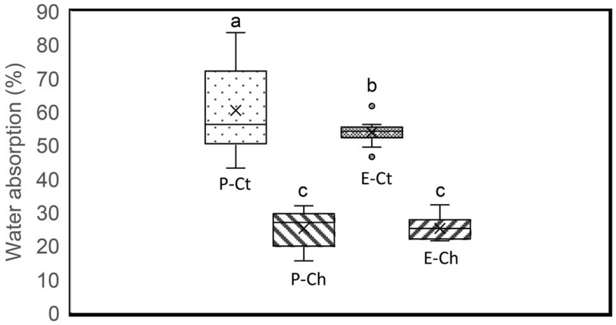

Figure 9. Water absorption of test specimens immersed in water for 120 h (P-Ct: *P. taeda* control; P-Ch: *P. taeda* charred; E-Ct: *E. bosistoana* control; E-Ch: *E. bosistoana* charred). Bars with the same letters indicate that there was no statistical difference between the specimens according to Duncan's multiple range test ($p \leq 0.05$). (*n*: 10)

elevated temperature used (400°C) for charring, caused surface cracking which increased porosity, and diminished contact angles of both species. In our study, oil application following the charring process was used to prevent cracking or splitting from forming on charred wood surfaces due to shrinkage and loss of moisture, and thus we show much better water absorption rates in charred specimens. In a more recent study by Kymäläinen *et al*. (2018), one-sided charred spruce specimens were studied for their wettability, dynamic vapor sorption and surface chemistry by FT-IR and the reduction of OH groups on charred wood surfaces were correlated with decreased sorption capacity of charred wood. While Tjeerdsma *et al*. (1998) and Wannapeera *et al*. (2011) suggest cross-linking in the carbohydrate-lignin matrix during thermal degradation, Rautkari *et al*. (2010) indicates the migration and enrichment of extractives on the wood surface as another factor affecting water absorption. The softening of lignin in the thermal degradation process may have the potential to form hydrophobic surface in thermally modified wood (Nuopponen *et al*. 2003; Kymäläinen *et al*. 2018). In the present study, total carbohydrates of charred wood specimens decreased by about 30% in both *P. taeda* and *E. bosistoana* specimens after charring. Along with use of oil, chemical modifications and decreases in OH content by thermal degradation in the hemicelluloses contributed to the decreased water absorption in the study. Čermák *et al*. (2019) found that reductions in equilibrium moisture content levels due to decreases in hydroxyl groups and cross-linking of lignin were observed on one-sided surface charred wood specimens. In addition, the increases in soluble carbohydrates and phenolic compounds significantly diminished the water absorption properties of the charred wood specimens. Machová *et al*. (2021) also determined that total carbohydrates showed significant alterations in wood after one-sided charring. Kymäläinen *et al*. (2022c) suggested that even though water behavior in charred wood was similar to un-charred wood, affinity of charred wood to water is decreased because of removal and conversion of carbohydrates (Gosselink *et al*. 2004), and elevated hydrophobicity and modifications in surface functional groups (Gomez-Serrano *et al*. 1996; Labbé *et al*. 2006; MacBeath *et al*. 2011; Kymäläinen *et al*. 2015). They also suggest that extractives evaporating during charring may condense on the wood surface and further support hydrophobicity resulting in plasticization of wood surfaces. Šeda *et al*. (2021) studied wettability and water absorption of one-sided charred beech wood. Their studies revealed that the highest contact angle values were obtained at the lowest temperature conditions (200°C) and as the temperature increased up to 400°C, the contact angle values decreased. On the other hand, the water absorption of the radial specimens submerged in water for 72 h was lower than the un-charred controls suggesting that the decreased water absorption is a result of decreases in the number of OH groups in the celluloses and hemicelluloses. In addition to this phenomenon, increases in cellulose crystallinity and cross-linking in lignin are considered other factors affecting hydrophobicity of charred wood.

Conclusion

The carbonization of *P. taeda* and *E. bosistoana* woods with surface charring using a hot plate was investigated to evaluate biological performance against fungi and termites along with changes in moisture and total carbohydrate contents. No surface cracks or splits were seen on the charred wood surfaces due to even treatment and oil application in the carbonization process. Charred specimens of both species also demonstrated decreased absorption during 120-h-immersion in water. Chemical analysis showed a significant decrease in total carbohydrate contents after charring due to thermal degradation of wood compounds. Significant improvements were achieved in fungal decay resistance when the charred specimens were subjected to *T. versicolor*, *C. puteana* and *T. palustris*. Charred *P. taeda* specimens showed increased mold resistance; however, no mold growth was seen on *E. bosistoana* specimens in either un-charred or charred specimens. Surface carbonization also results in increased durability against the subterranean termites although wood material is still highly susceptible to termite feeding. Underlying changes in water absorption and total carbohydrate content likely help explain enhanced biological performance of charred wood specimens. This correlation indicating chemical modifications and degradations in wood structure might be the most reasonable theory to explain the improvements in biological performance. It is possible that since charring theoretically removed/changed surface carbohydrates, the fungi are in a state of starvation while attacking, which wouldn't necessarily prevent deterioration, but would likely halt decay progression resulting in considerably less mass loss in charred samples compared to the controls as seen in this study. The degradation of wood carbohydrates of charred wood is generally considered as an important reason for decreased water uptake due to decreased hydrophilic OH groups and for increased resistance against fungi since such compounds are important nutritious source for the establishment and progression of wood decay and mold fungi. The study states that a proper regulation of temperature and time is needed for surface charring process. Suitable charring treatment is a capable of resulting in more carbohydrates affected, facilitating lower water sorption; however, extremely elevated temperatures and durations might result in surface cracks further leading to water uptake through the checks. In this study, oil application along with surface charring provided more even charring without crack formation on the wood surfaces. Results suggest that all-surface-charring process followed in this study might be another factor in increased biological performance when compared to most previous studies where one-side charred wood specimens have been employed. Further studies are in progress to evaluate more detailed chemical characteristics and anatomical properties of charred wood; physical and mechanical changes after carbonization process and accelerated and natural weathering characteristics of charred wood surfaces.

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Instituto Superior de Estudios Forestales, Sede Tacuarembó Cenur Noreste, Universidad de la República, Uruguay: 31° 44′11.8"S 55°58′37.1"W.

USDA Forest Service Forest Products Laboratory, Madison, WI, USA: 43°04′29.0"N 89°25′39.0"W.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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Data availability statement

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

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