Article

Bolt-Bearing Yield Strength of Three-Layered Cross-Laminated Timber Treated with Phenol Formaldehyde Resin

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Abstract: The effect of impregnation treatment of low molecular weight phenol formaldehyde (MWPF) resin on the bolt-bearing yield strength (BBYS) of a three-layered cross-laminated timber (CLT) composed of two face layers of southern pine (Pinus taeda) and one core layer of sweetgum (Liquidambar styraciflua) was investigated together with two additional factors of material type and loading direction. Experimental results indicated that the amount of low MWPF resins penetrating into sweetgum was more than the ones into southern pine. Sweetgum had more uniform distribution of low MWPF resins penetrating longitudinally than southern pine. Impregnation treatment using a low MWPF resin at a concentration of 20% can enhance the BBYS of three-layered CLTs with a 60% increase. The BBYS of a core layer material in three-layered CLTs can significantly alter the overall BBYS of the three-layered CLTs.

Keywords: southern pine; sweetgum; bolt-bearing yield strength; cross-laminated timber; phenol formaldehyde resin

1. Introduction

The Southern pine (Pinus taeda) and sweetgum (Liquidambar styraciflua) are two of most common wood species in the south of the United States. The southern pine has been widely used in engineered wood timber and composites. Sweetgum is moderately heavy and hard, and often has a form of cross grain called interlocked grain that makes the wood deform and crack easily if not dried slowly. Sweetgum is mainly for veneer, plywood, slack cooperage, fuel, pulpwood, boxes, and crates [1]. Table 1 summarizes the basic physical and mechanical properties of southern pine and sweetgum at 12% moisture content (MC) [1], including specific gravity (SG), modulus of rupture (MOR), modulus of elasticity (MOE), compression strengths, and tension strength perpendicular to grain.

| Species          | MC (%) | SG  | MOR  | MOE  | Compression Parallel to Grain | Compression Perpendicular to Grain | Tension Perpendicular to Grain |
|------------------|-------|-----|------|------|-------------------------------|-----------------------------------|-------------------------------|
| Sweetgum         | 12    | 0.52| 86   | 11,300| 43.6                          | 4.3                               | 5.2                           |
| Southern pine    | 12    | 0.51| 88   | 12,300| 49.2                          | 5.4                               | 3.2                           |

Cross-laminated timber (CLT), a new generation of engineered wood product that originated in Europe, then spread to North America, Australia, Japan, and China. Recently, CLT has been widely used in the field of wood construction, such as for single-family houses, residential buildings,
multistory public buildings, industrial and commercial buildings, and bridge structures [2]. CLT panels are usually made of three to seven layers of lumber of conifer wood species, and every layer is at a right angle to the adjacent layer. Typically, there is an odd number of layers arranged symmetrically to the middle layer. Currently, in the context of adopted standardization, the thickness of a CLT layer ranges from 12 to 45 mm [3].

Connections in a CLT system may be classified into three categories of (1) wall-to-wall or floor-to-floor, (2) wall-to-floor, and (3) wall to foundation. Commonly used mechanical fasteners in connecting CLT products are dowels, bolts, screws, and nails. The performance of these solid timber structures using CLT products highly depends on the strength and stiffness of the connections used. Specifically, the lateral load-bearing capacity of a dowel-type connection mainly depends on the dowel-bearing strength in CLT materials and the fastener’s yield moment capacity [4]. Johansen’s yield theory is widely used for estimating the lateral load-bearing capacity of dowel-type connections in wooden materials [5–7]. In this theory, dowel-bearing strength in wooden connection members and the yield moment capacity of the dowel-type fastener govern the lateral load-bearing capacity. Therefore, the dowel-bearing strength in the wooden materials used for connection members such as CLT is crucial for the structural design of wooden construction [8]. The dowel-bearing strength in wood construction can be calculated using the empirical equations provided in the National Design and Specification (NDS) [9] and Eurocode5 (EC5) [10]. All these equations indicate that the dowel-bearing strength in wood and wood-based materials is governed by the specific gravity or density of the wood and wood-based materials used in the construction. Therefore, increasing the wood material density is one way to enhance the dowel-bearing strength of the material, therefore increasing the lateral load-bearing capacity of a dowel-type connection in wood and wood-based materials.

Wood is a porous material containing two types of internal voids: large voids, such as cell lumina and pit openings, and cell wall microvoids. Chemical modification of wood through the impregnation of resins such as phenol formaldehyde (PF) is a typical method to improve the physical and mechanical properties of wood [11,12]. PF resins are a water solution of polymer molecules (oligomers) that differ in molecular weight (MW) and shape. During the impregnation process, treating solutions with oligomers of different average MW will reach the large pore system (the lumens of different fiber types, vessels, rays, libriforms, etc.) first, and then diffuse into the cell wall [13].

Aizat et al. [14] reported that laminated compreg oil palm wood composites treated with PF resin had significantly improved dimensional stability, MOE, and MOR than untreated material. Furuno et al. [15] investigated the penetration of PF resin into wood cell walls in relation to the dimensional stability and decay resistance, and found that low (290 g/mol) MW resin penetrated more easily into cell walls and formed a polymer-layered wall inside wood cell walls than medium (470 g/mol) and high (820 g/mol) MW resins. Biziks et al. [13] used light microscopy to examine the distribution and penetration depth of PF resin impregnated into beech (Fagus sylvatica) wood, and observed that the PF resin mainly penetrated into fiber lumens, and that specimens with a concentration of 27% had more uniformly distributed resin in fiber lumens than specimens with concentrations of 9% and 18%. All these observations indicated that a low MWPF resin was more effective for treating the wood.

There is limited literature related to how resin impregnation affects the dowel-bearing strength of wood species, such as southern pine and sweetgum, that are commonly used as raw materials in manufacturing of CLT products. Therefore, the main objective of this study was to investigate the effects of impregnation with low MWPF resin on the bolt-bearing yield strength (BBYS) of CLT composed of southern pine and sweetgum.
2. Materials and Methods

2.1. Materials

The two wood species used in this experiment were southern pine and sweetgum purchased from East Mississippi Lumber (Starkville, MS, USA). The adhesive used in bonding the CLT components was commercial phenol resorcinol formaldehyde (PRF) resin (Table 2). The impregnation was carried out using a commercial low molecular weight phenol formaldehyde (MWPF) resin (Table 3) purchased from a local store (Starkville, MS, USA).

### Table 2. The formulation and physical properties of the phenol resorcinol formaldehyde (PRF) adhesive used in this experiment for bonding cross-laminated timber (CLT) components.

| Parameters                                      | Values  |
|-------------------------------------------------|---------|
| Phenol resorcinol adhesive (CASCOPHEN® LT-75C)   | 100     |
| Hardener (CASCOSET® FM-282) (parts)             | 15–17   |
| Operational time at room temperature (21 °C)     | 2.5     |
| Gel time at 105 °C (minutes)                    | 6.03    |
| Viscosity (cps)                                 | 6280    |
| Clamp time (hours)                              | 9       |

### Table 3. Information of low molecular weight phenol formaldehyde resin used in this experiment for impregnation treatment of two wood species.

| Parameters                     | Values |
|--------------------------------|--------|
| Nonvolatile content (%)        | 48.42  |
| Specific gravity               | 1.150  |
| Viscosity (cps)                | 125    |
| pH                             | 8.5    |
| Free Phenol (%)                | 8.0    |
| Free Formaldehyde (%)          | 0.15   |
| Molecular weight (Mn)          | 310    |

2.2. CLT Design

Figure 1 shows the layout of the three-layered CLT used in this experiment to test the products. The CLT is composed of two face layers of southern pine timber and one core layer of sweetgum timber, which are arranged crosswise to each other at a 90 degree angle according to the CLT handbook [16].

![Figure 1. The general layout of three-layered cross-laminated timber (CLT) used for the construction of CLT products evaluated in this experiment.](image)

2.3. Experimental Design

A complete 3 × 2 × 2 factorial experiment with 12 replications per combination was implemented to investigate the factors influencing the BBYS of the CLT products constructed in this experiment. The three factors were material type (southern pine, sweetgum, and CLT), loading direction (longitudinal and radial), and impregnation treatment (control and treated). The control was the block without
the low MWPF resin impregnation, while the treated block was the one with the low MWPF resin impregnation. Figure 2 shows the general configurations and detailed dimensions of half-hole blocks evaluated in this study according to American Society for Testing and Materials (ASTM) D 5764-97 [17]. There were 144 bolt-bearing tests performed in this study.

2.4. Specimen Preparation and Testing Method

The full-size 420 mm long × 305 mm wide × 60 mm thick CLT board was fabricated first, followed by cutting 60 mm × 60 mm × 50 mm half-hole bolt-bearing blocks (Figure 2) from the full-size board. The fabrication process of the CLT board started with cutting eight pieces of southern pine quarter-sawn lumbers measuring 1280 mm × 70 mm × 20 mm (length × width × thickness) and six pieces of sweetgum quarter-sawn lumbers measuring 1140 mm × 70 mm × 20 mm (length × width × thickness) per the cutting patterns (Figure 3), respectively. Meanwhile, single wood species half-hole bolt-bearing blocks (Figure 2) measuring 60 mm × 50 mm × 20 mm (length × width × thickness) were cut at the two ends of southern pine and sweetgum lumbers per cutting pattern (Figure 3). The CLT fabrication was completed with sanding, adhesive application, cross-laminating (Figure 1), and cold pressing. A low MWPF resin loading of 323 g/m² was applied on the contacting surfaces of two adjacent material surfaces. The three-layered CLT was pressed using 450 Ton press machine (Dieffenbacher, Eppingen, Germany) under the pressure of 1.03 MPa for 6 h.

All treated blocks were impregnated with a low MWPF resin with a concentration of 20%. The low MWPF resin was obtained by diluting the low MWPF with 48.42% solid content (Table 3). The impregnation process started with vacuuming the pressure of the cylinder of a lab-made vacuum
was calculated using the following equation:

\[ F_{emm,y} = \frac{P}{D_t} \]  

(a) 

(b) 

Figure 3. Cutting pattern of southern pine (a) and sweetgum (b) lumbers.

Figure 4. Diagram illustrating where a slice was taken from a treated block for observing low molecular weight phenol formaldehyde (MWPF) resin penetration distribution along its longitudinal direction using a confocal laser-scanning microscope (CLSM).

All half-hole bolt-bearing tests were performed on a hydraulic SATEC (Norwood, MA, USA) universal testing machine. Figure 5 shows the testing setup for determining the half-hole BBYS of evaluated materials per ASTM D5764-97 [16]. The loading speed was 1 mm/min. The ultimate loads and failure modes of all tested blocks were recorded. The BBYS in tested materials, \( F_{emm,y} \) (N/mm²), was calculated using the following equation:

\[ F_{emm,y} = \frac{P}{D_t} \]
where $P$ is the compressive bolt-bearing yield load (N); $D$ is the bolt diameter (mm); $t$ is the thickness of a tested block (mm).

Load–deformation curves of all tested materials were recorded. The bolt-bearing yield load of a tested material was determined by fitting a straight line to the initial linear portion of a recorded load–deformation curve, and offsetting this line by a deformation equal to 5% of the loaded bolt diameter [16]. If the point of intersection of this offset line with the curve did not reach the maximum load, then the load at the intersection point was considered as the yield of the tested material; but, if the intersection point passed the peak load, then the peak was considered as the yield of the tested material. The SG and MC values of evaluated materials were determined through cutting off specimens from tested half-hole blocks as shown in Figure 4 according to ASTM D2395–93 [18] and ASTM D4442-92 [19], respectively.

**Figure 5.** Setup for evaluating the half-hole bolt-bearing yield load of evaluated wood materials in this study: front view (a), side view (b), and photo of the setup (c).

### 2.5. Statistical Analysis

The effects of material type and impregnation treatment and their interaction on mean SG values, and the effects of material type, loading direction, and impregnation treatment and their interactions on mean BBYS values were analyzed using the analysis of variance (ANOVA) general linear model (GLM) procedure. Mean comparisons using the protected least significant difference (LSD) multiple comparison procedure for equal size replicates or least squares means (LSMEAN) procedure for unequal size replicates were performed if any significant interaction was identified; otherwise, the main effects were concluded. All statistical analyses were performed at the 5% significance level.

### 3. Results and Discussions

#### 3.1. Specific Gravity

The MC values of southern pine and sweetgum averaged 12.01% and 10.41%, respectively, and their corresponding coefficient of variance (COV) averaged 1.54% and 4.31%, respectively. Table 4 summarizes the mean values of the SG of wood materials evaluated in this experiment.

ANOVA results (Table 5) indicated that the two-way interaction is not significant for SG. Further checking the magnitudes of F values indicated that impregnation had a larger F value of 37.67 than material type with an F value of 6.76. This could mean that the significance of impregnation treatment on SG was much stronger than material type. Therefore, the impregnation treatment effect on SG was performed based directly on mean comparisons of the main effect, while the material type effect on SG was analyzed by considering the non-significant two-way interaction using the LSMEAM multiple comparison procedure. This is because the nature of conclusions from interpretation of main effects also depends on the relative magnitudes of the interaction and individual main effects [20].

Tables 4 and 6 summarize the mean comparisons of SG values for impregnation treatment and material type, respectively. The main effect mean comparisons indicated that each of the three treated
materials had significantly higher SG values than their corresponding control (Table 4). The ratios of treated material SG values to their control values were calculated and are shown in Table 4. The ratios range from 1.08 to 1.15 and indicate that sweetgum had a higher ratio (1.15) than the other two materials, indicating that the low MWPF resin penetrated further on average into sweetgum than the other two materials. Mean comparisons of SG values for material type (Table 6) support this observation. There is no significant difference in SG values among the three control materials. Treated sweetgum had a significantly higher SG value than the other two materials. There was no significant difference in the SG values between the treated CLT and southern pine materials.

Table 4. Summary of mean specific gravity (SG) values of materials used in this experiment with and without impregnation.

| Material Type | Impregnation Treatment | Control | Treated | Ratio |
|---------------|------------------------|---------|---------|-------|
| Southern pine | 0.53 (6.86) (12) B *   | 0.57 (12.04) (24) A | 1.08   |
| Sweetgum     | 0.55 (3.16) (12) B     | 0.63 (3.41) (24) A | 1.15   |
| CLT          | 0.54 (4.23) (12) B     | 0.59 (3.95) (24) A | 1.09   |

* The values in the first and second parentheses are coefficients of variation in percentage and number of replications, respectively. Two means in each row not followed by a common letter are significantly different one from another at the 5% significance level.

Table 5. Summary of analysis of variance (ANOVA) results for specific gravity values for materials evaluated through the general linear model (GLM) procedure performed on two factors.

| Source                        | F Value | p Value |
|-------------------------------|---------|---------|
| Material type                 | 6.76    | 0.0017  |
| Impregnation treatment        | 37.67   | <0.0001 |
| Material type × Impregnation treatment | 1.51    | 0.2253  |

Table 6. Mean comparisons of specific gravities of materials used in this experiment by material type.

| Impregnation Treatment | Material Type  |
|------------------------|----------------|
|                        | Southern Pine | Sweetgum | CLT    |
| Control                | 0.53 A *      | 0.55 A   | 0.54 A |
| Treated                | 0.57 B        | 0.63 A   | 0.59 B |

* Means in each row not followed by a common letter are significantly different one from another at the 5% significance level.

The SG value of treated sweetgum was significantly higher than southern pine, because the amount of low MWPF resin penetrating into sweetgum was more than that penetrating into southern pine. This observation was supported by CLSM images (Figure 6), indicating more resin in vessel cells of sweetgum than tracheid ones of southern pine, i.e., more low MWPF resin penetrated into the middle section of sweetgum than southern pine. This could be because the average diameter of sweetgum vessel cells (Figure 6b) is much bigger than that of southern pine tracheids (Figure 6a), and sweetgum has more rays in the radial direction (Figure 6b) than southern pine (Figure 6a), leading to more resin penetration into sweetgum.

3.2. Bolt-Bearing Yield Strength

Figure 7 shows two typical load–deformation curves (Type A and Type B) observed in half-hole bolt-bearing tests performed in this study. In a Type A curve, the load at the peak is the yield load of the tested material. For a Type B curve, the load at the point of intersection between the 5% offset line and load–deformation curve is the yield load of the tested material. Table 7 summarizes the mean BBYS values of the materials evaluated in this experiment.
Table 9, and Table 10 compare the BBYS values of the impregnation treatment, loading direction, and material type, respectively. These results are based on the LSMEAN procedure performed at the 5% significance level.

3.3. Mean Comparisons

ANOVA results (Table 8) indicated that the three-way interactions were significant. Table 7, Table 9, and Table 10 compare the BBYS values of the impregnation treatment, loading direction, and material type, respectively. These results are based on the LSMEAN procedure performed at the 5% significance level.

Table 7 indicates that, in general, impregnating low MWPF resin into the evaluated materials can significantly increase their BBYS [21]. This is mainly because the low MWPF resin fills the empty large pore systems of the wood [13]. However, different percentages of increase in BBYS could happen. Specifically, the BBYS ratio of treated to control southern pine samples in radial direction was 1.31,....
which was relatively lower than the other ratios in the radial direction, which ranged from 1.65 to 1.74. This could be mainly because southern pine (Figure 6a) has fewer rays in the radial direction than sweetgum (Figure 6b), resulting in a lower penetration of the low MWPF resin.

Table 8. Summary of analysis of variance (ANOVA) results obtained from the general linear model (GLM) procedure performed on three factors.

| Source                                      | F Value | p Value |
|---------------------------------------------|---------|---------|
| Material type                               | 257.38  | <0.0001 |
| Loading direction                           | 659.98  | <0.0001 |
| Material \times loading direction           | 77.41   | <0.0001 |
| Impregnation treatment                      | 864.52  | <0.0001 |
| Material type \times impregnation treatment | 22.26   | <0.0001 |
| Loading direction \times impregnation treatment | 67.94   | <0.0001 |
| Material type \times loading direction \times impregnation treatment | 11.92   | <0.0001 |

Table 9. Mean comparisons of bolt-bearing yield strengths for loading direction within each combination of material type and impregnation treatment.

| Material Type | Impregnation Treatment | Loading Direction | Ratio of Longitudinal to Radial |
|---------------|------------------------|------------------|---------------------------------|
|               |                        | Longitudinal (MPa) | Radial (MPa) |                                |
| Southern pine | Control                | 38.18 A *         | 21.15 B      | 1.83                            |
|               | Treated                | 68.96 A           | 27.79 B      | 2.48                            |
| Sweetgum      | Control                | 56.88 A           | 34.60 B      | 1.64                            |
|               | Treated                | 95.52 A           | 60.15 B      | 1.59                            |
| CLT           | Control                | 38.16 A           | 33.68 B      | 1.13                            |
|               | Treated                | 64.11 A           | 55.46 B      | 1.16                            |

* Two means in each row not followed by a common letter are significantly different one from another at the 5% significance level.

Table 10. Mean comparisons of bolt-bearing yield strengths for material type within each combination of impregnation treatment and loading direction.

| Impregnation Treatment | Loading Direction | Material Type |
|------------------------|-------------------|--------------|
|                        | Longitudinal (MPa)| Southern Pine| Sweetgum   | CLT         |
| Control                | 38.18 B *         | 38.16 B      | 38.18 B    | 38.16 B   |
| Radial                 | 21.15 B           | 34.60 A      | 33.68 A    | 33.68 A   |
| Treated                | 68.96 B           | 95.52 A      | 64.11 C    | 64.11 C   |
| Radial                 | 27.79 C           | 60.15 A      | 55.46 B    | 55.46 B   |

* Three means in each row not followed by a common letter are significantly different one from another at the 5% significance level.

Table 9 indicates that, in general, the BBYS values in the longitudinal direction were all significantly higher than those in the radial direction [22], as expected. Specifically, in the case of southern pine, the BBYS value in the longitudinal direction was 1.83 times greater than that of the radial direction in control wood, and 2.48 times greater than in the treated wood. The increase in the BBYS value in the control specimens was similar to the value found by Sawata and Yasumura [6]. The much greater increase in the BBYS of treated samples, i.e., a ratio of 2.48, was because the amount of low MWPF resins penetrating radially was lower than in the longitudinal direction. This caused a smaller increase of the BBYS in the radial direction, i.e., the ratio of treated to control wood was 1.31 (Table 7). In the case of sweetgum, both the control and treated materials had similar longitudinal to radial BBYS ratio. Both control and treated CLTs had similar longitudinal to radial BBYS ratios (close to 1), but less than the two other tested materials, indicating that the evaluated CLT materials tended to show an isotropic mechanical property in terms of BBYS in both longitudinal and radial directions.

Table 10 shows that the BBYS values of sweetgum materials were significantly higher than southern pine materials in all combinations of impregnation treatment and loading direction. However, according
to the Wood Handbook (Table 1) [1], the compressive strength of sweetgum in both longitudinal and radial directions is lower than those of southern pine, but the tensile strength of sweetgum is higher than that of southern pine. This implies that the wood’s tensile strength perpendicular to grain governs its BBYS rather than its compressive strength. The untreated and treated CLTs loaded in the radial direction had significantly higher BBYS values than southern pine loaded in the radial direction. This observation implies that the core layer of sweetgum with a significantly higher BBYS value than two face layers of southern pine could significantly increase the overall BBYS of the CLT. The treated CLTs loaded in the longitudinal direction had a significantly lower BBYS value than the treated southern pine loaded in the longitudinal direction. This observation implies that a core layer of sweetgum in the radial direction with a face layer of southern pine in the longitudinal direction would significantly lower the overall BBYS of a CLT composed of two face layers of treated southern pine in the longitudinal direction and one core layer of treated sweetgum in the radial direction. There was no significant difference between the BBYS values of untreated CLT and southern pine materials loaded in the longitudinal direction. This observation implies that a core layer of sweetgum in the radial direction and a face layer of southern pine in the longitudinal direction would not significantly lower the overall BBYS of a CLT composed of two face layers of untreated southern pine in the longitudinal direction and one core layer of untreated sweetgum in the radial direction. All these mean comparisons of BBYS values indicate that the BBYS of the core layer material significantly alters the overall BBYS of the CLT.

4. Conclusions

This study investigated the BBYS of three-layered CLTs composed of two face layers of southern pine and one core layer of sweetgum, considering the effects of low MWPF resin impregnation treatment, material type, and loading direction. The following conclusions can be drawn:

1. The amount of low MWPF resins penetrating into sweetgum was more than that penetrating into southern pine;
2. The distribution of low MWPF resin penetrating along longitudinal sweetgum was more uniform than in southern pine;
3. The BBYS of three-layered CLTs evaluated in this study can be improved by 60% by using a low MWPF resin impregnation treatment;
4. The BBYS of the core layer material in a three-layered CLT can significantly alter the overall BBYS of the CLT;
5. The three-layered CLTs evaluated this study showed an isotropic mechanical property in terms of BBYS along their longitudinal and radial directions.

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