

Analysis of Bonding Mechanism of Glass Fiber-reinforced Bamboo Plywood

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Bamboo curtains, coarse glass fiber cloth, bamboo mat, poplar veneer, and fine glass fiber cloth impregnated with phenolic resin adhesive were sequentially laid from the inside to the outside, and the symmetrically balanced material was sent to the hot press. The bonding mechanism of the resulting glass fiber-reinforced bamboo plywood (GFRBP) was analyzed using environmental scanning electron microscopy (ESEM) and Fourier transform infrared (FTIR) spectroscopy. The bonding between the phenolic resin and fiberglass cloth was achieved with a coupling agent, which reacted chemically with both the fiberglass cloth and phenolic resin. ESEM showed that glass fiber was bonded strongly with phenolic resin after treatment with coupling agents such as KH-550, KH-560, and KH-792. When the GFRBP was damaged, there was no breakage at the bonding surface between the glass fiber and phenolic resin. During the hot-pressing process, the phenolic resin easily migrated to the middle of the gap between plywood plates, which promoted better integration and properties. FTIR results showed that the longitudinal sides of the GFRBP pressed by the fiberglass cloth that had been treated by the coupling agents KH-550 and KH-792 were similar to each other, but those treated with KH-792 contained more -CH₂- and had a longer carbon backbone.

Keywords: Bamboo; Glass fiber (cloth); Phenolic resin glue; Coupling agent; Molecular structure; Bonding mechanism

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INTRODUCTION

Bamboo is a popular building material for traditional Asian buildings and furniture. The vast territory, complicated topography, and various climate and soil conditions in China increase the diversity of bamboo (Li *et al.* 2015). Over the past 20 years, bamboo has been widely employed in daily life, and new fields are being explored, such as the garment and automotive industries, flooring boards, veneers, *etc.* Floors and plywood are especially increasing in demand all over the world because they have the texture of marble and the elegance of wood. In addition, bamboo materials are strong, durable, smooth, clean, non-sliding, and resistant to humidity (Anwar and Hiziroglu 2011; Liu *et al.* 2015). Bamboo has great potential for development, so it is essential to study bamboo products and pay attention to using bamboo timber and small caliber raw bamboo as major materials to produce various bamboo products, which are often aligned with people's ideas for adornment of their lives and a return to nature (Tian *et al.* 2015; Hong and Lei 2019). Rich bamboo resources and improved bamboo processing industries play a unique role in promoting the development of mountainous rural communities. Glass fiber-reinforced bamboo plywood (GFRBP) has excellent mechanical properties, good environmental

adaptability, economic diversification, and reduces the cost of cement concrete templates and container floors. The development of bamboo plywood can improve the added value of the processing and utilization of bamboo resources and broaden the range of application of bamboo. Bamboo-based composites are a growing trend in the development of bamboo-based materials (Thwe and Liao 2003; Bal *et al.* 2015). It is proposed that in line with the development of bamboo-based composites and the forestry sustainable development strategies, GFRBP has great potential application in China.

Many studies related to glass fiber-reinforced plywood have been reported (Thwe and Liao 2003; Abdul Khalil *et al.* 2012). The resulting material can be used as a building template or container floor. Glass fiber has been used as a reinforcing material, and other materials (non-bamboo) acted as substrates. Additionally, other materials can be used as reinforcing materials and bamboo as substrates. Bamboo is used to produce cement templates or containers flooring directly without addition of enhanced material such as glass fiber. For example, Ata *et al.* (2019) investigated the failure mode and failure load of adhesively bonded composite joints made by glass fiber-reinforced polymer. Bank *et al.* (2009) used glass fiber, polypropylene fiber, flame retardant viscose fiber, (two-way) fiber-reinforced plastic mesh, and non-oriented fiber reinforced plastic strips to design and prototype different cement templates that are suitable for bridge plywood. In recent years, a number of researchers have been studying the application of wood with various species, especially wood-based composites. Ashori *et al.* (2016) used carbon fiber (CF) and waste rubber (WR) as reinforcing agents for plywood. The differences in the mechanical and physical properties of plywood composite panels found in the study were associated with the three variable parameters used. The addition of the CF enhanced the mechanical properties of the plywood specimens. The modulus of rupture (MOR) properties of specimens did not improve with the addition of the WR content. Kim *et al.* (2011) impregnated cardboard with water-based phenolic resin to produce plywood cement templates by hot pressing. Experiments showed that the products were resistant to hot and cold cycles, as well as alkalinity and wear. Sundaram (2009) pointed out that when compared with the wood or steel template as a cement template, the glass fiber-reinforced plastic had better size stability and greater strength, and the cost was greater than the wood template, but lower than that of steel template. With the promotion and development of the bamboo plywood in the market, a growing number of them are used as packing and building materials.

Recently, good progress has been made in exploiting and utilizing bamboo materials, and new products including bamboo plywood board, bamboo scrimber, and bamboo-based composites have been manufactured. The productive technology of bamboo plywood form is presented. Anwar and Hiziroglu (2011) used 4-year-old *Gigantochloa scortechinii* grown in Kuala Lumpur to produce a resin-treated plywood type panel. Some of the products were made from low molecular weight phenol formaldehyde (LMwPF)-treated bamboo strips. Experiments showed that the surface-treated bamboo plywood was less rough than the control, and the modulus of elasticity (MOE), MOR, compression hardness, and dimensional stability were less affected. Bamboo scrimber (Shangguan *et al.* 2014) is an engineered material, where bundles of bamboo fiber are arranged in parallel, with fibers glued to each other using adhesive under a hot press after drying. Shangguan *et al.* (2014) provides a theoretical foundation for the rational use of bamboo scrimber. Chen *et al.* (2015) developed a new type of bamboo fiber-reinforced polypropylene composite. The new composite has good tensile strength, tensile modulus, and impact strength. To improve the bamboo plywood performance, a large number of studies have focused on a

variety of mechanical and physical properties of bamboo-based panels and much of this research up to now has been descriptive in nature. However, few scholars debated the impact of phenolic resin and coupling agent on the bamboo-based panels. The current study explored the bonding mechanism of GFRBP that can provide a reference for further improvement for bamboo-based panels.

EXPERIMENTAL

Materials

Materials used in this study include fiberglass cloth, phenolic resin, bamboo curtain and mat, poplar veneer, and organosilane coupling agent.

The mesh size of fiberglass cloth (Zhongsheng Glass Fiber Products Co., Ltd., Taizhou, China) used was 6 mm × 10 mm, 436 g/m², and the diameter of a single fiber was 20 μm. The other fiberglass cloth was 14 × 14-mesh, 148 g/m², and the diameter of a single fiber was 7 μm. The solid content of phenolic resin (cure temperature and time of phenolic were 120 °C and 5 min, respectively.) raw material (Xinguo Artificial Plates Co., Ltd., Yongan, Fujian, China) was 47.03%, viscosity was 143.0 s (TU-4 viscometer; below 20 °C), pH value of 11.02, mass fraction of free aldehyde ≤ 0.40%, and mass fraction of free phenol ≤ 2.0%. *Phyllostachys heterocycla* (Carr.) Mitford was supplied (Pushang Forest Farm, Nanping, China) as raw material for bamboo curtain and bamboo mat (after drying twice), the moisture content of 4- to 6-year-old bamboo was ≤ 15%, and the dimensions were 19 mm × 1.8 mm (width × thickness). The dimensions of *Populus tomentosa* Carr. Veneer (Xinguo Artificial Plates Co., Ltd., Yongan, Fujian, China) used was 50 cm × 50 cm × 0.1 cm (length × width × thickness), and moisture content was ≤ 12%.

Three types of organosilane coupling agents (Aocheng Chemical Co., Ltd., Nanjing, China) KH-550, KH-792, and KH-560 were used and their corresponding structures are shown in Table 1.

Table 1. Sample Preparation Conditions

No.	Sample Information	
	Sample opening	Coupling agents
A1	Longitudinal	None
A2	Transverse	
B1	Longitudinal	Type: KH-550 $\text{H}_2\text{NCH}_2\text{CH}_2\text{CH}_2\text{-Si} \begin{cases} \text{OCH}_3 \\ \text{OCH}_3 \\ \text{OCH}_3 \end{cases}$
B2	Transverse	
C1	Longitudinal	Type: KH-560 $\begin{array}{c} \text{CH}_2\text{CH} \\ \diagdown \quad \diagup \\ \text{O} \end{array} \text{CH}_2\text{O-CH}_2\text{CH}_2\text{CH}_2\text{-Si} \begin{cases} \text{OCH}_3 \\ \text{OCH}_3 \\ \text{OCH}_3 \end{cases}$
C2	Transverse	
D1	Longitudinal	Type: KH-792 $\text{H}_2\text{NCH}_2\text{CH}_2\text{-HNCH}_2\text{CH}_2\text{-Si} \begin{cases} \text{OCH}_3 \\ \text{OCH}_3 \\ \text{OCH}_3 \end{cases}$
D2	Transverse	
Note: The numbering of the following scanning electron microscopy (SEM) images follows the numbers of this table		

Methods

Preparation of GFRBP

The method of preparing the sample is shown in Fig. 1. The specific steps in the preparation process are as follows:

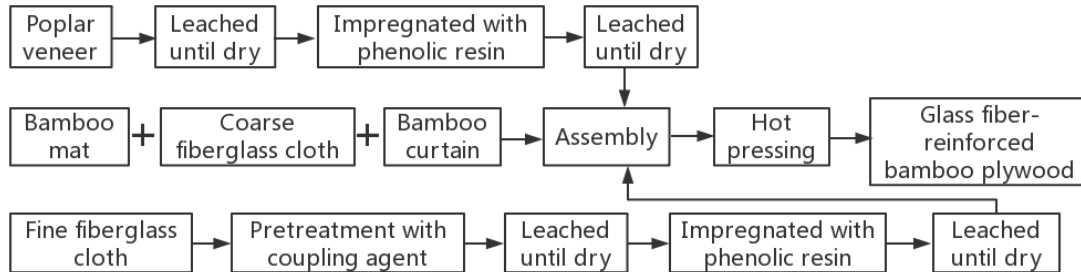


Fig. 1. Production of glass fiber-reinforced bamboo plywood

Step 1 - Pretreatment of fiberglass cloth with coupling agent: Coupling agent and alcohol were mixed at a ratio of 1:2 (V/V). After mixing, all fine and coarse fiberglass cloths were leached, then impregnated with phenolic resin solution (volume ratio of phenolic resin to water of 1:0.5), and then leached until dry.

Step 2 - The three principles of molding were followed (symmetry principle, odd number principle, and thickness principle). The mold structure is shown in Fig. 2, in which the coarse fiberglass cloth of the inner layer was of 6×10 -mesh and the outer fiberglass cloth was of 14×14 -mesh. The thickness of each bamboo layer was approximately 1.8 mm.

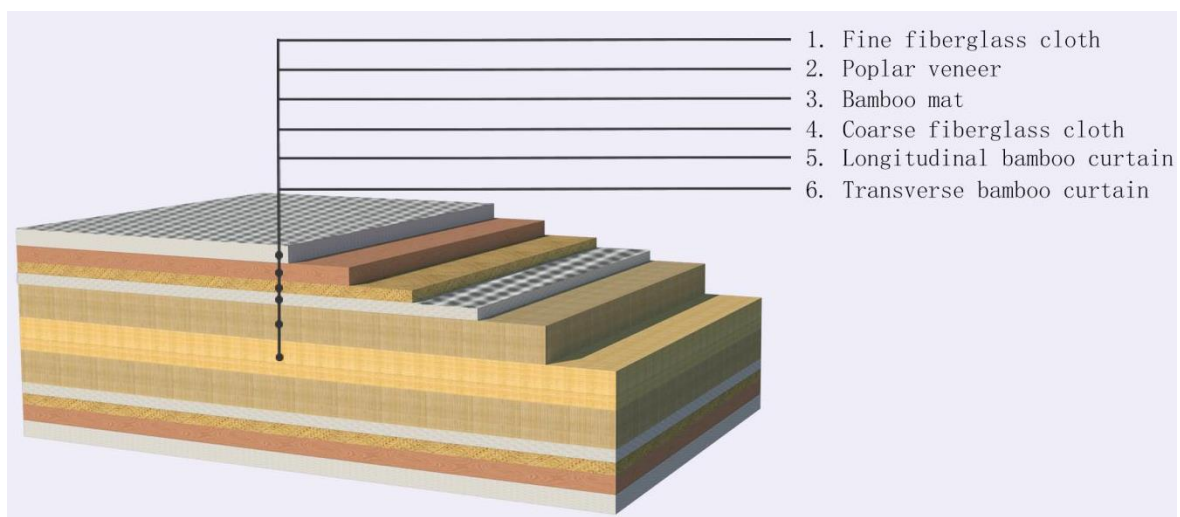


Fig. 2. Billet structure of GFRBP (1. Fine fiberglass cloth; 2. Poplar veneer; 3. Bamboo mat; 4. Coarse fiberglass cloth; 5. Longitudinal bamboo curtain; and 6. Transverse bamboo curtain)

Step 3 - Hot press molding: The pressure, temperature, and time were set to 2.2 MPa, 150 °C, and 1.1 min/mm. The total thickness of the sample was approximately 15 mm.

Step 4 - Numbering the samples: Different samples were prepared according to the specific coupling agent and fiber cloth used with the samples of bamboo sheets, and the control samples were numbered A1 and A2, as shown in Table 1.

The mechanical properties (MOE and MOR) of GFRBP were determined by Chinese standard “Test methods of evaluating the properties of wood-based panels and surface decorated wood-based panels” (Standard No: GB/T17657-2013).

Table 2. Fourier Transform Infrared (FTIR) Results of the Longitudinal Side of Four Types of GFRBP

Wavenumber (cm ⁻¹)				Attribution of Main Adsorption
No. 0	No. 1	No. 2	No. 3	
3433	3431	3431	3408	O-H Stretching vibration
2922	2920	2920	2918	C-H Stretching vibration
	2850		2849	C-H Stretching vibration
2026				Overtone of C-H on benzene ring
			1736	C=O Stretching vibrations of ketones, aldehydes, esters, and carboxylic acids
1636				C=O Stretching vibration
			1593	C=O Stretching vibration (C=O is coupled with benzene ring)
	1581	1581		C=C Skeletal vibration of benzene ring
			1463	Asymmetrical stretching vibration of CH ₃ /symmetrical deformation vibration of CH ₂ (scissoring vibration)
	1412	1412	1424	Deformation vibration of alcoholic O-H
			1375	Deformation vibration of alcoholic O-H/Bending vibration of C-H
			1330	Skeletal vibration of CH ₃ /in-plane bending vibration of O-H
			1242	Asymmetrical stretching vibration of ether R-O-R' /Ar-O-R
1046	1057	1051	1053	Stretching and asymmetrical vibrations of ether C-O-C in R-O-R'/Ar-O-R
			897	Asymmetrical out-of-plane stretching vibration of cyclic C-O-C/Wagging vibration of CH ₂ (CH ₂ OH)

Fourier transform infrared spectroscopy

The FTIR spectra were recorded using a Nicolet 380 FTIR (Thermo Nicolet Corporation, Madison, WI, USA) spectrophotometer between 375 and 7800 cm⁻¹ with a resolution of 0.5 cm⁻¹ using attenuated total reflectance (ATR) spectroscopy. The sample was prepared by a tableting method: 1 mg of the sample was ground into a fine powder in an agate mortar and mixed with dry potassium bromide powder (approximately 100 mg, particle size 200-mesh), uniformly filled, filled into a mold, and compressed. The machine (Thermo Nicolet Corporation, Madison, WI, USA) pressed the powder into tablets.

Scanning electron microscopy

To investigate the influence mechanism of different coupling agents' treatment of glass fiber cloth on the performance of GFRBP, an XL30D environment scanning electron microscope was used (model S 4800, Japan) to observe the interface of four kinds of bamboo plywood pressed by fiber cloth that had KH-550, KH-560, and KH-792 coupling agents-treated glass fiber cloth and unadded glass fiber.

RESULTS AND DISCUSSION

Mechanical Properties Test

The samples were tested for mechanical properties and the test results are shown in Table 3.

Table 3. Mechanical Properties of Glass Fiber-Reinforced Bamboo Plywood

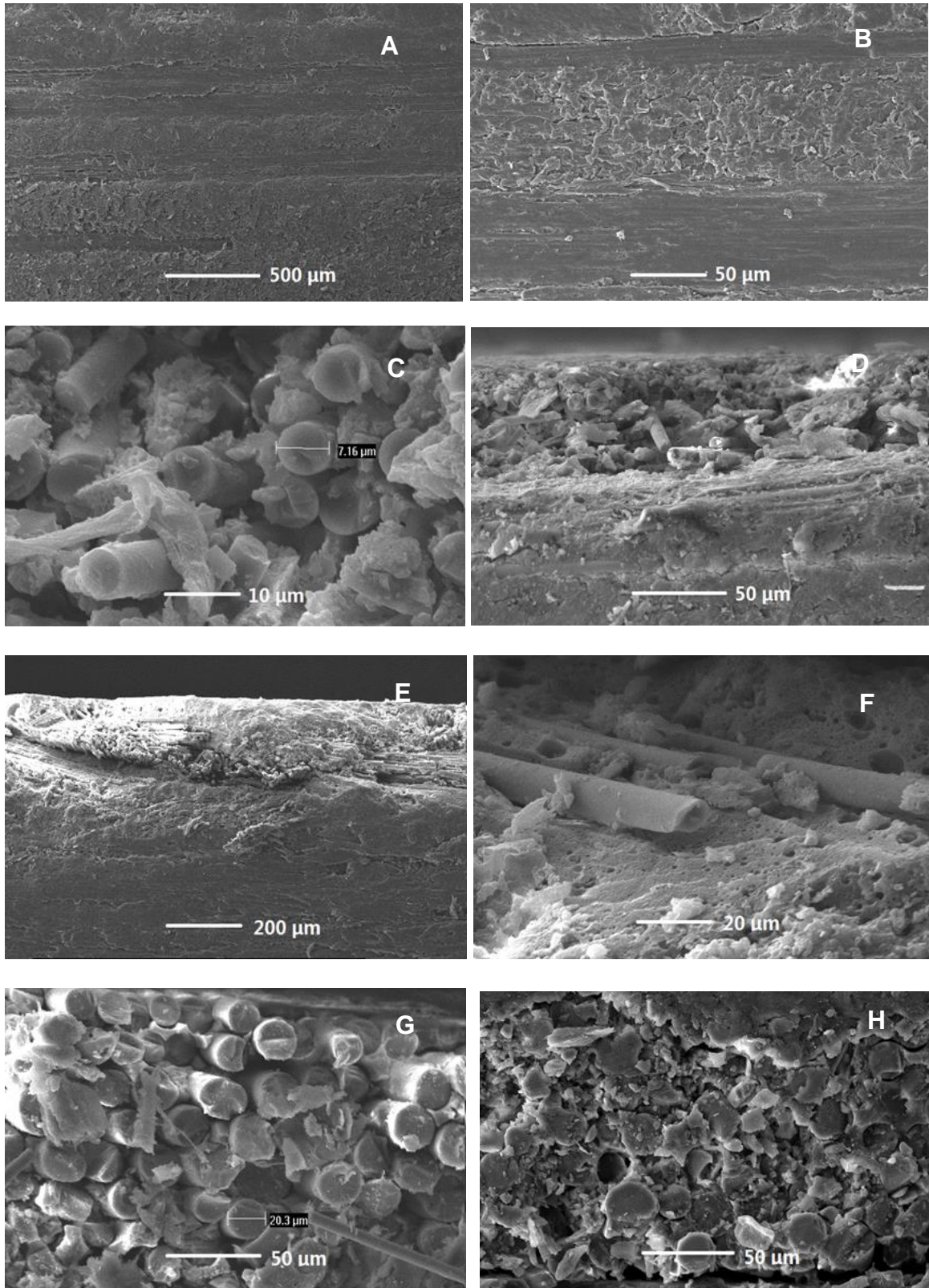
No.	Sample Information			
	Longitudinal MOE (MPa)	Transverse MOE (MPa)	Longitudinal MOR (MPa)	Transverse MOR (MPa)
A1	7352	-	68.2	-
A2	-	5258	-	47.1
B1	10650	-	72.6	-
B2	-	6270	-	53.6
C1	12010	-	117.3	-
C2	-	7200	-	67.1
D1	11300	-	77.1	-
D2	-	5930	-	52.6

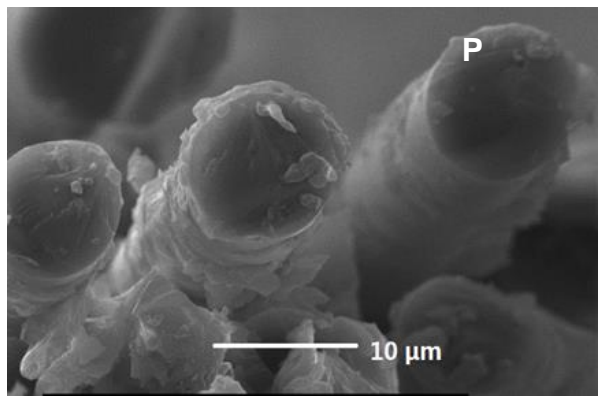
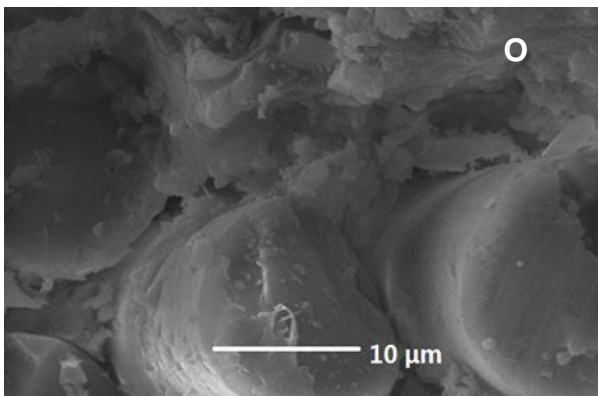
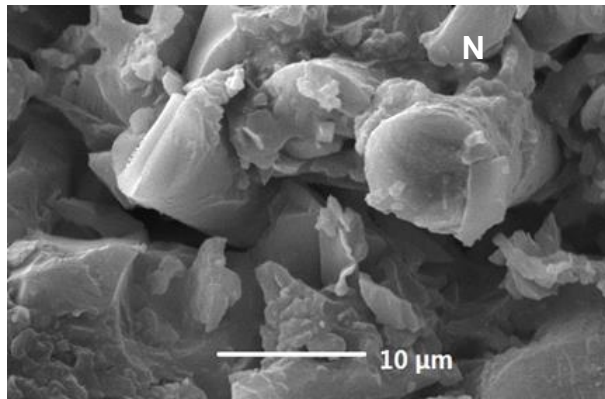
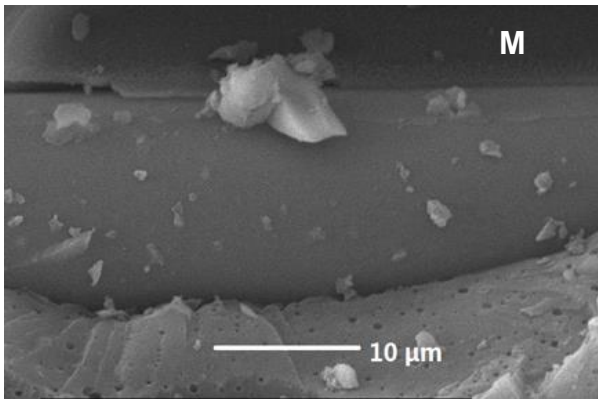
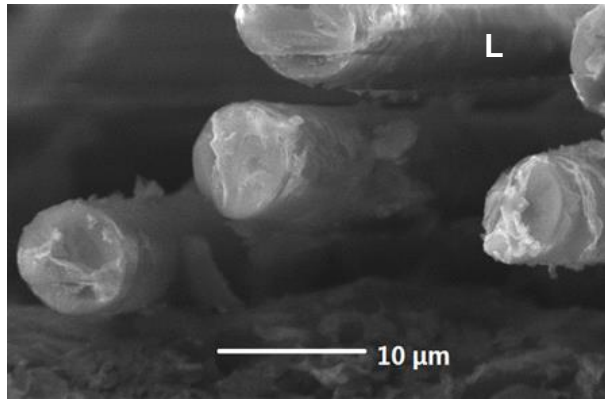
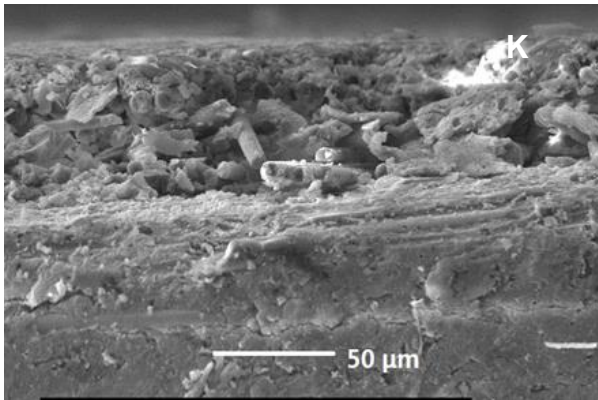
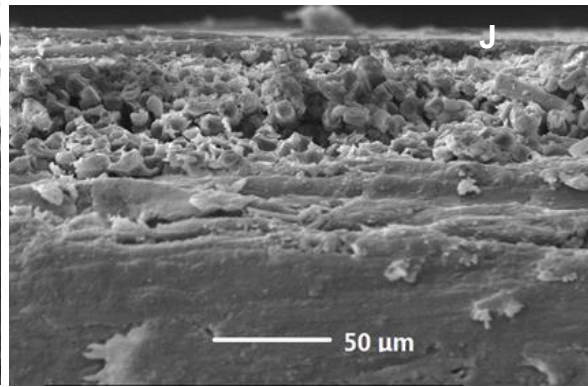
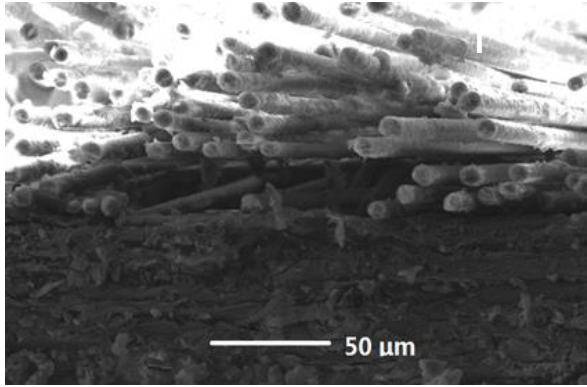
Characterization of the Surface by ESEM

The internal bonding of the sample was observed by environmental scanning electron microscopic (ESEM) analysis. Interface images of the four kinds of GFRBP (the interfacial bonding of fiberglass cloths treated with various coupling agents, KH-550, KH-560, and KH-792, and untreated fiberglass cloth) in the longitudinal section and the cross section were taken.

As shown in Fig. 3a and 3b, the longitudinal section of the bamboo plywood pressed without fiberglass cloth had tight, strong binding in the bamboo interface. However, local cracks can still be seen inside the plate layers. The results from Fig. 3c and 3d revealed that the fine fiberglass cloth consisting of 18 layers of glass fibers had a total thickness of approximately 60 μm , or up to nine layers. All layers were nearly perpendicular to each other, and each of the fine glass fibers had a diameter of approximately 7 μm . Figure 3e and 3f show that even if the surface of the poplar veneer was still relatively uneven, the upper surface of the plate was eventually filled in by solidified phenolic resin. The bond between the bamboo curtains was due to the adhesion of cured phenolic resin, which formed a relatively continuous glue layer. The phenolic resin can penetrate the surface of the bamboo cell under the high temperature and pressure conditions produced during hot pressing that causes a gluing nail effect (Wolfrum and Ehrenstein 2015; Chadha and Bast 2017). As shown in Fig. 3g and 3h, the coarse fiberglass cloth consisting of 16 layers of glass fibers had a total thickness of approximately 276 μm . The upper and lower 8 layers were perpendicular to each other, each with a diameter of approximately 20 μm , which was approximately 2/3 of the outer diameter of the cell wall of bamboo fiber.

There was a large gap in the interior of the coarse fiberglass cloth, probably because of the relatively short time the fiberglass cloth was dipped in coupling agent and phenolic resin glue during the sample preparation stage, so that the coarse fiberglass cloth between the upper and lower layers was not fully impregnated with glue (Shudo *et al.* 2017). There was a small contact area between the coupling agent, glass fiber, and phenolic resin, and the bond was not strong, so the structure was susceptible to mechanical damage.





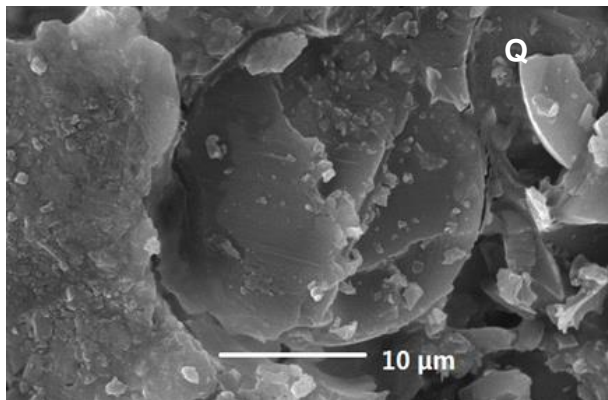


Fig. 3. SEM images and correlation analysis of different samples (A. No. A1 (×100), B. No. A2 (×100), C. No. B1 (×2000), D. No. D1 (×2000), E. No. B2 (×100), F. No. B2 (×1000), G. No. B1 (×500), H. No. C1 (×500), I. No. B1 (×500), J. No. C1 (×500), K. No. D1 (×500), L. No. B1 (×3000), M. No. B1 (×3000), N. No. C1 (×3000), O. No. C1 (×3000), P. No. D1 (×3000), and Q. No. D1 (×3000))

As shown in Figs. 3i to 3k, the bond between fine fiberglass cloth and poplar veneer treated by KH-550 coupling agent was not strong and far weaker than KH-560 or KH-792, thus affecting the strength of the bond between fine fiberglass cloth and poplar veneer. From Figs. 3l to 3q, it was observed that the bonds between coarse glass fiber and phenolic resin and between coarse glass fiber and bamboo fiber were relatively strong. The glass fiber was strongly bonded to the phenolic resin gel after the treatment with coupling agent, and it was not broken at the bonding surface between the glass fiber and the phenolic resin when the GFRBP was damaged (Wang *et al.* 2015a; Song *et al.* 2017). There were obvious fusions on the surface of coarse glass fiber and cured phenolic resin treated with KH-560 and KH-792, forming defects on the surface after the collapse of the sample. The bonds between the fine glass fiber and phenolic resin, and between fine glass fiber and poplar wood were not strong (Rizvi and Sernalul 2008). There were also some fusions between fine glass fiber and phenolic resin glue. After the sample was destroyed, the residual phenolic adhesive remained on the surface of the glass fiber (Wu *et al.* 2015).

FTIR Analysis of GFRBP

The chemical composition of the sample surface was analyzed by FTIR spectroscopy. The spectra of GFRBP samples made with fiberglass cloth that had been treated with various coupling agents (KH-550, KH-560, and KH-792) are shown in Fig. 4. The FTIR of the longitudinal side of the GFRBP pressed by the fiberglass cloth treated by KH-550 and KH-792 were consistent, but the latter had more $-CH_2-$ and a longer carbon backbone. The FTIR of the longitudinal side of the GFRBP pressed by KH-560-treated fiberglass cloth contained relatively rich information, indicating that the oxygen atoms in the epoxy-propyl group in KH-560 molecule were active (Kim *et al.* 2015).

Figure 4 is an integrated FTIR spectra of the longitudinal side of four types of bamboo plywood with and without glass fiber reinforcement. Table 2 gives the attribution of the corresponding major absorption peaks. The molecules of the coupling agents (KH-550 and KH-792) have the same functional group. Figure 4 indicates that the $-O-H$ absorption peak in the plate was weakened by the coupling agents (spectra of samples No. 0 to 3) and No. 4 sample indicated a new absorption peak at 2918 cm^{-1} and 2849 cm^{-1} , which was attributed to the C-H stretching vibration of $Si-CH_3$ and the symmetrical stretching vibration of $-CH_2-$. This indicated that the silane molecule formed a chemical

bond with the active hydroxyl group on the surface of glass fiber, rather than a simple physical adsorption or coverage (Mandal and Alam 2012; Carotenuto and Nicolais 2015).

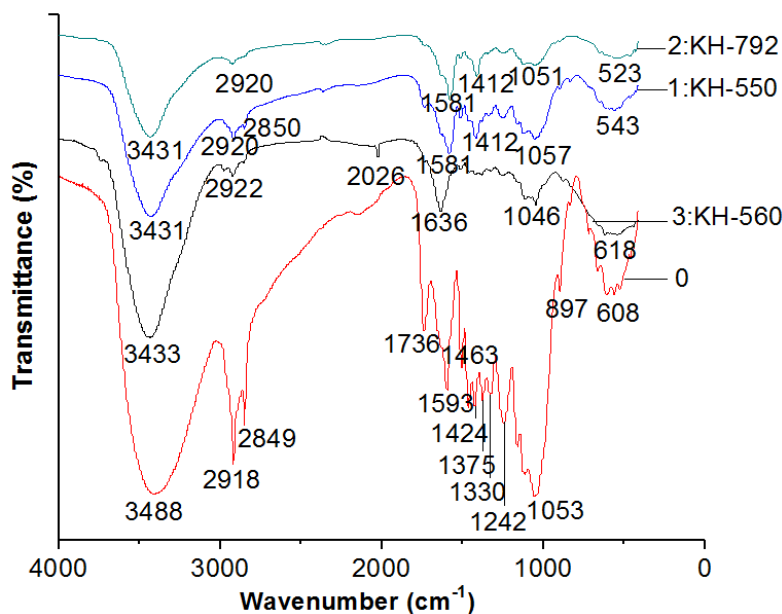


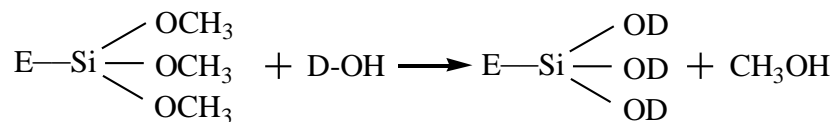
Fig. 4. FTIR of the longitudinal sides of four types of glass fiber-reinforced bamboo plywood

The FTIR of sample No. 3 was relatively complex, mainly due to the chemical reaction between oxygen atoms and other groups in KH-560, which formed new ether bonds, aldehydes, and esters. The oxygen atoms in the glycidyl groups in the KH-560 molecule were active. Oxygen atoms in the epoxy-propyl group after ring-opening are prone to chemical reaction with other molecules in the plate. This reaction produced more branches and increased the reticulation of molecular structures, which improved the overall mechanical properties of the plate. The adsorption peak at 1593 cm^{-1} was lower than that of the normal C=O group (1740 cm^{-1} to 1720 cm^{-1}), indicating the conjugation between C=O and a benzene ring (in phenolic resin macromolecule and bamboo lignin). The absorption band at 1330 cm^{-1} corresponded to the skeletal vibration of -CH₃, which showed that more branches formed after the chemical reaction. A highly reticulated molecular structure was generated and rendered the molecules less prone to slide (Zięba-Palus *et al.* 2015; Li *et al.* 2017). Therefore, sample No. 3 had better mechanical properties than the other samples.

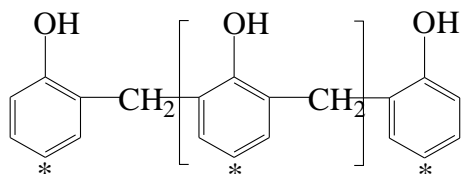
The Reaction Mechanism of Coupling Agent

To better understand the mechanism of underlying bonding in GFRBP, the effect of various coupling agents in promoting many elements of the plate combined with the relevant chemical reaction (Huang *et al.* 2002; Wang *et al.* 2015b) are discussed. The main chemical composition of glass fiber is silica (with small amounts of calcium oxide, magnesium oxide, and sodium oxide). Glass fiber possesses many -OH groups on its surface, and it has a body-centered structure. An oxygen atom is linked to two silicon atoms, and a silicon atom is linked to four oxygen atoms. The molecular formula of this was marked as D-OH, and the general molecular structure of three silane coupling agents

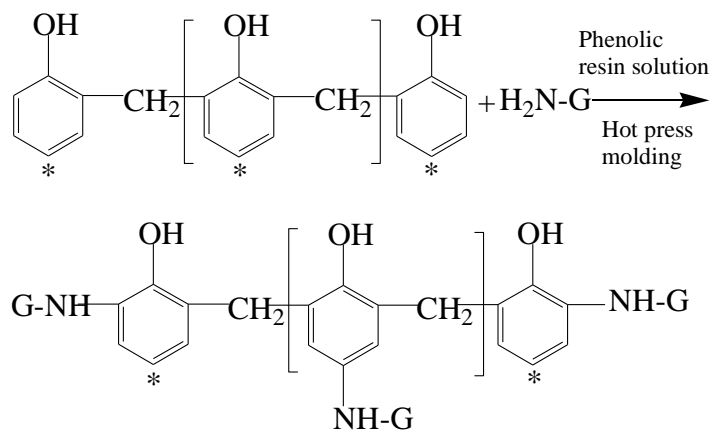
(KH-550, KH-792, and KH-560) were labelled as E-(OCH₃)₃. The chemical reaction involved in the process of impregnating coupling agent was given as follows:



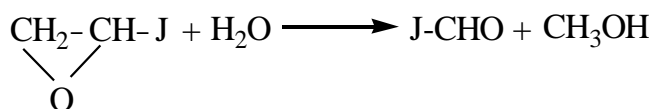
After impregnation with the coupling agent, the fiberglass cloth was leached and dried and then impregnated with phenolic resin. The phenolic resin used was a polymer compound composed of formaldehyde and phenol. Its structural formula is complex and diverse. There are three active sites on the benzene ring in the phenol molecule. The methylene groups on the benzene ring can be in the *ortho* or *para* positions. For the analysis, the following shows the molecular formula only for the *ortho* polycondensation (when the phenol-aldehyde ratio is 1:1; * indicates possible locations of -CH₂- group):



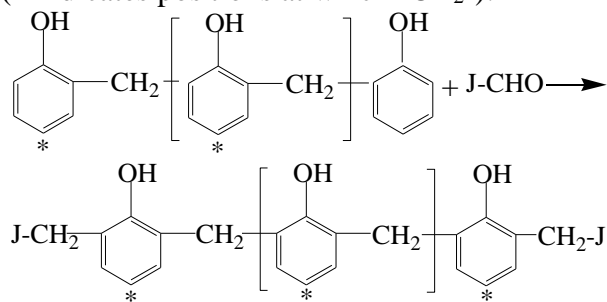
The general molecular formulas of two coupling agents KH-550 and KH-792 are shown as H₂N-G, where -NH₂ is alkaline (as a nucleophile). The lone pair of electrons in N-atom can absorb the hydrogen atom of the phenol hydroxyl group in phenolic resin. Due to structural instability, the -NH₂ group is rearranged to the *ortho* or *para* position of the phenolic hydroxyl group in the benzene ring (* indicates positions at which -CH₂-). The chemical reaction involved is illustrated as follows:



The molecular structure of KH-560 coupling agent is shown as , and the hydrolysis reaction with water occurs as follows:



The J-CHO is an aldehyde compound that can continue to react with the phenolic resin (* indicates positions at which -CH₂-):



The main component of bamboo is cellulose, containing a large amount of hydroxyl groups. The hydroxyl groups on the surface of wood fibers can form intermolecular hydrogen bonds. A portion of the unreacted coupling agents reacted with these hydroxyl groups and hydrogen bonds to promote adhesion between adhesives and bamboo. This increased the degree of aggregation of bamboo fibers to a certain extent, and the stress was dispersed.

CONCLUSIONS

Glass fiber-reinforced bamboo plywood (GFRBP) was prepared using bamboo-based materials including bamboo curtain and bamboo mat as the substrates, fiberglass cloth impregnated with the coupling agents as reinforcing material, and phenolic resin as the adhesive agent. The bonding of GFRBP was explored, and the following conclusions are drawn:

1. The combination of phenolic resin and fiberglass cloth took place as a coupling reaction mainly through the effect of the coupling agent. The coupling agents reacted with both the fiberglass cloth and the phenolic resin, forming strong chemical bonds.
2. During the process of hot pressing, the phenolic resin easily penetrated the gap between plates, creating more contact area between the layers in the plate, rendering the combination stronger and improving the mechanical properties. After the ring-opening reaction of KH-560, the epoxyethyl group reacted with phenolic resin, glass fiber, and cellulose or hemicellulose in the substrate. The reaction produced more branches, generating a highly reticulated molecular structure and making the molecules difficult to slide between each other. In this way, the overall GFRBP mechanical performance was improved.

ACKNOWLEDGMENTS

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