

Bonding Performance of Preservative-Treated Cross-Laminated Timber (CLT) Posttreated with CU-Based Preservatives

Franklin Quin, Jr. Samuel Ayanleye Tamara S. F. A. França
Rubin Shmulsky Hyungsuk Lim

Abstract

To expand the use of cross-laminated timber (CLT) to exterior applications, there is a need to protect the panels from biodegrading agents such as fungi and termites. Pressure treatments are effective methods of increasing the durability of wood and wood-based products; however, studies on pressure-treated CLT are limited. In this study, preservative-treated CLT samples from prefabricated CLT panels were prepared and impregnated with Cu-based preservatives through a conventional vacuum-pressure process. The effects of panel layup (3-ply parallel, 3-ply perpendicular, and 5-ply parallel) and preservative treatment (untreated [control], copper azole-type C [CA-C], and micronized copper azole-type [MCA]) on the bonding performance were investigated. Panel layup and preservative treatment had a significant influence on the block shear strength and percentage of wood failure (WFP) of the treated panels. Overall, approximately 60 percent of the block shear specimens had a WFP of >75 percent. However, fewer than 10 percent of the delamination specimens met the ASTM D2559 (2018) limitation of 1 percent for softwood used in outdoor applications. ASTM D2559 counts shallow wood failure as delamination, which could have been a reason for the high delamination rate. The percentage of wood failure and the high rate of delamination could be due to the moisture-induced adhesion failure resulting from the pressure-treatment process. The preservative pressure-treatment of the CLT panels increased the moisture content (MC) from 12–15 percent to approximately 85 percent MC, and the severe swelling of the panels during treatment might have imposed a high stress on the bond line. However, no noticeable delamination of the panels was observed during the actual treating phase of the study. These results show the feasibility of treating prefabricated CLT panels with CA-C and MCA preservatives without compromising the bonding strength.

Cross-laminated timber (CLT) is an engineered wood product manufactured mostly from softwood dimension lumber or structural composite lumber. CLT is made by laminating and gluing adjacent layers of lumber together with the layers bonded at 90° to each other (Wang et al. 2018). Lamination is by adhesive, nails, wooden dowels, or a combination thereof. CLT panels are typically manufactured with 3 to 9 layers. CLT, which was first developed in Austria and Germany in the 1970s and 1980s, has enjoyed a major level of success in the European construction market and is establishing a presence in North American construction projects (Brandner et al. 2016).

CLT panels are usually covered for protection from the environment when transported from the manufacturing company to the construction site. Weather-resistant barriers are also used in building envelope systems to keep CLT panels dry. In order to expand the use of CLT in exterior applications the question of durability must be addressed when exposed to moisture (weathering), decay, and wood-attacking insects such as termites (França et al., 2020; Lim et al. 2020).

Preservative treatment of wood using copper compounds for wood protection has been around for >200 years (Nguyen

et al. 2012). Within the United States, the volume of wood products treated with copper-based preservatives grew exponentially during the 1970s and 1980s and remains high today (De Groot and Woodward 1999). The tolerance of some fungi to copper-based preservatives led to the development of new preservatives with other co-biocides for improved efficacy against biodeteriorating agents (Freeman and McIntyre 2008, Ayanleye et al. 2022).

The authors are, respectively, Research Associate III (fq3@msstate.edu), Graduate Research Assistant (soa48@msstate.edu), Assistant Professor (tsf97@msstate.edu [corresponding author]), and Head and Professor (rs26@msstate.edu), Dept. of Sustainable Bioproducts, Mississippi State Univ., Mississippi State, Mississippi; and Senior Lecturer (thomas.lim@canterbury.ac.nz), School of Forestry, Univ. of Canterbury, Christchurch, New Zealand. This paper was received for publication in May 2023. Article no. FPJ-D-23-00031.

©Forest Products Society 2023.

Forest Prod. J. 73(4):326–338.

doi:10.13073/FPJ-D-23-00031

Other co-biocides are added to copper compound preservative systems to make them effective against copper-tolerant fungus species. Moreover, the Environmental Protection Agency (EPA) announced a voluntary agreement with wood treaters to discontinue the use of chromated copper arsenate (CCA)—treated wood in certain residential applications because the possible health and environmental impacts necessitate the development of environmentally friendly alternatives such as alkaline copper quaternary (ACQ) and copper azole (CA; Townsend et al. 2005).

Lee et al. (2006) using ASTM D905 (2021) reported on the dry block shear strength (BSS) of two softwood species (Korean pine [*Pinus koraiensis*] and Japanese larch [*Larix kaempferi*]) treated with four levels of waterborne preservatives (untreated; CCA; CD-HDO [copper, boric acid, and N-cyclohexyldiziniumdioxide]; and CA) bonded together with four different adhesive systems (urea-melamine formaldehyde [UMF], melamine formaldehyde [MF], phenol formaldehyde [PF], and resorcinol formaldehyde [RF]). The dry shear values ranged from 0.52 MPa to 5.50 MPa with no evident trends.

Lisperguer and Becker (2005) compared the bond strength durability of two different adhesives (commercial and laboratory-modified phenol-resorcinol-formaldehyde (PRF) when bonding radiata pine (*Pinus radiata*) wood treated with different retention levels (4 and 6 kg/m³) of chromated copper arsenate (CCA). The modified PRF passed the minimum requirements for ASTM D2559 (2018), which was <1 percent delamination rate.

Lim et al. (2020) measured the block shear strength (BSS) and delamination rate of 3-ply southern yellow pine (*P. echinata*) CLT panels fabricated from micronized copper azole-type C (MCA-C) lumber treated to 1.0 kg/m³ and 2.4 kg/m³ preservative retention levels bonded with three different adhesive systems (melamine formaldehyde [MF], resorcinol formaldehyde [RF], and one-component polyurethane [PUR]). The results showed that BSS and delamination rate were influenced by preservative treatment and the adhesive system. The study showed that the PUR adhesive yielded a higher bonding performance than did the MF and RF adhesives.

These previous studies dealt with fabricating CLT panels with treated lumber. Taylor et al. 2022 investigated the postpreservative treatment of CLT specimens cut from commercial-size spruce (*Picea* spp.), Douglas-fir (*Pseudotsuga menziesii*), and radiata pine CLT panels. In this study, they used a borate solution applied under vacuum pressure in flexible bags. Most industrial cylinders at commercial treating facilities are not designed to handle full-size CLT panels. Their experimental study showed success in the potential for treating CLT specimens.

There is a lack of data on the effects of posttreatment of southern yellow pine CLT panels when treated with micronized copper azole (MCA) or copper azole-type C (CA-C) to the retention level of 2.4 kg/m³ (UC4A [ground contact or fresh water] applications specified by AWP Standard U1-22) on the adhesive performance. The objective of this study was to evaluate the bonding performance and durability of post-layup-treated southern yellow pine CLT panels. A one-component polyurethane (PUR) adhesive was used; and also MCA and CA-C, which are both commonly used commercial preservative systems, were used for treating the CLT panels.

Table 1.—Summary statistics of moisture content (MC) and oven-dry specific gravity (SG) of lumber laminates used in cross-laminated timber (CLT) fabrication.

	MC (%)			SG _{oven-dry}		
	Mean	SD ^a	COV ^b	Mean	SD	COV
3-layer parallel CLT						
Control	10.78	1.36	12.67	0.52	0.03	5.35
CA-C ^c	11.19	1.22	10.91	0.50	0.04	8.90
MCA ^c	13.04	1.60	12.30	0.48	0.04	7.51
3-layer perpendicular CLT						
Control	10.77	1.09	10.13	0.51	0.02	3.89
CA-C	11.28	1.13	10.01	0.48	0.04	9.32
MCA	12.02	0.86	7.19	0.47	0.03	7.21
5-layer parallel CLT						
Control	10.70	0.82	7.70	0.50	0.04	7.02
CA-C	13.98	1.10	7.88	0.46	0.03	7.33
MCA	13.19	1.18	8.93	0.46	0.03	6.21

^a SD is standard deviation.

^b COV is coefficient of variation.

^c CA-C is copper azole-type; and MCA is micronized copper azole-type.

Materials and Methods

Materials

Visually graded Select Structural (SS) southern yellow pine (SYP) lumber (38 mm by 140 mm by 2,438 mm) was supplied by a regional sawmill. The SS lumber was chosen in order to minimize lumber defects as specified in the southern yellow pine grading rules (SPIB 2014). Each piece of lumber was weighed with an electronic scale and grouped into four weight classes. The weight classes were (1) <6,000, (2) 6,000 g to 7,000 g, (3) 7,000 g to 8,000 g, and (4) >8,000 g. The density of wood influences its shrinkage from green to oven-dry condition, with the denser wood shrinking more than the less dense wood. The shrinkage and swelling of wood also depend on the direction, with wood shrinking more in the tangential and radial directions as compared with the longitudinal direction (Schulgasser and Witztum 2015). This sorting was done to control the lumber density range for the laminates and to minimize the variability within samples. The wood laminates for CLT construction were cut from lumber in weight classes 2 and 3. The clear wood sections of the lumber were labeled and divided into 762-mm and 508-mm sections. As specified in ASTM D2559 (2018), only flat-grain (wood with growth rings that make an angle <45° with the wide surface) laminates with no pith were chosen as laminates. The wood laminates were planed on each face to a final thickness of 36 mm before gluing. Oven-dry specific gravity (SG) and moisture content (MC) specimens were cut from each section according to ASTM D2395 (2017) and ASTM D4442 (2020), respectively. Table 1 shows the average MC and the oven-dry specific gravity (SG_{oven-dry}) of the laminates. The average SG of the laminates was 0.49 with an average MC of 11.89 percent.

CLT manufacturing

The adhesive used for this study was a single-component polyurethane adhesive (LOCTITE PUR HB X602) supplied by Henkel Corporation. Before applying the adhesive, a wood primer was sprayed (spreading rate of 20 g/m²) onto the surface of the wood ≥10 minutes before adhesive application. The wood primer used was LOCTITE PR 3105

(specifically for bonding southern pine), also supplied by Henkel Corporation. The primer was mixed with tap water before use at a concentration of 10 percent by weight (9 parts by weight of tap water and 1 part by weight of primer). After the 10-minute curing of the primer on the wood surface, the PUR adhesive was applied with a spreading rate of 180 g/m² according to adhesive product specifications. The CLT panels were fabricated within 8 hours of the laminates being planed.

Three CLT panel configurations were constructed in this study (Fig. 1). Configurations 1 and 2 were 3-ply, while Configuration 3 was 5-ply construction. The main difference between Configurations 1 and 2 is that Configuration 1 has the top- and bottom-layer laminates parallel to the panel long direction, with the middle-layer laminates perpendicular to the panel long direction. Configuration 2 consisted of the top- and bottom-layer laminates perpendicular to the panel long direction, with the middle-layer laminates parallel to the panel long direction. The reasoning for Configurations 1 and 2 addresses the issue of moisture movement in wood. The movement of moisture within wood is faster parallel to the fiber direction as compared with the perpendicular direction, so the panel configuration could have an influence on preservative penetration and retention. This was investigated in another study (Ayanleye et al. 2023). Configuration 3 consisted of the top-, middle-, and bottom-layer laminates parallel to the panel long direction with the other two-layer laminates perpendicular to the panel long direction. The parallel layers were composed of three laminates (edge to edge), while the perpendicular layers were composed of five laminates (edge to edge). The final panel dimensions were 404 mm by 676 mm by 104 mm for the 3-ply CLT panel. The final panel dimensions were 404 mm by 676 mm by 175 mm for the 5-ply CLT panel.

The panels were manufactured using a Dieffenbacher laboratory hydraulic press (Fig. 2). The pressing parameters

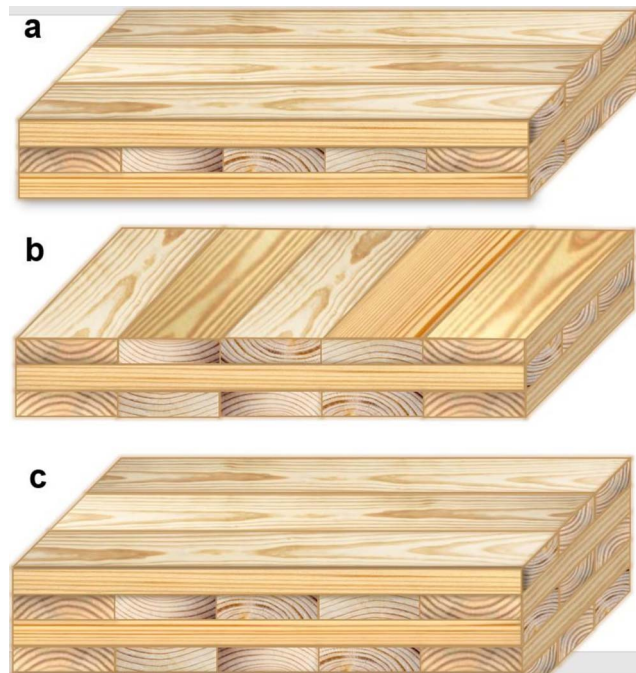


Figure 1.—Cross-laminated timber (CLT) panel configurations: (a) 3-ply, longitudinal, LT, (b) 3-ply, crosswise, CS, and (c) 5-ply, longitudinal, LT.

for the panels follow: Step 1—Press 0.207 MPa for 1 minute, Step 2—Press 0.345 Mpa for 1 minute, Step 3—Press 0.689 Mpa for 180 minutes, and Step 4—Press 0.345 Mpa for 0.5 minutes. A total of 27 panels were manufactured (10 CLT panels for Configuration 1, 10 CLT panels for Configuration 2, and 7 CLT panels for Configuration 3). The panels were sorted into three treatment groups and stored indoors until they were sent out for preservative treatment at the treating facilities.



Figure 2.—3-ply cross-laminated timber (CLT) panels in Dieffenbacher laboratory hydraulic press.

Preservative treatment of CLT

The three groups were as follows: copper azole – type C (CA-C) treatment group, micronized copper azole (MCA) treatment group, and an untreated (control) group. Eleven CLT panels (4 panels for Configuration 1, 4 panels for Configuration 2, and 3 panels for Configuration 3) were treated at Deforest Wood Preserving (1400 Industrial Drive, Bolton, Mississippi) for the CA-C. Eleven CLT panels (4 panels for Configuration 1, 4 panels for Configuration 2, and 3 panels for Configuration 3) were treated at Koppers Performance Chemicals (1016 Everee Inn Road, Griffin, Georgia) for MCA. Two panels each were left untreated as controls for Configurations 1 and 2, respectively. One panel was left untreated as a control for Configuration 3. There were no water-treatment control samples used in this study. The sampling design is similar to that reported in a previous publication (Ayanleye et al. 2023).

The panels were treated at the treating facilities according to 2-inch-thick lumber protocols through a modified full-cell process. The modified full-cell process consisted of a shorter vacuum time than the normal full-cell process, which has an initial vacuum of ≥ 30 minutes. The target retention was 2.4 kg/m^3 for the panels. This retention level is specified by the AWWPA U1-22 for UC4A (ground contact or freshwater) applications (AWPA 2022). Table 2 shows the preservative treating cycles as reported by the treating facilities. Both facilities confirmed the target retention of 2.4 kg/m^3 . After preservative treatment, the CA-C and MCA panels were allowed to air dry for 1 and 14 days, respectively, before being transported back to our laboratory. The treated and untreated panels were placed under the breezeway at the Franklin Center at Mississippi State University Department of Sustainable Bioproducts to air dry or absorb moisture as the case for the untreated panels (Fig. 3).

Block shear test method

Specimens were air-dried following treatment. Each CLT panel was cut into 15 square blocks measuring 133 mm by

Table 2.—Preservative treating cycle as reported by treating facility.

Parameters	Preservative treatment ^a	
	CA-C	MCA
Initial vacuum (in.-Hg)	18	18
Hold time (minutes)	3	5
Pressure (MPa)	1.07	1.03
Hold time (minutes)	11	15
Final vacuum (in.-Hg)	20	26

^a CA-C is copper azole-type; and MCA is micronized copper azole-type.

133 mm by 104 mm for 3-ply panels and 133 by 133 mm by 175 mm for 5-ply panels (Fig. 4).

Shear block specimens were prepared by referencing ASTM D2559 (2018). For each panel configuration, the shear block specimens were cut from five different locations (1, 2, 7, 8, 15), representing the corner, center, and edge of the panels (Fig. 4). The shear block specimens were stair-stepped with a shearing area of 51 mm by 38 mm (Figs. 5a and 5b). The shear block specimens were conditioned at 21°C and 65 percent relative humidity (RH) for ≥ 5 months before testing.

The tests were carried out per ASTM D905 (2021). The experimental setup is shown in Figure 6. The shearing tool applied a force through adjacent laminations at a rate of 5.08 mm/min until failure. Images of the failure shear plane were scanned using a Canon CanoScan LiDe 400 scanner, which has a maximum optical resolution of 4,800 by 4,800 dots per inch (dpi). The shear plane was analyzed using ImageJ2, an image processing software. This method has been used by other researchers in estimating wood failure (Lim et al. 2020, Alade et al. 2022). Block shear strength (BSS; f_v) was calculated as follows:



Figure 3.—Cross-laminated timber (CLT) panels under breezeway air-drying.

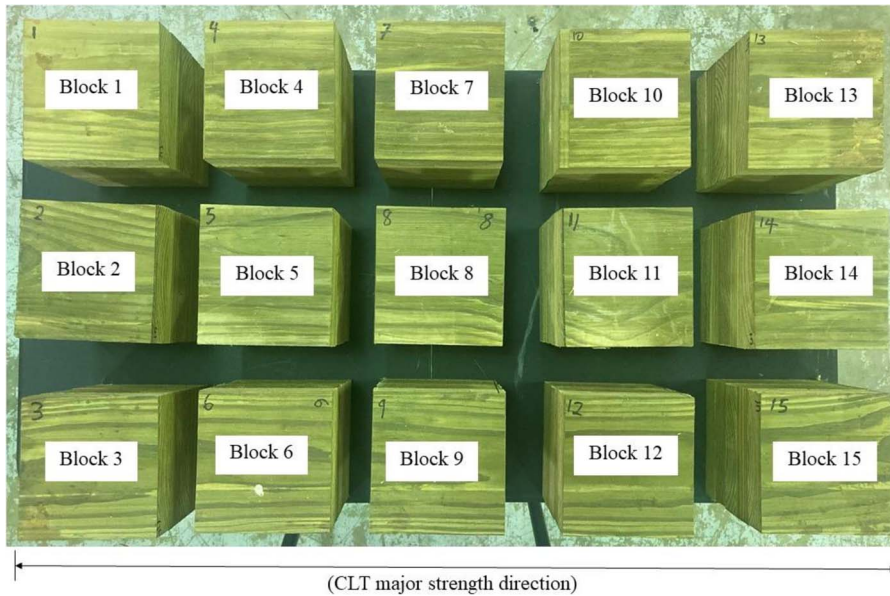


Figure 4.—One cross-laminated timber (CLT) panel cut into 15 square blocks.

$$f_v = F_u/A \quad (1)$$

where F_u = failure load (N) and A is the sheared area (mm^2). The percentage of wood failure (WFP) on the shear block failure plane was also measured using ImageJ2 software (Rueden et al. 2017). The shear blocks were analyzed for the modes of failure: adhesive failure (AD), failure parallel-to-grain (PAR), and failure perpendicular-to-grain (PER, rolling shear). The WFP was measured by dividing the wood failure area by the tested shear bonded area.

Delamination test method

Delamination specimens were prepared by referencing ASTM D2559 (2018). For each panel configuration, delamination specimens were cut from five different locations (1, 2, 7, 8, 15; similar to the sampling protocol for the shear block specimens). The delamination specimens were cut to 76 mm by 127 mm for all three configurations

(Figs. 5a and 5b). The delamination specimens for Configurations 1 and 2 were cut to the same orientation as shown in Figure 5a. The delamination specimens were conditioned at 21°C and 65 percent RH for ≥ 5 months. Three sides of each specimen were digitized using a Canon CanoScan LiDE 400 scanner, which has a maximum optical resolution of 4,800 by 4,800 dpi.

The delamination specimens were weighed to the nearest 1 g before testing. The specimens were placed in the wire mesh basket. The wire mesh basket was then placed in the pressure vessel. The pressure vessel was sealed and filled with water at a temperature of 21°C and placed under vacuum for 5 minutes at 0.207 Mpa. After the vacuum was released, the specimens were placed under air pressure for 1 hour at 0.517 Mpa. The vacuum–pressure cycle was repeated, the specimens had to increase in weight by ≥ 50 percent. The specimens were removed from the pressure vessel and placed in the oven at 65°C for approximately 22 hours with the test bond lines parallel to the airflow

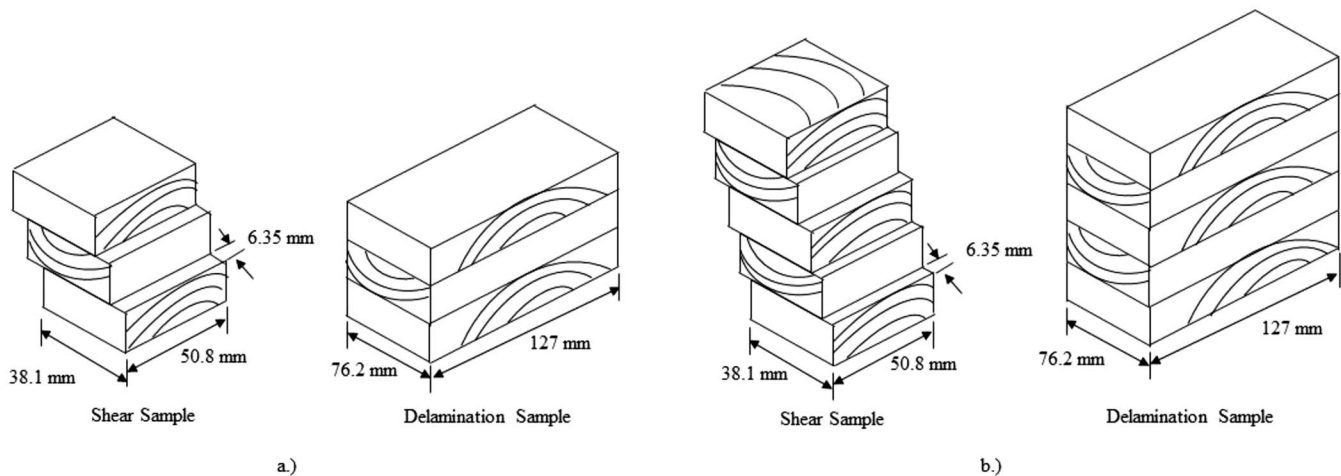


Figure 5.—(a) 3-ply cross-laminated timber (CLT) shear and delamination specimens; and (b) 5-ply CLT shear and delamination specimens.



Figure 6.—Block shear specimen in block shear test setup.

in the oven. The specimens were dried to within 15 percent of their original weights. The specimens were placed back into the wire mesh basket and placed back in the pressure vessel and sealed to be subjected to steam at 100°C for 90 minutes. The pressure vessel was cooled by flushing with tap water at 21°C until the temperature thermocouple on the pressure vessel displayed ambient temperature. The pressure vessel was then filled with water and placed under air pressure for 40 minutes at 0.517 Mpa. The specimens were then removed from the pressure vessel and placed back in the drying oven at 65°C as described above. After returning to within 15 percent of their original weight, the specimens were placed back in the pressure vessel for a repeat of Day 1 vacuum–pressure cycles. The specimens were removed from the pressure vessel and placed back in the oven. After drying to within 15 percent of the original weight, the delamination was measured along the test bond lines, and recorded. Knots, grade defects, and wood failure were excluded from delamination measurements. Figure 7

shows the bondlines of the delamination samples, which were labeled across the length (labeled “A” and “B” for 3-ply; labeled “A,” “B,” “C,” and “D” for 5-ply). Delamination was measured immediately after the specimens underwent cycles of vacuum, soaking, and oven-drying procedures as specified in ASTM D2559 (2018).

The total delamination $Delam_{tot}$ of a test piece was calculated as follows:

$$Delam_{tot} = 100 (l_{tot, delam}) / (l_{tot, glue line}) \text{ in } \% \quad (2)$$

where $l_{tot, delam}$ = the total delamination length and $l_{tot, glue}$ = the sum of the glue lines for five specimens in a panel.

Statistical Analysis

The effects of panel layup (3-ply parallel, 3-ply perpendicular, and 5-ply parallel) and preservative treatment (untreated,

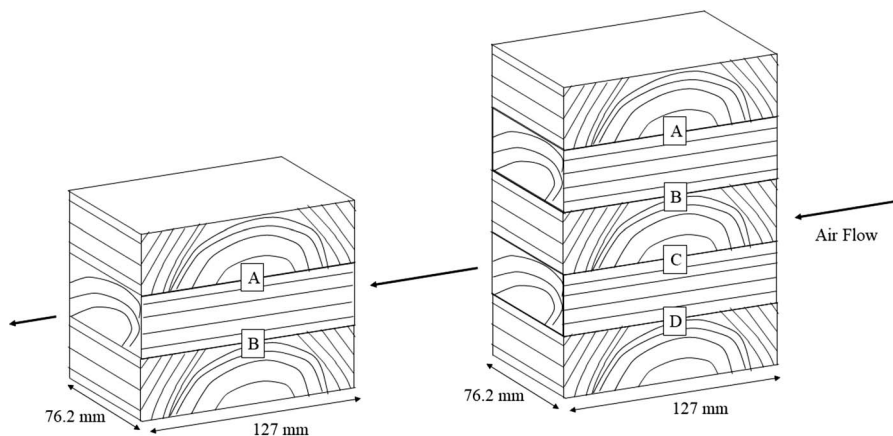


Figure 7.—Orientation of the delamination test specimen during the oven-drying procedure. A and B denote two bond lines for 3-ply cross-laminated timber (CLT). A, B, C, D denote four bond lines for 5-ply CLT.

Table 3.—Descriptive statistics of BSS (block shear strength) and WFP (wood failure percentage) for nine cross-laminated timber (CLT) groups.

CLT group ^a	Sample size	BSS (MPa)		BSS (MPa) COV (%)	WFP (%)		No. <75% WFP ^c
		Mean [95% CI ^b]	Median [range]		Mean	Median [range]	
1C	20	4.44 [3.95–4.93]	4.31 [3.03–7.52]	25.64	78.75	85.00 [20.00–100.00]	8
1CAC	40	3.26 [2.83–3.69]	3.27 [0.37–5.10]	30.45	79.13	80.00 [30.00–100.00]	13
1MCA	40	2.94 [2.54–3.34]	2.90 [0.62–4.60]	30.79	74.00	77.50 [5.00–100.00]	17
2C	20	3.08 [2.81–3.35]	2.84 [2.17–4.73]	20.27	85.00	100.00 [30.00–100.00]	5
2CAC	40	3.38 [3.04–3.72]	3.15 [1.96–6.36]	22.76	66.00	65.00 [15.00–100.00]	28
2MCA	40	2.93 [2.44–3.42]	3.02 [0.10–5.64]	37.76	70.25	72.50 [5.00–100.00]	20
3C	20	3.13 [2.65–3.61]	3.42 [0.53–5.52]	34.79	68.75	70.00 [0.00–100.00]	11
3CAC	40	2.78 [2.52–3.04]	2.81 [1.44–4.05]	21.18	81.00	80.00 [45.00–100.00]	13
3MCA	40	3.01 [2.69–3.33]	2.93 [1.90–4.76]	24.53	81.37	95.00 [30.00–100.00]	14

^a CA-C is copper azole-type; and MCA is micronized copper azole-type. 1C is Configuration 1 Control, 1CAC is Configuration 1 CA-C, 1MCA is Configuration 1 MCA, 2C is Configuration 2 Control, 2CAC is Configuration 2 CA-C, 2MCA is Configuration 2 MCA, 3C is Configuration 3 Control, 3CAC is Configuration 3 CA-C, 3MCA is Configuration 3 MCA.

^b CI—confidence interval.

^c Number of specimens.

CA-C treated, and MCA treated) on the bonding performance (block shear strength and wood failure percentage) and durability (delamination) was studied in posttreated CLT panels. Shapiro-Wilk test and Levene’s test were used to test for normality and homogeneity of variance on the raw data, respectively. If the assumptions of normality and homogeneity of variance were not met, the data were transformed and retested. The Kruskal-Wallis *H* test, a nonparametric equivalent of ANOVA (analysis of variance) was used to analyze the significance of the main effects if data could not be normalized after transformation. If the main effects proved to be significant, then Dunn’s pairwise test for multiple comparisons was used to compare observation groups. If assumptions of normality and homogeneity of variance were satisfied, a one-way ANOVA and the Tukey Honestly Significant Difference (HSD) test were performed for mean separation within the main effects (Lim et al. 2020). The data were analyzed using SAS 9.4 at a 5 percent significance level (SAS Institute 2016).

Results and Discussion

Panel air drying

Before treatment, the MC% of all CLT panels was 12 percent, which is within the target MC range for the adhesive specifications (Purbond X602). The average moisture content (MC%) of the panels after returning from the treaters was >85 percent. The CLT panels were inspected and weighed upon being returned from the treating facilities. There was no noticeable delamination in the gluelines. The average equilibrium moisture content (EMC) conditions to which the panels were subjected at Franklin Center at Mississippi State University was approximately 17 percent (measured from March 28, 2021 to July 30, 2021). After treatment, the air-dried MC% of the treated CLT panels for both CA-C and MCA after 4 months was 25 percent, which was close to the estimated target of 20 percent. There was some noticeable delamination or shallow wood failure on some of the treated CLT panels as the panels dropped below the fiber saturation point. The MC% of untreated panels remained 12 percent, but these panels were placed outdoors under a protected roof along with the treated CLT panels in order to moisture-equilibrate at approximately 18 percent. The EMC of the CLT panels was based on weight. The

average original MC of each panel was based upon the average moisture content of each laminate used for that panel fabrication.

Block shear test

The descriptive statistics of the calculated BSS and WFP for the nine CLT groups are shown in Table 3. Even though the block shear test was conducted by referencing ASTM D2559 (2018), the BSS values were not compared with the standard requirement, which is based upon the parallel-to-grain shear strength of wood. The major governing failure mode for CLT shear blocks was shear perpendicular-to-grain (rolling shear), which is less than the parallel-to-grain direction shear (Gong et al. 2016). The average BSS of the control samples ranged from 3.13 Mpa to 4.44 Mpa with coefficients of variation (COVs) ranging from 20.27 percent to 34.79 percent. These results are similar to those reported by other researchers on the bonding performance of CLT (Sharifnia and Hindman 2017, Lim et al. 2020). The CA-C- and MCA-treated samples had lower measured BSS values than did the control group for Configuration 1. For Configurations 2 and 3, the BSS values were similar for the control group and the CA-C and MCA groups. The average WFP ranged from 66 percent to 85 percent. The only control group that had a WFP <75 percent was Configuration 3. Overall, about 129 out of 300 block shear test specimens had WFPs <75 percent. Approximately 60 percent of the block shear specimens passed the minimum WFP requirement of 75 percent per ASTM D2559 (2018). Of those 129 block shear specimens that fell short of 75 percent WFP (Table 3), most of the specimens were close to meeting the minimum WFP requirements (88 had WFP between 50% and 75%).

Effect of preservative treatment.—For the 3-ply parallel configurations, a one-way ANOVA was used to compare the effects of preservative treatment on the mean BSS values because the data sets passed the normality and equality of variance tests. For the 3-ply cross configurations and the 5-ply parallel configurations, the data sets passed the normality tests but failed to pass the equality of variance tests ($P = 0.0051$ for 3-ply cross configurations and $P = 0.0137$ for 5-ply configurations); therefore, the mean BSS ranks

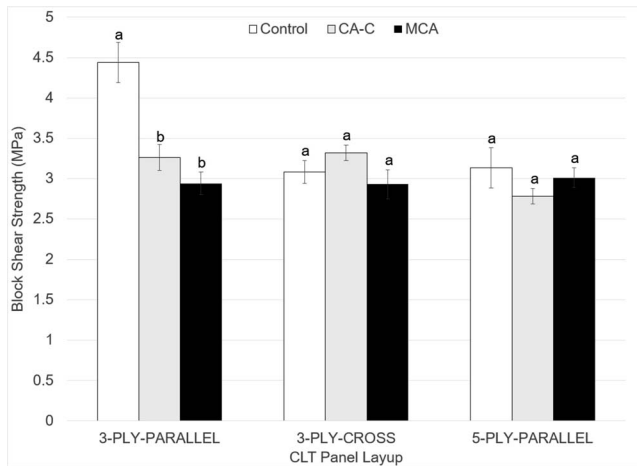


Figure 8.—Mean block shear strength of the cross-laminated timber (CLT) treatment by panel layout. Bars represent standard error; different letters above the bars indicate significant differences ($P < 0.05$) among the treatment means for within-panel layout. For pairwise comparisons, Tukey HSD was used for 3-ply parallel configuration; and Dunn's test with P values adjusted by the Bonferroni correction was used for 3-ply cross and 5-ply parallel configurations.

were tested using the Kruskal-Wallis H test. The only configuration group that was influenced by preservative treatment was the 3-ply parallel configuration ($P < 0.0001$). The mean BSS value for the untreated controls was statistically higher than the mean BSS values for the CA-C and MCA treatments (Fig. 8). There was no difference between the BSS values for the preservative treatments for the 3-ply cross configuration and the 5-ply parallel configuration ($P = 0.1286$ and $P = 0.1986$, respectively). PUR adhesives have a strong affinity to water, so extra care must be taken during storage to prevent exposure to moisture, which could cause premature gelling of the adhesive (Shirmohammadi 2023) This could explain the higher BSS values for the untreated specimens in 3-ply parallel configuration because these were the initial panels fabricated. This is further seen in the effects of the panel layout section in which the BSS of 3-ply parallel configuration control (untreated) specimens were significantly higher than the control specimens for 3-ply cross and 5-ply parallel configurations but no difference between 3-ply cross and 5-ply parallel configurations. Table 1 shows that the SG of the laminates used for the 3-ply parallel configurations was higher than the laminates used in the 3-ply cross and 5-ply parallel configurations. Studies have shown that the BSS of treated CLT could be lower than untreated CLT or higher, but this is when the panels are fabricated using preservative-treated laminates (Lim et al. 2020, Adnan et al. 2021). Dias et al. (2020) reported that the BSS was slightly lower for glued laminated timber specimens treated after gluing as compared with panels fabricated from preservative-treated lumber. The study showed some delamination in some of the glue lines during the drying process after preservative treatment. The adhesives used in that study were PRF and melamine urea-formaldehyde (MUF), while in our study the adhesive was PUR. PUR adhesives are prone to be more influenced by moisture than are PRF and MUF adhesives. The superficial delamination in the glue line during the drying process could have

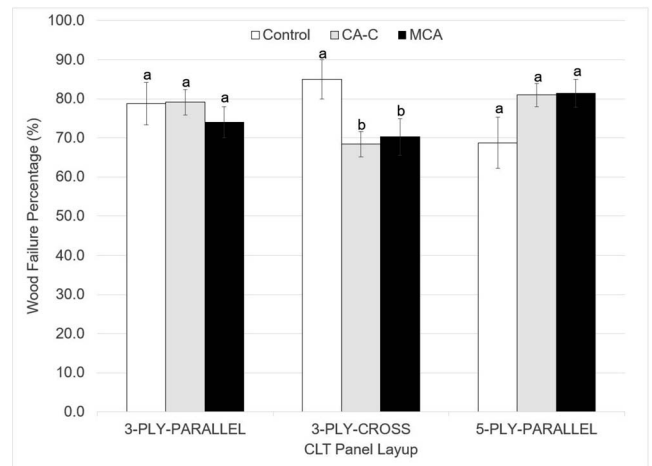


Figure 9.—Mean wood failure percentage of cross-laminated timber (CLT) treatment by panel layout. Bars represent standard error, different letters above the bars indicate significant differences ($P < 0.05$) among the treatment means for within-panel layout. For pairwise comparisons, Dunn's tests with P values adjusted by the Bonferroni correction was used for all three configurations.

influenced the results in that study. Some superficial delamination was noticed in our study, and it seems that the BSS was not significantly influenced by the preservative treatments for 3-ply cross and 5-ply parallel configurations. The SG of the laminates could have influenced the results. The SG of the laminates used for different configurations showed some differences, which ranged from 0.44 to 0.53; therefore, the amount of preservative penetration within the CLT panel could have influenced the BSS as shown in the study by Lim et al. (2020). Ayanleye et al. (2023) showed that the preservative penetration is dependent upon panel orientation, preservative formulation (CA-C and MCA), and the location within the panel.

To compare the effects of preservative treatment on the WFP, the Kruskal-Wallis H test was used because all the data sets failed to pass the normality and the equality of variance tests. The effect of preservative treatment on the WFP of specimens was observed in the 3-ply cross configuration where the untreated control samples showed a significantly higher percentage of wood failure at 85 percent, while the CA-C and MCA treatments showed lower wood failure of approximately 70 percent. Moreover, there was no significant influence of preservative treatment on the WFP of the 3-ply parallel and the 5-ply parallel configurations, with the WFP ranging from 70 percent to 80 percent (Fig. 9). The differences in wood failure percentage for the 3-ply cross configuration could have been influenced by the difference in SG of the laminates. The average SG was 0.51 with a COV of 3.9 percent for the 3-ply cross untreated samples, while the average SG for the 3-ply cross CA-C and MCA samples was 0.48 with a COV of 9.3 percent and 0.47 with a COV of 7.2 percent, respectively. The higher SG for the 3-ply cross configurations as compared with the 3-ply cross CA-C and MCA could have allowed deeper penetration of the adhesive, which would require confirmation with other scientific techniques such as light microscopy. WFP reported by Lim et al. (2020) on CLT panels fabricated with MCA-C-treated laminates glued with PUR adhesive showed no significant difference

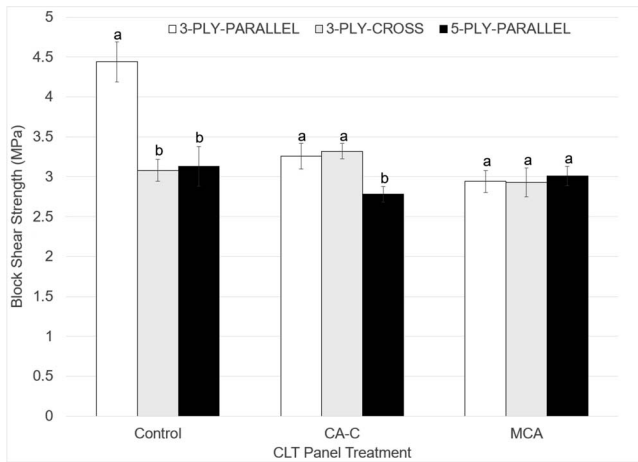


Figure 10.—Mean block shear strength of cross-laminated timber (CLT) panel layout and thickness by preservative treatment. Bars represent standard error; different letters above the bars indicate significant differences ($P < 0.05$) among the panel layout and thickness within treatments. For pairwise comparisons, Tukey HSD was used for untreated controls and the micronized copper azole-type (MCA) treatment; while Dunn's test with P values adjusted by the Bonferroni correction was used for copper azole-type (CA-C) treatment.

between treated and untreated panels with a mean WFP >89 percent. The high levels of wood failure for our results shows that the BSS was heavily dependent upon the shearing strength of the laminates. Another factor to consider was that we had more observations for the treated specimens than the control specimens.

Effect of panel layout.—For the untreated control samples and the MCA-treated samples, a one-way ANOVA was used to compare the effects of panel layout on mean BSS values because the data sets passed the normality and equality of variance tests. For the CA-C-treated samples, the data set passed the normality tests, but failed to pass the equality of variance tests; therefore, the mean BSS ranks were tested using the Kruskal-Wallis H test. The results showed that the control samples and the CA-C were influenced by the layout of the panels ($P = 0.0001$ and $P = 0.0011$, respectively). For the control samples, the 3-ply parallel sample measurements demonstrated a significantly higher mean BSS than did the 3-ply cross and the 5-ply parallel. For the CA-C samples, the 5-ply parallel sample measurements demonstrated a significantly lower mean BSS than did the 3-ply parallel and 3-ply cross. The mean BSS of the MCA-treated samples were not significantly influenced by the panel layout ($P = 0.0756$; Fig. 10). As mentioned before with the control samples and the CA-C samples being influenced by panel layout, the PUR adhesive may have been compromised during the CLT fabrication process.

The WFP of the CA-C samples were the only ones influenced by the layout of the panels. The 3-ply cross panel measurement showed significantly lower WFP than those of the 3-ply parallel and the 5-ply parallel. The WFP for the control and the MCA samples were not significantly influenced by the panel layout (Fig. 11). The SG of the laminates could not explain why the 3-ply cross panel measurements showed a significantly lower WFP than did the 3-ply parallel and the 5-ply parallel samples.

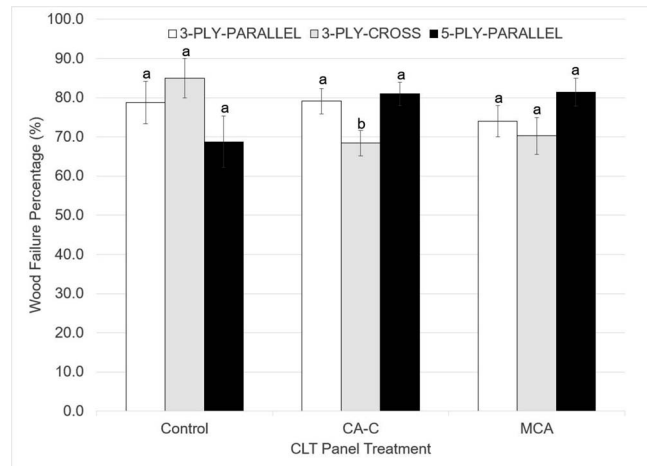


Figure 11.—Mean wood failure percentage of cross-laminated timber (CLT) panel layout and thickness by preservative treatment. Bars represent standard error, different letters above the bars indicate significant differences ($P < 0.05$) among the panel layout within treatments. For pairwise comparisons, Dunn's test with P values adjusted by the Bonferroni correction for all three treatments.

Failure modes.—The three failure modes recognized for the block shear tests are shown in Figure 12. AD (adhesive failure) occurred when the adhesive to wood bond shear strength was weaker than the wood shear strength. PER (perpendicular-to-grain [rolling shear]) and PAR (parallel-to-grain) occurred when the adhesive bond was stronger than the wood. Most of the specimens ($\geq 50\%$) had PER failure because the shear strength of wood is significantly lower perpendicular to the grain as compared with parallel to the grain (Kretschmann 2010). The untreated control groups had the smallest percentage of adhesive failure as the controlling failure mode. Table 4 list a breakdown of the observed controlling failure modes for each specimen. These results are similar to failure modes described by Lim et al. (2020).

Delamination test

Delamination is defined as a separation of layers at the interface between the adhesive and the adherent (Gong et al. 2016). Delamination in adhesive joints is influenced by internal stresses that are produced from dimensional changes during the shrinking and swelling of the adherents. Wood is orthotropic, so the dimensional changes are dependent upon its grain orientation. The shrinkage and swelling of wood in the tangential and radial directions are significantly more than in the longitudinal direction (Fig. 13). The criteria for delamination were based upon the ASTM D2559 standard, which includes shallow wood failure as delamination.

Table 5 shows the results of the delamination test. The delamination rates ranged from 0.0 percent (5-ply parallel Glues Line C and D, control specimens) to 25.8 percent (3-ply cross Glue Line A, control specimens). The untreated control specimen measurements showed the smallest average delamination rate.

On average the preservative treatment increased the delamination rate; the CA-C and MCA treatment measurements showed higher delamination rates than did the control specimens for the 3-ply parallel and the 5-ply parallel configurations. For the 3-ply cross configurations the average delamination

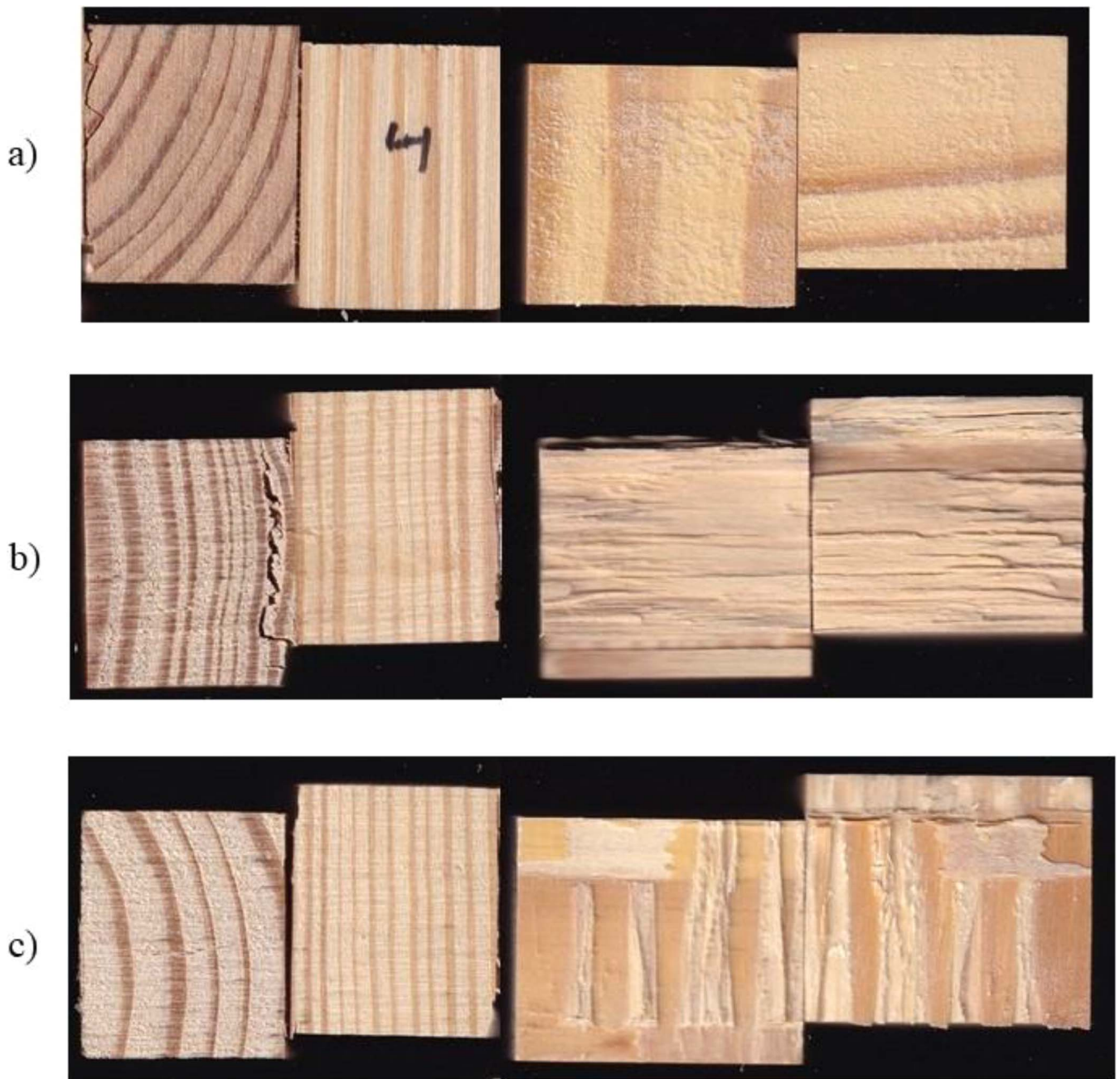


Figure 12.—Failure modes of block shear specimens: (a) AD—adhesive failure, (b) PER—perpendicular-to-grain failure (rolling shear), (c) PAR—parallel-to-grain failure.

rates were similar for the control specimens and the CA-C and MCA specimens. The delamination specimens for the 3-ply cross configuration (Configuration 2) were prepared the same way as the 3-ply parallel configuration (Configuration 1), as shown in Figure 7. The delamination specimens cut from locations 1 and 7 of Configuration 1 share the same surface laminations, which governs the dimensional changes during delamination tests. The delamination specimens cut from locations 1 and 7 of Configuration 2 do not share the same surface laminations. Configuration 2 results could have been more representative of delamination behavior. Lim et al. (2020) reported an increase in the delamination rate for CLT panels constructed with a PUR adhesive and treated with copper-based preservative treatment as compared with untreated panels. The CA-C-treated panels on average had a

better delamination rate than did the MCA-treated panels. Dias et al. (2020) presented data that showed less delamination for glued-laminated timber treated after gluing as compared with panels fabricated from preservative-treated laminates.

The 5-ply parallel configuration measurements showed a better average delamination rate than did the 3-ply panels across the three treatment combinations. This is in contrast to results reported by Knorz et al. (2017), who reported that the delamination rate increased when comparing a 3-ply and 7-ply spruce CLT panel. The rationale for this difference in their study was that it takes longer to produce the 7-ply panel so that could have influenced the bonding conditions.

Based on the results, we found that preservative treatment negatively influenced the delamination results except for

Table 4.—Block shear test results by controlling failure mode.

Cross-laminated timber group ^a	No. of observations (mean BSS in MPa) ^b				
	AD	PAR	PER	PAR/PER	Total
1C	3 (4.90)	4 (4.64)	13 (4.27)	0 (N/A)	20 (4.44)
1CAC	6 (3.46)	9 (3.45)	24 (3.06)	1 (5.10)	40 (3.26)
1MCA	12 (2.72)	4 (3.33)	24 (2.99)	0 (N/A)	40 (2.94)
2C	3 (2.85)	0 (N/A)	17 (3.12)	0 (N/A)	20 (3.08)
2CAC	10 (3.45)	6 (3.56)	24 (3.21)	0 (N/A)	40 (3.38)
2MCA	13 (3.11)	7 (3.01)	19 (2.79)	0 (N/A)	40 (2.93)
3C	5 (2.11)	1 (2.63)	12 (3.65)	2 (2.77)	20 (3.13)
3CAC	6 (2.83)	5 (2.84)	29 (2.75)	0 (N/A)	40 (2.78)
3MCA	10 (3.24)	6 (3.38)	22 (2.76)	2 (3.54)	40 (3.01)

^a CA-C is copper azole-type; and MCA is micronized copper azole-type. 1C is Configuration 1 Control, 1CAC is Configuration 1 CA-C, 1MCA is Configuration 1 MCA, 2C is Configuration 2 Control, 2CAC is Configuration 2 CA-C, 2MCA is Configuration 2 MCA, 3C is Configuration 3 Control, 3CAC is Configuration 3 CA-C, 3MCA is Configuration 3 MCA.

^b AD—adhesive failure; PAR—parallel-to-grain wood failure; PER—perpendicular-to-grain wood failure; BSS—block shear strength.

the 3-ply cross configuration. A possible explanation could be the moisture-induced adhesion failure as a result of the treatment. The vacuum impregnation of the CLT panels increased the MC from 12–15 percent to approximately 85 percent MC; and the severe swelling of the panels during treatment might have imposed high stress on the bond line, thus leading to the increased delamination observed in the treated CLT. This statement holds true for the 3-ply parallel and 5-ply parallel configurations; however, the effect of preservative treatment was not significant in the case of 3-ply crosswise configuration. Without a deeper understanding of the preservative dispersion in the 3-ply parallel configuration and the 3-ply crosswise configuration, there is no explanation for the difference.

Conclusions

The effect of the MCA and CA-C preservative treatment and CLT panel layup on the bonding performance of posttreated SYP CLT panels manufactured using a one-component PUR adhesive was investigated by conducting

Table 5.—Summary of delamination test results.

Cross-laminated timber group ^a	Bondline	Bondline delamination ^b	Bondline length ^c	Delamination rate ^d (%)
		(mm)	(mm)	
1C	A	219.1	2,540	8.6
	B	139.7	2,540	5.5
1CAC	A	727.1	5,080	14.3
	B	266.7	5,080	5.3
1MCA	A	974.7	5,080	19.2
	B	995.4	5,080	19.6
2C	A	654.1	2,540	25.8
	B	301.6	2,540	11.9
2CAC	A	871.5	5,080	17.2
	B	808.0	5,080	15.9
2MCA	A	800.1	5,080	15.8
	B	1162.1	5,080	22.9
3C	A	74.6	1,270	5.9
	B	25.4	1,270	2.0
	C	0.0	1,270	0.0
	D	0.0	1,270	0.0
3CAC	A	509.6	2,540	20.1
	B	303.2	2,540	11.9
	C	195.3	2,540	7.7
	D	388.9	2,540	15.3
3MCA	A	376.2	2,540	14.8
	B	331.8	2,540	13.1
	C	290.5	2,540	11.4
	D	134.9	2,540	5.3

^a CA-C is copper azole-type; and MCA is micronized copper azole-type. 1C is Configuration 1 Control, 1CAC is Configuration 1 CA-C, 1MCA is Configuration 1 MCA, 2C is Configuration 2 Control, 2CAC is Configuration 2 CA-C, 2MCA is Configuration 2 MCA, 3C is Configuration 3 Control, 3CAC is Configuration 3 CA-C, 3MCA is Configuration 3 MCA.

^b Sum of delamination length on two sides of all specimens for each bond line.

^c Sum of bond line length on two sides of all specimens for each bond line.

^d Bond line delamination divided by total bond line multiplied by 100.

block shear and delamination tests. The only configuration group for BSS that was influenced by preservative treatment was the 3-ply parallel configuration. The 3-ply parallel configurations were the first to be fabricated, so the PUR adhesive strength may have been compromised.

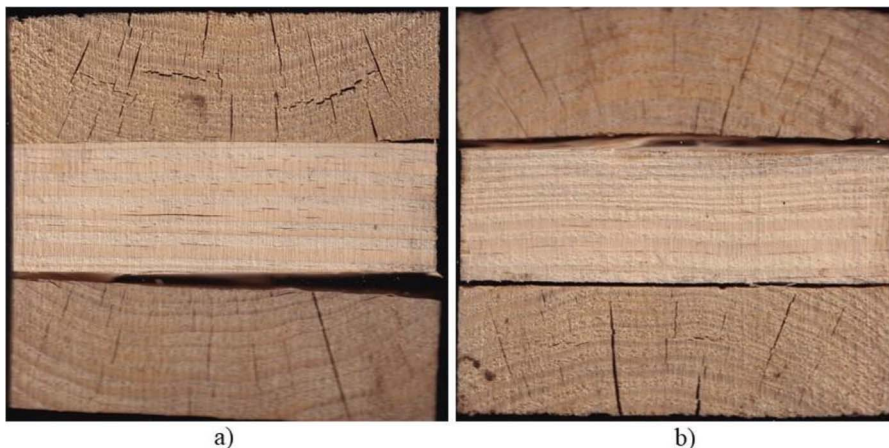


Figure 13.—Wood laminates after accelerated weathering cycles: (a) dimensional changes out of plane and (b) dimensional changes in-plane.

Shirmohammadi (2023) stated that care must be taken in the storage of PUR adhesives to prevent gelling of the adhesive because of its affinity for moisture. There was statistically no significant difference between the treatments within the 3-ply cross and the 5-ply parallel configurations for BSS. The mean WFP was >70 percent for all the configurations except for the 3-ply cross configuration and the 5-ply parallel configuration. The panel layup influenced the BSS, with the 5-ply parallel configuration measured as having lower BSS. It should be noted that the locations and the amount of preservative penetration within the panels may have influenced the results in this study. Correlating the preservative penetration with BSS and delamination rate could account for some of the variability in the results. The perpendicular to grain was the major failure mode observed for the block shear samples. Most of the specimens had some amount of delamination. The delamination rate could have been lower if shallow wood failure was not counted as delamination. Based upon the results of this study, which showed WFP to be ≥ 70 percent for the treated samples for both CA-C- and MCA-treated specimens, the potential in posttreating SYP CLT panels after fabrication is feasible. The issue of being able to treat a commercial-sized CLT at a commercial treating facility still has to be addressed in future studies.

Acknowledgments

This research was funded by a Wood Innovation Grant from the USDA Forest Service. In accordance with Federal law and U.S. Department of Agriculture policy, this institution is prohibited from discriminating on the basis of race, color, national origin, sex, age or disability. (Not all prohibited bases apply to all programs.) To file a complaint of discrimination: write USDA, Director, Office of Civil Rights, Room 326-W, Whitten Building, 1400 Independence Avenue, SW, Washington, D.C. 20250-9410 or call (202) 720-5964 (voice and TDD). USDA is an equal opportunity provider and employer. This publication is a contribution of the Forest and Wildlife Research Center, Mississippi State University. This paper was approved as journal article SB1109 of the Department of Sustainable Bioproducts, MSU. This publication is a contribution of the Forest and Wildlife Research Center, MSU.

Literature Cited

Adnan, N. A., P. Tahir, H. Husain, S. H. Lee, M. K. A. Uyup, M. N. M. Arip, and Z. Ashaari. 2021. Effect of ACQ treatment on surface quality and bonding performance of four Malaysian hardwoods and cross laminated timber (CLT). *Eur. J. Wood Wood Prod.* 79:285–299. <https://doi.org/10.1007/s00107-020-01609-7>

Alade, A. A., Z. Naghizadeh, and C. B. Wessels. 2022. A new method for estimating wood failure percentage in adhesive-bonded shear specimens. *Int. J. Adhes.* 112:103028. <http://doi.org/10.1016/j.ijadhadh.2021.103028>

American Society for Testing and Materials (ASTM). 2017. D2395. Standard Test Methods for Density and Specific Gravity (Relative Density) of Wood and Wood-Based Materials. ASTM International, West Conshohocken, Pennsylvania.

American Society for Testing and Materials (ASTM). 2018. D2559. Standard Specification for Adhesives for Bonded Structural Wood Products for Use Under Exterior Exposure Conditions. ASTM International, West Conshohocken, Pennsylvania.

American Society for Testing and Materials (ASTM). 2020. D4442. Standard Test Methods for Direct Moisture Content Measurement of Wood and Wood-Based Materials. ASTM International, West Conshohocken, Pennsylvania.

American Society for Testing and Materials (ASTM). 2021. D905. Standard Test Method for Strength Properties of Adhesive Bonds in Shear by Compression Loading. ASTM International, West Conshohocken, Pennsylvania.

American Wood Protection Association (AWPA). 2022b. American Wood Protection Association Book of Standards. Use Category System: User specifications for treated wood (U1-22). AWPA, Birmingham, Alabama, USA.

Ayanleye, S., K. Udele, V. Nasir, X. Zhang, and H. Militz. 2022. Durability and protection of mass timber structures: A review. *J. Build. Eng.* 46:103731. <https://doi.org/10.1016/j.job.2021.103731>

Ayanleye, S., F. Quin, X. Zhang, H. Lim, and R. Shmulsky. 2023. Preservatives penetration and retention in post-treated cross-laminated timber panels with different layup and thickness. *J. Build. Eng.* 67(5):106009. <https://doi.org/10.1016/j.job.2023.106009>

Brandner, R., G. Flatscher, A. Ringhofer, G. Schickhofer, and A. Thiel. 2016. Cross laminated timber (CLT): Overview and development. *Eur. J. Wood Wood Prod.* 74:331–351. <https://doi.org/10.1007/s00107-015-0999-5>

De Groot, R. C. and B. Woodward. 1999. Using copper-tolerant fungi to biodegrade wood treated with copper-based preservatives. *Int. Biodeterior. Biodegrad.* 44(1):17–27. [https://doi.org/10.1016/S0964-8305\(99\)00047-5](https://doi.org/10.1016/S0964-8305(99)00047-5)

Dias, A., C. Martins, and A. Dias. 2020. Influence of the treatment phase on the gluing performance of glued laminated timber. *BioResources* 15(3):5725–5736.

França, T. S. F. A., C. E. Stokes, J. D. Tang. 2020. Development of a modified standard termite test for mass timber products. *Wood and Fiber Science.* 54(1): 24–34.

Freeman, M. and C. McIntyre. 2008. A comprehensive review of copper-based wood preservatives. *Forest Prod. J.* 58(11):6–27.

Gong, Y., G. Wu, and H. Ren. 2016. Block shear strength and delamination of cross-laminated timber fabricated with Japanese larch. *BioResources* 11(4):10240–10250.

Knorz, M., S. Torno, and J. Kuilen. 2017. Bonding quality of industrially produced cross-laminated timber (CLT) as determined in delamination tests. *Construct. Build. Mater.* 133(2):219–225. <https://doi.org/10.1016/j.conbuildmat.2016.12.057>

Kretschmann, D. E. 2010. Chapter 5: Mechanical properties of wood. *In: Wood Handbook. Wood as an Engineer Material*, FPL-GTR-190. US Department of Agriculture, Forest Service, Forest Products Laboratory, Madison, Wisconsin. 508 pp. <https://doi.org/10.2737/FPL-GTR-190>

Lee, D., M. J. Lee, D. Son, and B. D. Park. 2006. Adhesive performance of woods treated with alternative preservatives. *Wood Sci. Technol.* 40:228–236. <https://doi.org/10.1007/s00226-005-0036-7>

Lim, H., S. Tripathi, and J. Tang. 2020. Bonding performance of adhesive systems for cross-laminated timber treated with micronized copper azole type C (MCA-C). *Constr. Build. Mater.* 232:1–10. <https://doi.org/10.1016/j.conbuildmat.2019.117208>

Nguyen, T., J. Li, and S. Li. 2012. Effects of water-borne rosin on the fixation and decay resistance of copper-based preservative treated wood. *BioResources* 7(3):3573–3584.

Rueden, C. T., J. Schindelin, M. C. Hiner, B. E. DeZonia, A. E. Walter, E. T. Arena, and K. W. Eliceiri. 2017. ImageJ2: ImageJ for the next generation of scientific image data. *BMC Bioinform.* 18:529. <https://doi.org/10.1186/s12859-017-1934-z>

SAS Institute. 2016. SAS Software, version 9.4. SAS Institute Inc., Cary, North Carolina.

Schulgasser, K. and A. Witztum. 2015. How the relationship between density and shrinkage of wood depends on its microstructure. *Wood Sci. Technol.* 49:389–401. <https://doi.org/10.1007/s00226-015-0699-7>

Sharifnia, H. and D. P. Hindman. 2017. Effect of manufacturing parameters on mechanical properties of southern yellow pine cross laminated timbers. *Construct. Build. Mater.* 156(12):314–320. <https://doi.org/10.1016/j.conbuildmat.2017.08.122>

Shirmohammadi, M. 2023. Study of the hygroscopic properties of three Australian wood species used as solid wood and composite products. *Eur. J. Wood Wood Prod.* (2023) 81:1495–1512.

- Southern Pine Inspection Bureau (SPIB). 2014. Standard Grading Rules for Southern Pine Lumber. Southern Pine Inspection Bureau, Pensacola, Florida.
- Townsend, T., B. Dubey, T. Tolaymat, and H. Solo-Gabriele. 2005. Preservative leaching from weathered CCA-treated wood. *J. Environ. Manag.* 75(2):105–113. <https://doi.org/10.1016/j.jenvman.2004.11.009>
- Taylor, A., M. Denavit, J. Lloyd, L.-W. Kim, Kirker, G., Mankowski, M. 2022. Borate treatment of CLT panels using vacuum: a proof of concept. *Forest Prod. J.* 73(1):24–30.
- Wang, J. Y., R. Stirling, P. I. Morris, A. Taylor, J. Lloyd, G. Kirker, S. Lebow, M. E. Mankowski, H. M. Barnes, and J. J. Morrell. 2018. Durability of mass timber structures: A review of the biological risks. *Wood Fiber Sci.* 50:119–127.