Fluorescence Microscopy Characterization of the Bonding Interface of Scrimbers Made of Alkali-Treated Finely Fluffed Poplar Veneers

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To improve the penetration of adhesives on finely fluffed poplar veneer (FFPV), a 3% NaOH solution was used to treat FFPV heartwood and sapwood. The treated FFPVs were made into engineered wood composites, otherwise known as scrimbers, with phenol-formaldehyde (PF) resin. The static contact angle and the resin weight gain rate of the FFPV were tested. The shear strength of the FFPV scrimbers was investigated, and the bonding interface was characterized by fluorescence microscopy. The results revealed that the static contact angle of the treated FFPV decreased, but the resin weight gain rate increased. The shear strength of the treated FFPV scrimbers was lower than that of the untreated scrimbers. Fluorescence microscopy revealed that the bondline morphology of the treated FFPV scrimbers changed, with a thinner bondline, a deeper penetration distance, and a smaller glue stain. This result illustrated that alkali treatment can enhance the penetration of PF resin on the FFPV effectively, while excessive penetration of the adhesive should be avoided to ensure adequate bonding properties of the scrimber.

Keywords: Alkaline treatment; Finely fluffed poplar veneer; Fluorescence microscopy; Bonding interface; Wettability

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INTRODUCTION

Scrimbers are a new type of engineered wood composite made of finely fluffed poplar veneer (FFPV) all stacked in the same direction. (Zhang *et al*. 2017). Scrimbers have excellent mechanical performance and dimensional stability, which meet the market demand for structural materials. In addition, scrimbers can reduce the inherent variability in the mechanical properties of natural wood and enhance the effective utilization of low-cost and fast-growing wood in China (Li *et al*. 2015). Finely fluffed poplar veneers have been universally used in making scrimbers, but their low density and the differences between heartwood and sapwood affect the permeability of adhesives, which affects the physical and mechanical performance of scrimbers (Zhang *et al*. 2018). Therefore, it is necessary to modify the wettability of FFPVs. The alkali treatment of veneer is a widely used method to improve the physical and mechanical properties of wood composites. Song *et al.* (2017) reported that plywood composites manufactured by alkali-treated eucalyptus veneers had better flexural properties. Fluorescence microscopy is a popular method to study the bonding interface of composite materials by microscopic morphology. Gruver and Brown (2006) investigated the permeability and bonding performance of polymeric diphenylmethane diisocyanate (pMDI) adhesive in three different species by fluorescence microscopy. However, the effect of alkali treatment on FFPVs obtained by means of fluorescence microscopy is unknown.

To evaluate the bonding mechanism of FFPVs after alkali treatment, and level out the difference surface wettability of heartwood and sapwood to create a uniform interface with phenol formaldehyde (PF) resin, the surface wettability of PF resin on FFPVs was examined. Then, the FFPVs were made into scrimbers, and the shear strength of the scrimbers was measured. In addition, the penetration of the PF on the bonding interface was characterized by fluorescence microscopy to understand the bonding mechanism.

EXPERIMENTAL

Materials and Methods

The FFPVs were obtained from the Shandong Province in China. (Yellow River Delta Jingbo Chemical Research Institute Co., Ltd, Shandong, China). The FFPVs had an average air-dry density of 0.40 g/cm^3 ± 0.25 g/cm^3, a thickness of 1.25 mm, and an initial moisture content (MC) of 8% to 12%. The pH values of the heartwood and sapwood were 7.40 and 5.75, respectively. The PF resin was made in the laboratory, with a 24.5% solids content, a viscosity of 17 mPa·s, a pH of 10.45, a 0.61% free aldehyde content, and a 0.02% free phenol content. The sodium hydroxide (NaOH) solution had a concentration of 3% and a pH of 11.0.

For the alkali treatment, the FFPV heartwood and sapwood were immersed in 3% NaOH solution for 10 h. The samples were marked as follows: HC was the untreated FFPV heartwood, HB was the alkali FFPV heartwood, SC was the untreated FFPV sapwood, and SB was the alkali FFPV sapwood.

Scrimber Manufacturing

The FFPVs were dried to a MC of 6% to 8% before and after the alkali treatment, impregnated in the PF resin to achieve a 20% resin content, and dried again to 6% to 8% MC. Twenty-layer FFPVs were arranged parallel to the length and then cured for 22 min at 5 MPa and 140 °C to obtain an average density of 0.85±0.05 g/cm^3. Then, the scrimbers were conditioned at 65% RH and 20 °C until the MC was stable (8% to 12%). The samples were marked as follows: HCS was the untreated FFPV heartwood scrimber, HBS was the alkali FFPV heartwood scrimber, SCS was the untreated FFPV sapwood scrimber, and SBS was the alkali FFPV sapwood scrimber.

Characterization

Contact angle analysis

To evaluate the wettability of modified FFPVs with PF resin, the contact angle test was carried out. A JC2000C contact angle measuring instrument (Powereach, Shanghai, China) was used to measure the contact angles of the FFPVs before and after the alkali treatment with PF resin as the reagent. Repeated samples are 5.

PF weight gain rate

The dried FFPVs before and after the alkali treatment were impregnated in the PF resin and were then oven-dried at 60 °C for 2 h. The PF weight gain rate of the FFPVs was calculated using Eq. 1,

$$W= \frac{(m_2-m_1)\times S}{m_1} \times 100\% \tag{1}$$

where W is the PF weight gain rate; m_1 is the weight of the oven-dried specimens before impregnation (g); m_2 is the weight of the oven-dried after impregnation (g); and S is the solid content (%). When conducting the calculations, five replications were tested for each treatment.

Shear strength

In order to evaluate the bonding properties of scrimbers prepared from alkali-treated FFPVs, the shear strength of scrimbers was tested. According to the ASTM D2344-13 standard (2013), the shear strength of the poplar scrimbers was tested with a bending span of 60 mm. Six specimens with a size of 90 mm (length) × 50 mm (width) × 16 mm (thickness) were prepared. The load drop-off was 30% at the crosshead speed of 1.0 mm/min. The result was the average derived from the six replications.

Fluorescent characterization

To understand the morphology of bonding interface, fluorescence microscopy was used to investigate the bonding interface microstructure between heartwood scrimbers and sapwood scrimbers before and after alkali treatment. A fluorescence microscope (BX51, Olympus, Tokyo, Japan) and Image-Pro Plus 6.0 software (Media Cybernetics, Rockville, USA) were used to observe and analyze the bonding interface of the scrimbers (Johnson and Kamke 1992). Three bondlines in one image were analyzed and 20 images for each sample.

RESULTS AND DISCUSSION

Wettability of FFPV

Figure 1 shows the static contact angle of the FFPVs. The wettability of the FFPV sapwood and heartwood samples was improved after alkali treatment. The static contact angle of the alkali-treated FFPV heartwood was 81.5°, which was 10° lower than that of the untreated heartwood.

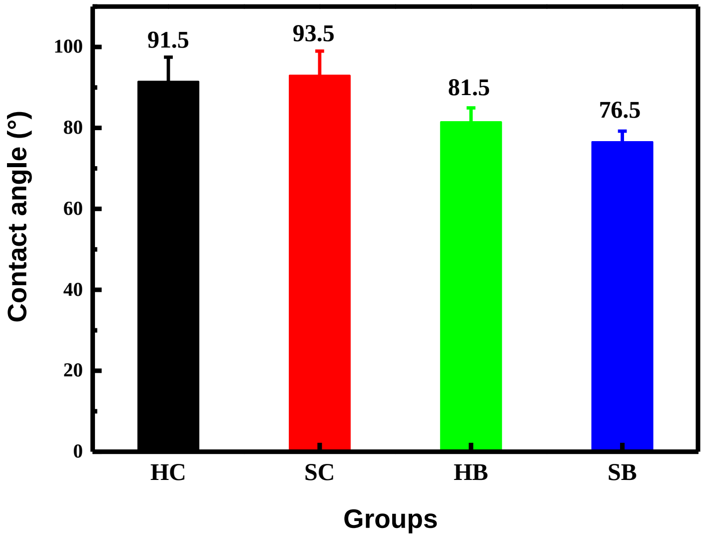


Fig. 1. The static contact angle of the FFPVs

The static contact angle of the treated FFPV sapwood was 76.5°, which was 17° smaller than that of the untreated FFPV sapwood. These results show that the alkali treatment enhanced the wettability of the FFPV sapwood better than that of the heartwood.

Figure 2 illustrates the relationship between the PF weight gain rate and the impregnation time of the FFPV. When the impregnation time was more than 0.5 h, the PF weight gain rate of the treated FFPV increased faster than that of the untreated FFPV. After 3 h, the PF weight gain rate of the treated FFPV leveled off. After the alkali treatment, the PF weight gain rates of the FFPV increased, especially for the treated FFPV sapwood. The PF weight gain rate of the treated FFPV sapwood was 6% to 11% higher than that of the untreated FFPV sapwood. Alkali treatment improves the PF weight gain rates of veneers, which is beneficial for increasing the properties of scrimbers (Chen *et al*. 2014).

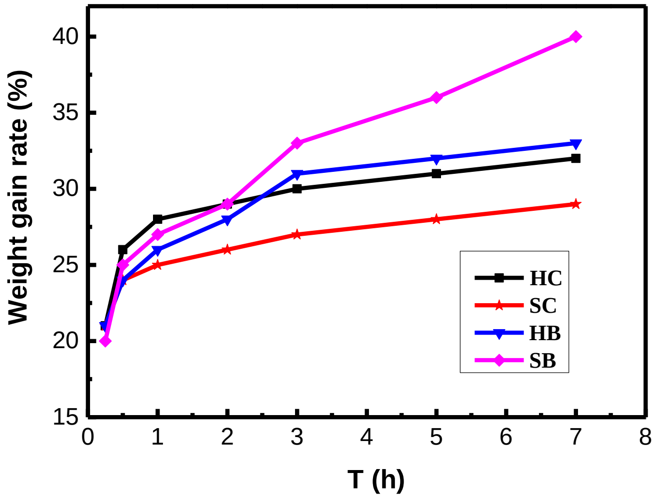


Fig. 2. The PF weight gain rate of the FFPVs

Shear Strength of Scrimbers

The perpendicular shear strength testing setup of the panels is shown in Fig. 3.

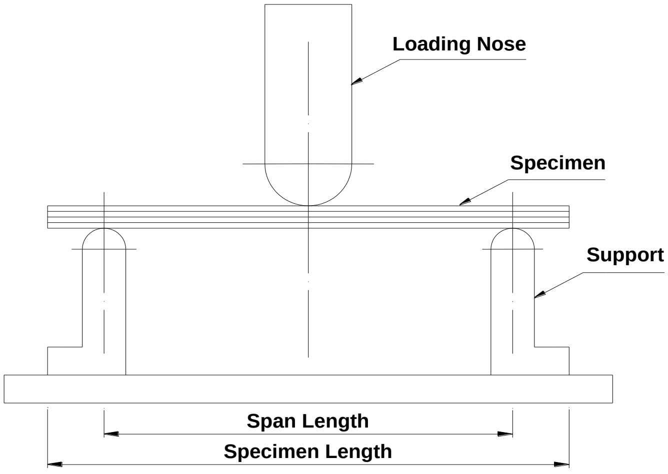


Fig. 3. The perpendicular shear strength testing setup of the panels

As shown in Fig. 4, the mean shear strength values of the four-group scrimbers were greater than 9 MPa, which exceeds the maximum required value (6.5 MPa, 65V-55H) of the Chinese standards for construction materials (GB/T 20241 2006). However, compared with the untreated FFPV, the shear strength of the treated FFPV scrimbers decreased by 20% to 28%, mainly because of the excessive penetration of the PF adhesive on the FFPVs after the alkali treatment. In addition, the shear strength of the scrimber is also related to the PF resin distribution on the gluing interface (Wang *et al*. 2017).

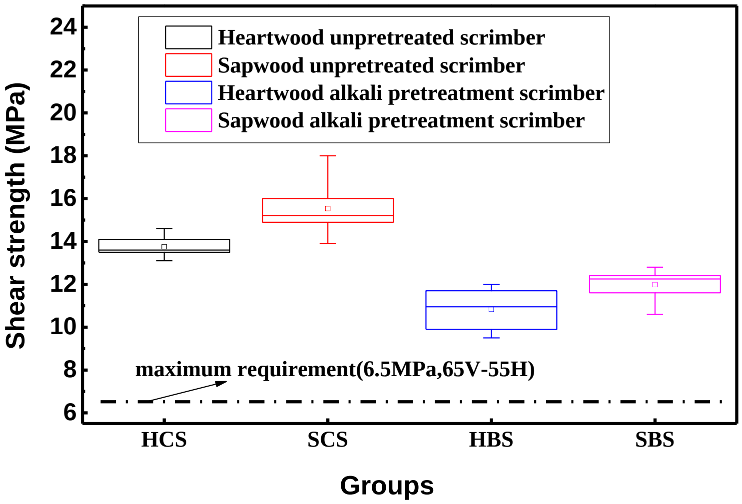


Fig. 4. The shear strength of FFPV scrimbers

Fluorescent Characterization of FFPV Scrimbers Bonding Interface

Figure 5 shows the distribution of the PF resin on the bonding interface of the scrimbers. The average penetration depth (AP) and the effective penetration depth (EP) are two important indexes to assess the permeability of the adhesive at the wood bonding interface (Guan *et al*. 2013; Qin *et al*. 2015). Meanwhile, the average glueline thickness was also measured in this study. The calculation for the AP is shown in Eq. 2,

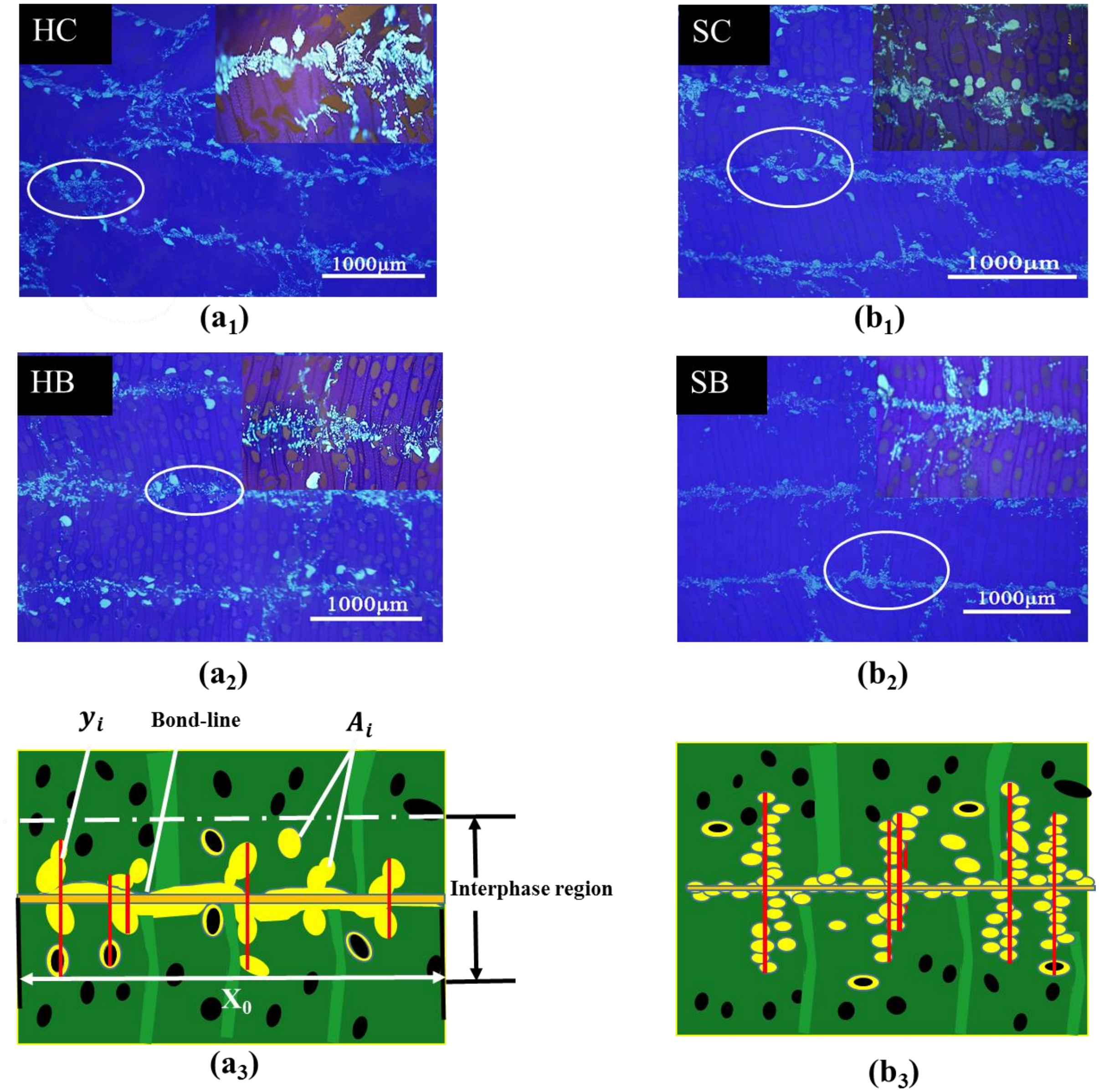
$$AP = \frac{\sum_{i=1}^{n}(y_i)}{5} \tag{2}$$

where AP is the average value of the five farthest penetration distances (μm), and y_i is the farthest penetration distance (μm). The calculation for the EP is shown in Eq. 3,

$$EP = \frac{\sum_{\mathrm{i}=1}^{\mathrm{n}}(\mathrm{A_i})}{X_0} \tag{3}$$

where EP is the effective penetration depth (μm), A_i is the area of the adhesive object i (μm^2), and X_0 is the total length of the glueline in the objective image. The bondline thickness is measured with the same method of EP, but the objective bondline only included pure phenol formaldehyde center line and penetration on wood surface 3 to 5 parenchymal cells in distance, ignoring penetration in vessels and ray cells. The measurement parameters used in Eqs. 2 and 3 are illustrated in Fig. 5(a_3), and the calculated results are shown in Table 1. As shown in Fig. 5(a_1) and Fig. 5(b_1), the PF resin is distributed on the interface of the untreated FFPV scrimbers with a large amount of fully filled vessels and wood rays around the parenchymal cell near the pure PF bondline.

Compared with the untreated FFPV, Fig. 5(a_2) and Fig. 5(b_2) show that the treated FFPV scrimbers had thinner bondlines, deeper penetration distances, and smaller glue stains in the parenchymal cell and less so in vessels and wood rays. The untreated FFPV scrimber exhibited a fish-bone glueline shape with a “glue nail,” which has a strong mechanical locking mechanism, as shown in Fig. 5(a_3). However, the glueline of the treated FFPV scrimber is dispersive, similar to the dendritic pattern, leading to weak mechanical locking (Fig. 5(b_3)). The bondline interfacial morphology difference illustrated the bondline of pure PF decreased, which should be the main reason caused the shear strength of the treated FFPV scrimber to decrease. The penetration morphology of heartwood scrimber and sapwood scrimber after NaOH treatment became similar and the interface penetration seemed level.



(a_1) (b_1)

(a_2) (b_2)

(a_3) (b_3)

Fig. 5. (a_1, a_2, b_1, b2) The fluorescent photomicrographs and partial enlargement of the four kinds of scrimbers, (a_3) the schematic diagram of the PF resin bondline on the FFPV before and (b_3) after alkali treatment

Table 1. Penetration Parameters of PF Resin in the FFPV Scrimbers

Scrimber Group	AP (μm)	EP (μm)	Bondline Thickness (μm)
HCS	72.0	61.6	58.8
SCS	124.2	75.3	71.1
HBS	90.2	63.2	48.4
SBS	127.8	74.5	57.9

As shown in Table 1, the AP and EP of the treated FFPV scrimbers were greater than those of the untreated scrimbers. The AP increased by 25.3% in the HBS and 2.9% in the SBS, while the EP increased by 7.4% in the HBS and 4.8% in the SBS. The bondline thickness of the treated FFPV scrimbers decreased by 21.5% in the HBS and 23.2% in the SBS. Figure 4, Fig. 5, and Table 1 illustrate that the overpenetration of the adhesive will lead to a lower bondline thickness, thus decreasing the bonding strength of the scrimber.

CONCLUSIONS

1. The wettability and PF weight gain rate of the FFPV scrimbers all increased after the alkali treatment, especially for the FFPV sapwood.
2. The shear strengths of the treated and untreated FFPV scrimbers were all greater than the required value, although the values of the treated FFPV scrimbers were lower than those of the untreated scrimbers.
3. The fluorescence analysis showed that the alkali-treated FFPVs had better permeability, greater penetration depths, and thinner bondline thickness, which may slightly reduce the shear strength of the scrimbers.

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