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Improving durability of cross laminated timber (CLT) with borate treatment

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Abstract

Borate solution was used to treat two sets of Douglas-fir wood samples, one by spraying cross-laminated timbers (CLT) and another set by dip-treating wood in solutions at different retentions. A novel model was developed to explain and predict borate uptake based on dip-treatment parameters. Small-scale CLT samples were prepared using commercial emulsion polymer isocyanate (EPI) and polyurethane (PU) adhesive with dip-treated wood. The effect of adhesive and borate retention on CLT samples were evaluated through adhesion, fire, termite, and decay tests. The adhesion strength of wood was statistically unaffected by borate treatment. Statistical analysis showed that both spray- and dip-treated samples had significantly higher termite and decay resistance and fire performance than the untreated boards. Untreated CLT samples bonded with PU showed a considerably higher inherent decay and termite resistance than untreated specimens bonded with EPI adhesive.

Keywords: Mass-timber, Borate, Durability, Flammability, Termite resistance, Decay resistance, Spray treatment

Introduction

Cross-laminated timber (CLT) is a modern engineered wood product gaining popularity in residential and non-residential applications due to its high load-bearing behavior, dimensional stability, strength to weight ratio, and significantly shorter construction time [1]. In addition, stored carbon in the wood components of CLT buildings contributes to carbon sequestration, and CLT has been shown to reduce the global warming potential compared to other building materials [2]. However, wood is susceptible to biodegradation, and prolonged exposure to moisture increases the potential for structural damage caused by decay and termite attacks on wood components, especially in humid conditions, both during and after construction [1]. Borates, which are known to have low mammalian toxicity, as well as fungicidal, insecticidal, and increased flame retardancy properties, may be useful in protecting CLT from biological

degradation [1]. In addition, wood is a combustible material, and the structure may become involved in the event of fire [3]. Compared with copper-based preservatives, borates are colorless, odorless, low cost, and protect the wood against a variety of wood-destroying organisms if used indoors that are not exposed to direct rain to leach out from the wood [4]. In an outdoor field study, where wood was sheltered from rain to prevent excessive leaching, inorganic borates with target retention of 2–3% equivalent to boric acid (11 kg/m³) were also shown to increase the average service life of wood for up to 10 years in above-ground applications [5]. Morris et al. [6] also reported that borate-treated lumber showed no or very small traces of subterranean termite attack after 10 years of exposure in above-ground conditions in Canada, Hawaii, and Japan. Borate-based treatments are a commonly used fire retardant and have been shown to decrease the flame spread and mass loss when exposed to a flame [4].

Disodium octaborate tetrahydrate (DOT) is one of the most common inorganic borate sources used in the wood preservation industry due to its high solubility in water and simple treatment process compared to other

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borate derivatives, such as borax and boric acid. DOT's high mobility and water solubility allow the use of more concentrated solutions, resulting in higher uptake into the wood, even at low moisture content. These advantages make DOT a good choice for hard-to-treat, high-density woods, such as Douglas-fir [7]. Although borates' high water solubility and diffusion properties are considered advantages for efficient treatment, they also increase the risk of leaching preservative chemicals from the treated wood [8]. Since DOT is not chemically fixed in the wood, borate-treated wood components are susceptible to leaching when exposed to high humidity environments or liquid water [5, 9]. Nevertheless, many studies conducted in Japan and Hawaii showed that inorganic borate preservatives are effective enough for structural wood products, as long as the surface of the wood is not repeatedly exposed to wetting and does not remain wet for extended periods [5, 10].

While imparting some desirable characteristics, some studies reported that borate treatment negatively affects the surface properties of wood, resulting in poor adhesion [9, 11–13]. The rough surface could potentially expedite panel delamination, decreasing mechanical strength. Therefore, sanding off the well-treated and preservative-rich surface of the wood is recommended before gluing borate-treated wood materials [1]. Compared to other glue-laminated beams, it is difficult to treat CLT panels, because the assembled panels are often too big to fit into standard pressure-treatment cylinders. In addition, pressure-treatment of glue-laminated members (*i.e.*, after gluing) is typically done with oil-based preservatives to avoid damage from the swell/shrink that can result from waterborne treatments. Stirling et al. [11] investigated the effect of borate dip-treatment on planed hem-fir CLT samples joined with polyurethane (PU) adhesive. Their results showed that the measured penetration thickness was less than 125 mm, which does not meet the required 10 mm penetration for borate-treated hem-fir. They also pressure-treated Douglas-fir samples without planning after treatment using three types of adhesives to make CLT samples and reported that only the one-component polyurethane (PU) adhesive met the shear strength criteria on both untreated and treated samples, while an emulsion polymer isocyanate (EPI) adhesive worked only on untreated samples and melamine-formaldehyde did not work on either treated or untreated wood samples [12].

Due to possible issues regarding over or under-treating CLT (*e.g.*, loss of mechanical strength, surface roughness, limited fungicidal and insecticidal activity), it is critical to optimize treatment parameters. By modeling the diffusion process of the borate solution into the wood, it will be possible to more accurately predict treatment efficacy

based on solution concentration and size of the wood samples. It should be noted that understanding the diffusion mechanism of borate solution during the treatment will also help to predict its leaching mechanics. The penetration and leaching mechanics of borates in wood have been studied by numerous researchers [14–17]. However, modeling and precise mathematical formulas are missing in the literature. Koumbi-Mounanga et al. [8] modeled the effect of long-term borate concentration gradients in brush-treated black spruce and the effect of glue-line on the diffusion of borate in CLT. At the beginning of their experiment, the glue line hindered borate penetration, but eventually, borate was able to penetrate through the glue line because of its high mobility. Waldron et al. [14] also developed a model for predicting borate leaching compared with chemically fixed preservatives. They showed that, in contrast to copper-based preservatives that chemically bond into the wood, borate continues to leach out at a decreasing rate while in contact with water [14].

In this paper, the effect of borate treatment on the adhesion strength of two commercially available adhesives was studied. Efficacy of borate treatment, both dip- and spray-treated, in improving decay, termite, and fire performance of CLT samples were also investigated. Dip-treatments were done at two different retention levels of 2.7 and 4.5 kg/m³ (0.17 and 0.28 pcf, respectively) as recommended by American Wood Protection Association (AWPA) [18] for above-ground applications in highly susceptible subterranean termite regions. The spray treatment was used to evaluate the possibility of spraying borate solution over CLT panels before construction or on-site after installation. In addition, a novel model was developed using MATLAB software to optimize the treatment parameters and evaluate the impact of the different treatment factors.

Materials and methods

Wood properties

Forty pieces of flat-sawn Douglas fir boards typical of those used in CLT construction (2–3 average annual rings per cm) with dimensions of 610 mm × 610 mm × 100 mm, and also 3-Ply CLT samples made with the same lumber were purchased from SmartLam (Columbia Falls, MT, USA). Boards were conditioned at 20 °C and 50% relative humidity (RH) for 4 weeks to reach equilibrium with average moisture content (MC) of 9% before borate treatment.

An additional 20 commercial flat-sawn Douglas-fir borate-treated boards (1–2 average annual rings per cm) with dimensions of 1830 mm × 150 mm × 50 mm (longitudinal × tangential × radial) were kindly supplied by Matheus Lumber Company Inc. (Woodinville, WA,

USA). DOT ($\text{Na}_2\text{B}_8\text{O}_{13} \cdot 4\text{H}_2\text{O}$ 98%) was provided by the American Borate Company (Virginia Beach, VA, USA), which was used for all borate treatments.

The average density of the untreated boards was measured by the water immersion method as specified in ASTM D2395 (Method B) [19]. This method utilized 20 mm Douglas-fir wood cubes obtained from the purchased boards. Their initial oven-dry mass was obtained, and then the wood cubes were soaked in water to reach equilibrium, ensuring MC was above fiber saturation point ($\text{MC} = 36 \pm 3\%$). After 72 h, the wood samples were wiped with a paper towel before measuring the weight. A 250 mL beaker was filled with 200 mL of distilled water and placed on a balance tared to zero. Using a sharp long needle, a wood sample was completely submerged in the water without touching the container. After reaching equilibrium (approximately 30 s), the mass of the displaced water was recorded. The density of the greenwood sample (based on the volume of displaced water) was calculated by dividing the mass of oven-dry wood by green volume.

The moisture content measurement was done using the oven-dry procedure described in ASTM D4442 [20] with slight modification. Briefly, 20 mm Douglas-fir cubes obtained from the purchased boards were placed in an oven at 105°C for 5 h when the samples reached constant weight (*i.e.*, the weight measurement in an interval was equal to or less than twice the balance sensitivity of 10 mg). For further confirmation, the samples remained in the oven for an additional 3 h, and the mass was unchanged. The samples were removed from the oven and placed in a desiccator to cool down to room temperature before recording their final oven-dry mass. The percent moisture content was calculated using the following equation:

$$\text{Moisture Content (\%)} = \frac{\text{Initial mass} - \text{oven dried mass}}{\text{oven dried mass}} \times 100. \quad (1)$$

Measurements for density and moisture content were done in ten replicates.

The boards were kept in the same condition as the CLT boards to reach the moisture content of about 9% before

testing. It is important to note that the moisture content of wood samples for achieving the strongest bond was recommended to be between 6 and 16% by the adhesive manufacturers.

CLT samples

The CLT samples using two commercial adhesives were prepared to meet the different requirements of the fire, termite, and decay standard test methods. The dimensions of test specimens are provided in Table 1. Additional CLT samples with the same dimensions were made from untreated and commercially treated boards. Figure 1 gives details regarding the CLT samples used in decay, termite, and fire performance tests.

Adhesives

To investigate the effect of different adhesives on the bond performance of treated and untreated samples, two commercial cold-curing adhesives, one-component PU and EPI, were provided by adhesive producers. The viscosity of the adhesives was measured using a Brookfield DV2T viscometer at a shear rate of 1000 s^{-1} in three replicates. The solid content was measured by adding approximately 1 g of adhesive to an aluminum pan and placing it in an oven at 105°C . The samples were kept in the oven for 1 h, and then the mass was measured every 10 min afterward until reaching a constant mass. The solid content was calculated using the following equation:

$$\text{Solid content (\%)} = \frac{\text{Initial weight} - \text{weight after drying}}{\text{Initial weight}} \times 100. \quad (2)$$

Borate treatment

The dip-treated samples were prepared using 7 wt.% aqueous solutions of DOT submerged for 8, 12, 16, 20, or 24 h at room temperature. Treatment time could be reduced using commercial pressure treatment techniques for individual non-CLT panels. In this study, dip-treatment at room temperature was performed due to ease of treatment and unavailability of commercial treatment instruments. The amount of treatment solution was considerably more than the volume of wood to ensure that

Table 1 Wood sample dimensions for each test

Test method	Dip-treated board dimensions before CLT assembly (mm)	CLT dimensions after treatment and assembly (mm)	Number of Layers
Decay Resistance	$152 \times 19 \times 5.8$	$19 \times 19 \times 19$	3
Termite Resistance	$200 \times 25 \times 3$	$25 \times 25 \times 6$	2
Fire Performance	$100 \times 50 \times 10$	$100 \times 100 \times 30$	3

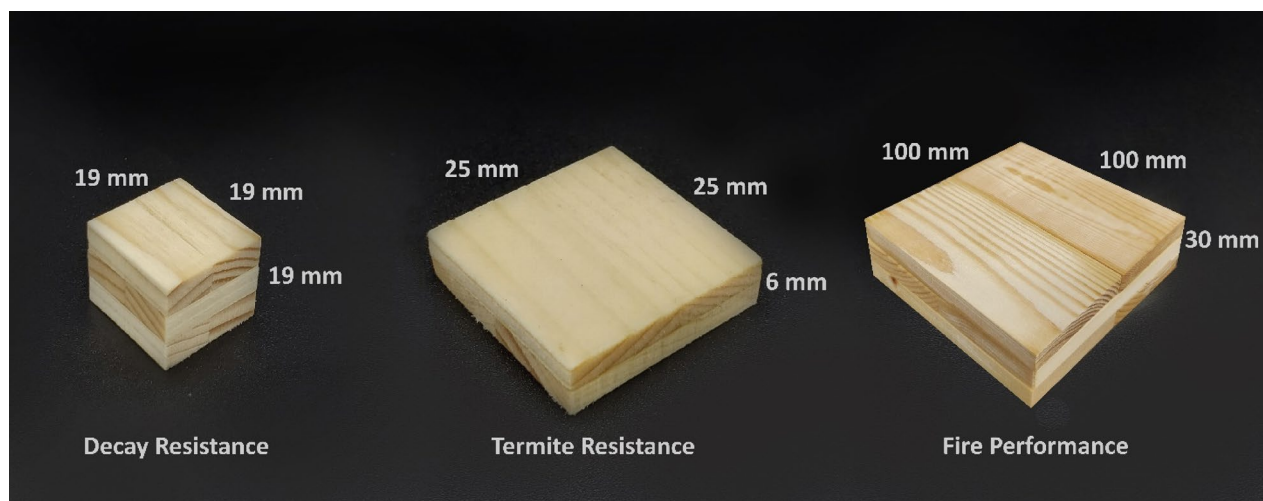


Fig. 1 Small-scale CLT samples and their dimensions were prepared for decay, termite, and fire tests

wood samples were completely submerged in water. Afterward, samples were left in a conditioning room ($\sim 20^\circ\text{C}$, $\sim 50\%$ RH) for 1 week until equilibrium.

For the spray-treated specimens, assembled CLTs prepared using untreated wood were sprayed one time using a 20% aqueous solution of DOT to cover the surface entirely. The samples were left in a conditioning room (20°C , 50% RH) for 1 week prior to testing.

Borate retention measurement

Whole laboratory dip-treated and large cross sections (length = 100 mm) from different parts of commercially treated were ground into fine particles ($< 1\text{ mm}$) for retention analysis, according to the AWWPA A40-18 test method [21]. A manual titrator (Brandtech Titrette 4,761,151, 25 mL) and a pH meter (Seven Compact S220) were used for the titration. For this method, 1 g of oven-dried wood dust was mixed with 40 mL of DI water and 4 drops of 2% Triton X-100 surfactant for 15–20 min. The mixture was then acidified with 2 mL of 0.5 N hydrochloric acid to convert all borates to boric acid, and then 6 g mannitol was added to the mixture to form borate esters. Finally, the sample was titrated with sodium hydroxide (0.1 N NaOH), and B_2O_3 retention was measured using the following equation:

$$R = \frac{N \times V \times 3.481}{W} \times \frac{\rho}{100} \quad (3)$$

where R is the B_2O_3 retention (kg/m^3), N is the normality of NaOH (0.1 N), V is the volume of NaOH used to titrate borate ester (mL), W is oven-dried wood dust weight (1.0 g), and ρ is the average wood density, *i.e.*, the

ratio of oven-dried weight to green volume ($450\text{ kg}/\text{m}^3$). Retention measurements for each treatment condition were done in triplicate. Whole samples were ground and analyzed following this method; therefore, the obtained retention data indicate the average retention throughout the depth of the sample.

Modeling and optimization

MATLAB software was used to model the borate retention based on treatment time and sample geometry. The B_2O_3 retention of 45 samples (triplicates) at five treatment times and three geometries (Table 1: column two) was measured and fitted to the general model equation (Eq. 4):

$$R = R_0 \left(1 - e^{-\frac{S}{V} D' t} \right) \quad (4)$$

where R is the retention of B_2O_3 in wood (kg/m^3), t is treatment time (h), D' is modified diffusion rate (m/h), S/V is surface to volume ratio of wood (m^{-1}), and R_0 is maximum retention that treatment solution can induce in wood (kg/m^3). This type of equation is commonly used to explain trends with an initial sharp increase/decrease, a slower rate, and finally, a plateau curve. The same behavior was observed for retention over time. Exponential regression was used to fit the B_2O_3 retention of wood samples on a 3D surface with a high coefficient of determination. By minimizing the sum of squared residual (least squares), equation constants were calculated. This model was then used to optimize the treatment time

for specific samples based on their sizes to achieve the required retentions of 2.7 and 4.5 kg/m³ B₂O₃.

Shear strength

The adhesion strength of each sample was assessed using single-lap-joint samples consisting of two panels with the dimensions of 102 mm × 25 mm × 3 mm (longitudinal × tangential × radial). The whole sample was treated and then glued at the 25 mm end of the panels according to ASTM D5868 [22], as illustrated in Fig. 2. The gluing parameters were obtained from the technical data sheet provided by the adhesive producers. The adhesive applied on the surface was approximately 0.15 g on the 25 mm² end section of lap shear veneer samples. After applying the adhesive, samples were pressed under 1.38 MPa for 4 h at room temperature as recommended by CLT manufacturers. The shear strength was measured (ten replicates) using an Instron 5565 universal testing machine. It is important to note that no machining or sanding was performed on the samples after treatment before gluing.

Thermal analysis

Thermal properties of untreated and treated samples were measured using thermogravimetric (TG) analysis (TGA Q50, TA Instruments) under a nitrogen atmosphere with a flow rate of 60 mL/min. Samples (~ 10 mg) were heated from room temperature (approximately 22 °C) to 600 °C with a heating rate of 10 °C/min. Char residue and maximum degradation temperature were recorded, and the derivative thermogravimetric analysis (DTGA) was used to obtain the rate of weight loss as a function of temperature.

Fire performance

To evaluate the fire performance, CLT samples were tested using the cone calorimeter located at the Forest Products Laboratory (FTT iCone Mini, East Grinstead, West Sussex, UK) in accordance with ASTM E1354 [23]. Prior to testing, samples were conditioned in a chamber at 21 °C and 50% RH for 3 months. Samples were exposed in the horizontal orientation with the conical radiant electric heater located 25 mm above the sample surface. The optional retainer frame was used to hold the sample during the test, the sides and bottom of the sample were wrapped in aluminum foil, and the sample rested on a ceramic fiber blanket. The conical heater was set to

produce a heat flux of 50 kW/m², and the spark igniter was used. In the cone calorimeter, the heat release rate (HRR) was measured as a function of time and calculated based on the measurements of oxygen depletion during the test.

Termite resistance

Termite resistance of CLT samples (made from the sapwood of borate-treated Douglas fir) was determined in a no-choice test according to ASTM D3345 [24]. Testing was done in clear plastic containers filled with 150 g of sterile, sifted sand, and 20 mL of sterile DI water. The test samples (one per jar) were placed on the sand surface; ten replicate samples were evaluated per treatment. Ten untreated southern yellow pine (SYP) (*Pinus* spp.) solid wood specimens were also included for comparison, as recommended by the testing standard. One gram of freshly collected eastern subterranean termites, *Reticulitermes flavipes* (Kollar), was then added to each jar. All jars were maintained at 60% RH/27 °C for the duration of the 4-week test. At the end of the test, CLT samples were removed, brushed free of debris, and dried in a hood for 1 week prior to reconditioning at 30% RH/27 °C for an additional week. Samples were weighed to determine the amount of weight loss from termite feeding.

Decay resistance

The white-rot fungus *Irpex lacteus* (MAD 517) and the brown rot fungus, *Serpula lacrymans* (ATCC 36,335) were obtained from the USDA–NRS–Center for Forest Mycology Research Herbarium Collection (Forest Products Laboratory, Madison, WI). Fungal cultures were maintained on malt extract agar (BD, ThermoFisher Scientific, Waltham, WA) with 0.5% yeast extract (Thermo Fisher Scientific) at 27 °C and 70% RH. Prior to fungal exposure, CLT test samples were conditioned for 2 weeks at 27 °C and 30% RH. Test blocks were sterilized for 24 h in 2 mL propylene oxide per liter volume of the vessel prior to fungal exposure.

Accelerated decay tests were performed on borate-treated douglas fir (sapwood) according to AWPA Standard E10–16 [25]. In each decay jar, an SYP wood wafer was initially colonized with *I. lacteus* or *S. lacrymans* for 3 weeks after inoculation by fungal mycelial plugs. Test samples were exposed to *I. lacteus* and *S. lacrymans* separately. Mycelial plugs were cut from malt extract agar

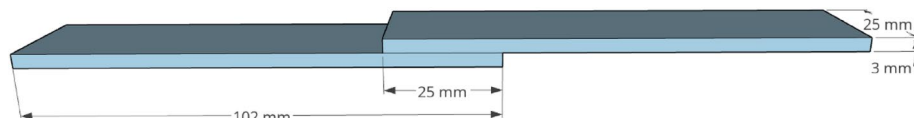


Fig. 2 Illustration of a specimen used in shear lap tests

plates with 0.5% yeast from a 2-week-old culture. One sterilized test block was aseptically placed in each jar on the colonized SYP wafer and incubated at 27 °C and 70% RH for 12 weeks. Following fungal exposure, test blocks were gently brushed free of fungal mycelia and oven-dried overnight (60 °C) overnight and reconditioned (27 °C and 30% RH) for 2 weeks. Blocks were weighed pre- and post-fungal exposure to calculate weight loss (%) due to fungal degradation, according to the following equation:

$$\text{Weight loss (\%)} = \frac{W_1 - W_2}{W_1} \times 100 \quad (5)$$

where W_1 is the conditioned block weight pre-fungal exposure, and W_2 is the conditioned block weight post fungal exposure. Weight loss was reported as an average of 5 replicates for each sample treatment configuration (SYP solid wood, control EPI, control PU, borate spray EPI, borate spray PU, dip (2.7 kg/m³ B₂O₃) EPI, dip (2.7 kg/m³ B₂O₃) PU, dip (4.5 kg/m³ B₂O₃) EPI, and dip (4.5 kg/m³ B₂O₃) PU).

Statistical analysis

The results were analyzed by statistical software (SAS, SAS Institute, Cary, NC, USA) using one-way analysis of variance (ANOVA), and significant differences between means were calculated using Tukey's honestly significant difference test with a 95% confidence level. The statistical test result was indicated as a letter, and the ones with the same letter are not significantly different and vice versa.

Results and discussion

The moisture content of Douglas-fir boards was around 9% after 4 weeks of conditioning at approximately 20 °C and 50% relative humidity. In addition, both lab-treated and commercially borate-treated wood samples were kept in the same conditions to reach the same moisture content as control. The average density of the untreated boards was 0.45 ± 0.01 g/cm³, while commercially treated boards had a significantly lower density (0.42 ± 0.02 g/

cm³). Figure 3 shows the cross section of the untreated and commercially treated boards. The average annual rings of untreated boards were approximately 2–3 per cm, while the average annual rings of commercially treated boards were between 1 and 2 per cm. Indicating that commercially borate-treated lumbers were possibly produced from fast-grown trees, explaining their lower density.

Retention modeling

The fitted model on the data set is shown in Eq. 6 and Fig. 4:

$$R = 7.53 \left(1 - e^{-0.17 \frac{S}{V} t} \right) \quad (6)$$

where R is the retention of B₂O₃ in wood (kg/m³), t is treatment time (h), and S/V is the surface to volume ratio of wood (m⁻¹).

All data points fell within the prediction confidence bound of 95%, with a coefficient of determination of 0.87. Figure 5 shows the two-dimensional contour plot of the model function. B₂O₃ retention is represented by a color (right), and dots represent the experimental retention data.

The B₂O₃ retention increased as treatment time increased in a non-linear manner. At the start of the treatment, borate migrates more rapidly due to the large difference between borate concentration in the treatment solution and the wood. Therefore, the driving force of the diffusion is greatest at the beginning of the treatment. As the migration of borate continues, the concentration of borate in the wood increases until it reaches an equilibrium condition, then the driving force dwindles, and the diffusion rate decreases. For the wood sample with a total treatment time of 24 h, more than 80% of the borate uptake happened in the first 12 h of treatment. Waldron et al. [15] observed a similar diffusion pattern in leaching experiments, where the leached amount of borate showed a similar trend with exposure time. In addition, an increase in wood samples' surface area to volume ratio



Fig. 3 Untreated (left) and commercially treated (right), showing a cross section of Douglas-fir lumbers

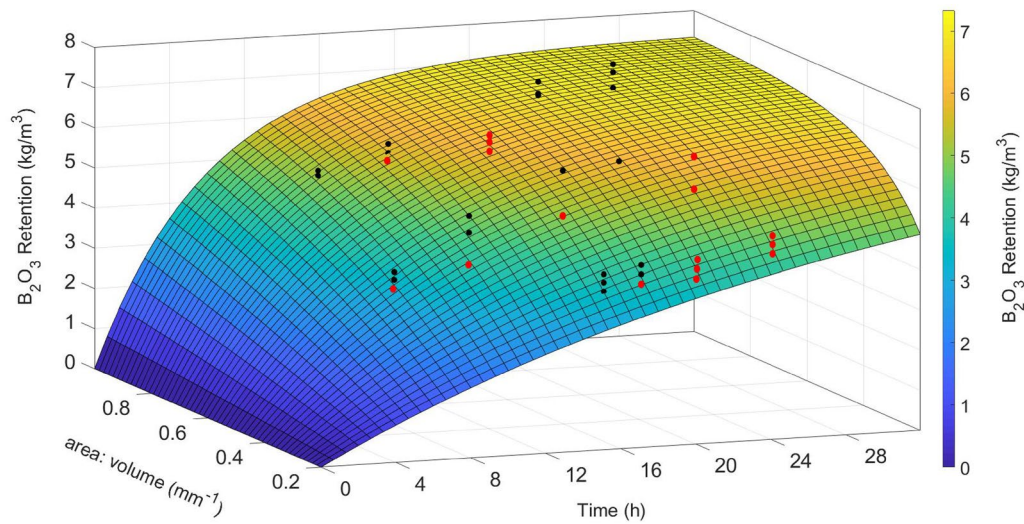


Fig. 4 Three-dimensional surface of the model equation of B_2O_3 retention in wood at different times and different surface area to volume ratios (experimental data points are shown as dots; black dots are data points that are above the surface, and red dots are data points that fall below the surface)

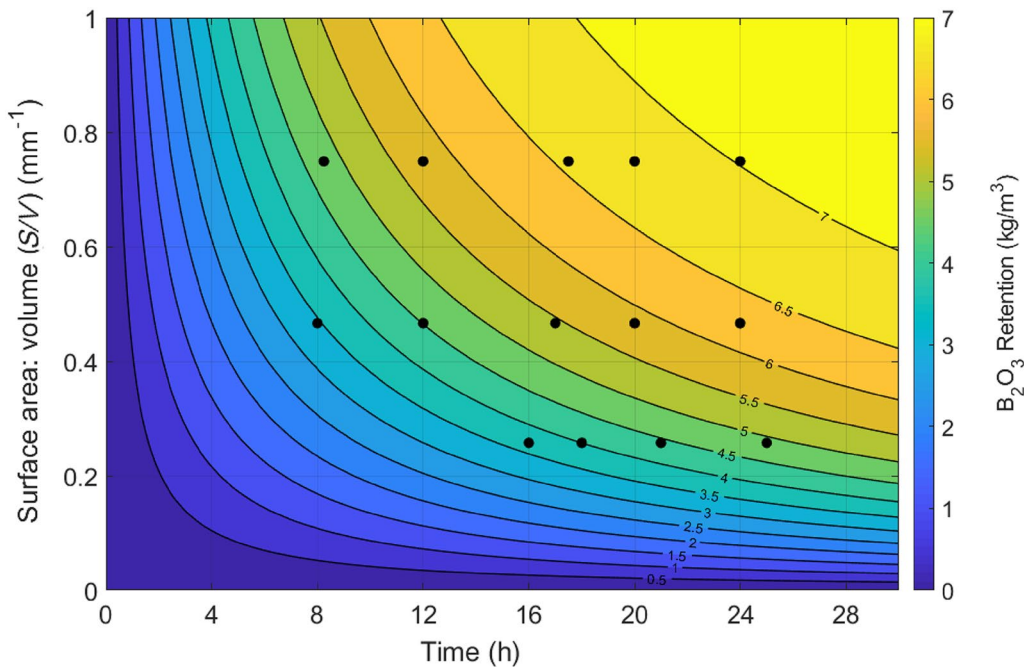


Fig. 5 Two-dimensional contour plot of retention vs. treatment time and surface area to volume ratio (Black dots show the experimental measured B_2O_3 retention)

resulted in more B_2O_3 retention, linearly. As expected, more surface area leads to increased borate migration. Using the model, given the desired retention and geometry of the samples, the optimized time for treatment can be determined following equation:

$$t = \frac{-1}{D' \frac{S}{V}} \ln \left(1 - \frac{R}{R_0} \right). \quad (7)$$

To validate the model, samples were dip-treated in the same condition for the period that was predicted

by the model. Then, B_2O_3 retention was measured (red squares in Fig. 6). Almost all samples reached the minimum required standard retention within the prediction bounds (95%), confirming the prediction model's accuracy for this study's conditions.

One of the limitations of this model (Eq. 6) is that input retention data are based on the laboratory-sized samples; thus, further confirmation of the accuracy of the model is required for commercial-sized samples. In addition, changing the treatment parameters outside of the range that was experimented with in this study most likely results in a change of model constants. However, borate and potentially other preservatives' diffusion in wood should follow Eq. 5, and with a large training set size, the model can be refitted and utilized in the new treatment parameters range. The outcome of this model is the total retention in a wood sample, and it does not provide any information about the penetration depth and the retention gradient in the wood sample.

Adhesion strength

Figure 7 shows the shear strength of variously treated samples compared to untreated (control) samples. Results showed that as the borate retention increased, the shear strength of samples bonded with EPI decreased, although this reduction was not statistically significant. However, borate treatment has been reported to reduce the surface energy of the wood, thus reducing the wettability of adhesive [26, 27]. Insufficient wetting reduces the contact area between the adhesive and wood surface, thus negatively impacting the adhesion strength [28]. However, a more in-depth study is needed to investigate the interaction effect between borate-treated woods and these adhesives.

EPI adhesive showed higher shear strength compared to PU adhesive for the commercially treated boards and untreated wood samples. During EPI curing, several reactions take place at various rates. Although high amounts of biuret and urethane reactions occur during EPI curing, the reaction of water with isocyanate (resulting in urea) is the most prominent reaction [29]. The EPI adhesive had a viscosity of $17,525 \pm 95$ mPa.s and solid

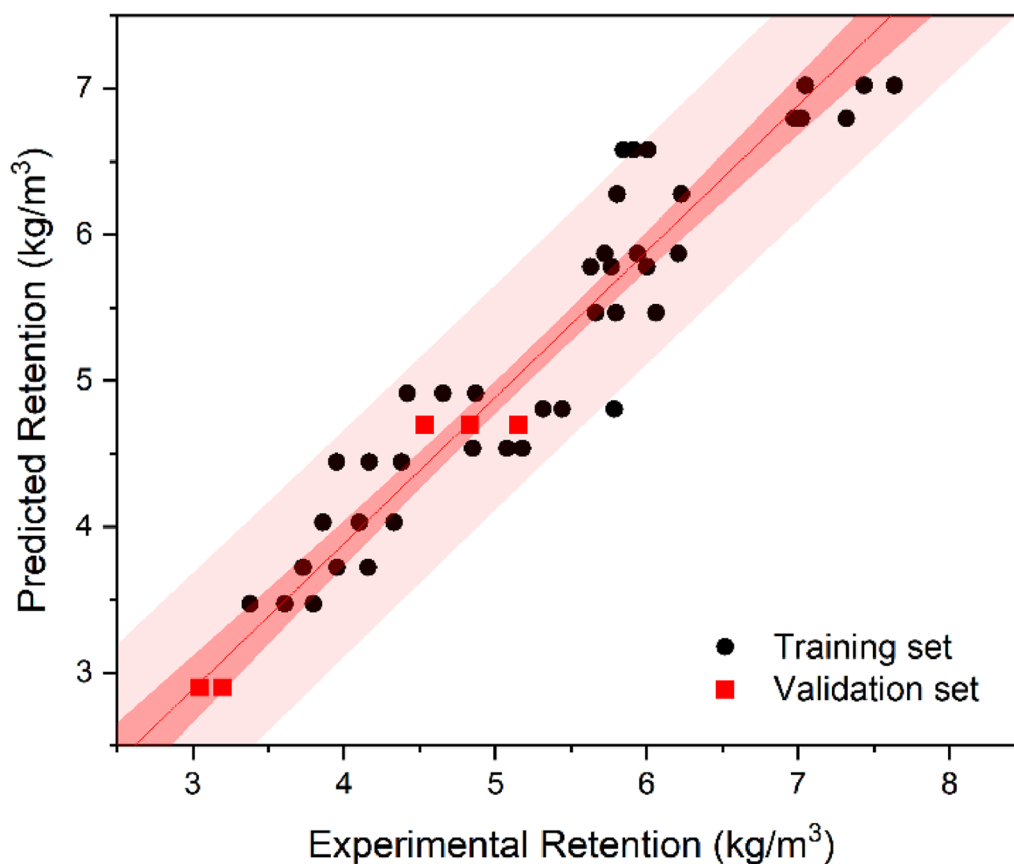


Fig. 6 Predicted B_2O_3 retention of samples vs. experimental B_2O_3 retention for the training and validation sets of the model ($R^2 = 0.91$)

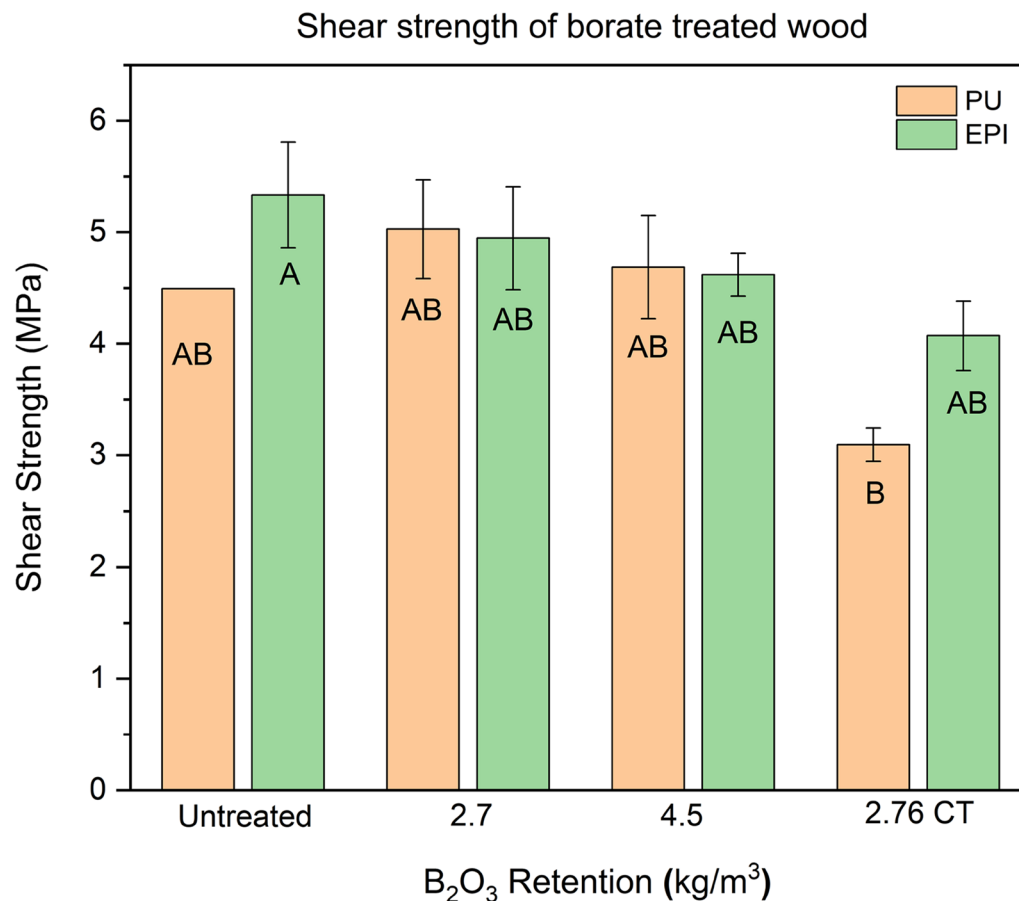


Fig. 7 Shear strength of untreated, borate-treated, and commercially dip-treated (CT) Douglas-fir samples bonded with PU and EPI adhesives. The borate retention of the CLT sample was obtained at 2.76 kg/m³. Means with the same letters are not significantly different at the 0.05 confidence interval (p values < 0.05)

content of $95.6 \pm 0.3\%$. The PU adhesive had a viscosity of $19,700 \pm 81$ mPa.s and solid content of $93.2 \pm 0.2\%$. The lower viscosity of EPI adhesive might have improved the penetration of EPI to open pores of untreated and low density (commercially treated) woods resulting in higher adhesion strength compared to dip-treated wood samples. The adhesive must have enough fluidity to penetrate the porosity of the wood but not too much fluidity (very low viscosity) to cause a starved glue line [28]. The commercially borate-treated wood had lower adhesion strengths mainly due to the lower density of these boards, causing wood failure at a lower force.

Thermal analysis

Several methods can be used to evaluate the efficacy of a fire retardant. Thermogravimetric analysis is one of the most common methods for assessing the impact of fire retardant preservatives on thermal decomposition behavior [30]. Figure 8 shows the thermal analysis of treated and untreated wood samples. When heating samples,

noncombustible products such as water vapor and carbon dioxide are produced in the temperature range of 100–200 °C. In the temperature range of 240–350 °C, degradation of cellulose and hemicellulose happens [31]. Decomposition of hemicellulose occurs at slightly lower temperatures than cellulose. The lignin degradation happens at above 400 °C, and at the end of the pyrolysis, char and more gases are produced [32].

Borate derivatives are considered fire retardant preservatives [33]. In one of the theories of fire retardancy (chemical theory), the retardant chemicals change the pyrolysis of the wood by increasing the char residue and decreasing the amount of volatile and combustible vapors [34]. Based on 1% mass loss, degradation happened at 265 °C for the untreated wood, while it occurred at 270 °C for treated samples. Through decomposition of the preservative, a protective layer is formed on the surface of the wood, increasing the initial degrading temperature and char residue [35]. From Fig. 8a, b, the temperature at which the maximum rate of mass loss occurred for the

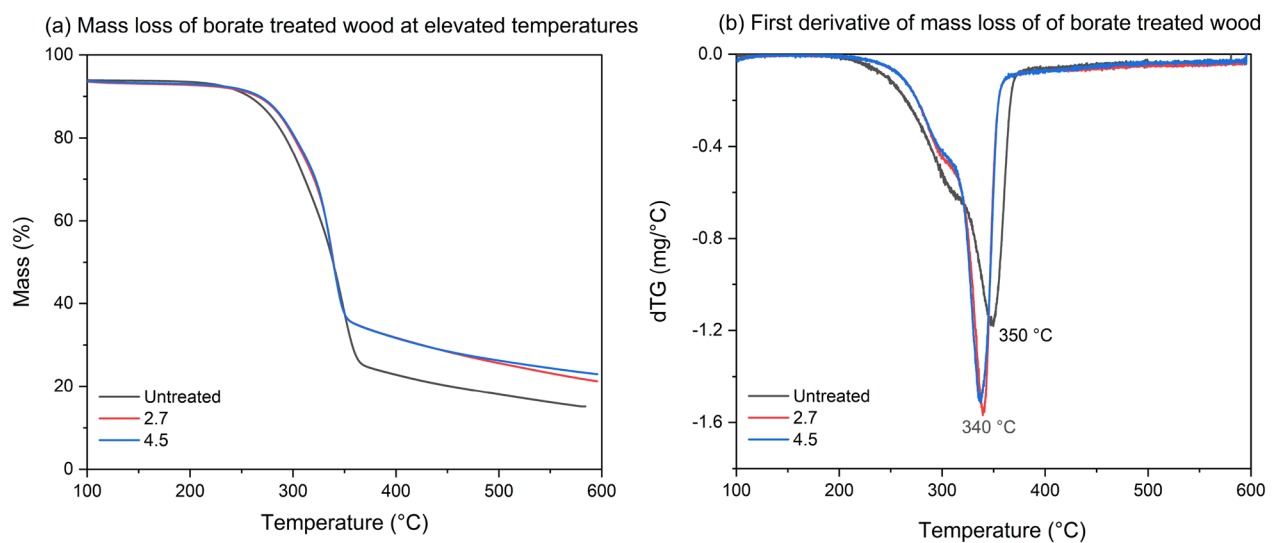


Fig. 8 Thermogravimetric analysis (TGA) (a) and derivative thermogravimetric analysis (DTGA) (b) curves of untreated and samples with 2.7 and 4.5 kg/m³ B₂O₃ retention

untreated sample was 350 °C with a char residue of 15%. However, the average maximum degradation temperatures of the treated samples were 340 °C with a higher char residue than untreated samples (~24%).

Material flammability

To evaluate the flammability of the samples, the HRR for each treatment and adhesive combination were measured (Fig. 9). The HRR curves exhibited two peaks, typical of wood materials. The initial peak is attributed to the rapid HRR for uncharred wood, which is reduced once the protective char layer forms. The second peak reflects the thermal wave reaching the unexposed side of the sample and can be affected by many variables, including the density, heat flux, material thickness, and backing product [36–38].

From Fig. 9, the untreated Douglas-fir heat release rate curve is comparable to results obtained for untreated wood [39], with a rapid increase to a peak HRR around 160 kW/m². Figure 10a shows the average time to initial peak heat release rate (pHRR) for each sample type. Although it seems that the spray- and dip-treated samples at a retention level of 2.7 kg/m³ B₂O₃ have improved time to initial pHRR, only treatment at 4.5 kg/m³ showed a statistically significant increase.

In Fig. 10b, the initial pHRR shows the amount of heat that was released at the initial peak. The amount of released heat was affected by the treatment. All treated specimens had a lower average initial pHRR than the control specimens. Although spray-treated wood did not perform as well as dip-treated samples, it performed

better than control untreated samples. This shows that penetration and retention are crucial to positively impact the initial heat released. In general, as the borate retention increased, the total amount of released heat decreased. Spray treatment decreased the amount of released heat compared to control samples, but the dip-treated samples had superior fire performance.

The adhesive type had no effect on the initial pHRR. However, beyond the initial pHRR, the adhesives did affect the HRR results when the butt-joint of the exposed surface opened due to adhesive failure at the bond line, causing fresh wood from the next ply to be exposed.

After the combustion of samples, the residual mass fraction (RMF) was measured. The mean result for the untreated specimens was 0.17 (±0.06). In contrast, the results for the B₂O₃ dip-treated samples ranged from 0.20 to 0.27, with a mean result of 0.23 (±0.02). Higher RMF is associated with fire retardant treatments for wood products, because the treatments reduce the production of volatile flammable gases, thereby reducing the amount of material consumed. The RMF result of spray-treated specimens were similar to untreated specimens, with an average RMF of 0.18 (±0.05). RMF results obtained from the cone calorimeter were in great agreement with the residual obtained from the TGA.

Levan and Tran [40] noted that wood flame retardants typically work by causing acid-catalyzed dehydration reactions that facilitate the formation of char, reduce the effective heat of combustion, and result in lower heat release and flame spread. Boron compounds have also been shown to form a film that may inhibit the flow of

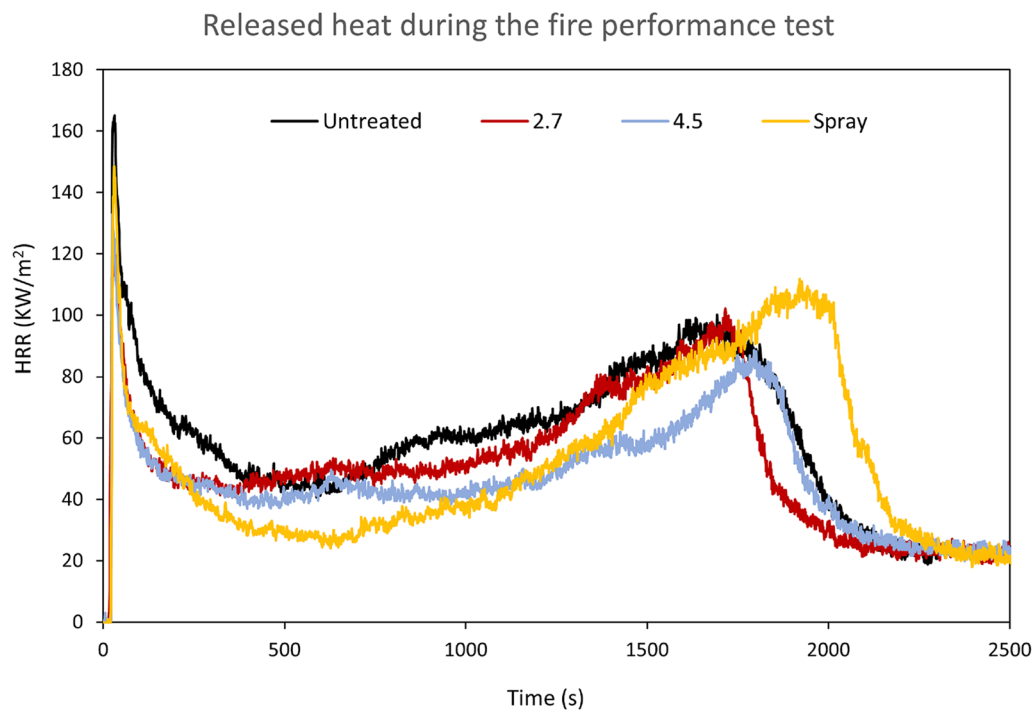


Fig. 9 Heat release rate curves for representative CLT samples bond with Polyurethane (PU) adhesive at different treatments. “Untreated” refers to the control CLT sample with no treatment, “2.7” and “4.5” refer to the dip treated CLT samples with B_2O_3 retention of 2.7 and 4.5 kg/m^3 , respectively. “Spray” refers to the commercial CLT sample sprayed over with borate solution

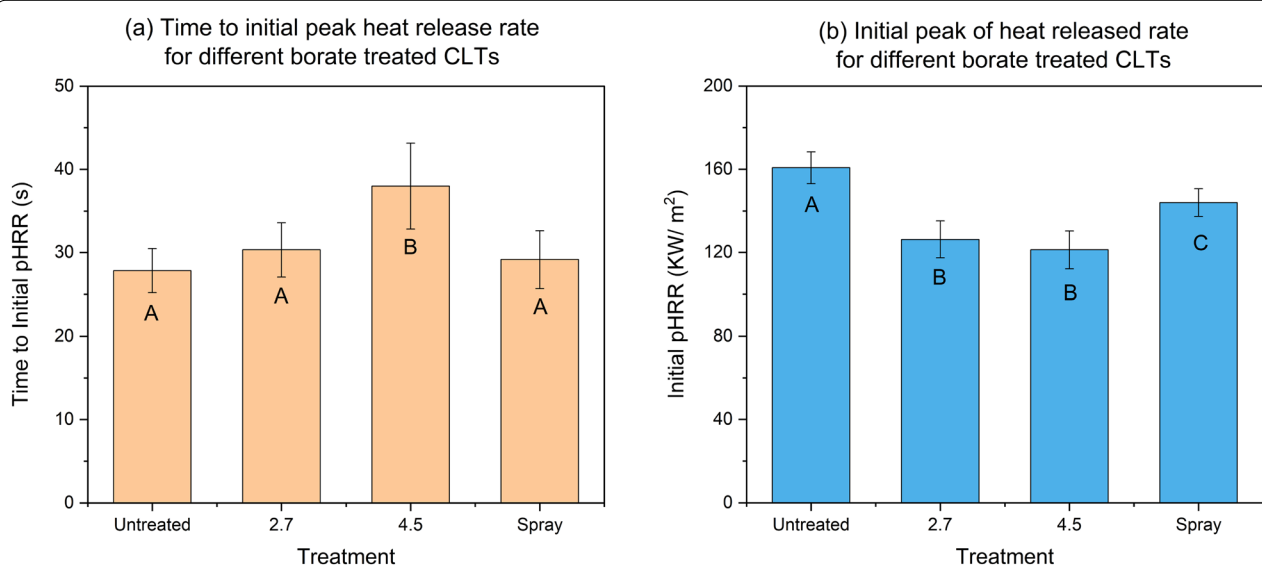


Fig. 10 Average time to initial peak heat release rate (pHRR) (a) and initial peak heat release rate (pHRR) (b) untreated and dip-treated samples to retention levels of 2.7 and 4.5 kg/m^3 B_2O_3 and spray treated triplicate samples (error bars show standard deviation). Means with the same letters are not significantly different at the 0.05 confidence interval (p values < 0.05)

combustible vapors to the fire-exposed surface, decreasing the reactions. Similar to other studies that confirmed the reduction in heat release from wood treated with various borate compounds [4], the dip- and spray treatments used here led to a significant reduction in heat released from the CLTs as expected.

Termite resistance

The termite resistance of borate-treated and untreated samples are shown in Fig. 11. All borate-treated samples showed significantly higher resistance levels to subterranean termite feeding than the untreated CLT and SYP solid wood control samples. The results of spray- and dip-treatment were not significantly different, with weight loss between 1 and 2% for PU and EPI adhesives. In most cases, complete termite mortality was observed in all treated samples occurring by week 2. However, this is not unexpected as borates have repeatedly been shown to kill termites [1, 41].

Within the untreated control blocks, untreated southern pine specimens showed only slight levels of termite mortality, while untreated CLT samples bonded with

PU showed noticeable resistance to termites with only 10% weight loss to the CLT blocks and 100% termite mortality by the end of the test. This suggests that PU alone has insecticidal activity. In contrast, EPI control CLT samples were readily consumed by termites with the highest woodblock weight loss of 60% and resulted in only slight termite mortality. Weight loss in these specimens was nearly 20% greater than that observed in the SYP solid wood control sample.

PU adhesive is one component (1 K) adhesive made of an NCO-terminated prepolymer with about 10–15% unreacted isocyanate that can bond with hydroxyl groups of the wood to form even some urethane linkages at the interface. In addition, the higher unreacted isocyanate in the 1 K formulation could result in some NCO reacting with water forming urea linkages at the bond line. The formulation might have contained zinc borate or flame retardant that are toxic to termite.

In contrast, the EPI is a two-component emulsion polymer with the main ingredient of polyvinyl alcohol (PVOH), latex, and fillers. The water-soluble PVOH will crosslink by reacting with isocyanate after being

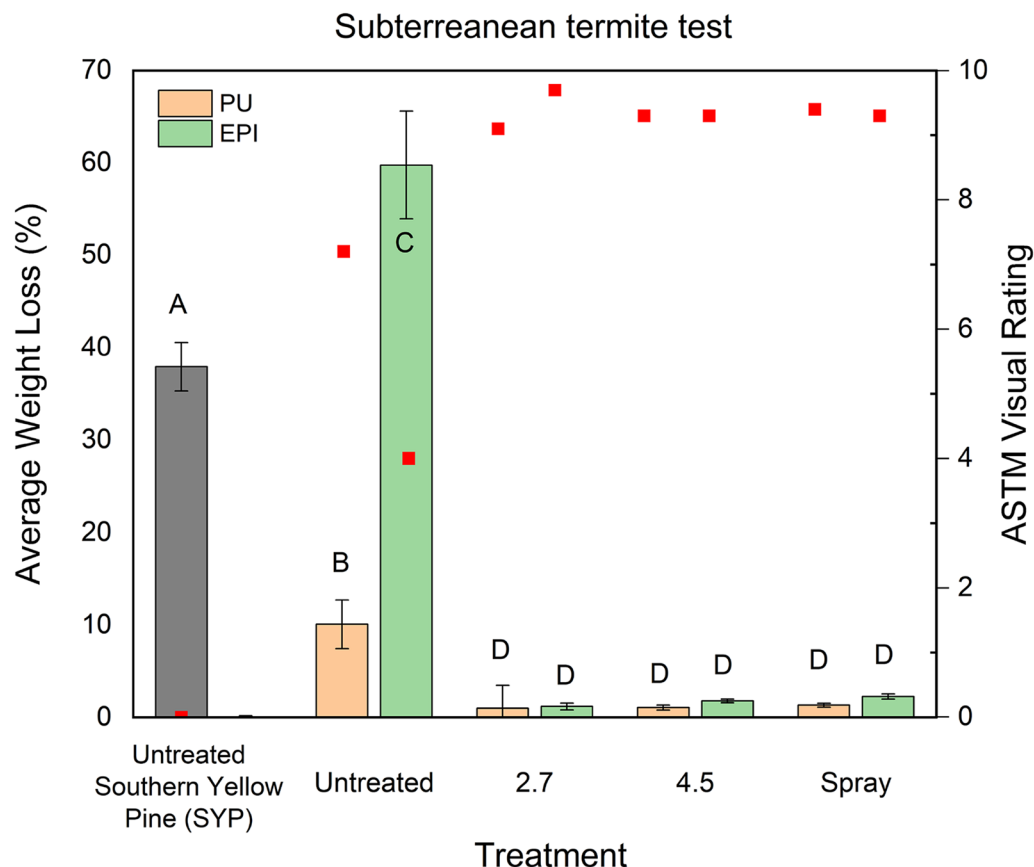


Fig. 11 Average weight loss (bars) and ASTM D3345 [24] visual rating (red squares) of CLT test samples after subterranean termite attack. Error bars show standard deviation. Means with the same letters are not significantly different at the 0.05 confidence interval (p values < 0.05)

mixed with a polymeric methylene diphenyl diisocyanate (MDI) as a hardener in a ratio of 100:15. Since the exact formulations of these commercial adhesives are unknown, more information is needed to accurately explain the surprising effect of EPI that seemed to serve as food for termites when used on untreated wood samples.

Figure 12 shows the selected samples after 4 weeks of exposure to termite feeding. While dip- and spray-treated blocks showed little to no termite damage, the feeding pattern in untreated samples seems to demonstrate the toxicity of the PU adhesive. Termites appeared to have consumed a few millimeters on the first layer of CLT samples bonded with PU until they reached the penetration depth of the adhesive. Mortality likely occurred soon after consuming a small amount of PU material. Since the amount of available moisture for curing a small amount of PU adhesive is more than enough, the time between CLT assembly and testing was more than 2 weeks. It is safe to assume that PU adhesive was cured entirely, and incomplete curing was not a factor in these results.

In untreated EPI bonded CLT, as termites reached the penetration depth of the EPI, they seemed to either consume a small amount of the adhesive or avoid it entirely, consuming the surrounding uncontaminated area. As a result, many samples showed just a thin layer of wood and glue-line remaining at the end of the 4-week test, without resulting in any noticeable termite mortality. While the PU adhesive provided some termite resistance in these small test specimens, this effect would be negligible for thicker commercial CLT samples as the amount of wood available for termite consumption before reaching the glue line is much greater.

In this research, the difference between the adhesives, treatment process, and borate retention were investigated as a comparative study using small-scale CLT

samples. However, as test sample geometry, block configuration, and environmental conditions are likely to be different for larger CLT blocks in-service, it will be crucial to consider all factors when comparing the laboratory scale samples with the large scale commercial CLTs. Particularly as these factors have been shown to influence termite management and prevention [42, 43].

Decay resistance

The decay resistance of CLT samples was evaluated based on the resistance of fully prepared small-scale test samples exposed to two species of wood decay fungi, *I. lacteus* (white-rot) and *S. lacrymans* (brown-rot), based on the approved test fungi listed in the AWP A E10–16 standard. The *I. lacteus* was chosen because of the high rates of decay caused by this white-rot fungus, and *S. lacrymans* was chosen due to its ability to translocate water during the attack and high rates of decay. In addition, *S. lacrymans* fungus is prevalent in Europe, where CLT structures are more common.

Figure 13 shows the average weight loss of treated samples compared to untreated samples and a comparison between the effect of the adhesive on the decay resistance of samples. Control samples bonded with PU adhesive showed more resistance compared to EPI samples. It has been reported that some microorganisms were able to degrade polyvinyl acetate (PVA), which is one component of EPI adhesive, and the most damage was recorded caused by fungi. Still, yeasts, algae, lichens, and bacteria could also damage PVA [44]. When exposed to *I. lacteus* (Fig. 13a), untreated SYP solid wood showed more than 45% mass loss, while EPI control samples showed less than 30% mass loss, although insignificant, offering only slight protection from white-rot attack and likely not enough to warrant resistance. PU control sample showed less than 12% weight loss, which indicates PU provides a decent amount of protection against *I. lacteus* over the course of this study. When exposed to *S. lacrymans* (Fig. 13b), control samples show similar behavior, where SYP and PU control samples show 40% weight loss and EPI control samples show 50% weight loss indicating no protection against brown-rot attack.

According to Fig. 13, after dip treatment and reaching the standard retention goal, there was no significant difference between adhesive types and treatment types. In addition, dip-treated samples, as well as spray-treated samples, showed minimal percent weight loss after exposure to *I. lacteus* and *S. lacrymans*. There was no significant difference between decay resistance of spray treated and dip-treated samples, which emphasizes the potential effectiveness of spray treatment even during the construction of mass timber buildings. Since the CLT panels are much larger, thicker, and drier than the samples

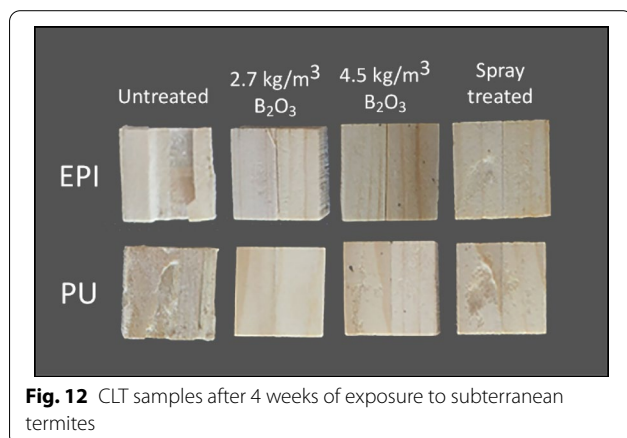


Fig. 12 CLT samples after 4 weeks of exposure to subterranean termites

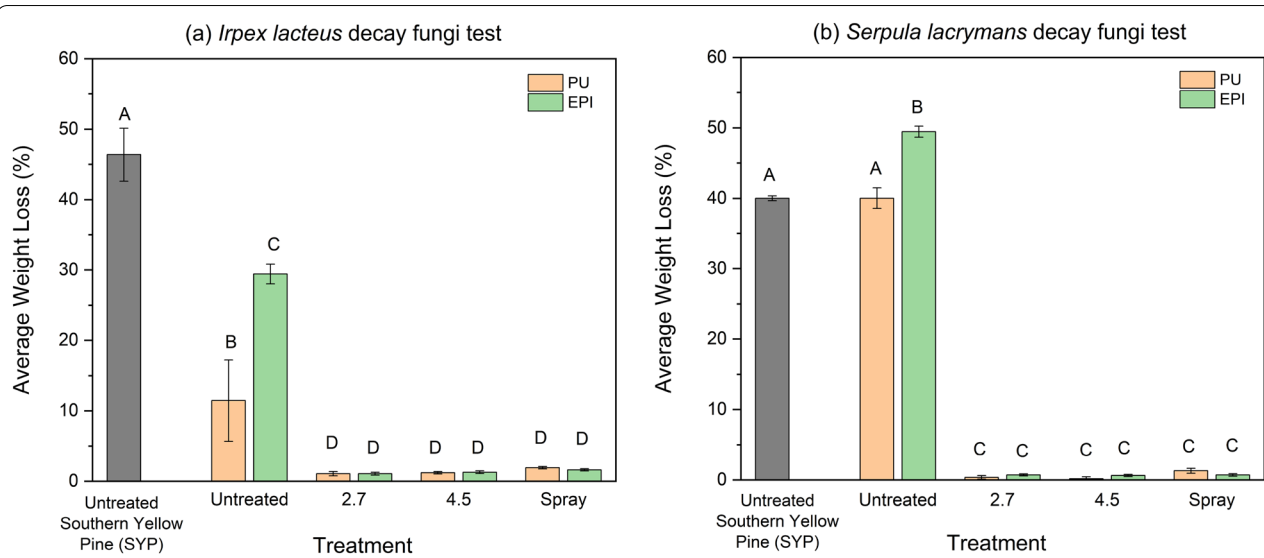


Fig. 13 Effect of treatment and adhesive type on weight loss of CLT samples exposed to *Irpex lacteus* (a) and *Serpula lacrymans* (b). PU and EPI indicate the adhesives that were applied to the sample. Spray indicates the spray treatment with a borate solution. 2.7 and 4.5 show the B_2O_3 retention (kg/m^3) of dip-treated samples. Means with the same letters are not significantly different at the 95% confidence interval (p values < 0.05)

used in this project, further studies are required to evaluate the efficacy of spray treatment on large-scale CLT constructions.

No visible sign of fungal growth was observed on borate-treated samples (Fig. 14). However, according to weight loss data (Fig. 13), all samples showed better performance against *S. lacrymans* than *I. lacteus*. In addition, when exposed to *S. lacrymans*, at high borate retention levels, samples lost less than one percent weight, whereas dip-treated samples bonded with PU seemed to have at least twice the resistance of EPI samples.

Although the samples used in this study were small-scale CLT tested in a laboratory setting, the information generated is useful for baseline data on the effectiveness of treatments on CLT, and it can be expanded to large-scale, outdoor studies in the future.

The treatment time was the most influential factor at the start of the borate-treatment process. However, as time goes on, the surface area to volume ratio seems to significantly impact B_2O_3 retention. Treatment time and surface area to volume ratio were incorporated into an exponential function to quantify boron uptake in the wood. Dip-treated CLT samples performed marginally



Fig. 14 Decay tests (AWPA E10–16) showing no visible fungal growth on borate-treated CLT blocks exposed to *S. lacrymans* for 12 weeks (left) and fungal growth on untreated CLT blocks exposed to *S. lacrymans* for 12 weeks (right)

better than spray-treated and untreated control samples. Spray-treatment provides a thin layer of well-protected wood, which could be potentially beneficial, providing further surface modifications, such as machining, sanding, etc., are prevented after treatment. Fast spray-treatment results in samples with comparable properties to dip-treated samples. However, commercial-sized CLT samples are larger and have a smaller available surface-to-volume ratio compared to laboratory-sized samples. This would result in overall lower borate uptake and potentially higher borate loading near the surface than the center. This effect is more desirable for fire performance and less desirable for decay and termite resistance. To reduce this effect, spray treatment could be performed in several stages at different times to ensure flattening of the steep borate gradient in wood.

Untreated samples bonded with PU adhesive showed increased durability against termites and decay fungi, although not to the extent observed with treatment. TGA and cone calorimeter were in good agreement to test the efficiency of the borate treatment, which resulted in increased char residue compared to the untreated specimens.

Conclusions

This study was one of the first to test protective treatments against termite and decay attacks on CLT. The CLT blocks were assembled using borate-treated wood, and mechanical strength, decay, termite resistance, and fire performance of CLT samples bonded with two commercial adhesives were evaluated. A prediction model was developed to optimize the treatment process to achieve the B_2O_3 retentions required by the standards-based on sample size and treatment time. The developed model can be implemented with a few changes based on the training set samples to predict the borate retention of samples with larger dimensions, different layout geometries, and potentially different treatment techniques for laboratory and commercial-scale wood. Although the adhesion strength of samples slightly decreased as retention increased in borate dip-treated samples, the differences were not statistically significant. Both spray-treated and dip-treated samples showed significantly higher resistance against decay and termites, which indicates spraying prefabricated CLT panels with borate solution even during the construction could improve the biological durability of CLT. Untreated samples bonded with EPI adhesive showed significantly lower resistance against decay and termite tests which could be due to the PVA (one component of EPI adhesive formulation), which is known to be susceptible to biodeterioration. Further in-depth study is needed to better explain why PU adhesive

performed better than EPI and what made PU adhesive appear more toxic to termites.

Abbreviations

CLT: Cross-laminated timber; EPI: Emulsion polymer isocyanate; PU: Polyurethane; DOT: Disodium octaborate tetrahydrate; AWWPA: American wood protection association; RH: Relative humidity; NaOH: Sodium hydroxide; TGA: Thermogravimetric analysis; DTGA: Derivative thermogravimetric analysis; HRR: Heat release rate; pHRR: Peak heat release rate; SYP: Southern yellow pine; ANOVA: Analysis of variance; CT: Commercially dip-treated; RMF: Residual mass fraction; PVOH: Polyvinyl alcohol; MDI: Methylene diphenyl diisocyanate; PVA: Polyvinyl acetate.

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Author contributions

SB and MA: data curation (Borate treatment, CLT samples preparation, retention, shear strength, and TGA analysis), modeling, investigation, methodology, validation, visualization, and writing—original draft, KO, LH, and RA: data curation (conducting decay, termite and fire analysis), writing—review and editing, MN: conceptualization, funding acquisition, methodology, project administration, resources, supervision, validation, visualization, and writing—review and editing. All authors read and approved the final manuscript.

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Declarations

Competing interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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