Effect of the ACQ Preservative on the Bonding Strength of Aqueous Polymer Isocyanate Bonded Masson Pine Joints and on the Adhesive Penetration into Wood

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The influence of alkaline copper quaternary (ACQ) preservative was studied relative to the bonding strength of Masson pine joints and the penetration of the adhesive into wood. Masson pine specimens treated with ACQ of three concentrations (0.1%, 0.5%, 1.0%) were bonded with aqueous polymer isocyanate (API) adhesive, and the shear strength, wood failure percentage, bondline thickness, average penetration depth (*AP*), and effective penetration depth (*EP*) of the wood joints were evaluated. The shear strength (6.34 to 6.85 MPa) and the wood failure percentage (87.0% to 87.8%) of the three series of treated joints were significantly lower than that of the untreated samples, while the treated specimens showed significantly larger bondline thickness (43.3 to 47.2 μm), smaller *AP* (30.6 to 35.8 μm), and smaller *EP* (24.7 to 25.7 μm) than the untreated specimens. The increase of ACQ concentration from 0.1% to 1.0% had no significant impact on the bonding strength and adhesive penetration parameters. The correlation between shear strength and penetration depths of treated joints was significant at the 0.01 level based on Pearson correlation analysis, while the coefficient of determination (R^2) of the shear strength resulted from least squares regression analysis was 0.250 and 0.143 for *AP* and *EP*, respectively.

Keywords: ACQ preservative treatment; Aqueous polymer isocyanate; Masson pine; Bonding strength; Adhesive penetration

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INTRODUCTION

Preservative treatments are a widely used approach to increase the service life of wood and wood products (Yang *et al.* 2012). Although various chemicals such as creosote oil, coal tar, and pentachlorophenol prevent wood degradation by white and brown rot fungi, termites, and marine borers, the low-toxicity and environmentally friendly water-based preservatives have gradually become the leading products. Chromated copper arsenate (CCA) has high efficiency and low cost as a commercial wood preservative, but its high toxicity has decreased its use (Onuorah 2000; Antwi-Boasiako and Damoah 2010). Alkaline copper quaternary (ACQ) has become one of the most promising preservatives, for its good permeability in wood, resistance to leaching in water, long-term efficiency, low toxicity, and the absence of arsenic, chromium, and phenols (Goodell *et al.* 2007).

Masson pine (*Pinus massoniana* Lamb.) is the most widely distributed pine species in China and has become one of the dominant forest plantation species in southern China. It has good mechanical properties, with a bending strength of 87.9 MPa and elastic modulus of bending strength of 13.8 GPa for mature plantation wood (Bao *et al.* 1998). Its growth rate is more rapid than other pine species in China, and thus it could be an important raw material for structural timbers. However, the sapwood of Masson pine is easily subjected to wood-decaying fungi and insect attack, and it must be pre-treated with preservatives for outdoor and construction uses. In addition, Masson pine wood has more large knots and lower trunk straightness than pine species in colder areas of northern China (Hu *et al.* 2018). Processing it into glued laminated timbers has become, therefore, an important way to improve the utilization rate of logs, and to manufacture high-performance structural products.

Preservative treatments change both the physical (such as surface free energy and surface roughness) and chemical (such as pH and buffer capacity) properties of wood, and thus could affect the bonding strength of glued products. The influence mechanism differs according to different categories of wood, preservatives, and adhesives. Soluble copper and monoethanolamine (MEA) in ACQ accelerate the cure of phenol-resorcinol-formaldehyde resin (PRF) and reduce its bonding strength with southern pine (Lorenz and Frihart 2006). Studies on Japanese larch concluded that ACQ had no influence on both bonding strength and delamination (Miyazaki and Nakano 2003). Moreover, Shukla and Kamdem (2012) reported that ACQ treatment showed no significant effect on most of the properties (such as density, flexural modulus of rupture (MOR), and modulus of elasticity (MOE) of southern pine laminated veneer lumber (LVL). The results from Jin *et al.* (2010) showed that the mechanical properties of bamboo oriented strand board (OSB) were not affected by pre-treatment with ACQ within the retention level of 1.52 to 4.67 kg/m^3. Up to now, there has been a lack of published research involving the bonding strength of treated Masson pine with ACQ.

During the formation of wood/adhesive glue joints, adhesives undergo five distinct motions (Marra 1992), the penetration of adhesives into the porous structure of wood is essential because it influences the bond quality and subsequently the performance of the whole structure. Mechanical interlocking and chemical bonds are most important and common among all bonding mechanisms. According to the mechanical interlocking mechanism, the bonding strength depends on the penetration of adhesives in wood, and thus a deeper penetration contributes to a higher bonding strength. Therefore, adhesive penetration is an important predicator of bond strength and durability (Modzel *et al.* 2011).

Adhesive penetration of wood occurs on micrometer level (gross penetration), a nanometer level (cell wall penetration), or other smaller levels of penetration (Frihart 2004). The gross penetration has been studied qualitatively and quantitatively by various methods such as transmitted light microscopy (Hare and Kutscha 1974), fluorescent microscopy (Johnson and Kamke 1992), scanning electron microscopy (Koran and Vasishth 1972), and X-ray microtomography (Modzel *et al.* 2011), while the cell wall penetration has only been qualitatively detected by techniques such as ultraviolet microscopy (Gindl *et al.* 2002), X-ray energy dispersive spectrometer (Buckley *et al.* 2002), confocal Raman microscopy (Gierlinger *et al.* 2005), and nanoindentation (Konnerth and Gindl 2006). Generally, gross penetration is measured by penetration depth of the adhesive, and it is strongly influenced by wood factors (wood species, anatomical orientation and surface roughness), adhesive factors (adhesive type and viscosity), and process factors (applied pressure and temperature) (Kamke and Lee 2007). The effects of preservatives on wood surface properties and curing characteristics of adhesives might lead to different penetration depths between treated and untreated wood. Although various analytical techniques have been used to determine the depth of the adhesive penetration in untreated wood as well as several categories of modified wood, a quantitative characterization on adhesive penetration in preservative treated wood has not been reported.

In this study, the effects of the ACQ preservative on the bonding strength of aqueous polymer isocyanate (API) bonded Masson pine joints and on the adhesive penetration into wood was investigated. Masson pine wood samples were treated with ACQ of three concentrations (0.1%, 0.5%, 1.0%) and then bonded with the API adhesive. The bonding strength and adhesive penetration of glue joints were investigated with a mechanical testing machine and optical microscope, respectively. The difference between treated and untreated wood/API glue joints and the correlation between bonding strength and adhesive penetration for the treated joints were analyzed.

EXPERIMENTAL

Materials

Masson pine logs with a length of 2 m were collected from a 22-year-old progeny test forest of primary seed orchard in Nanning Forestry Division. The logs were sawn into timbers with a thickness of 35 mm and dried in a conventional kiln. The dried timbers were further processed into boards with a size of 300 mm × 50 mm × 20 mm (longitudinal × tangential × radial). The final wood moisture content and air-dried density were 11.10% and 0.58 g/cm^3, respectively.

The wood preservative solution, with 15.4% ACQ-D (68.4% copper amine expressed as copper oxide to 31.6% didecyldimethylammonium chloride (DDAC) quaternary ammonium compound), was purchased from Green Thai Environmental Technology Co., Ltd. (Shenzhen, China).

Two-component API adhesive was provided by Xin Mei Environmental Technology Co., Ltd. (Guilin, China), and the weight ratio of main agent to curing agent was 100:20. The main agent was a mixture of poly (vinyl alcohol) (PVOH) solution, emulsifiers including poly(styrene-*co*-butadiene) (SBR) and poly(ethylene-*co*-vinyl acetate) (EVA), and filler of calcium carbonate ($CaCO_3$). The main agent was white viscous liquid with a solid content of 60%, and its viscosity and pH were 20 Pa·s and 7.0, respectively. The curing agent (polymeric methylene *bis*-(phenyl isocyanate) (pMDI)) was brown liquid with a viscosity of 0.3 Pa·s.

Methods

Preservative treatment

Masson pine boards were placed into an impregnation vessel with ACQ solution (0.1%wt, 0.5%wt, 1.0%wt), and the vessel was moved into a pressure tank. A preliminary vacuum was applied at -0.08 to -0.09 MPa for 30 min, and pressure was then applied up to 1.2 MPa for 60 min. The retention of wood preservatives was calculated as follows,

$$R = \frac{(m_1 - m_2) \times C}{V} \times 1000 \tag{1}$$

where R is the retention of wood preservatives (kg/m^3), m_1 is the mass of the wood specimens after impregnation treatment (kg), m_2 is the mass of the wood specimens before impregnation treatment (kg), C is the concentration of preservation solution (%), and V is the volume of wood specimens before impregnation treatment (m^3).

The treated wood specimens were transferred to an oven at 40 °C for drying. After drying for 1 week, the specimens were transferred to a humidity chamber (temperature of

20 ± 1 °C, relative humidity of 65 ± 5%) for another two weeks. The penetrating of wood preservatives was measured according to GB/T 31761 (2015).

Bonding test

The gluing surface of specimens was sanded before being pressed into 2-layer wood joints under unit pressure of 1.0 MPa for 8 h. The API adhesive was applied with a spreading ratio of 250 g/m^2. Each set of tests was repeated 10 times, and two samples were cut from each joint (Fig. 1). A few samples with processing defects were abandoned, and 17 to 20 samples were obtained for each set of tests. The shear strength and wood failure percentage were measured by a universal mechanical testing machine with loading speed of 0.5 kN/s, according to the GB/T 26899 (2011).

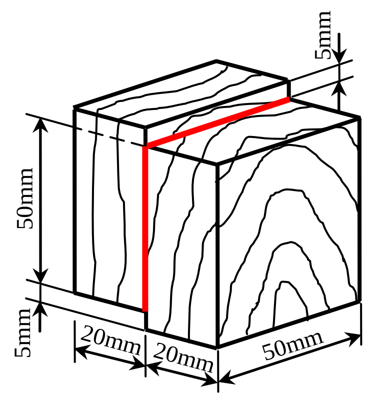


Fig. 1. An illustration of laminate with a 50 mm-glued surface and a 5 mm-unglued portion

Adhesive penetration measurement

For each wood joint, one block with length of 20 mm was first cut across the longitudinal direction. The block was then processed to a thinner block with a thickness of 7 mm, ensuring that the bondline located nearly the central of the block. Two small blocks with a width of 7 mm were finally cut from the thinner block, and in total 20 samples were obtained for each set of tests. The samples were impregnated in water for 3 d. Transverse sections with a thickness of 25 μm were cut from the samples using a sliding microtome and dehydrated by placing them in an alcohol solution under progressively increasing concentrations (30%, 50%, 70%, 95%, 100%) for 10 min. The dehydrated slices were fixed between a glass slide and a cover glass with a drop of Canada balsam.

The bondline thickness and gross penetration were measured by a Nikon microscope (Eclipse Ni-E, Japan). The average penetration depth (*AP*) calculated using Eq. 2 represents the average depth of penetration for several column tissues within the entire measurement length, while the effective penetration depth (*EP*) calculated using Eq. 3 represents the total adhesive area detected in the interphase region divided by the width of the bondline (Qin *et al*. 2016),

$$AP = \sum_{i=1}^{N} y_i / N \tag{2}$$

$$EP = \sum_{i=1}^{N} A_i / X_0 \tag{3}$$

where y_i is the penetration depth of on column tissue (μm), and N is the total column number of tissues in measurement length (μm). A_i is the area of adhesive object i (μm^2), and X_0 is the length of the bondline in the measurement area (50 measurement areas; bondline length of each area was 1400 μm in this article).

An illustration of measurement parameters in Eq. 2 and Eq. 3 is shown in Fig. 2. Image processing and analysis software are usually used to measure these three parameters (Johnson and Kamke 1992). X_0 and y_i could be easily acquired with Image-Pro Plus software (Media Cybernetics Incorporated, Rockville, USA). A_i was usually measured by circling the adhesive area using MATLAB software (Math Works Incorporated, Natick, MA, USA), which was proved to be a highly efficient and simple way.

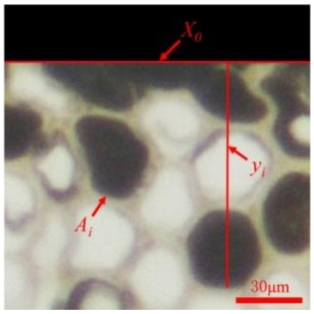


Fig. 2. Measurement parameters in experimental image

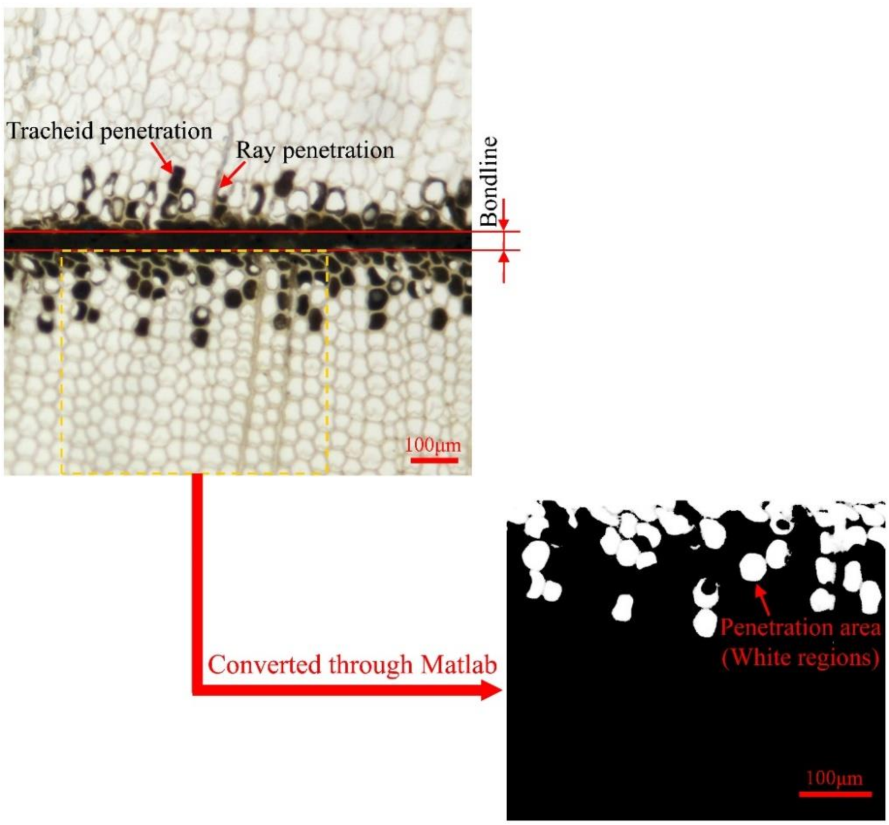


Fig. 3. Calculation of penetration area (A_i) by MATLAB software

To be specific, the target area was first chosen from the photomicrograph, and then it was converted to binary images using MATLAB software (Fig. 3). Consequently, adhesive regions were converted to white areas, while the remaining regions were converted to black areas. A_i was finally obtained by counting the pixel elements of white areas and a simple manual calculation.

Statistical analysis

Pearson correlation analysis and least squares regression analysis were used to investigate the relationship between penetration parameters and bonding strength with SPSS Statistics (International Business Machines Corporation, Armonk, NY, USA).

RESULTS AND DISCUSSION

Preservative Retention

As shown in Fig. 4, the retentions of ACQ with concentrations of 0.1%, 0.5% and 1.0% were 0.597 kg/m^3, 3.051 kg/m^3, 6.320 kg/m^3, respectively, and the retention had a highly linear correlation with concentration. The penetration of ACQ in Masson pine wood was larger than 90% for the all three set of tests. Above all, ACQ penetrated well in Masson pine sapwood under conventional vacuum-pressure treatment, and its retention was easily adjusted by controlling the ACQ concentration.

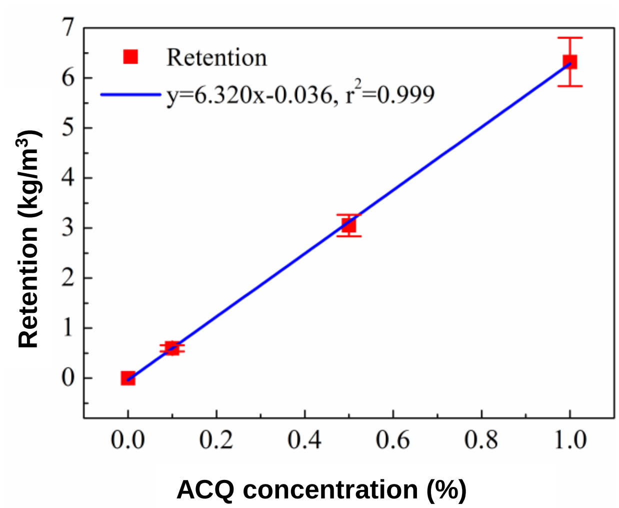


Fig. 4. Retention of ACQ in Masson pine wood at different concentrations

Bonding Strength

As shown in Fig. 5, the shear strength of the three series of ACQ treated wood/API glue joints were 6.34 to 6.85 MPa, which were 16.6% to 22.8% lower than that of the untreated sample. The wood failure percentage of the three series of ACQ treated wood/API glue joints were 87.0% to 87.8%, which were 11.8% to 12.6% lower than that of the untreated sample. The measured shear strengths were further compared on the basis of concentrations by analysis of variance (ANOVA) and Fisher LSD multiple range test (Fig. 5). The results showed that both the shear strength and the wood failure percentage of the ACQ treated samples were significantly different from that of the untreated sample, while there was no significant difference in that between the three treated groups.

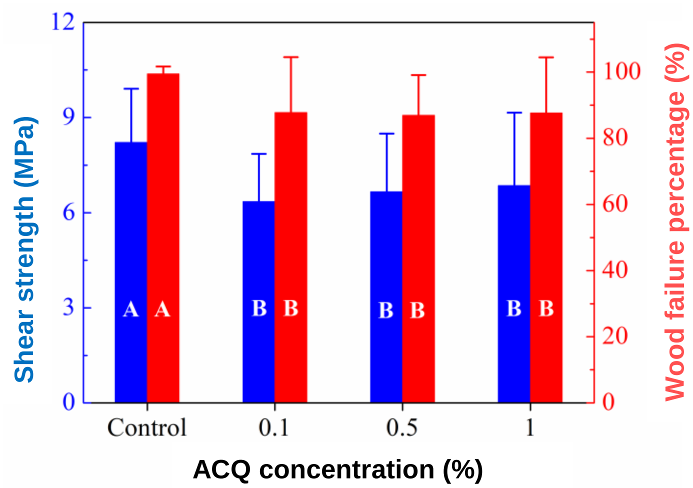
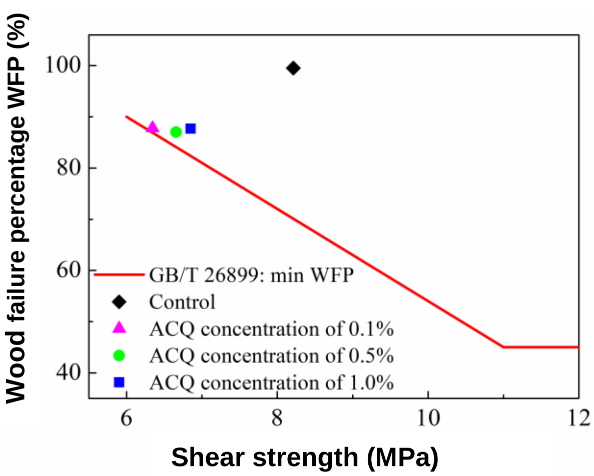
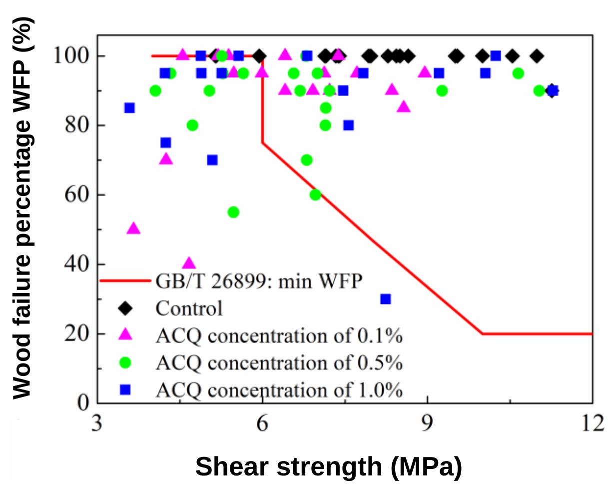


Fig. 5. Bonding strength of Masson pine/API joints with different ACQ concentrations (Statistically homogenous groups determined using LSD's significance test)

The average and single values of shear strength and wood failure percentage of untreated Masson pine joints met the criteria of GB/T 26899 (2011) (Fig. 6). Although the average shear strength and wood failure percentage of treated joints met the criteria, some of the single values failed to meet the criteria. This result suggested that the bonding strength of ACQ treated Masson pine joints need to be improved by optimizing the processing parameters, adding primers, or replacing the adhesive.



(a) Average value



(b) Single value

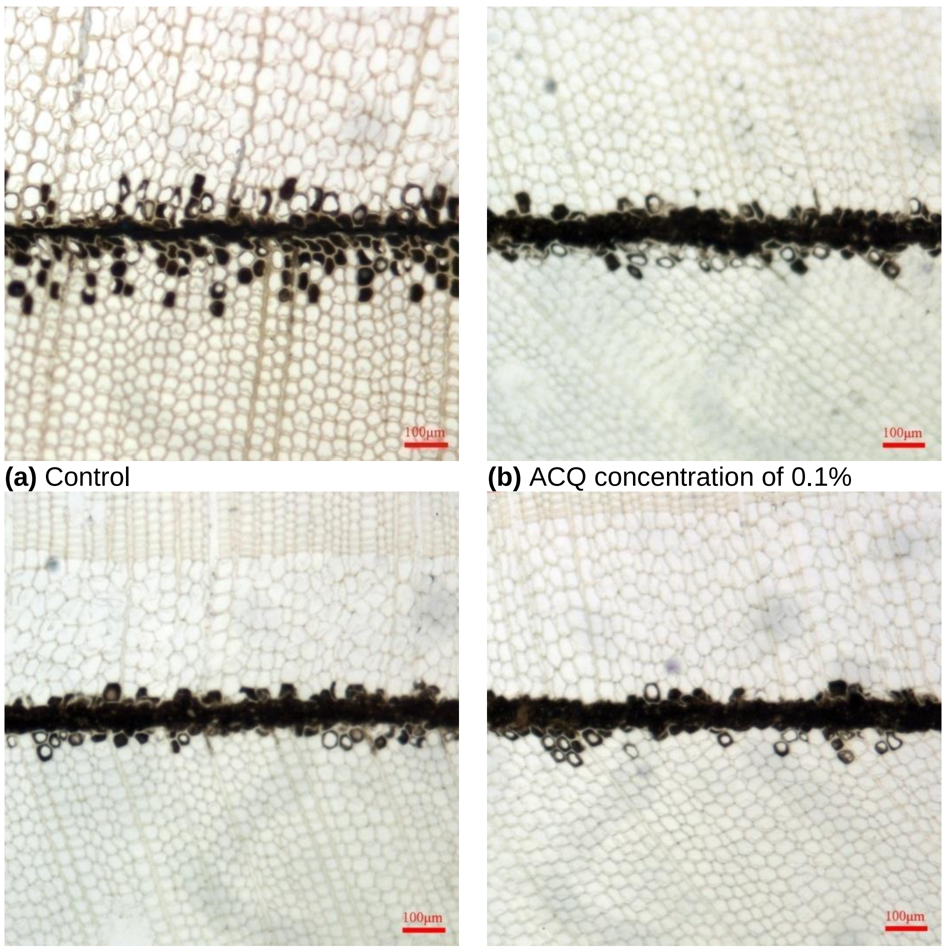
Fig. 6. Correspondent relation between shear strength and wood failure of Masson pine/API joints with different ACQ concentrations

Adhesive Penetration

Micrographs of the bonding interphase of wood/API glue joints are shown in Fig. 7. The dark area represented the adhesive zone. The treated samples had smaller penetration depth and larger bondline thickness than the untreated sample, which indicated that the API adhesive showed poorer penetrability in ACQ treated Masson pine wood than in untreated wood.

The penetration parameters of the API adhesive in glue joints are given in Table 1.

The bondline thickness of the three series of ACQ treated wood joints was 43.3 to 47.2 μm, which was 63.7% to 78.6% higher than that of the untreated sample. Average penetration depth and effective penetration depth of the treated samples were 30.6 to 35.8 μm and 24.7 to 25.7 μm, respectively, which were 44.7% to 52.9% and 29.2% to 31.9% lower than that of the untreated sample. The measured penetration parameters were further compared on the basis of concentrations by analysis of variance (ANOVA) and Fisher LSD multiple range test (Table 1). The penetration parameters of the ACQ treated samples were significantly different from the untreated sample, while there was no significant difference between the three treated groups.



(a) Control **(b)** ACQ concentration of 0.1% **(c)** ACQ concentration of 0.5% **(d)** ACQ concentration of 1.0%

Fig. 7. Micrographs of the bonding interphase of wood/API glue joints

Table 1. Bondline Thickness and Penetration Depth of API Adhesive

ACQ concentration	Bondline Thickness (μm)	Average Penetration Depth (μm)	Effective Penetration Depth (μm)
Control	26.44 ± 5.25* B	64.86 ± 22.60 A	36.28 ± 5.88 A
0.1%	47.21 ± 11.19 A	30.57 ± 9.65 B	25.70 ± 6.70 B
0.5%	43.28 ± 21.32 A	32.51 ± 10.42 B	24.88 ± 5.57 B
1.0%	43.46 ± 12.34 A	35.84 ± 10.11 B	24.71 ± 7.16 B

* Standard deviation. Statistically homogenous groups determined using LSD's significance test are indicated by the same letter.

The Fisher LSD multiple range test indicated that the addition of ACQ had significant negative effect on bonding properties as well as on penetration parameters of glue joints. The reducing of adhesive penetration depth could be a reason for the decrease of shear strength and wood failure percentage of glue joints. The addition of ACQ might change the surface activation energy of wood surface (Cao and Kamdem 2006), and affect the curing of API (Miyazaki and Nakano 2002), thus reducing the flow of API into the wood and forming less bonding force between wood and adhesive.

Correlation Analysis between Shear Strength and Penetration

The three series of ACQ treated groups were merged into one group for Pearson correlation analysis, given that there were no significant differences in bonding strength and penetration parameters among them. The obtained Pearson correlation coefficients were listed in Table 2. The shear strength showed a significant correlation with average penetration depth and effective penetration depth at the 0.01 level, while there was no significant correlation between shear strength and bondline thickness.

Table 2. Pearson Correlation Coefficient between Shear Strength and Penetration Parameters for ACQ Treated Glue Joints

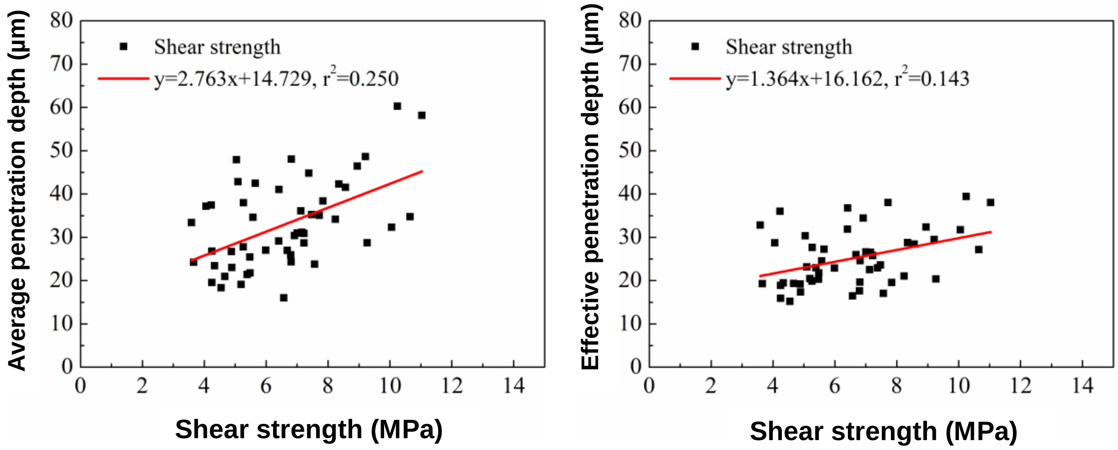
Parameters	Pearson Correlation Coefficient
Shear strength × bondline thickness	-0.173
Shear strength × average penetration depth	0.516**
Shear strength × effective penetration depth	0.401**

**Correlation is significant at the 0.01 level (2-tailed).

For ACQ treated glue joints, the variation of bonding properties and adhesive penetration parameters were mainly ascribed to structural difference of wood and uneven distribution of ACQ in wood. The shear strength showed positive correlation with penetration depths but negative correlation with bondline thickness, according to the results from Pearson correlation analysis. This result suggested that a better penetration of API in ACQ treated Masson pine could contribute to a better bonding performance of glue joints. Furthermore, the relationship between penetration depth and shear strength was more remarkable than that between bondline thickness and shear strength, as the penetration depth directly determined the contact area and interaction between adhesives and wood in the bondline (Kamke and Lee 2007).

Least squares regression analysis of shear strength and penetration parameters was further conducted for ACQ-treated samples, as shown in Fig. 8. The coefficient of determination (R^2) for average penetration and effective penetration depth were 0.250 and 0.143, respectively.

Although the correlation between shear strength and penetration depths was significant at the 0.01 level, the coefficient of determination (R^2) was small, probably due to the complicated influence factors (such as variability of wood properties and curing degree of adhesives) for shear strength of wood joints (Albuquerque and Latorraca 2000; Hass *et al*. 2009; Uysal 2010). Further studies involving more factors affecting adhesive penetration and bonding strength should be done to clarify the relationship between bonding properties and adhesive penetration.



(a) Average penetration **(b)** Effective penetration

Fig. 8. Linear regression analysis for shear strength and penetration parameters

CONCLUSIONS

1. The average shear strength (6.34 to 6.85 MPa) and wood failure percentage (87.0% to 87.8%) of the ACQ treated wood joints met the criteria of GB/T 26899 (2011), while some of the single values failed to meet the criteria.
2. The addition of ACQ showed a significant negative effect on both bonding strength and adhesive penetration parameters of glue joints, while the increase of ACQ concentration from 0.1% to 1.0% had no significant impact on the properties of treated glue joints.
3. The correlations between shear strength and penetration depths were significant at the 0.01 level, and the coefficient of determination (R^2) for shear strength with average penetration depth and effective penetration depth were 0.250 and 0.143, respectively.

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