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Bonding performance of adhesive systems for cross-laminated timber treated with micronized copper azole type C (MCA-C)

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HIGHLIGHTS

- Cross-laminated timber (CLT) can be manufactured with preservative treated wood.
- Lumber treated with Micronized Copper Azole-Type C (MCA-C) was used as CLT lamstock.
- Bonding performance of CLT made using three adhesive systems were evaluated.
- Polyurethane adhesive appeared to be suitable for cross-laminating MCA-C treated lumber.

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ABSTRACT

The feasibility of manufacturing cross-laminated timber (CLT) from southern yellow pine (United States grown) treated with micronized copper azole type C (MCA-C) preservative was evaluated. Lumber (2x6 visually graded no. 2 boards) was treated to two retention levels (1.0 and 2.4 kg/m^3), planed to a thickness of 35 mm, and assembled along with an untreated control group using three adhesive systems following product specifications: melamine formaldehyde (MF), resorcinol formaldehyde (RF), and one-component polyurethane (PUR). Block shear and delamination tests were conducted to examine the bonding performance in accordance with ASTM D905 and ASTM D2559 Standards, respectively. One-way analysis of variance and Kruskal-Wallis H test were conducted to evaluate the effects of preservative retention and adhesive type on block shear strength (BSS) and wood failure percentage (WFP). Regardless of adhesive type, the 1.0 kg/m^3 retention treatment significantly lowered BSS compared to the untreated control. CLT composed of the laminations treated at 2.4 kg/m^3 maintained BSS when PUR and RF were used but not MF. The average WFP of each CLT configuration ranged from 89% to 99%. The untreated CLT specimens did not experience any delamination under accelerated weathering cycles. The delamination rates of the treated specimens assembled using MF and RF increased with the preservative retention level, while PUR provided delamination rates less than 1% to the laminations treated at both levels. These combined data suggest that, under the conditions tested, PUR provided overall better bonding performance than MF and RF for MCA-C treated wood.

1. Introduction

Cross-laminated timber (CLT) is an engineered wood product composed of layers of structural lumber assembled in directions perpendicular to adjacent layers. The product concept was first developed in Austria and Germany in the 1970s and 1980s [1]. After 30 years of further development through research and applications, CLT has successfully penetrated the European construction market and established an emerging market in North America [1]. A factor that has contributed to the increasing popularity of CLT for mid- and high-rise wood buildings is its flexible construction, which combines outstanding structural performance with aesthetic design and environmental sustainability. Economic savings are also achieved because onsite construction takes minimal time and space, while generating minimal waste.

In North America, CLT panels have the potential to complement the existing light frame and heavy timber options in indoor construction and has the capacity to replace concrete, steel, and masonry in some applications [2]. Looking to the future, the CLT

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market would be further expanded if it could be used for exterior applications such as balconies and ground-contact walls [3,4]. Wood, though, is a biological material that can be degraded in its end use condition when exposed to weather, decay fungi, and wood-attacking insects. Fortunately, there are effective methods that can be used to protect wood from degradation, one of which is to pressure treat wood with chemical preservatives.

Copper compounds have been used for more than two centuries for wood protection [5]. However, there are several species of brown-rot fungus that can tolerate high levels of copper [6]. Consequently, copper-based preservative systems traditionally combine copper with a co-biocide to increase efficacy against copper-tolerant fungi [5]. For example, two water-borne preservative systems marketed for residential applications of pressure treated wood in the USA are alkaline copper quat (ACQ) and copper azole (CA) [7]. In both these formulations, the copper component is dissolved in ethanolamine and/or ammonia in water [8]. When moisture content in the treated wood exceeds 15–18%, corrosion of metal fasteners occurs by reduction of cupric ions from the copper-based preservative in the treated wood [9]. Some newer particulate formulations are made with insoluble copper carbonate. The copper carbonate is milled into an emulsion of fine particles that becomes micronized or dispersed with surfactants in the water-borne preservative system. Studies comparing ACQ with MCQ (micronized copper quat) have shown that MCQ causes less corrosion to stainless steel and hot-dipped galvanized fasteners than ACQ [10,11]. As far as performance efficacy is concerned, particulate copper systems appear to perform as well as comparable ionic copper-based systems for protecting wood from biodegradation [12].

Treating wood with preservatives, however, can adversely affect adhesive bonding performance with wood at the submicroscopic [13,14] and microscopic [15] levels, which in turn can affect bonding strength and rates of delamination [16,17]. Most notably, the preservative can physically and chemically block surfaces where the intermolecular forces of bonding develop [13], reduce wettability of the wood, which reduces adhesion, penetration, and spreading of the adhesive [14,15] and alter adhesive cure rates [16,17]. The complexity of the multi-level interactions among preservative, adhesive, and wood species has led to a recurring theme in the literature that bond strength is best determined empirically. For example, Lee et al. [18] used ASTM Standard D905, a compressive dry shear test, to evaluate bond strength of two softwood species (Korean pine and Japanese larch) treated with four levels of waterborne preservatives (untreated; chromated copper arsenate, CCA; copper HDO, which has copper, boric acid, and HDO or N-cyclohexyldizeniumdioxide, CB-HDO; and CA) for each of four different adhesive systems (urea-melamine formaldehyde, UMF; melamine formaldehyde, MF; phenol formaldehyde, PF; and resorcinol formaldehyde, RF). The most obvious trend was that the highest dry shear strength values (4.05–5.69 MPa) were obtained with RF resin for all species and preservative treatment except for CCA-treated larch (2.78 MPa). All remaining combinations of resin, preservative treatment, and species exhibited shear strength values ranging between 0.52 and 2.18 MPa, with no evident trends observed.

The durability of bond strength, which refers to its environmental resistance to prolonged weather exposure, is another important consideration in extending treated laminate products to exterior applications. Lisperguer and Becker [17] used ASTM Standard D2559, which measures delamination after three harsh cycles of wetting and drying, to compare durability of bond strength between commercial and laboratory-modified phenol-resorcinol-formaldehyde (PRF) adhesive systems. Laminated specimens were made from radiata pine wood treated with chromated copper arsenate (CCA) preservative at two retentions (4 and 6 kg/m^3). Although both types of PRF adhesive systems performed similarly in dry shear strength tests with mean values>10 MPa, only the laboratory-modified system, which had a higher percentage of resorcinol (25% versus 16% for the commercial PRF) passed the minimum ASTM Standard D2559 requirements. These findings emphasize that although increasing resorcinol content did not change bond strength, it significantly increased bond strength durability.

The effects of preservative retention level on bond adhesion is another factor to consider because the amount of preservative in wood used in outdoor applications increases with the degree of environmental exposure. In many cases, retention has been found to exert little effect on bond strength or durability. In the aforementioned study by Lisperguer and Becker [17], retention level of CCA (4 and 6 kg/m^3) had no effect on bond durability. Vick et al. [19] also found no effects of retention (3.2, 6.41, and 9.61 kg/m^3) on PRF bond adhesion for aspen veneer treated with alkyl ammonium chloride alone or with copper, but did find increasing bond strength with increasing retention for alkyl ammonium chloride with carbamate, and decreasing bond strength with retention for ammoniacal copper zinc arsenate.

There is a lack of data on the effects of micronized copper-based preservatives on adhesive performance in wood. Therefore, the objective of this study was to evaluate the potential of using micronized copper azole – type C (MCA-C) treated southern yellow pine (SYP) wood for CLT production. Wood was treated with MCA-C to two retention levels (1.0 and 2.4 kg/m^3) and assembled along with an untreated control group using three adhesive systems (MF; RF; and polyurethane, PUR). MCA-C is in current commercial use to pressure-treat lumber for residential applications, and the three adhesives are in current use for manufacturing lumber-based composite products. Both block shear and delamination tests were conducted to examine the bonding performance of the CLT following ASTM Standards D905 [20] and D2559 [21] as guidelines, respectively. The ultimate goal of this research was to examine the feasibility of extending the use of CLT to exterior applications.

2. Material

Visually graded No. 2 SYP lumber (2 × 6 normal × 3 m long) were supplied by Shuqualak Lumber Co. located in Mississippi, which has design modulus of elasticity of 9,653 MPa [22]. Defect-free portions of flat-grained lumber were chosen following ASTM Standard D2559 [21] as a guideline, and cut to an average length of 1.5 m. Consistent specific gravity (SG) was expected considering the lumber selection criteria, which was measured after the block shear tests. Two hundred fifty-six pieces of the defect-free lumber were treated with MCA-C. The American Wood Protection Association (AWPA) Standard P62-16 lists the percent active ingredients of MCA-C as: 96.1% copper, 1.95% propiconazole, and 1.95% tebuconazole [23]. Boards were treated to two target MCA-C retention levels (1.0 and 2.4 kg/m^3) at a commercial facility (Tri-state lumber Company, Fulton, MS) using a modified full-cell process. These levels were chosen to meet the preservative treatment requirements for UC3B (exterior above ground, exposed or poor water runoff) and UC4A (ground contact or fresh water) applications specified by the AWPA Standard U1-18 [24]. The retention levels were confirmed by following AWPA Standard A9-18 for analysis of treated wood by X-ray spectroscopy [25]. Following ASTM Standard D4442 [26], MC of treated and untreated lumber was measured after kiln drying at dry bulb temperature of 100 °C and after indoor storage for more than one month, respectively (Table 1). The average MCs was within the optimum MC range of 12 ± 3% recommended in the CLT Handbook [27]. The untreated

Table 1
Moisture contents of untreated and treated lumber.

	Retention level (kg/m^3)		
	0	1.0	2.4
Sample size (n)	20	20	20
Mean (%)	11.09	9.32	9.63
COV (%)	7.86	5.64	8.28

and treated lumber was stored indoors for at least two weeks before CLT assembly.

Lumber was planed to a final thickness of 35 mm and width of 133 mm. The three-layered CLT panels were constructed using the laminations planed within 12 h and three commercial adhesive systems, MF (Hexion Cascomel™ 4720 with Wonderbond™ Hardener 5025A), RF (CASCOPHEN® Adhesive System G-1131) and one-component PUR (PURBOND® HB E452).

In total, nine CLT configurations were constructed, which were identified using two letters. The first letter indicated the adhesive type ("M" for MF, "R" for RF, and "P" for PUR resin), and the second letter represented the retention level ("C" for no treatment, "L" for the retention level of 1.0 kg/m^3, and "H" for the retention level of 2.4 kg/m^3). The CLT panels with dimensions of 667 mm in length, 400 mm in width, and 107 mm in thickness were manufactured using a Dieffenbacher laboratory hydraulic press. Two CLT panel replicates were constructed for each configuration using the manufacturing parameters described in Table 2 including single-face glue-spread rates, which were adopted from the adhesive product specifications [28–31]. Afterward, the CLT panels were conditioned at 65 ± 2% relative humidity and 21 ± 2 °C for at least 14 days before preparation of block shear and delamination test specimens.

3. Methods

3.1. Block shear test

For each configuration, stair-stepped CLT specimens (7 from one panel replicate and 8 from the other) with glue-bond areas of 51 mm × 38 mm (2 bond areas per specimen) were prepared in accordance with ASTM Standard D2559 [21], which made a total of 15 specimens and 30 bond areas tested per configuration. The test specimens were conditioned at 21 ± 2 °C and a relative humidity of 65 ± 5% for a minimum of 14 days prior to the tests. As shown in Fig. 1, the specimens were cut so that the grain of the outer laminations was perpendicular to the loading direction, while that of

Table 2
CLT manufacturing parameters.

Adhesive system	Parameters	Retention level (kg/m^3)		
		0	1.0	2.4
MF	Temperature (°C)	23	26	28
	Glue-spread rate (g/m^2)	440	440	440
	Clamping pressure (MPa)	0.86	0.86	0.86
	Clamping time (h)	11.25	10.5	8
RF	Temperature (°C)	23	26	28
	Glue-spread rate (g/m^2)	342	342	342
	Clamping pressure (MPa)	0.86	0.86	0.86
	Clamping time (h)	11.25	10.5	8
PUR*	Temperature (°C)	23	26	28
	Glue-spread rate (g/m^2)	147	147	147
	Clamping pressure (MPa)	0.69	0.69	0.69
	Clamping time (h)	3	3	3

*A commercial primer (LOCTITE PR 3105) solution was diluted to 5% with water by weight was applied on wooden face layers at the rate of 20 g/m^2 approximately 30 min. before application of PUR adhesive system.

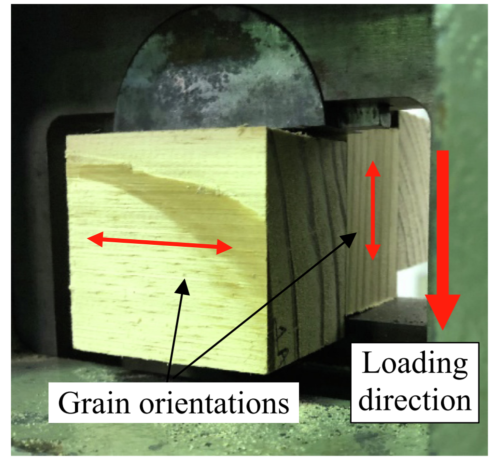

Fig. 1. Position of CLT specimen undergoing block shear testing.

core laminations was parallel to the loading direction. The core lamination was subjected to the shearing twice, when each outer lamination was placed under load once. Under this test setup, the outer laminations were more likely to get damaged than the core laminations due to the shear strength properties of the different wood grain orientations [32]; the shear strength properties of the perpendicular-to-grain direction are less than half of those of parallel-to-grain direction [33]. Planar stresses were applied to the bonded areas through adjacent laminations using a shearing tool at a rate of 5.08 mm/min (0.20 in./min) as recommended in ASTM Standard D905 [20] until failure. Block shear strength (BSS) was calculated as:

$$\tau_{BS} = \frac{F}{b \times t} \tag{1}$$

where τ_{BS} = BSS (MPa), F = failure load (N), b = sample width (mm), and t = sample thickness (mm).

The fractured bond areas were visually inspected to determine the types of failure modes: adhesive (AD), parallel-to-grain (PAR), and perpendicular-to-grain (PER). The images of the failure planes were digitized using a Canon CanoScan LiDE 120 scanner which had a maximum optical resolution of 2400 × 4800 dpi. Wood failure area of each scanned plane was measured using ImageJ2 [34], an image processing software. Wood failure percentage (WFP) was calculated by dividing the wood failure area by the tested bond area. The SGs were measured according to ASTM Standard D2395 [35] from 20 mm cube blocks cut from each lamination of the tested specimens after conditioning at 65 ± 2% relative humidity and 21 ± 2 °C for at least 14 days.

3.2. Delamination test

For each CLT configuration, 15 delamination test specimens (8 from one panel replicate and 7 from the other) with dimensions of 76 mm × 127 mm × 105 mm were prepared. Before conducting the tests, all six sides of specimens were digitized using the photo scanner described earlier. Only specimens with perfect glue bondlines, that is no visually apparent delamination, were selected for the test. Specimens with missing glue bondlines due to manufacturing defects were eliminated, which decreased the sample size of each configuration to ten. Delamination of bondlines along the length (labeled "A" and "C" in Fig. 2) were measured immediately after the specimens underwent three accelerated aging cycles, which consisted of vacuum, soaking, and oven-drying procedures, as specified in ASTM Standard D2559 [21].

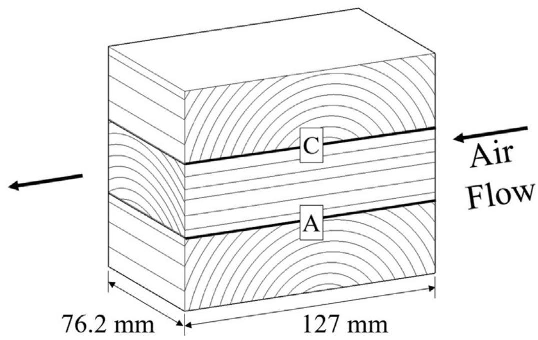

Fig. 2. Orientation of the delamination test specimen during the oven-drying procedure. A and C denote the two bond lines.

Delamination measurements were made by capturing the two sides of the tested specimens, where the A and C bondlines were located, with the photo scanner followed by analysis with ImageJ2 software [34]. Delamination was also visually examined using a fluorescent lamp and a magnifying lens (×10). When there was a confusion between delamination and wood failure, the laminations were separated using a chisel and hammer for a more detailed view. Delaminated surfaces with adhesive failures could be distinguished from fractured wood fibers because the former reflected more fluorescent light than the latter. Delamination rates of the A and C bondlines (Fig. 2) for each configuration were calculated as the total delaminated bondline length divided by the total tested bondline length. The total length of each bondline was 2540 mm for a total of 5,080 mm tested for each configuration.

3.3. Statistical analysis

The effects of preservative retention and adhesive on measured responses were analyzed using SPSS version 25.0 [36]. Assumptions of normality and homogeneity of variance were tested on raw data using the Shapiro-Wilk test and Levene's test, respectively. If the assumptions were not met, the data were normalized by logarithmic-transformation, i.e. log(x) [37], and tested again for normality and homogeneity of variance. If assumptions were satisfied ($p > 0.05$), one-way ANOVA followed by the Tukey Honestly Significant Difference (HSD) test was used for mean separation of the levels within the main effect being analyzed. Significance testing was done at $\alpha = 0.05$. If data could not be normalized, then the Kruskal-Wallis H test, a non-parametric equivalent of one-way ANOVA, was performed. If the effect being analyzed proved to be significant at $\alpha = 0.05$, then mean rank separation was done using Dunn's pairwise test adjusted by the Bonferroni correction.

4. Results and discussion

4.1. Block shear test

The descriptive statistics of the calculated BSS and WFP are presented in Table 3 for each configuration. Although the tests were prepared and conducted according to ASTM D2559 [21], the mean BSS values were not compared against the standard requirements which were established for structural glued laminated timber. The required BSS specified in the standard is based on parallel-to-grain shear strength of wood. However, the governing failure mode of CLT shear block test specimens was a perpendicular-to-grain shear failure, as discussed in later sections. The mean BSS of the control configurations, which ranged from 3.57 MPa to 3.76 MPa. The variation of the presented BSS data sets, which had COVs ranging from 19.98% to 37.56%, were similar to those reported in previous CLT bond performance studies [32,38,39]. All nine configurations exceeded the minimum average WFP of 75% required by ASTM D2559 [21]. Regardless of the adhesive type, the treated configurations had a greater number of specimens not meeting the minimum average WFP than the untreated control (2 for control, 11 for the low retention, and 6 for the high retention).

The summary statistics of SG measurements are provided in Table 4 for each configuration. The SG at equilibrium moisture content (EMC) describes the physical conditions of the block shear specimen at the time of testing, while oven-dry SG can be used to evaluate the consistency in the quality of lumber used for making CLT. The average EMC of the all the conditioned specimens was 12.7% with a 7.94% COV. Based on one-way ANOVA analysis comparing all nine CLT configurations, neither SG_{EMC} nor $SG_{oven\text{-}dry}$ means showed significant differences ($p > 0.05$), indicating that variation in SG of the wood from which the laminations were made was unlikely to have affected the block shear test results.

Table 3
Descriptive statistics of BSS and WFP for the nine CLT configurations.

CLT Config.	Sample size (n)	BSS (MPa)			WFP (%)		No. < 75% WFP**
		Mean [95% CI*]	Median [range]	COV (%)	Mean	Median [range]	
MC	30	3.76 [3.42–4.09]	3.66 [2.42–5.70]	23.86	98.88	100.00 [84.19–100.00]	0
ML	30	2.90 [2.59–3.21]	2.73 [1.76–4.87]	28.58	91.27	99.63 [47.61–100.00]	5
MH	30	3.02 [2.74–3.30]	2.90 [1.58–4.48]	24.68	93.22	99.88 [52.13–100.00]	2
RC	30	3.57 [3.31–3.83]	3.31 [2.48–5.35]	19.98	99.36	100.00 [92.57–100.00]	0
RL	30	2.80 [2.41–3.20]	2.95 [0.73–4.74]	37.56	91.10	99.93 [37.10–100.00]	5
RH	30	3.67 [3.27–4.07]	3.40 [2.17–6.18]	29.15	99.06	100.00 [88.30–100.00]	0
PC	30	3.68 [3.31–4.04]	3.50 [2.18–6.28]	26.83	93.60	96.63 [60.00–100.00]	2
PL	30	2.95 [2.62–3.27]	2.80 [1.52–4.82]	29.84	94.13	100.00 [73.05–100.00]	1
PH	30	4.02 [3.62–4.42]	4.08 [2.25–5.89]	26.72	89.49	97.70 [21.70–100.00]	4

*CI, confidence interval.
**Number of specimens with < 75% WFP.

Table 4
Summary statistics of SGs for nine CLT configurations*

CLT Config.	Sample size (n)	SG_{EMC}		$SG_{oven\text{-}dry}$	
		Mean	COV (%)	Mean	COV (%)
MC	45	0.47	13.69	0.49	13.95
ML	45	0.46	10.94	0.49	11.29
MH	45	0.49	9.86	0.51	10.59
RC	45	0.48	15.51	0.51	16.19
RL	45	0.47	13.59	0.50	14.16
RH	45	0.46	10.15	0.48	10.62
PC	45	0.47	11.20	0.50	11.44
PL	45	0.46	11.80	0.49	12.38
PH	45	0.47	10.36	0.50	10.94

*No significant difference ($p > 0.05$) among the nine CLT configurations for either SG_{EMC} or $SG_{oven\text{-}dry}$.

4.1.1. Effect of preservative treatment

For the MF and PUR configurations, one-way ANOVA was used to compare the effect of preservative retention on mean $\log_{10}$BSS values since transformed datasets passed normality and equality of variance tests. For the RF configurations, data were not normal even after transformation so mean BSS ranks were tested using the Kruskal-Wallis H test (Table S1). Regardless of adhesive type, statistical analyses showed that retention level significantly affected BSS (Fig. 3). The low and high retentions of the MCA-C treatment for the MF resin were not significantly different from each other, but both exhibited significantly reduced BSS compared to the untreated controls. For the RF and PUR resins, however, only the low retentions showed a significant reduction in BSS; the untreated and high retention treatments were statistically similar.

The observed reductions in shear strength of the laminations could not be explained by the SG measurements, which were found to be statistically similar for all nine configurations (Table 4). Another potential factor that could have affected the BSS is the preservative chemistry, itself. Some preservatives like CCA and the amine-based copper systems have been shown to decrease the mechanical properties of wood [40]. Research on three different micronized copper systems used to treat southern yellow pine sapwood, however, has indicated that they cause no or only a minor decrease (up to 5%) on modulus of rupture (MOR) compared to water-treated controls, where post-treatment drying conditions were 24 °C in a room held at constant relative humidity [41].

The increase in BSS of the high retention back to untreated control levels in the RF and PUR configurations is not readily explained without a better understanding of the effects of kiln-drying on micronized copper systems and the interactions that different types of resin may have with the micronized copper preservative as retention increases. Positive effects of increased retention on wood mechanical properties for a waterborne-preservative using a soluble copper system has been reported by Yildiz et al. [42]. They observed that a low retention ACQ-1900 treatment on Scots pine sapwood decreased bending MOR and modulus of elasticity (MOE) with strength properties increasing back to untreated control levels as retention increased. ACQ-2200, however, produced different results. MOE decreased with increasing retention, while MOR was reduced regardless of retention relative to untreated controls [42]. The two formulations both have copper but have different quaternary ammonium components.

Significance testing of WFP showed that only RF configurations were significantly affected by preservative retention according to the Kruskal-Wallis H test (Table S2). The RL configuration had lowest mean WFP rank, which was significantly different from the RC configuration but not RH. For the MF and PUR configurations, no significant effect of retention was detected (Fig. 4). All configurations had mean WFP values 89%, thereby meeting the minimum WFP requirement of 75% stated in ASTM D2559 [21]. Of those nineteen specimens that fell short of 75% WFP (Table 3), the majority were close to meeting the WFP requirement (13 had WFP between 50 and 75%).

4.1.2. Effect of adhesive system

The effect of the adhesive type on BSS was only statistically significant for the high-retention treatment, where PUR and RF provided significantly larger BSS values than MF (Table S3). These results indicate that all three adhesives had similar interactions with the untreated and low retention treated laminations in terms of mechanical interlocking and intermolecular attractions [43]. On the other hand, the $\log_{10}$BSS means of the PH and RH configurations were significantly larger than those of the MH as illustrated in Fig. 5. This phenomenon might be explained by the differences in interactions of the adhesives and treated laminations, which determine adhesive-preservative compatibility. It has been reported that soluble metallic components of preservative systems would enhance curing rates of adhesive systems as catalysts, while

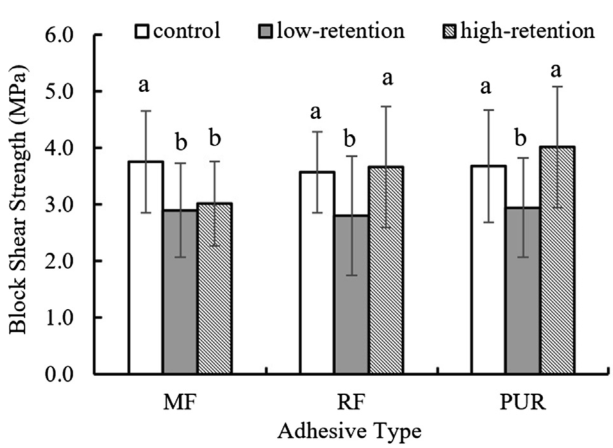

Fig. 3. Mean BSS of the CLT configurations by adhesive types (bars represent standard deviation; different letters above the bars indicate significant differences ($p < 0.05$) among the treatment means for within adhesive comparisons; for pairwise comparisons, Tukey HSD was used for MF and PUR while Dunn's test with p-values adjusted by the Bonferroni correction was used for RF).

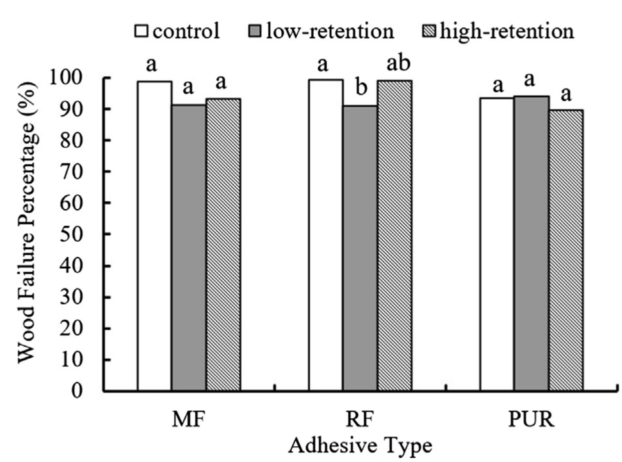

Fig. 4. Mean WFP of the CLT configurations by adhesive types (different letters above the bars indicate significant differences ($p < 0.05$) among the adhesive means for within retention level comparisons; for pairwise comparisons, Dunn's test with p-values adjusted by the Bonferroni correction was used).

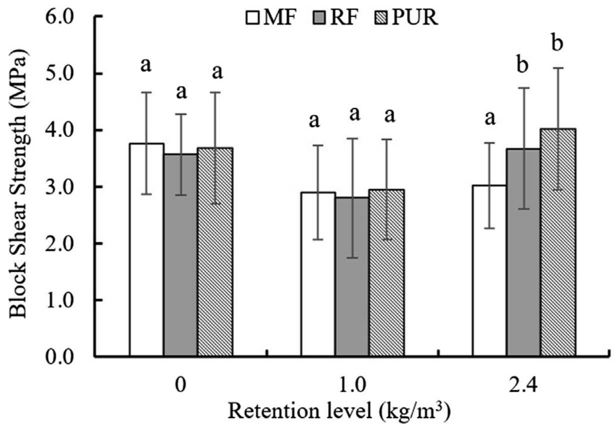

Fig. 5. Mean BSS of the CLT configurations by retention levels (bars represent standard deviation; different letters above the bars indicate significant differences ($p < 0.05$) among the treatment means for within adhesive comparisons; for pairwise comparisons, Tukey HSD was used for 0 kg/m^3 and 2.4 kg/m^3 retention levels, while Dunn's test with p-values adjusted by the Bonferroni correction was used for 1.0 kg/m^3 retention level).

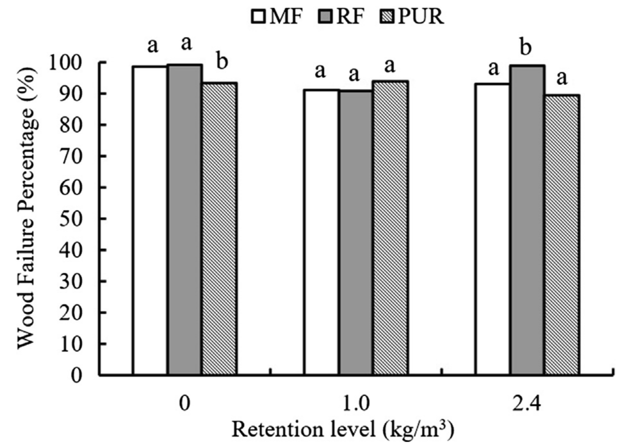

Fig. 6. Mean WFP of the CLT configurations by retention levels (different letters above the bars indicate significant differences ($p < 0.05$) among the adhesive means for within retention level comparisons; for pairwise comparisons, Dunn's test with p-values adjusted by the Bonferroni correction was used).

insoluble metallic components, which MCA-C is mostly composed of, might hinder formation of strong adhesion by impeding intermolecular attraction between adhesives and wood fibers [13,16,44]. Direct comparisons between particulate and micronized and soluble copper systems have shown that the amount of extractable soluble copper (Cu^{2+}) from wood treated for commercial applications after air-drying was less for the micronized copper system (15–30 ppm) than for the soluble copper system (100–450 ppm) [45]. Considering that greater BSS can be achieved with RF and PUR at ground contact retentions, it appears that these two adhesives would be more compatible with MCA-C treated southern pine laminations than MF. It is also possible that, at the higher retention, the polymeric dispersants of the MCA-C formulation might have partially interfered with the normal condensation and curing reactions of the MF resin. The fact that MF resin can interact with surfactants, however, is well established since modern methods of microcapsule production use emulsifiers and pH, albeit at higher concentrations than in the treated wood, to control shell formation in terms of quality, quantity, shape, and size [46]. Future studies that implement analytical chemistry techniques such as Fourier-transform infrared spectroscopy and differential scanning calorimetry, however, will be needed to further investigate the underlying mechanisms at play between the treated wood and resin.

From the wood failure perspective, the adhesive type significantly affected the WFP only in the control and high retention configurations according to the Kruskal-Wallis H test (Table S4). As illustrated in Fig. 6, the descending rank order for WFP control was: RC > MC > PC with MC and RC significantly greater than PC, but not significantly different from each other. The descending rank order for WFP high retention was: RH > MH > PH with RH significantly higher than MH and PH but MH and PH not significantly different from each other. Thus, the WFP was more affected by the adhesive type than BSS.

4.1.3. Failure modes

In general, the fibers of CLT laminations aligned perpendicular to the loading direction were damaged more than those aligned in parallel due to the differences in shear strength described earlier. Three failure modes (AD, PAR, and PER) were observed during the block shear tests (Fig. 7). Since the laminations were flat-sawn, the PAR failures occurred along radial-longitudinal planes. The PER failures were induced by the rolling of wood fibers against each other under shear stresses applied along tangential-radial and radial- tangential planes, which are also known as rolling shear failures [47]. Most of the specimens fractured in combinations of the three failure modes with the controlling failure mode determined by their relative contributions. For instance, if a tested glue-bond area showed 50% AD, 20% PER, and 30% PAR, the controlling failure mode was AD. Controlling failure modes are shown in Table 5. Since the AD failure occurred when the interface between the adhesive layer and wood fibers had lower shear strength than the wood substrate, it yielded relatively low BSS values. On the other hand, the BSS properties of the specimens failed in PAR were relatively high due to the contribution of the parallel-to-grain shear strength of wood.

a)

b)

c)

Fig. 7. Failure modes of block shear specimens: a) AD b) PER, and c) PAR.

Table 5
Block shear test results by controlling failure mode.

Config.	Number of observations (mean BSS in MPa)			
	AD	PAR	PER	Total
MC	0 (N/A)	8 (4.27)	22 (3.57)	30 (3.76)
ML	1 (2.37)	8 (3.32)	21 (2.77)	30 (2.90)
MH	0 (N/A)	8 (3.33)	22 (2.91)	30 (3.02)
RC	0 (N/A)	9 (4.06)	21 (3.36)	30 (3.57)
RL	2 (0.98)	5 (3.78)	23 (2.74)	30 (2.80)
RH	0 (N/A)	6 (5.25)	24 (3.28)	30 (3.67)
PC	0 (N/A)	6 (4.33)	24 (3.55)	30 (3.68)
PL	0 (N/A)	7 (3.59)	23 (2.75)	30 (2.95)
PH	3 (3.70)	2 (4.49)	25 (4.02)	30 (4.02)

Overall, the governing failure mode (i.e., majority of controlling failure modes) was PER which occurred more than 60% in each configuration while less than 7% of the treated specimens failed in AD. The block shear test results indicate that there was a relationship between controlling failure mode and BSS. Regardless of the adhesive type and preservative retention level, the largest mean BSS values were obtained with the PAR failures. However, even these mean BSS values failed to reach the parallel-to-grain shear strength of any major SYP species (i.e. longleaf pine, shortleaf pine, slash pine, and loblolly), which have parallel-to-grain shear strength values ranging from 9.6 to 11.6 MPa [48]. In addition to the observations of the mixed failure modes described earlier, this comparison suggests that the BSS of the glue-bond area failed in the PAR mode was also affected by the perpendicular-to-grain shear strength.

The characteristics of annual rings appeared to determine the patterns of the PER or rolling shear failures. Narrow annual rings tended to prevent propagation of rolling shear cracks (Fig. 8b), while wide annual rings promoted cracks growth (Fig. 8a). The laminations with annual rings oriented at steeper angles from the shear planes had more severe rolling shear failures (Fig. 8c and d, respectively). This was also observed in block shear tests conducted by Gupta and Sinha [49]. During the shear failures, the wood fibers fractured towards the pith locations of the laminations (Fig. 8e and f). Specimens that failed in PER, as a controlling mode, also had fibers fail in PAR, and their BSS properties were larger, on average, than previous test of rolling shear strength of SYP obtained by conducting two-plate or short-span bending tests, which ranged from 1.47 MPa to 2.40 MPa [47].

4.2. Delamination test

In a wood-based composite system, delamination is affected by internal stresses induced by dimensional changes and adhesion performance of a composite systems. Cylindrical anisotropy induces internal stresses along the planes tangential and normal to its annual rings when wood undergoes dimensional changes. Orthotropic characteristics and orientations of the fibers make wood more dimensionally stable in the longitudinal direction than either the tangential or radial directions. For instance, the tangential and radial shrinkage percentages (i.e., green to oven-dry moisture content) of loblolly pine are 7.4 and 4.8, respectively, while the longitudinal shrinkage is between 0.1 and 0.2 % [50]. These differences during the delamination tests led to the disparity between the dimensional changes of the orthogonally assembled laminations. Thus, the internal stresses induced by the dimensional changes provoked failures on wood fibers, adhesive layers, or linkages between the adhesives and wood fibers. The wood failures occurred when the bonding established by the adhesive with the fibers was stronger than the natural bonding between the fibers. Adhesive systems used in wood composites are designed to have larger shear strength properties than wood substrates. As a result, failures associated with the adhesives, which were considered as delamination, would occur when their linkages with the wood fibers are disrupted.

As shown in Table 5, delamination of bondlines was only observed in the treated configurations with a maximum rate of 2%. This may have been caused by interference from insolubles in the MCA-C preservative in intermolecular interactions between wood and adhesives as discussed earlier and/or physical blockage by the preservative to adhesive penetration [13]. Confirmation, though, would require techniques like light microscopy and scanning electron microscopy [51], which were outside the scope of this investigation.

From the preservative retention perspective, delamination rates were increased, on average, as the retention level was increased for the MF and RF configurations, which agreed with results reported by Gaspar et al. [52] on the glulam composed of CA treated maritime pine and PRF resin. Although an opposite trend was observed in the PUR configurations, both bondlines of the PL configuration exhibited delamination less than 1%, which could have been caused by natural variation in percent extractives and earlywood/latewood ratio.

From the adhesive perspective, the PUR showed the least percent delamination across both retentions indicating that the PUR configurations (Table 6) withstood moisture-driven dimensional changes better than MF and RF, although all three adhesive systems are known to perform well under wet conditions [43]. Mechanical testing has shown that MF and RF adhesives are more rigid and brittle than PUR adhesives [53], while PUR systems are capable of absorbing additional energy upon deformation, a favorable characteristic when wood is exposed to repeated cycles of

a) b) c) d) e) f)

Fig. 8. Rolling shear failure patterns of cross laminations with: a) wide annual rings, b) narrow annual rings, c) annual rings oriented at steep angles, d) annual rings oriented at shallow angles, e) pith located towards the adhesive layer, and f) pith located away from the adhesive layer.

Table 6
Summary of delamination test result.

Config.	Bondline	Bondline delam. (mm)	Delam. rate (%)
MC	A	0	0
	C	0	0
ML	A	36	1.42
	C	51.5	2.03
MH	A	47	1.85
	C	45.5	1.79
RC	A	0	0
	C	0	0
RL	A	8	0.31
	C	3	0.12
RH	A	36	1.42
	C	0	0
PC	A	0	0
	C	0	0
PL	A	13	0.51
	C	12	0.47
PH	A	0	0
	C	0	0

wetting and redrying [54]. This suggests that flexibility of adhesive would govern adhesion performance of CLT rather than rigidity of adhesive.

Delamination rates suggest that PUR is the most suitable adhesive for assembling CLT panels made with laminations treated with low and high retentions of MCA-C preservative. These retentions correspond to current above ground and ground contact retentions set by AWPA Use Category system (UC3B and UC4A retentions, respectively [24]). Any of the three adhesives (MF, RF, and PUR) would be suitable for indoor applications of CLT made with untreated laminations. These recommendations apply to 3-ply 35 mm laminations since higher ply and thicker laminations could behave differently. Except for ML, MH, and one of the bondlines of the RH configuration, the delamination rates of all other bondlines were still less than the 1%, which is the maximum allowed delamination rate specified in the reference standard. The performed delamination tests were also probably more vigorous than specified in the standard [21] since the laminations used to construct the test specimens were thicker than recommended. The ground contact-retention encouraged more delamination for the MF and RF adhesive systems. Moreover, the delamination rates were substantially smaller than previous results [39,55] obtained following the European CLT product standard, EN 16351[56], which has a different delamination criterion from ASTM D2559. The European standard considers the wood failures occurring on cells adjacent to the adhesive layers as delamination.

As shown in Fig. 9, the out-of-plane dimensional changes caused partial separations of the laminations by inducing tensile stresses perpendicular to the bondline, while the shear stresses introduced by in-plane dimensional changes separated laminations along the entire test bondline. The mix of these dimensional changes in two directions damaged the fibers of both surface and core layers. Thus, governing failure modes could not be determined.

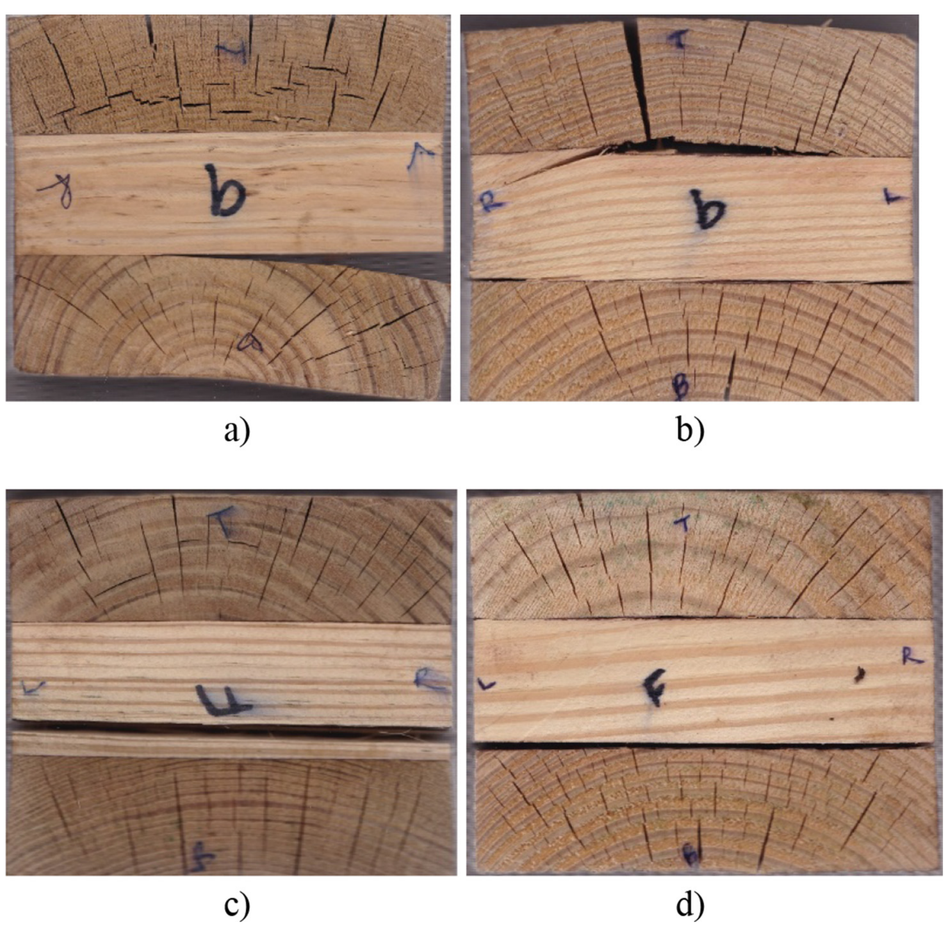

Fig. 9. Distortion of laminations after accelerated weathering cycles: a) and b) are out-of-plane dimensional changes, c) and d) are in-plane dimensional changes.

5. Conclusion

The effect of MCA-C retention levels on the bonding performance of SYP CLT panels manufactured using three adhesive systems, MF, RF, and PUR, were investigated by conducting block shear and delamination tests. Both BSS and WFP were significantly affected by the retention level and adhesive type. Although all the preservative treated configurations achieved mean WFPs more than the minimum required by ASTM D2559, the treatment at a retention level of 1.0 kg/m^3 lowered BSS significantly regardless of adhesive type, while the 2.4 kg/m^3 retention level treatment did not affect BSS when the RF or PUR adhesive was applied compared to untreated controls. After exposure to accelerated weathering cycles, only the PUR resin provided satisfactory bonding performance for all the treatment levels. Thus, based on the experimental results of this study, one component PUR provided better overall bonding performance compared to MF and RF for manufacturing 3-ply CLT (35 mm laminations) with SYP lumber treated with MCA-C at retentions used for exterior above ground or ground contact general use applications.

Examination of the failure modes of the block shear and delamination test specimens showed that although the block shear specimens were fractured in mixed failure modes, the perpendicular-to-gain or rolling shear failure was the governing mode for all configurations. Thus, the establishment of face-joint shear strength requirement for CLT products can be considered based on the minimum WFP requirement and the characteristic rolling shear strength of wood. Severe damages of laminations were observed after the delamination tests. However, they did not cause any delamination of the tested bond lines of the untreated configurations. Along with the block shear test results, these findings support that all three adhesive systems can be used for manufacturing untreated CLT.

Declaration of Competing Interest

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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Appendix A. Supplementary data

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