Effect of Copper Azole Preservative on the Surface Wettability and Interlaminar Shear Performance of Glulam Treated with Preservative

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The application of copper azole (CA) preservative on glulam can improve the durability of the material. The effect of CA preservative was evaluated relative to the surface bonding quality of laminates with different surface conditions. The surface morphology and wettability of CA preservative-treated laminates were analyzed. A comparison was made on the interlaminar shear strength of preservative-treated glulam under varying environmental conditions. The atomic force microscope (AFM) diagrams showed that CA preservative adhered to the wood fibers' surface and filled some wood cell cavities, thus reducing the water permeability of the treated laminate. Planing effectively enhanced the surface wettability of the preservative-treated laminate. The tiny particles of CA preservative on the planed laminate surface were distributed relatively uniformly, with a proportion smaller than that of the unplaned laminate. Furthermore, the interlaminar shear strength of preservative-treated glulam using planed laminates was at least 15% higher than that of unplaned laminates. In both hot and humid environments and natural aging tests, preservative-treated glulam bonded with resorcinol formaldehyde (RF) adhesive outperformed those bonded with polyurethane (PUR) adhesive. To ensure reliable quality of preservative-treated glulam, it is recommended to plane the laminate by 0.6 mm and use RF adhesive.

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Keywords: Preservative treated Glulam; Copper azole preservative; Surface wettability; Shear performances

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INTRODUCTION

Engineered wood products provide material sources for the development of modern timber construction and meet the requirements of today's society for high-quality, personalized, aesthetic, energy saving, and environmentally friendly building materials (Aicher and Tapia 2018; Currie *et al*. 2021). They have played a positive role in promoting the rapid development of the modern timber construction industry (Fu *et al*. 2014). Glulam (glued laminated timber) is a type of engineered wood product made of finger-jointed laminates (Aicher and Tapia 2018).

While inheriting the characteristics of solid wood, glulam effectively overcomes the shortcomings of solid wood, such as easy deformation, difficult drying, and many knot defects (Walsh-Korb *et al*. 2019).

Glulam is widely used in modern timber construction due to its excellent mechanical properties, good dimensional stability, and various product specifications. However, when glulam is used in engineering projects such as the bottom of wood buildings, wood bridges, or landscape architecture, the glulam components are prone to mildew, decay, cracking, and delamination due to long-term environmental erosion, posing severe security risks to the buildings.

The natural durability of glulam largely depends on the wood species. Zarah *et al*. (2019) studied the compressive strength of foundation piles made of oak (*Quercus*), pine (*Pinus*), spruce (*Picea*), fir (*Abies*), alder (*Alnus*), and poplar (*Populus*) with varying service lives. They found that bacteria only eroded alder and white oak, while spruce and pine exhibited good performance. Thaler *et al*. (2013) investigated the effects of natural aging on the durability of oak (*Quercus* sp.), beech (*Fagus sylvatica*), and Norwegian spruce (*Picea abies*) that had been in use for 130 to 200 years. They discovered that the mechanical properties and anti-fungal properties remained unchanged for oak and spruce, whereas beech displayed poor decay resistance against fungal invasion. In addition, compared to fresh samples, aged specimens had a lower resistance to fungal attacks.

Impregnating wood with preservatives can significantly enhance its durability compared to naturally durable wood (Charles *et al*. 2016). It is an effective technical measure to improve the durability and environmental adaptability of ordinary glulam by impregnating the laminates with preservatives before composite production (Wang *et al*. 2015). The quality of bonding between the glulam laminates is crucial to the production of preservative-treated glulam (Garfner *et al*. 2014). According to ASTM D905-08 (2013), Lee *et al*. (2006) evaluated the bond strength of preservative-treated glulam manufactured from two softwood species (Korean pine and Japanese larch), three different water-based preservatives [copper chromate arsenate (CCA), double-(N-ring) cyclohexyl diazene dioxide (CuHDO), copper azole preservative (CA)], and four different adhesives [urea-melamine formaldehyde (UMF), melamine-formaldehyde (MF), phenolic glue (PF), and resorcinol formaldehyde glue (RF)]. They observed that the shear strength of preservative-treated wood decreased to some extent compared to untreated wood, and the shear strength was influenced by the types of adhesives and preservatives.

Copper azole (CA) preservatives, known for their antifungal properties, bleed resistance, and environmental protection, are widely used in wood preservation today (Maldas and Kamdem 1998; Schmitt *et al*. 2014; Alade *et al*. 2022). Submicroscopic and microscopic observations of preservative-treated wood revealed that preservatives could physically and chemically block the surface where intermolecular forces develop (Maldas and Kamdem 1998). The divalent copper ions and azolane compounds in copper azole preservatives form copper salt complexes and “nitrogen-copper-oxygen” complexes with carboxyl groups in hemicellulose and hydroxyl groups in lignin, respectively, thereby reducing wood water absorption and contributing to dimensional stability and durability. However, the presence of preservatives in treated wood may not be conducive to bonding between treated wood components (Lim *et al*. 2020; Adnan *et al*. 2021).

This study analyzed the impact of CA preservative on the surface bonding performance of laminates under various surface conditions. The study utilized CA preservative for the treatment of glulam laminates through high-pressure impregnation. The surface morphology and wettability of the CA preservative-treated laminate were examined, and a comparison was made of the interlaminar shear performance of preservative-treated glulam under different environmental conditions.

EXPERIMENTAL

Materials

The wood species used in the study were lodgepole pine (*Pinus contorta* Douglas ex Loudon) from Canada and scotch pine (*Pinus sylvestris* L.) from Europe. The air-dry density of lodgepole pine and scotch pine was 0.693 and 0.478 g/cm^3, respectively. The selected wood samples consisted of sapwood, featuring some live knots, with an average moisture content of 13% following kiln drying. The preservative used in the tests was copper azole (CA) preservative, which is composed of divalent copper, azolane compound, ammonia or amine, and water. Two types of adhesives were used in the tests: one-component polyurethane (PUR) adhesive (manufacturer: Franklin International Co. Ltd.) with an initial viscosity of 6000 mPa·s (23 °C) and two-component resorcinol formaldehyde (RF) adhesive (manufacturer: Franklin International Co., Ltd.) with an initial viscosity of 5000 mPa·s (23 °C) after mixing.

Preparation of Preservative Treated Glulam Laminate

The CA preservative (0.3 wt%) was impregnated into lodgepole pine and scotch pine laminates (dimensions of 1 m in length, 100 mm in width, and 30 mm in thickness) using Bethell's full-cell process (Frias *et al*. 2021). The full-cell process consisted of three stages. In the initial stage, the vacuum pressure was set to -0.075MPa and maintained for 30 min to evacuate the air outside the wood. During the high-pressure impregnation stage, the pressure was set to 0.8 MPa and maintained for 30 min to impregnate the preservative into the wood. In the final stage, the vacuum pressure was set to -0.07 MPa and maintained for 20 min to extract the excess preservatives from the wood and the high-pressure tank. After impregnation, the laminates were dried in a drying kiln. After drying, the preservative treated laminates had a moisture content of approximately 15%, with a preservative retention of 4 kg/m^3 (standard deviation 0.16), and the depth of preservative penetration exceeds 85%.

Surface Wettability Test of Glulam Laminate

The surface wettability of the preservative-treated laminate was tested using the contact angle measuring instrument (OCA200, Krüss Scientific Instrument Co., Ltd., Germany) in an environment with a relative humidity (RH) of 65 ± 2% and a temperature of 21± 2 °C. Due to the water-soluble nature of the preservative, distilled water was chosen as the 5 μL test droplet, and the contact angle was measured after the droplet was in contact with the laminate sample for 1 second. The surface of the preservative-treated laminates exhibited three states: unplaned, planed to a thickness of 0.6 mm, and planed to a thickness of 1.2 mm. There were a total of 8 groups of samples, with 6 pieces in each group. The dimensions of the samples were 50 x 50 x 10 mm, and the measurement points were located in the center of the samples.

Interlaminar Shear Performance Test of Glulam

Defect-free laminates and defect-free preservative-treated laminates were selected respectively for fabricating glulams. The dimensions of glulams composed of two laminates were 1 m in length, 10 mm in width, and 50 mm in depth. The composite process of glulam was conducted under a pressure of 1.15 MPa at 26 °C for 24 h, with an adhesive application rate of 250 g/m^2. Glulams were then processed into block shear specimens for shear testing following ASTM D905 (2013). The shear tests were performed using a universal testing machine (AG-IC, Suns Technology Stock Co., Ltd., China) at 21 ± 2 °C and 65 ± 5% RH. For the damp-heat treatment of specimens, the block shear specimens were placed in a constant temperature and humidity chamber. After 36 h, the specimens were removed, and their shear strength was immediately measured. The damp-heat treatment environment was categorized into three states: environment A (50 °C, 95% RH), environment B (50 °C, 85% RH), and environment C (20 °C, 60% RH). The test parameters are detailed in Table 1, with 20 groups consisting of 6 pieces in each group.

Table 1. Design of Experiment

Set Code	Species	Preservative	Adhesive	Planed (mm)	Damp-heat treatment	Natural aging (day)
L1	Lodgepole pine	CA	PUR	0	None	0
L2	Lodgepole pine	CA	PUR	0	None	0
L3	Lodgepole pine	CA	RF	0	None	0
L4	Lodgepole pine	CA	RF	0.6	None	0
L5	Lodgepole pine	CA	PUR	0.6	None	30
L6	Lodgepole pine	CA	PUR	0.6	None	60
L7	Lodgepole pine	CA	PUR	0.6	None	90
L8	Lodgepole pine	None	PUR	0	None	0
L9	Lodgepole pine	None	PUR	0	None	30
L10	Lodgepole pine	None	PUR	0	None	60
L11	Lodgepole pine	None	PUR	0	None	90
L12	Lodgepole pine	None	RF	0	None	0
S1	Scotch pine	CA	PUR	0	None	0
S2	Scotch pine	CA	PUR	0	None	0
S3	Scotch pine	CA	RF	0.6	None	0
S4	Scotch pine	CA	RF	0.6	None	0
S5	Scotch pine	CA	PUR	0.6	Environment C	0
S6	Scotch pine	CA	PUR	0.6	Environment A	0
S7	Scotch pine	CA	PUR	0.6	Environment B	0
S8	Scotch pine	CA	RF	0.6	Environment C	0
S9	Scotch pine	CA	RF	0.6	Environment A	0
S10	Scotch pine	CA	RF	0.6	Environment B	0

Natural Aging Treatment of Specimens

The specimens underwent natural aging treatment on the campus of Nanjing Forestry University (45° 39′54″N, 25° 33′27″E, Elevation 27.3 m). Table 2 details the weather conditions in Nanjing from October 2019 to January 2020. Subsequently, the bonding performance of the samples was evaluated.

Table 2. Climate Conditions During the Experiment

Year	Month	Average Temp (°C)	Maximum Temp (°C)	Minimum Temp (°C)	Relative Humidity (%)	Total Rainfall (mm)	Average Cloud Cover (%)
2019	10	20.2	30.0	13.0	47.1	0.6	26.5
	11	14.8	26.0	-1.0	49	26.1	35
	12	8.7	17.0	-2.0	67	89.4	47
2020	1	5.5	11.0	-3.0	82	167.8	83

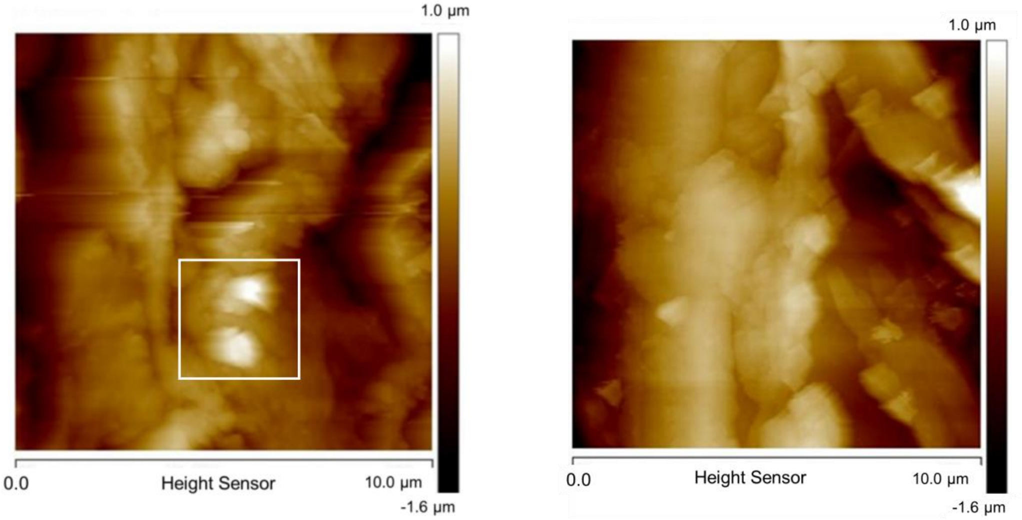
Data Analysis Procedure

All data analyses regarding interlaminar shear performance of the glulams were performed using SPSS statistical software. The test results were subjected to one-way analysis of variance (ANOVA) at a 5 percent significance level. For the bond shear strength of each group of glulams made of planed or unplaned laminations, multiple comparisons were tested by ANOVA at $p < 0.05$.

RESULTS AND DISCUSSION

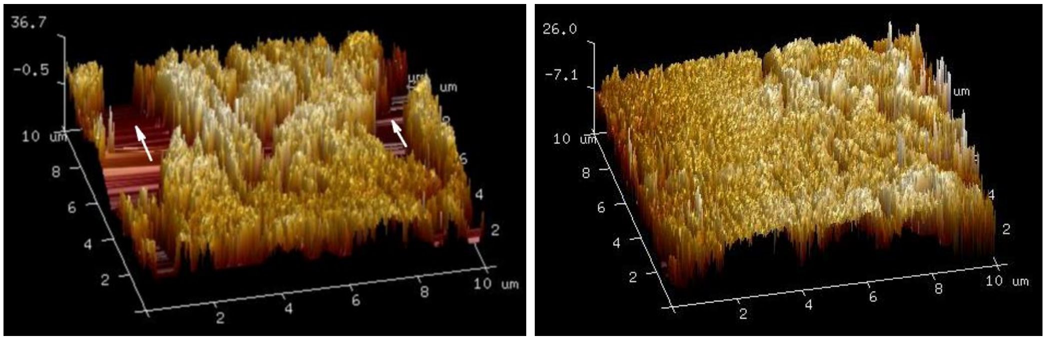
Surface Characteristics of Preservative Treated Glulam Laminate

The emergence of the atomic force microscope (AFM) has provided a new technical means for studying the ultrastructure of natural biomaterials (Zheng *et al*. 2019). The AFM device (ICON, Bruker Co., Ltd., Germany) was used to image the surface of preservative-treated glulam laminates before and after planing (thickness approximately 0.6 mm) in the air. The AFM image could reveal the coverage of particulate matter on the fiber surface (Li *et al*. 2009). The structures on the surface of preservative-treated glulam laminates are shown in Figs. 1, 2, 3, and 4.



(a) Before planing **(b)** After planing

Fig. 1. Typical height diagram of scotch pine preservative treated laminate's surface

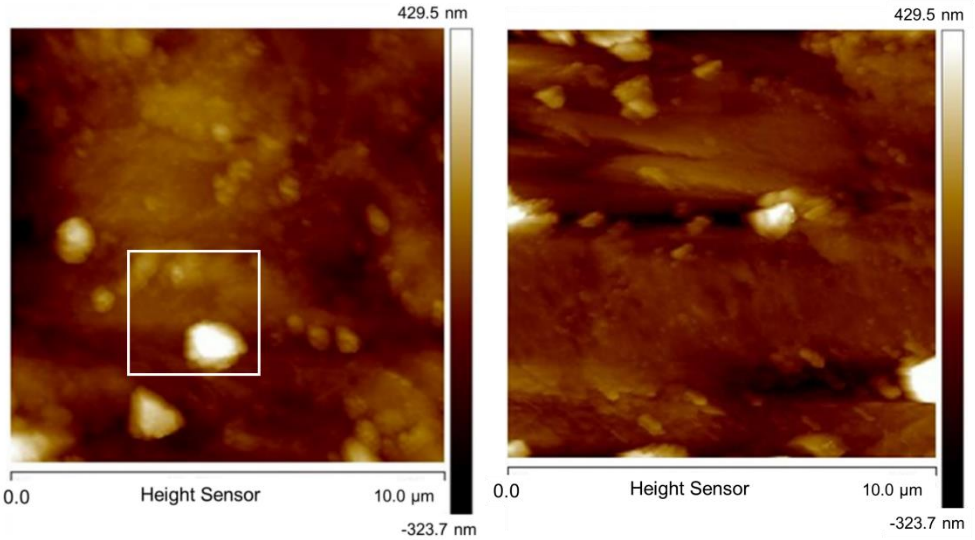


(a) Before planing **(b)** After planing

Fig. 2. Typical three-dimensional phase diagram of scotch pine preservative treated laminate's surface

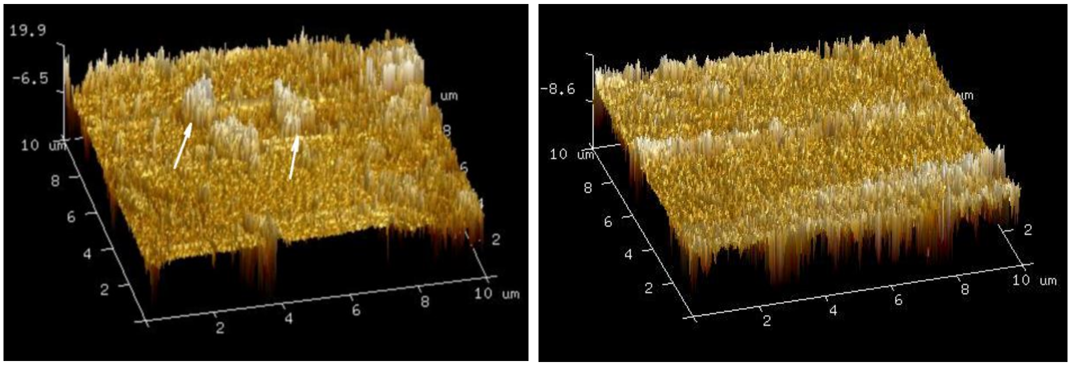
As shown in Figs. 1 and 2, the aggregation of particles on the surface of scotch pine preservative-treated laminate before planing (as marked in the box in Fig.1) was significantly greater than that after planing. The three-dimensional phase diagram of the surface underwent a sharp change before planing (as marked by the arrows in Fig. 2). The preservative-treated glulam laminate was planed smooth prior to preservative treatment, with a substantial amount of preservatives adhering to the surface post-treatment, reflecting the discontinuous coating characteristic of the preservatives. Following planing, the particles gathered on the surface were noticeably reduced in quantity and size, indicating a relatively uniform distribution of wood cells coexisting with preservatives attached to the cell wall and cavity.

The AFM diagrams of lodgepole pine preservative treated laminate's surface in Figs. 3 and 4 exhibit similar characteristics to the Scotch pine laminate's surface. The surface of the lodgepole pine preservative treated laminate demonstrates the coexistence of wood and preservatives both before and after planing. However, the aggregation of particles is significantly larger before planing. The significant change in the three-dimensional phase diagram of the surface before planing also reflects this characteristic.



(a) Before planing **(b)** After planing

Fig. 3. Typical height diagram of lodgepole pine preservative treated laminate's surface



(a) Before planing **(b)** After planing

Fig. 4. Typical three-dimensional phase diagram of lodgepole pine preservative treated laminate's surface

In conclusion, the AFM images taken in the air clearly depict the surface structure of preservative-treated glulam laminate before and after planing. After planing, the preservative particles on the wood surface are relatively evenly distributed with a relatively low proportion.

Laminate Surface Wettability

The surface contact angles of preservative-treated glulam laminates are shown in Fig. 5. The contact angle of each group of laminates gradually decreased over time, indicating that the liquid gradually permeated the laminate surface. The surface contact angle of the preservative-treated laminate is larger than that of the untreated normal laminate, suggesting that the presence of preservatives hinders the surface wetting of the laminate.

The surface contact angles of lodgepole pine and scotch pine preservative-treated laminates were different before and after planing, with the contact angles decreasing after planing. During the high-pressure immersion process, the preservative permeates from the surface to the interior of the laminate, with a gradual decrease in concentration (Ganguly *et al.* 2024). The preservative concentration on the surface is higher than that in the inner layer, and surface planing effectively removes excess preservatives, exposing fresh wood in the inner layer and promoting liquid penetration of the wood cell wall. Laminates planed to 1.2 mm thickness exhibited better surface wettability than those planed to 0.6 mm, with lower preservative content in the former.

Considering that the loss of wood planing caused by planing 1.2 mm was greater than that of planing 0.6 mm, and the lower preservative content in the former is not conducive to the durability of laminates, the planing thickness of 0.6 mm for preservative treated glulam laminates was judged to be more economical and practical.

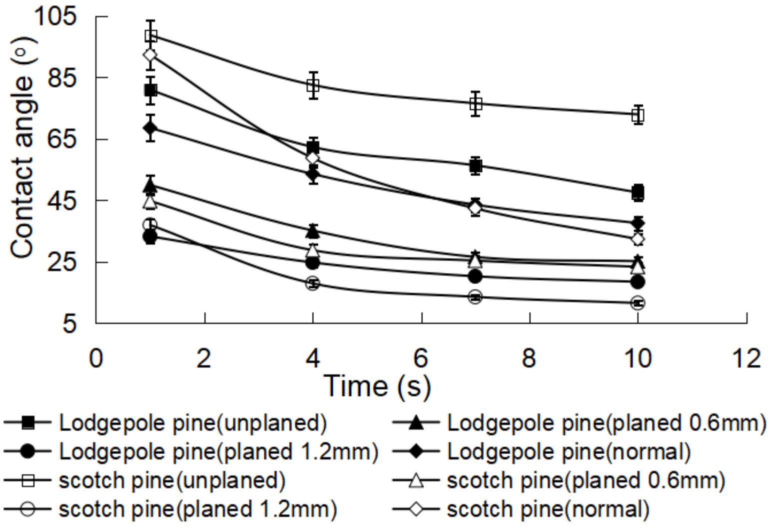


Fig. 5. Surface contact angle of preservative treated glulam laminate

Effect of Planing on Shear Performance of Preservative Treated Glulam

For the bond shear strength of glulams made of planed or unplaned laminations, multiple comparisons were tested by ANOVA at $p < 0.05$. The interlaminar shear strength of glulam made of planed laminates is depicted in Fig. 6. The values were significantly higher than that of glulam made of unplaned laminates, with the former showing an increase of more than 15% in shear strength. Planing plays a crucial role in the composite process of preservative-treated glulam. By planing the surface of the preservative-treated laminate, the influence of a large number of preservatives attached to the surface is removed, resulting in relatively even distribution of preservative particles on the surface with a smaller proportion (Tascioglu 2007). Additionally, planing can cause damage to the cell wall on the wood surface, thus opening up the cells and facilitating the penetration and bonding of adhesives. Therefore, planing the laminate surface before gluing contributes to enhancing the bond shear performance of preservative-treated glulam.

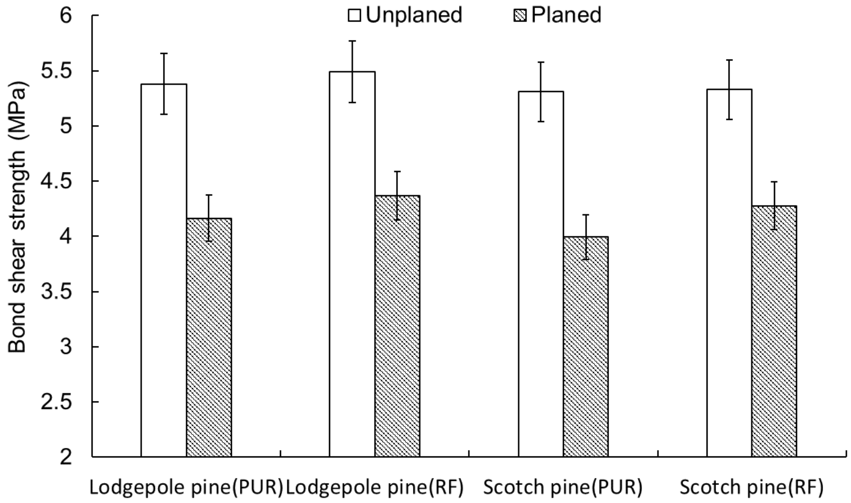
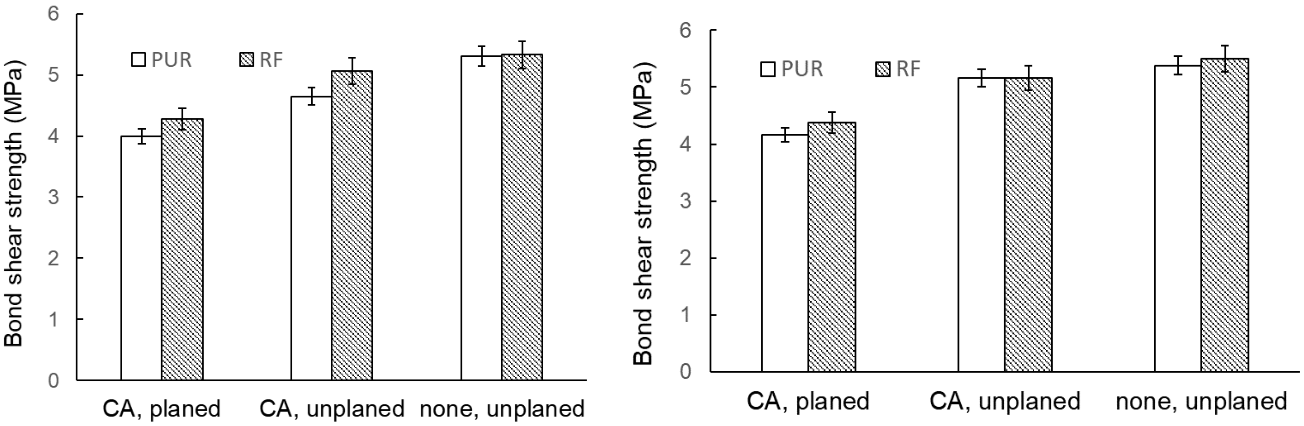


Fig. 6. Shear performance of glulam made of planed and unplaned laminates

Effect of Adhesive Type on Interlaminar Shear Performance

The effect of PUR adhesive and RF adhesive on interlaminar shear strength is depicted in Fig. 7. The shear strength of lodgepole pine and scotch pine glulam with different adhesives followed a consistent pattern, with RF displaying superior bonding performance compared to PUR. PUR and RF adhesives are commonly used as structural adhesives due to their excellent bonding properties.



(a) Lodgepole pine preservative glulam **(b)** Scotch pine preservative glulam

Fig. 7. Shear strength of different adhesive test groups

During the curing process of PUR, water in the wood reacts with isocyanate (-NCO), which then decomposes into amine and carbon dioxide after carbamate formation. Additionally, the decomposed amine reacts with unreacted isocyanate group (-NCO), facilitating the formation of a network structure in the adhesive molecular chain and enhancing bonding strength. The bonding strength of PUR is sensitive to the content of isocyanate (-NCO) and wood moisture content. Inadequate isocyanate (-NCO) content and excessive wood moisture content can compromise the chemical bonding between the adhesive and wood, thereby affecting bonding properties.

The moisture content of glulam laminates post-preservative treatment and drying treatment was higher than those without preservative treatment. Moreover, the preservatives present on the surface impede the penetration of PUR onto the laminate surface to some extent, which in turn affects the bonding of PUR with wood, indicating that the bonding performance of PUR was inferior to that of RF. RF adhesive is more suitable for manufacturing preservative-treated glulam than PUR adhesive.

For unplaned glulams, the interlaminar shear strength of the glulam without preservative treatment was superior to that of the preservative-treated glulam. The preservative-treated glulam was composed of unplaned preservative-treated laminates. The presence of a large amount of preservatives on the surface of the unplaned laminates hinders the adhesive penetration, making it challenging to achieve a reliable bond between the adhesive and the glulam laminate (Zhao 2020).

Effect of Wet Environment on Interlaminar Shear Performance

Scotch pine was chosen for testing in a wet environment. Figure 8 shows that increasing humidity and temperature had a negative impact on the interlaminar shear strength of both the scotch pine preservative-treated glulam and the untreated normal glulam. As humidity levels rose further, this negative impact increased, leading to a continuous decrease in interlaminar shear strength.

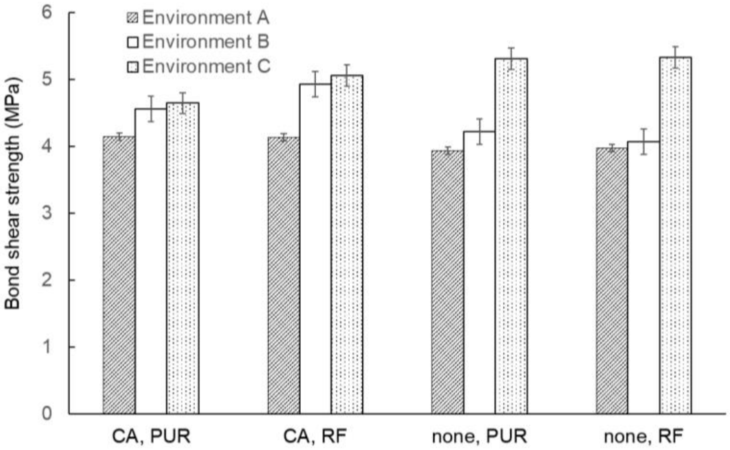


Fig. 8. Shear strength of different wet environment test groups

The interlaminar shear strength of scotch pine glulam without preservative treatment decreased in an environment with 85% RH and 50 °C compared to that in an environment with 60% RH and 20 °C. The interlaminar shear strength of two types of glulam with PUR and RF decreased by 21% and 24%, respectively. In high temperature and high humidity environments, wood absorbed moisture and softened, which reduced interlaminar shear strength to some extent. A slight decrease in the interlaminar shear strength of scotch pine glulam without preservative treatment was observed when the humidity increased from 85% RH to 95% RH, which was almost negligible. The change in interlaminar shear strength of scotch pine preservative treated glulam was relatively mild with the increase in environmental humidity and temperature. For scotch pine preservative treated glulam bonded with PUR, the shear strength decreased by 2% when the environment changed from 60% RH, 20 °C to 85% RH, 50 °C, and it further decreased by 9% when the humidity increased from 85% RH to 95% RH. In the case of preservative treated glulam bonded with RF, the shear strength decreased by 3% when the environment changed from 60% RH, 20 °C to 85% RH, 50 °C, and further decreased by 16% when the humidity increased from 85% RH to 95% RH.

Effect of Natural Aging on Interlaminar Shear Performance

Lodgepole pine was chosen for natural aging experiments. As shown in Fig. 9, the interlaminar shear strength of preservative-treated lodgepole pine glulam decreased in a polynomial manner ($y=0.0002x^2-0.302x+5.1115$, $R^2= 0.9432$) over time during natural aging. The interlaminar shear strength of untreated lodgepole pine normal glulam also exhibited a decreasing trend in a polynomial manner ($y=0.0002x^2-0.0036x+5.3315$, $R^2= 0.9621$). After 30 days of natural aging, there was a significant decrease in the interlaminar shear strength of both preservative-treated and normal lodgepole pine glulam, with further decreases observed at 60 and 90 days, although the difference was not pronounced.

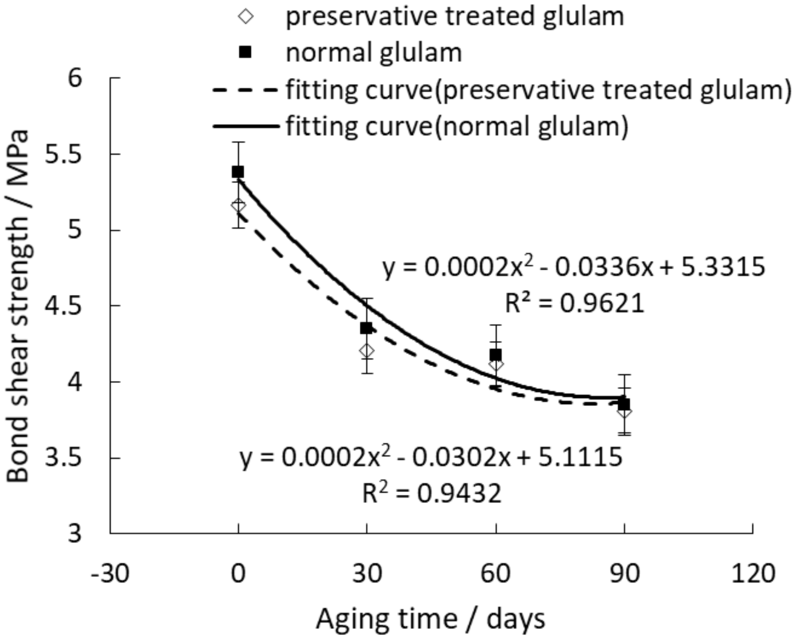


Fig. 9. Shear strength of glulam after natural aging treatment

During the initial 30 days of natural aging, the bonding strength of the samples decreased sharply as a result of the dry shrinkage and swelling of the wood, as well as the release of stresses caused by adhesive curing (Tascioglu, 2007). By the 60th and 90th day of natural aging, the shear strength of the preservative treated glulam decreased at a slower rate due to the gradual release of internal stresses. Exposure to outdoor environmental changes not only impacted the chemical bond between the adhesive and wood, but also altered the properties of both, leading to a reduction in bonding strength. For the bonding interface, aging has a greater impact on the edges of the interface than on the interior.

CONCLUSIONS

1. Using wood treated with preservatives to produce preservative-treated glulam is an effective method for improving the durability of glulam. This method overcomes the challenge of immersing large cross-section glulam in preservatives. After treatment, a large number of preservative particles are attached to the surface, with less evenly distributed preservative content. Planing can enhance the surface wettability of laminates. The presence of preservatives significantly reduces the interlaminar shear strength of glulam, but planing can effectively mitigate this impact on the bonding performance of glulam.
2. For CA-preserved glulam, the choice of adhesive significantly impacts the interlaminar shear strength of the glulam. RF adhesive outperforms PUR adhesive, making it more suitable for the technological bonding of CA-preserved glulam. Exposure to wet environments tends to decrease the interlaminar bonding performance of CA-preserved glulam. In high humidity conditions, the interlaminar shear strength of CA-preserved glulam is slightly higher than that of untreated glulam. Over time, the interlaminar shear strength of lodgepole pine CA-preserved glulam and untreated common glulam decreases in a polynomial function. To ensure high-quality CA-preserved glulam, it is recommended to plane the laminate to 0.6 mm and use RF adhesive.

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