Bonding Technology for Bamboo-based Fiber Reinforced Composites with *Phyllostachys bambusoides* f. *shouzhu* Yi

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The objective of this study was to investigate the mechanical and physical properties of bamboo-based fiber reinforced composites (BFRC) that were formed by using loose bamboo bundles (LBB) and phenol formaldehyde (PF) adhesive. Three resin content levels (10%, 14%, and 18%) and seven different assembly patterns were selected when the bundles were glued together. Board performance testing showed that the bond quality was improved by increasing the resin content. The board face layers impregnated with 18% resin content and core layers impregnated with 14% resin content had the best mechanical properties, and the boards impregnated with 18% resin content had the best dimensional stability properties. The mechanical and physical properties increased with increasing resin content and were affected by the assembly pattern. Various performance indices of the BFRC met the requirements of the China national standard GB/T 20241 (2006).

Keywords: Bamboo Fiber; Layered structures; Adhesion; Mechanical properties

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INTRODUCTION

Bamboo is an important biomass material for housing, construction, outdoor flooring, and other purposes in China. Due to the acute scarcity of timber for use in housing and construction, bamboo is becoming more and more important as a substitute in both urban and rural settings. In China, bamboo-based panel products have been adjusted and revised continually from bamboo weaving panel to bamboo plywood, bamboo scrimber, and bamboo-based fiber reinforced composites (BFRC). Bamboo scrimber has a desirable texture, high hardness, and excellent longitudinal strength properties (Zhang and Yu 2012; Yu *et al.* 2014; Yu *et al.* 2015; Zhu *et al.* 2015). It can be used for outdoor and indoor flooring, furniture, construction, and other civil engineering applications (Mahdavi *et al.* 2012; Shangguan *et al.* 2016). Hundreds of bamboo scrimber manufacturing enterprises currently exist in China with an annual production capacity of 900,000 m^3. Bamboo scrimber has been commercialized successfully and rapidly developed in China and has been studied since the 1980s. These studies have mainly focused on the use of bamboo without removing its waxy layers and the siliceous outer and inner layers to produce bamboo products. The use of such products should not be continued due to their poor bonding strength; the waxy and siliceous layers considerably affect adhesiveness and surface appearance (Nugroho and Ando 2000; Nugroho and Ando 2001). Bamboo scrimber is usually made from bamboo strips treated with phenol formaldehyde (PF) resin and compressed to the desired specific gravity and thickness. The forming process mainly includes hot pressing and cold molding with hot curing. The density of a bamboo scrimber can be controlled to solve the problems of the poor bonding strength and surface appearance. The bamboo bundle, bamboo sliver, and bamboo strip are produced using traditional mechanical separation processes (Li *et al.* 2016). During these processes, the outer and inner layers of the bamboo are removed to meet the requirement for the bonding of bamboo-based units and the mechanical properties of bamboo panels. This necessity to remove substances from bamboo material inevitably impedes the effective utilization of bamboo and negatively affects the yield of bamboo-based units.

BFRC is the latest generation of bamboo-based products. BFRC is an improved version of the scrimber fabrication process, which was developed in Australia. BFRC is a novel engineered composite made from loose bamboo bundles (LBB). Compared with other bamboo products, BFRC has a comparatively higher raw material utilization rate because it has a relatively higher utilization ratio in material sources, which include small-sized bamboo culm. BFRC was formed through hot-pressing several LBB together by using resin at high temperatures, and it is a novel scrimber produced using a mechanical process with a novel roll-pressing flattening without removing any inner layers and outer layers of bamboo. With this novel roll-pressing process, The bamboo is flattened into thick bundles with the thickness of 5 to 10 mm. Punctate or line segment cracks are formed by stress degradation using a stress degradation machine in the loose side of the bamboo bundles. After the drying treatment, the moisture content of the bundles is in a range of 7% to 15%. The BFRC produced from bamboo bundles has excellent performance. BFRC manufacturing is a new technology for improving the utilization of bamboo resources. This technology can utilize whole bamboo culms as a manufacturing unit, overcoming the bonding problems for outer and inner bamboo culms, and improving the bamboo utilization ratio to more than 90% (Qin and Yu 2009; Qin *et al.* 2012; Hu and Yu 2014; Qi *et al.* 2015; Hu *et al.* 2016). It has large potential markets in China and abroad.

Phenol-formaldehyde resins are frequently used adhesives for exterior and semi-exterior panels. They have excellent strength and weatherability, and they are widely used in bamboo scrimber manufacturing (Phong *et al.* 2013; Wang *et al.* 2016). Many studies have investigated how to improve the curing speed and further lower the cost of PF resins. There have been several studies on the resin loading method in lay-up of the bamboo veneer, showing that the resin cover situation and different adhesive load contents have a great influence on the microstructure, strain, and strength. The quality of bonding and gluing content are important for PF adhesive. Bond information between adhesive and lignocellulosic or porous material is important to determine the amount of adhesive penetration into a substance (Papadopoulos *et al.* 2006; Castro and Iwakiri 2014). Bamboo is a porous material formed by mainly parenchymatous tissues, which are also known as spongy tissues. This anatomical feature influences adhesive penetration into bamboo fiber. In BFRC manufacturing, one of the serious problems is high adhesive consumption due to the rough surface of LBB (Anwar *et al.* 2009, 2012). Decreasing adhesive consumption and maintaining the quality of the BFRC will require increased adhesive use. This will lead to an increase in the unnecessary cost of producing floor panels. Therefore, it is important to study the impact of gluing method and resin load on the material performance of the PF that is used to produce BFRC.

The resin load content of BFRC as a measure of the consumption of adhesive is one of the more important parameters to consider in the production of BFRC. Each solid content has a certain relationship to its resin load according to the impregnated experiments. Because of the great surface crack area of the roll-pressing flattening treatment, resin load increases notably. Even if the resin load is only a small proportion of the mass fraction of BFRC in the mixture, the high consumption of resin solid content in the fraction has a negative impact on the resin load of the coarse proportion. From a technological standpoint, producing a minimum amount of resin is necessary, and the desired properties of the boards result in sufficient bonding of the individual veneer. However, an excessive resin load imparts some technological disadvantages, such as high moisture content and possible problems with high vapor pressure during hot pressing. Furthermore, for economic reasons, the consumption of adhesive should be low as possible, as the resin contributes considerably to the costs of the finished boards. Because of the reasons discussed above, the authors decided to glue the core layers and face layers separately (Roffael and Dix 1991; Kamke 2007; Imam *et al.* 2013; Stoeckel *et al.* 2013).

This article describes the benefit of using long fiber bundles extracted from bamboo stems and water-soluble phenolic resins in the fabrication of BFRC for commercial and industrial purposes. The boards investigated had core layers and face layers loaded with different resin contents and different amounts of adhesive. The separate gluing enables the use of different compositions of glue mixes and different resin loads (gluing factors) for the seven layer types (Verma and Chariar 2012; Ashaari *et al.* 2016). This study explored novel BFRC products by treatment with different glue content and different gluing methods. The changes in the physical and mechanical properties of the BFRC boards from *Phyllostachys bambusoides* f. *shouzhu* Yi were also investigated. This study provides a reference for the study and production of BFRC.

EXPERIMENTAL

Materials

Phyllostachys bambusoides f. *shouzhu* Yi is a unique kind of economical bamboo species in Southwest China. It has the characteristics of fast vegetative growth, short growth cycle, excellent fiber state, and high cellulose content.

Bamboo culms were obtained from the hometown of Shouzhu bamboo in Liangping, located in northeast Chongqing in China. The bamboo was 4- to 5-years-old and as tall as 20 m, with a diameter of about 80 mm to 100 mm and a wall thickness of about 9 mm to 12 mm. Bamboo culms were cross-cut into two meter long culms. Using a bamboo culm splitter, each culm was split into three equal parts. The strips were then pressed and fibered into LBB (2000 mm × 100 mm ~ 200 mm × 3 mm ~ 5mm) by using a tailor-made flattening device and then cut to a size of 450 mm × 170 mm × 5 mm in accordance with Chinese patent No. 200920105914.9 (Yu *et al.* (2009)) and American patent US 20110274872 A1 (Yu *et al.* (2011)). The bamboo bundles were dried to a moisture content of about 10%.

Low-molecular weight phenol formaldehyde was obtained from Beijing Dynea Chemical Industry Co., Ltd. (Beijing, China) with a solid content of 44.9%, a viscosity of 33 CP·S, a pH of 10 to 11, and a water miscibility of 7 to 8.

Test Standards

The samples were cut from the composites according to the test standard GB/T 17657 (2013). The modulus of rupture (MOR), modulus of elasticity (MOE), and shear strength (parallel loading) (SS) were compared for different treatment composite samples based on China Standards GB/T 17657 (2013) and GB/T 20241 (2006). Nine samples were obtained for each group to evaluate the mechanical and physical properties. The thickness swelling rate (TS) and width swelling rate (WS) of the samples were determined with an accuracy of 0.001 mm at marked positions after immersion in boiling water for 4 h, drying in an oven at 90 °C for 20 h, and then immersion in boiling water again for 4 h. Dimensional stability was measured for nine samples in each group, and the results were averaged.

The LBB specimens were examined *via* a scanning electron microscope (JEDL JSM-5500LV, Beijing, China), and the surfaces of the LBB specimens after impregnation in PF were examined *via* a 3D digital microscope All-In-One Unit KH-8700 (Beijing, Country) for observing the resin penetration.

Methods

After the treatment, the LBB were reconditioned to reach equilibrium by immersing them in phenol-formaldehyde adhesive for a few minutes to make the BFRC. The target resin contents of the LBB were 10%, 14%, and 18% (ratio of the resin dry weight to the bamboo bundle dry weight) (Li *et al.* 2014a; Febrianto *et al.* 2015). The bundles were dried to achieve a moisture content of 12%.

The matching impregnated LBB material was assembled in a mixed mold with the grain in a parallel orientation and then pressed into the mold using a hot-pressing machine at 140 °C for 25 min (Rao *et al.* 2016). The thickness of the composites was set to 20 mm, and the target density was 1.1 g/cm^3. After conditioning for 1 d to 2 d to reach an equilibrium moisture content, the composite was cut and dimensioned. The dimensions of 420 mm × 170 mm × 20 mm were prepared. The specifics of the assembly pattern are shown in Fig. 1 and Table 1.

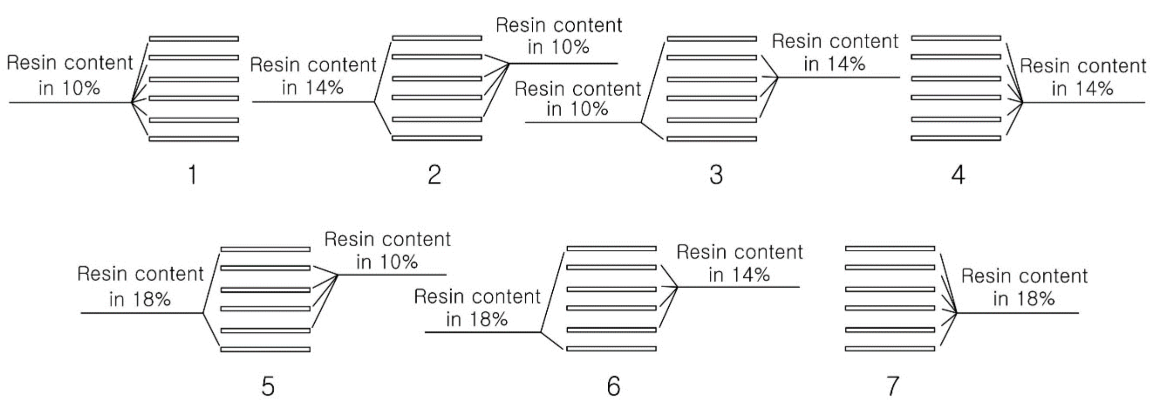


Fig. 1. Assembly design for LBB before hot-pressing

Table 1. Board Contents

Board Number	Face Layer Resin Content (%)	Core Layer Resin Content (%)
1	10	10
2	14	10
3	10	14
4	14	14
5	18	10
6	18	14
7	18	18

RESULTS AND DISCUSSION

Mechanical Properties of BFRC

The resin content of impregnation has an important influence for mechanical properties, especially for MOR and MOE. As shown in Fig. 2, with the increase of the resin load from 10% to 18%, the values of MOR and MOE increased from 160.34 MPa to 209.10 MPa and from 15.5 GPa to 20.3 GPa, respectively. The board face layers impregnated with an 18% resin content had the best mechanical properties, with an MOR of 209.10 MPa and an MOE of 20.3 GPa.

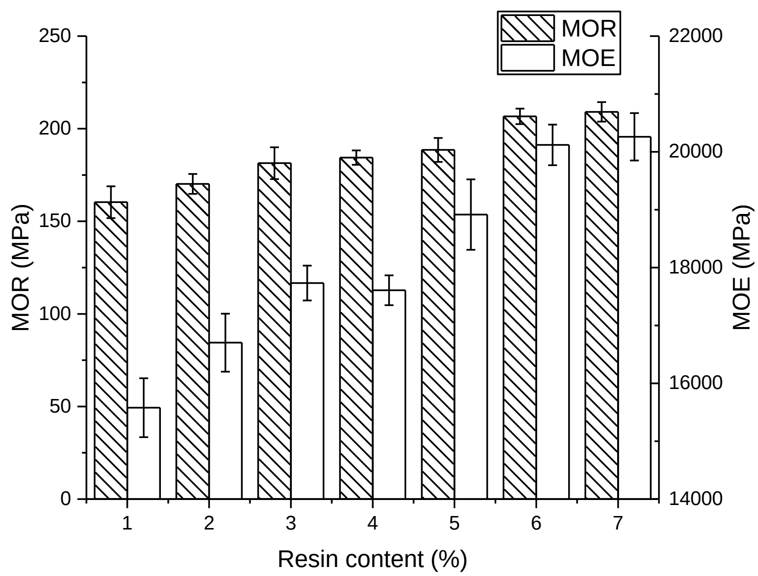


Fig. 2. MOR and MOE of BFRC. The resin content in the numbered columns is as follows: 1) 10% (all layers); 2) 10% and 14% (face and core); 3) 14% and 10% (face and core); 4) 14% (all layers); 5) 18% and 10% (face and core); 6) 18% and 14% (face and core); 7) 18% (all layers).

The face layers impregnated with a high resin content and the core layers impregnated with a low resin resulted in a stable resin content in a short time. Adhesive impregnating from low resin content PF did not have a good adhesive effect, but it enhanced the performance of the bamboo fiber to some extent. When the core bundles were initially impregnated with a low resin content, the core of the bamboo fiber was filled by the glue with low resin content. After the surface was impregnated with the high resin content glue, the surface of the bamboo fiber was filled by the glue with a high resin content. Therefore, the high resin content glue stayed on the boundary of bonding to bear external pressure, which is the main reason for the enhancements in the MOR and MOE of the board (Anwar *et al.* 2005; Milner 2006; Li *et al.* 2014b).

Compared with the specimen that was separately impregnated, the relationship of glue content to the MOR and MOE values had a good linear correlation. The MOR and MOE values of the board increased from 160.3 MPa to 188.5 MPa and from 15.5 GPa to 18.9 MPa with the face, and core layers were impregnated in 18% and 10% resin compared with the layers that were impregnated in 10% glue content. The results indicated that the board with a high glue content likely resulted in a stable cross-linked netlike structure. A stable cross-linked structure is the most influential factor in determining the mechanical properties of materials. Increases in MOE and MOR can result from a more stable cross-linked netlike structure when glue content is increased (Deng *et al.* 2012).

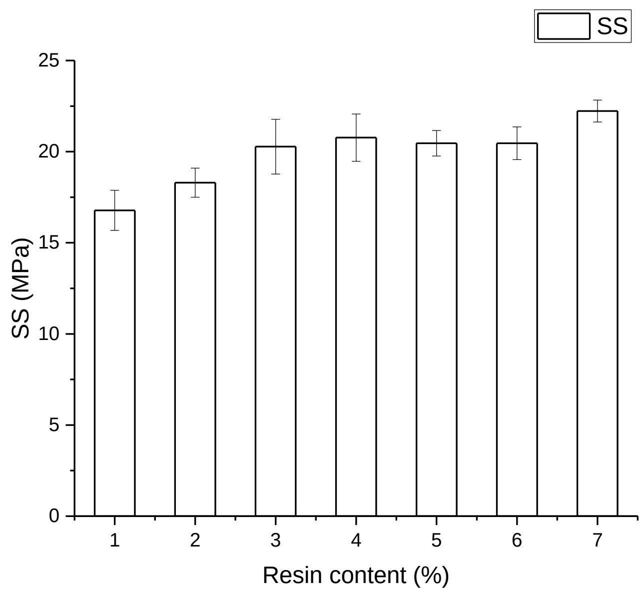
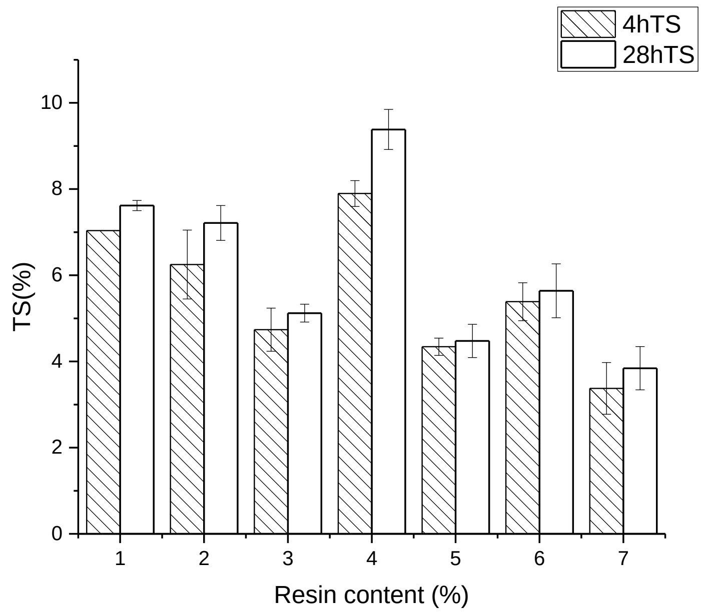


Fig. 3. SS of BFRC. The resin content in the numbered columns is as follows: 1) 10% (all layers); 2) 10% and 14% (face and core); 3) 14% and 10% (face and core); 4) 14% (all layers); 5) 18% and 10% (face and core); 6) 18% and 14% (face and core); 7) 18% (all layers).

The horizontal shear strength was used to evaluate the bonding properties of BFRC. The universal testing machine exerted force on the surface of specimens to produce shear stress on the panel, which led to the weak glue interface breaking, permitting the shear strength to be calculated. The boards impregnated once with a 10% resin content had the lowest horizontal shear strength, and the board surfaces impregnated with an 18% resin content had the best shear strength properties. These results reflected the changes in mechanical properties (Uysal and Yorur 2013). Clear differences in shear strength were observed in the specimens glued with different methods.

As depicted in Fig. 3, when the materials were the same, the shear strength of the BFRC with high resin content was higher than that of the glued board with low resin content. Increased bamboo resin content led to an increase in the shear strength of the BFRC. When assembly patterns were the same, the shear strength of the surface board impregnated with 18% resin content for producing BFRC was higher than that of the board impregnated with 14% resin content. The penetration effect of PF into the high resin content bamboo was greater than that of low resin content bamboo. However, there was no direct link between the assembly pattern and shear strength. The elements that influence shear strength include materials, density, resin content, and surface characteristics. The surface of LBB characteristics and mechanical properties increased with higher resin load gluing treatments. The SEM images from Fig. 5 indicated that BFRC parenchyma cells near the interface were deformed by squeezing and pressure, and the cell walls maintained their basic morphology (Sartori and Tomasi 2013). Some of the bamboo fiber cells were also crushed. Thus, it is possible that the difference in intensity between the different resin load glued-treatments influenced the bonding shear strength of the BFRC.



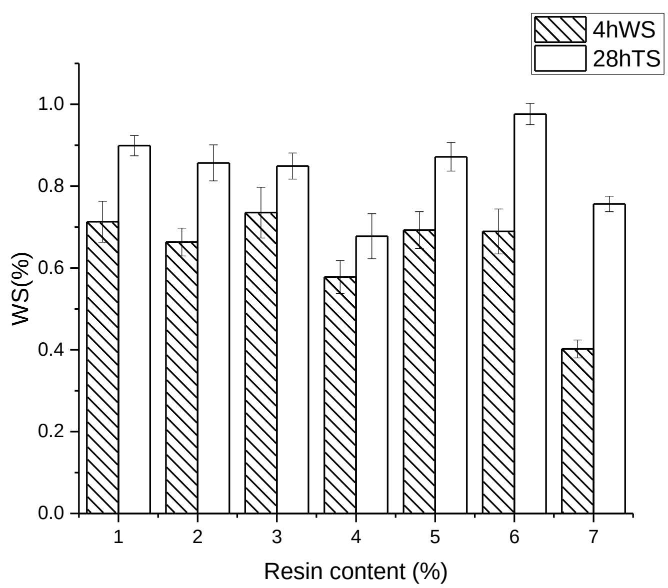


Fig. 4. TS and WS of BFRC. The resin content in the numbered columns is as follows: 1) 10% (all layers); 2) 10% and 14% (face and core); 3) 14% and 10% (face and core); 4) 14% (all layers); 5) 18% and 10% (face and core); 6) 18% and 14% (face and core); 7) 18% (all layers)

Dimensional Stability of BFRC

The PF resin diffused into the interface and then cured into a transparent, hard, and brittle material. Its compatibility with the bamboo micro-capillary system was insufficient. This linkage connected the bamboo fiber and resin but it was possibly considerably weakened after circular hygroexpansion during the drying and boiling treatment. This was because the stresses at the bond line increase during shrinking or swelling as the bound moisture content changes. The resin interface fails if the stresses exceed the interfacial strength.

The glue content is an obviously influential factor in determining the thickness swelling rate (TS) and width swelling rate (WS) at 28 h. In the separate assembly pattern of impregnation, the TS and WS at 28 h was reduced with increasing glue content, which had a resin content of 18%. In addition, the materials had good performance at 28 h. The results of this experiment can be explained by the fact that PF can form a cross-linked network involving insoluble synthetic thermosetting polymers (Fang *et al.* 2016; Wu *et al.* 2017). This network affects modulus, hardness, and tear strength and increases resistance to compression and extension. These polymers can effectively prevent the free hydroxyl radical of bamboo fiber cells from absorbing water.

A low glue content would cause the board to have a high mass of fiber when producing the same density board. Since the surface of bamboo fiber has many free hydroxyl groups, it easily absorbs water, with the formation of hydrogen bonds. The performance will be worse than others when this board sample is impregnated with water and dried in an oven. The glue line will crack when water swelling and dry shrinkage occurs. The dimensional stability of board will be reduced. When impregnated with enough high resin content from the PF adhesive with a high solid content, the cross-linked network insoluble synthetic thermosetting polymers have an adequate, homogenous, and uniform distribution on the surface of the fiber, which prevents the free hydroxyl groups of the board from absorbing water and prevents the board swelling. For this reason, the dimensional stability of board with high resin content on the surface will be better.

As application of glue increases, if the board has a low density, the fiber mass of the board will be reduced. The distance of the fiber will be increased so that the resin cannot bond well with the fibers. The inner loose structure space of the board will allow water to spread easily into the board, and the TS and WS will be increased. The high resin content in impregnation is superior to low resin content in impregnation in TS and WS. As a result, the board impregnated with an 18% resin content had the best properties of dimensional stability.

The assembly pattern of impregnation has an important and unique influence on dimensional stability properties. The board surface impregnated with a high resin content through dilution from the raw resin with high solid content had good performance compared to that of the board surface impregnated with low resin content. The TS of the face board impregnated with 14% resin content and the core layers with the resin content in 10% was higher than that of all the board impregnated with 14% resin content and all the board impregnated with 10% resin content. This result shows that the impregnated glue pattern is important for changing the TS. This is because cross-linked network polymers have an adequate and homogeneous uniform distribution on the surface of fiber when the solid content is increased. The cross-linked network polymers also increased in the face fiber layers. The point of junction between the glue and fiber in the face bundles will increase, and the contact path between water and fiber will decrease. This can explain why the bundle with the face layers of the fiber with 18% resin content and all the fibers with

18% resin content had better values in TS and WS.

Microstructure of BFRC and Glued LBB

During the preparation of LBB, the surface of a semicircular bamboo tube was split and extruded by a fluffing roller and fractionized with a driving roller. The semicircular bamboo tube was spread, and the waxy layers and the siliceous layers on the outer surface of bamboo culm were partly removed. Most of the outer layers of bamboo tube were preserved but were cracked, fragmented, or crushed to form the dotted-shaped/line-shaped cracks. Consequently, the performance of bamboo matrix impregnated with adhesive was improved obviously (Liu *et al.* 2016; Liang *et al.* 2017).

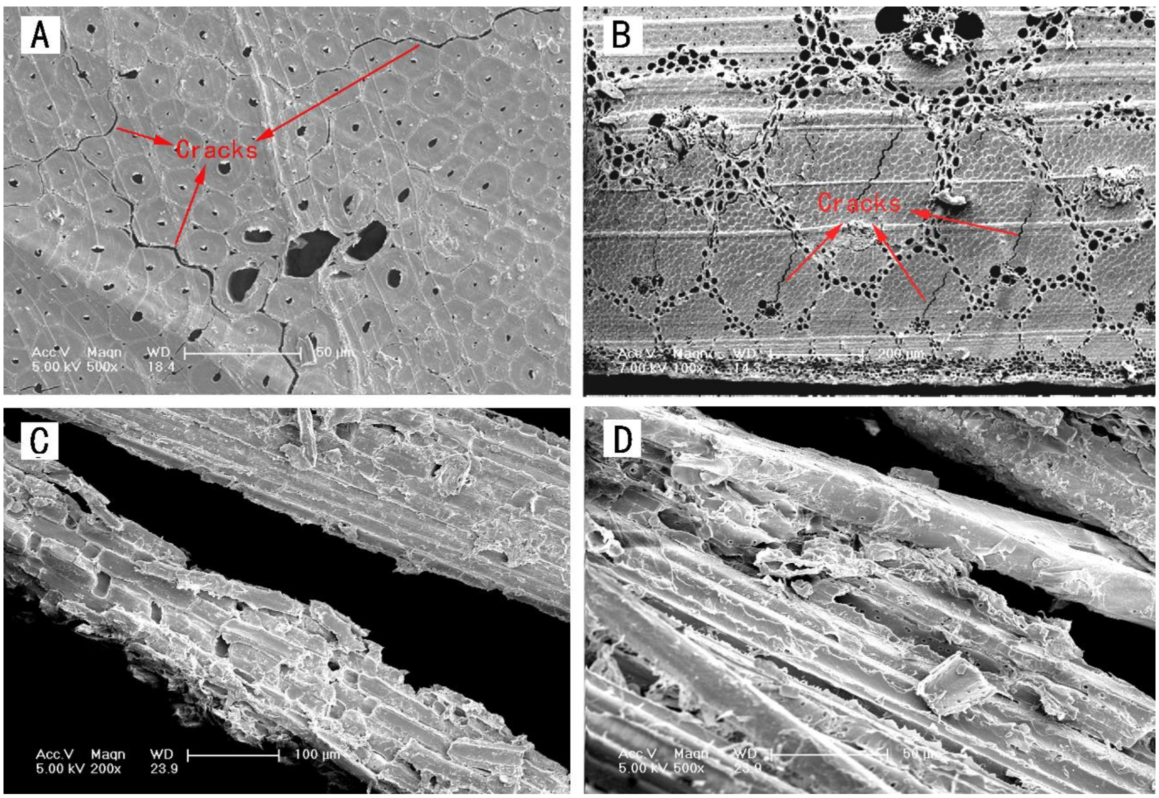


Fig. 5. (A) (B) SEM images of LBB after mechanical roll-pressing treatment: The fiber bundle is composed of several elementary fibers observed in the radial section. (C) (D) SEM images of bamboo showing the roughness of the fiber bundles after mechanical pressing observed in the transverse section.

Figures 5(A) and 5(B) display the microstructures on the transverse surfaces of LBB *via* stereomicroscopy images. As shown, a series of dotted and/or linear-shaped cracks formed on the inner and outer cylinder wall of the bamboo tube. The dotted and linear shaped cracks of LBB that formed due to the mechanical treatment indicate that it brought about enough depth for adhesives to permeate the bamboo during the formation of bamboo-based fiber composites (Khalil *et al.* 2012; Nguyen *et al.* 2013).

The main structure of bamboo consists of ground tissues, parenchyma cells, and bamboo fiber in vascular bundles. Bamboo fibers in vascular bundles are embedded in ground tissues. The lack of fiber cells results in the poor permeation of adhesive into bamboo materials. The morphology of parenchyma fiber cells after mechanical treatment is illustrated in Fig. 5. Fine, line-shaped cracks were present on the boundary of adjacent fiber cells. Therefore, the dilute phenolic resins could penetrate easily through the subtle cracks during the immersing process. As for fiber cells, their own framework structure was explored without severe rupturing along the longitudinal surface of fiber cells during mechanical treatment. This maintains the orientation of the natural bamboo fibers and the high mechanical performance (Zou *et al.* 2009; Kanzawa *et al.* 2012).

The changes in the outer surface of the bamboo tube were investigated by SEM analysis in Fig. 5(C) and Fig 5(D). The alternating smooth and irregular surfaces can be observed on the epidermis of the bamboo after mechanical treatment. It was noted that the longitudinal cracks present on the surface of the bamboo occurred during mechanical treatment. This is in agreement with the results of USM image analysis for the line-shaped cracks. As shown in Fig. 5(C) and Fig. 5(D), this indicates that the silicon and waxy content was dramatically removed during the mechanical treatment, and consequently, the main substance of the bamboo with good bonding performance was unmarked. Therefore, the effective bonding area of bamboo was augmented, and the matrix impregnation path of the bamboo units was improved without specially removing the outer portion of the bamboo (Kim *et al.* 2011).

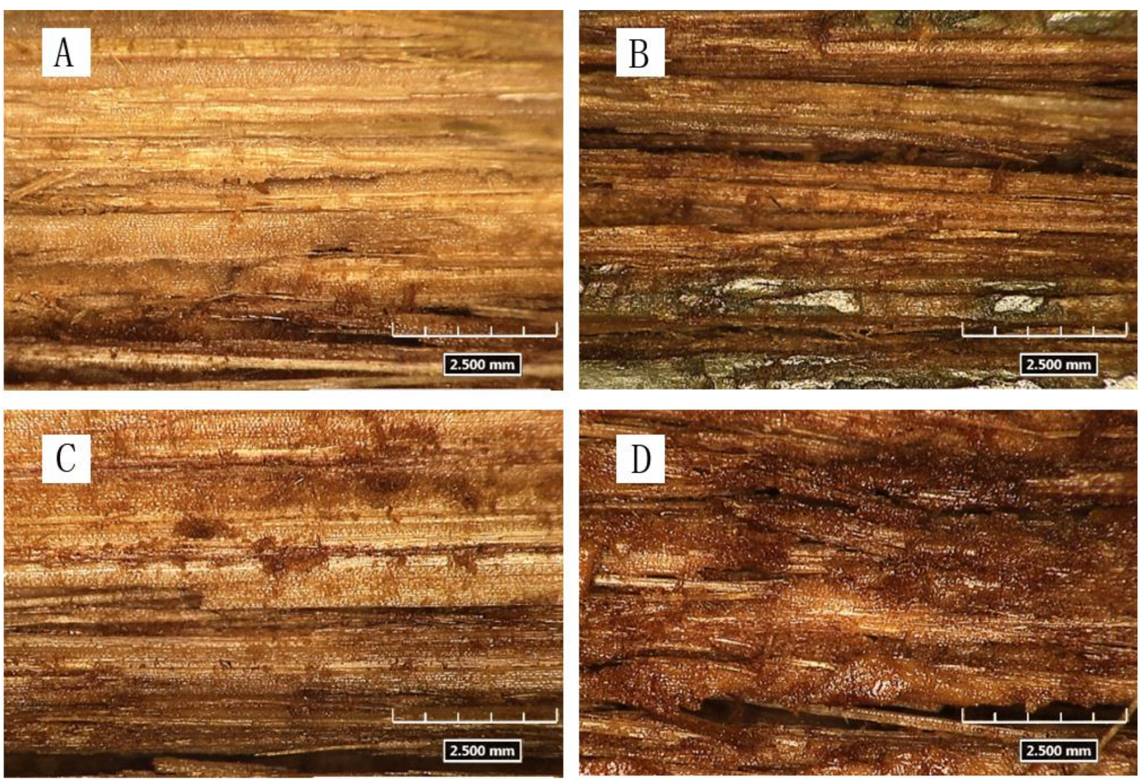


Fig. 6. (A) The bundles were examined *via* 3D digital microscope for the resin content of 10%. (B) The bundle was impregnated with resin of a high solid content and a resin content of 18%. (C) The bundle was impregnated with resin with a resin content of 14%. (D) The bundle was impregnated with raw resin with a solid content of 45.7% and a resin content of 14%.

As shown from Fig. 6(A), the glue sites of a BFRC prepared with PF adhesive that satisfies the relevant requirements of grown glue can be clearly observed in the 3D digital microscope image. According to the picture, with increasing resin content of the gluing treatment, the bonding interface of BFRC between bamboo fiber bundles became misty.

Consequently, the material became uniform. Moreover, the boards with high glue content have higher quality bonding than those boards with low glue contents.

The difference in the surface structure of the bamboo was evident when comparing bamboo impregnated with resin of low resin content to bamboo impregnated with resin of high resin content. As shown, the fine structure of the bamboo surface became partially masked by the PF resin. In Fig. 6, the crushed bamboo surface had jagged, fractured wall layers. In contrast, substrates with the bundles impregnated with resin of high solid content and resin content of about 30% appeared to be thickly coated with glue spots across the bamboo surface in Fig. 6(D). A thicker coating would allow greater interdiffusion of the resin at the interface when two blocks are placed in contact. Hence, a higher solid content of resin may serve to ensure bridging of the films between samples. Polymer entanglements between multilayers have been implicated in improving adhesion strength between multilayers, as an increase in layer number facilitates polymer interdiffusion (Singh *et al.* 2013; Konnerth *et al.* 2016).

Various shapes and sizes of adhersive particles were covered on the surface of bamboo fiber. The resin with higher resin content of 18% that covered the bamboo fiber surface contributed to the enhancement of the dimensional stability and mechanical properties of the samples. The photograph of the details also indicated that, although the same effect described above is less extensive and possibly less intense, it is nonetheless still clearly visible on the glue granules surrounding the bamboo fiber in the pictures. This clearly indicates that the effect described was only caused by the high resin content and solid content.

As illustrated in Figs. 6(B) and 6(D), the addition of the endowed PF resin became darker but achieved better surface coating. When it interacted with the bamboo substrate during hot pressing, there was synchronous crosslinking with the resin, resulting in better resin bonding distribution in the bamboo’s microstructure. Additionally, the pit capillaries on the bamboo cell walls became congested with the high resin content PF crosslinked polymer. Interaction or polymerization with the bamboo fiber cell wall components is an effective way to provide a stable interphase region at the layer structure. This is because the low-molecular PF resin better penetrated the cell wall.

CONCLUSIONS

1. The physical performance of LBB using PF adhesives was examined in this study. According to the results, all the fiber bundles impregnated with PF adhesive had higher strength and good dimensional stability after different resin content treatments. Bamboo bundles could be bonded satisfactorily with the PF adhesive with the exception of LBB with 10% resin content by weight. In exterior applications, the outdoor adhesive PF with high resin content exhibited the best combination effect for bonding performance. The penetration into the bamboo structure with a resin content of 18% in the face layers and a 14% resin content in the core layers had the best performance out of all methods. After treatment, the TS and WS of the boards impregnated with low resin content were higher than those of the boards impregnated with high resin content. The samples showed better performance in the multiple assembly pattern impregnated treatment. According to the data, the best dimensional stability was obtained from the resin content of 18% in the face layers.

2. According to the roll-pressing treatment, the crack surface provided the best interaction for the adhesive to penetrate into the substrate. The adhesive forms a molecular-level contact with the surface, and it should be easier to penetrate the bamboo cell and develop a close contact with the substrates. The face fiber in the radial section was treated more intensively than the core fiber, which generated obvious outer cracks and resulted in the face fiber being more easily penetrable than the inner fiber. Furthermore, the images show that the surface cells of the bamboo were crushed during the roll pressure preparation treatment, resulting in more penetration and differences in penetration regions.
3. The BFRC produced using LBB and PF resins through different gluing and laying-up methods are very meaningful. These results demonstrate that BFRC with good properties are promising and can be achieved with high bamboo utilization.

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