Effects of Surface Modification on the Physical, Mechanical, and Thermal Properties of Bamboo-polypropylene Composites

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To fabricate homogeneous bamboo fiber reinforced thermoplastic composites, polypropylene (PP) fiber and 3-aminpropyltriethoxysilane (APTES) modified bamboo fibers were first formed into mats by non-woven air paving technology, and then the composites were created by hot-pressing the mats. The modification of BFs was characterized by XPS and FTIR analyses, and the results confirmed that APTES had been grafted onto the surfaces of BFs. The effects of concentrations of APTES on the mechanical, physical, morphological, and thermal properties of the bamboo-polypropylene composites were examined by tests of bending strength, scanning electron microscopy (SEM), differential scanning calorimetry (DSC), and water absorption. The analysis of physical and mechanical properties revealed improved mechanical properties and water resistance (3 wt% of APTES). SEM images were used to assess the influence of modification treatment on the properties of the composites. The results confirmed that the modification of APTES improved the interfacial adhesion between BFs and PP matrix. DSC results indicated the melting point of composites decreased with an increase in concentration of APTES up to 3 wt%, while the melting point of composites increased when the concentration of APTES was higher than 3 wt%.

Keywords: Bamboo fiber; Polypropylene; Composite; 3-Aminpropyltriethoxysilane

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INTRODUCTION

With the growing demand for more comfortable, healthier, and environmentally friendly products, natural fibers have received increasing attention because they are biodegradable and renewable (John and Thomas 2008; Ku *et al*. 2011; Abdul Khalil *et al*. 2012). Natural fibers have many excellent properties, such as high strength and modulus, low cost, and biodegradability (Singha and Thakur 2009; Thakur *et al*. 2014; Fei *et al*. 2017).

Bamboo fibers (BFs) are thought to be a 'natural glass fiber' because of their high strength and stiffness (Okubo *et al*. 2004). Bamboo is abundant in many countries. Bamboo forests cover approximately 22 million hectares worldwide (Han *et al*. 2008). Furthermore, bamboo matures in three to four years and is regarded as one of the fastest growing plants. Bamboo possesses excellent mechanical properties due to its hierarchical structures. The

tensile strength and flexural strength of the natural bamboo are 298 and 148 MPa, respectively (Li *et al*. 2020). The BFs that are obtained from bamboo culms by mechanical grinding, chemical methods, or steam explosion are used to produce reinforced composites that display excellent mechanical properties (Wang *et al*. 2010).

In the past, the authors used solvent-extraction techniques to prepare BFs and non-woven air paving technology to prepare composite materials with good mechanical properties (Tang *et al.* 2018). This composite has a broad application prospect in the field of manufacturing interior-automotive parts (Ashori 2008) and furniture (Fei 2016). However, this composite has poor water resistance and interfacial adhesion between the matrix and the BFs. This is attributed to the fact that the main components of bamboo are cellulose, hemicellulose, and lignin, which are relatively hydrophilic (Wang *et al*. 2014). Thus, it is important to provide more hydrophobic chemical groups at the surface of bamboo fibers to strengthen the properties of BF composites. At present, the commonly used method is to modify the surface of bamboo fiber, and the modification method can be divided into physical, chemical, and coupling agent modification.

When polar bamboo fibers and non-polar materials are combined, the interfacial strength is generally low. However, the presence of coupling agent can solve this problem. Generally, there are two functional groups in silane coupling agent molecules: one has good compatibility with polymer plastics, and the other can form covalent bonds through chemical reactions with bamboo fibers. Therefore, silane coupling has been found to significantly improve adhesion in composites (Pickering *et al*. 2003; Park *et al*. 2004; Weyenberg *et al*. 2006; Thakur *et al*. 2010; Singha and Thakur 2009). Alkali treatment affects the swelling of cellulose fibers, altering their structure. Chemical modification of alkali-treated BFs with silane would increase their adhesion to the polymer matrix in composites (Zhang *et al*. 2015). The siloxy group in the silane coupling agents can hydrolyze to form the bond between the reactive siloxy group and hydroxyl group on the surface of bamboo fiber, which reduces the number of hydrophilic hydroxyl groups and improves the interface performance between bamboo fiber and plastic substrate (Miller and Berg 2003; Xie *et al*. 2010). There are many kinds of coupling agents for modified bamboo fiber, including aluminate ester (Chen *et al*. 2009), titanate ester (Wan *et al*. 2010), and silane coupling agent. Furtherly, silane coupling agent is most widely used in the world, and there are many kinds of silane coupling agents, such as $(RO)_3Si\text{-}(CH_2)_3\text{-}NH_2$ (Maldas *et al*. 1989; Abdelmouleh *et al*. 2005), $(RO)_3Si\text{-}CH{=}CH_2$ (Raj *et al.* 1989; Bengtsson and Oksman 2006), $(RO)_3Si\text{-}(CH_2)_3\text{-}SH$ (Ismail *et al*. 2002), and $(RO)_3Si\text{-}(CH_2)_{15}CH_3$ (Abdelmouleh *et al*. 2004). Haddou *et al*. (2016) studied the tetraethyl orthosilicate modified Vietnamese bamboo flour. Wang *et al*. (2014) used a coupling agent to treat BFs based on alkali treatment with 5% NaOH solution, which greatly increased the dispersion of BFs and decreased the surface energy of BFs. The mechanical property of the BFs reinforced polypropylene (PP) composite performed best when the silane coupling agent solution concentration was 3 wt%. Hong *et al*. (2018) developed a new pathway to enhance interfacial adhesion between the BFs and the PBs matrix by modifying BFs with the mussel-inspired hybrid coatings, and excellent static and dynamic mechanical properties and superior anti-water capability were achieved.

In this study the authors used a series of concentrations of the silane coupling agent 3-aminpropyltriethoxysilane (APTES) to treat the surface of BFs. Composite mats were prepared using a non-woven air method, which improved the BFs distribution remarkably. The mats were then hot pressed. The reaction mechanism between BFs and APTES was evaluated by Fourier transform infrared spectroscopy (FTIR) analysis and by X-ray

photoelectron spectroscopy (XPS) analysis. The main purpose of this study was to determine whether this modification method increases the mechanical strength of bamboo fibers/polypropylene (BFs/PP) composites.

EXPERIMENTAL

Materials

The BFs (moisture 6.5%) were provided by Haibosi Chemical Technology Co., Ltd. (Fujian, China). The PP with 11.11 dtex was from Taizhou Hailun Chemical Fiber Co., Ltd. (Guangdong, China). The APTES (Fig. 1) with a density of 0.934 g/cm^3 was purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China), and its chemical structure is shown in Fig. 1. Ethanol (99.7%) was obtained from Beijing Chemical Works (Beijing, China).

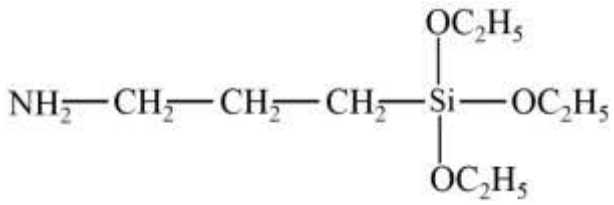

Fig. 1. Chemical structure of 3-aminpropyltriethoxysilane

Methods

Treatment of BF

The BFs were washed with distilled water to remove dust and then dried in an oven for 5 h at 103 °C. The BFs were proportionately added into ethanol and water solution (ethanol:water = 95:5 in vol%) containing APTES with different weight ratios: APTES/BFs = 0, 1, 3, 5, and 7 wt%. After 1 h, BFs were filtered and washed with ethanol to remove unreacted APTES. Then, the BFs were dried in an oven at 110 °C by air.

Composite preparation

The BFs treated with APTES were mixed with PP at a 50/50 weight ratio (BF/ PP) using a non-woven air paving technology. The composites were pressed into thin sheets (Fig. 2). They were subjected to a pressure of 4 MPa for 10 min at 180 ℃. Then, the composites were cooled down and kept in a cold press for 5 min under a pressure of 4 MPa before analysis.

Characterization

FTIR analysis

The FTIR analysis was performed on a Nicolet IS10 FTIR spectrometer (Thermo Fisher Scientific, Waltham, MA, USA) to characterize the functional group changes on the surface of BFs at room temperature over a scanning range from 4000 to 400 cm^{-1}.

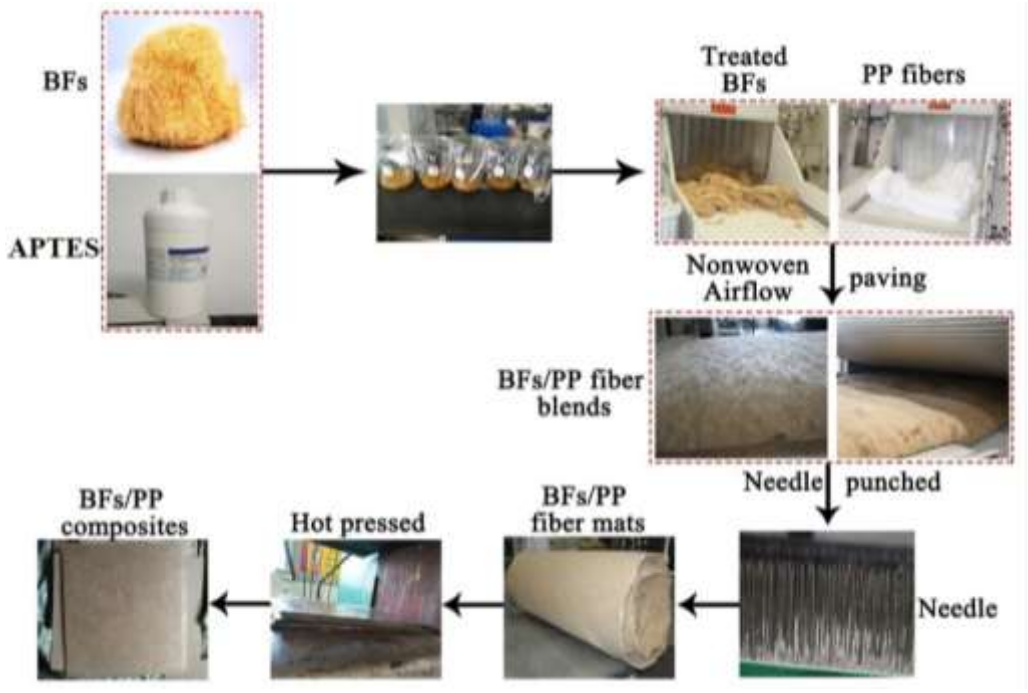

Fig. 2. Graphical illustration of the production process from BFs and PP fibers to composites

XPS analysis

The XPS experiments were conducted on an Escalab 250Xi apparatus (Thermo Fisher Scientific, Waltham, MA, USA) under vacuum of 2×10^{-10} mBar and a spot size of 500 μm with Al K_a source.

Differential scanning calorimetry (DSC)

Differential scanning calorimetry of PP and composites were characterized using a DSC-200F3 (NETZSCH Group, Selb, Germany). The samples approximately 5 to 10 mg were loaded in an aluminum pan. The samples were heated from 25 ℃ to 250 ℃ at a 10 ℃/min rate.

Mechanical characterization

The mechanical properties of the BF/PP composites were determined using a universal testing machine (Zwick/Roell Z030; Zwick Roell Group, Ulm-Einsingen, Germany). Flexural strength and modulus were tested with a minimum of five specimens ($127 \times 12.7 \times 4.4$ mm^3) using a three-point bending method according to ASTM D790-10 (2010).

Water absorption (WA) and thickness swelling (TS) of the composites was determined according to the ASTM D5229-10 (2010) standard. The WA of a specimen was obtained from the weight gain of the sample divided by its original dry weight. The ratio of WA was calculated using Eq. 1,

$$WA\ (\%) = \frac{m_2 - m_1}{m_1} \times 100 \quad (1)$$

where m_1 and m_2 are initial weight (g) and final weight (g), respectively.

The *TS* of a specimen was obtained from the thickness gain of the sample divided by its original dry thickness. The ratio of TS was calculated with Eq. 2,

$$TS\ (\%) = \frac{h_2 - h_1}{h_1} \times 100 \quad (2)$$

where h_1 and h_2 are initial thickness (mm) and final thickness (mm), respectively.

Interfacial morphology analysis

Scanning electron microscopy (SEM) was used to observe the cryo-fractured surfaces of BF/PP composites. The samples were introduced into liquid nitrogen and fractured. The fracture surfaces were subjected to a gold sputtering (e-1045; Hitachi Limited, Tokyo, Japan) and observed using a JEOL JM-6400 microscope (Hitachi Limited, Tokyo, Japan) operating at 40 kV.

RESULTS AND DISCUSSIONS

Surface Chemistry of BFs

To reduce the dust on the surface of BFs, BFs were treated with pure water. The APTES hydrolyzed in ethanol solution to generate silicon hydroxyl (Si-OH) (Gao *et al*. 2012); the BFs' hydroxyl groups reacted with the hydroxyl groups of the APTES and formed Si-O-C bonds on the fiber surface, as outlined in Fig. 3.

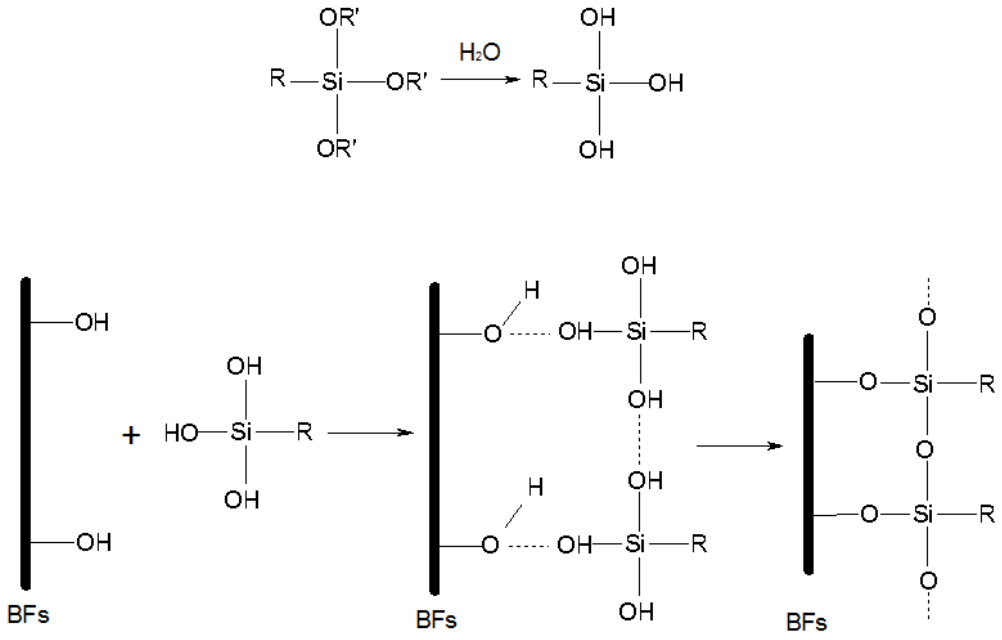

Fig. 3. The proposed chemical reactions between BFs and APTES

The reaction between BFs and APTES was verified through XPS and FTIR analyses. Figure 4 shows the XPS spectrum of untreated BFs and treated BFs with 3 wt% APTES (APTES-3-treated BFs). The XPS spectra of BFs indicated that treated BFs had a lower O/C ratio (0.82) than that of untreated BFs (0.98), because the APTES molecules with a lower O/C ratio (0.17) were grafted on the BFs surface. The chemical composition of the natural fibers surface can be estimated on the basis of the O/C ratio (Migneault *et al*. 2015). In treated BFs, the absorption peaks of Si and N in the XPS spectra appeared at 101

and 400 eV, respectively, indicating that nonpolar APTES was grafted onto the surface of BFs, which lowered the O/C ratio.

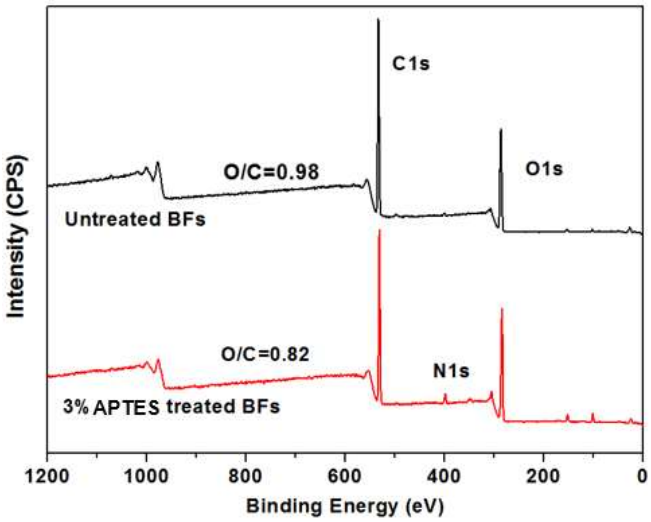

Fig. 4. XPS spectra of untreated BFs and treated BFs with 3 wt% APTES

Figure 5 shows the XPS high resolution spectrum of C1s on the surface of 2 samples. The high-resolution C1s spectra of both BFs were resolved into four characteristic functional groups: C1 (–C–H/–C–C, 284.6 eV), C2 (–C–O–, 286.5 eV), C3 (C=O, 287.8 eV), and C4 (–O–C=O, 288.8 eV) (Bulut and Aksit 2013). The C1 and C2 contents of APTES-3-treated BFs increased 16.9% and 3.35%, respectively, relative to untreated BFs (Fig. 5a), and the C3 and C4 contents were decreased by 15.7% and 4.47%, respectively (Fig. 5b). Increased C1 content demonstrated that the –C–C of APTES was grafted on the BFs surface so that the treated BFs contained a higher proportion of aliphatic carbons (Panthapulakkal and Sain 2007). The increase of C2 content indicated that the double bonds of C=O were opened to form a C-O, resulting in a decrease in C3 content and an increase in C2 (Chen *et al*. 2012). The decrease of extractives and carboxylic acids of BFs resulted in lower C4 contents.

The FTIR analysis was conducted to investigate chemical reaction between BFs and APTES in this study. Spectra of FTIR of raw BFs and treated BFs are presented in Fig. 6. A peak was observed at the 3427 cm^{-1} absorption band in the spectrum of BFs assigned to O-H stretching vibrations. A peak at 1597 cm^{-1} (bending vibration of N-H) was observed in the spectra of the APTES-treated BFs but not in the untreated BFs, which confirmed that N-H was grafted onto the BFs surface.

Mechanical Properties of Composites

The mechanical properties of five BF/PP composites are shown in Fig. 7. The flexural strength and flexural modulus of the composites increased as APTES concentration increased up to 3 wt%. The flexural strength increased 26% and flexural modulus increased 80%, compared to the untreated BF/PP composites. Moreover, the flexural strength was higher than that of the highest flexural strength of bamboo fiber composites reported by Tong *et al.* (2013) (39.8 MPa) and Lee *et al.* (2009) (53.1 MPa), respectively. The increase of mechanical properties with an increase in APTES concentration may have been due to enhanced fiber and matrix adhesion of the composites.

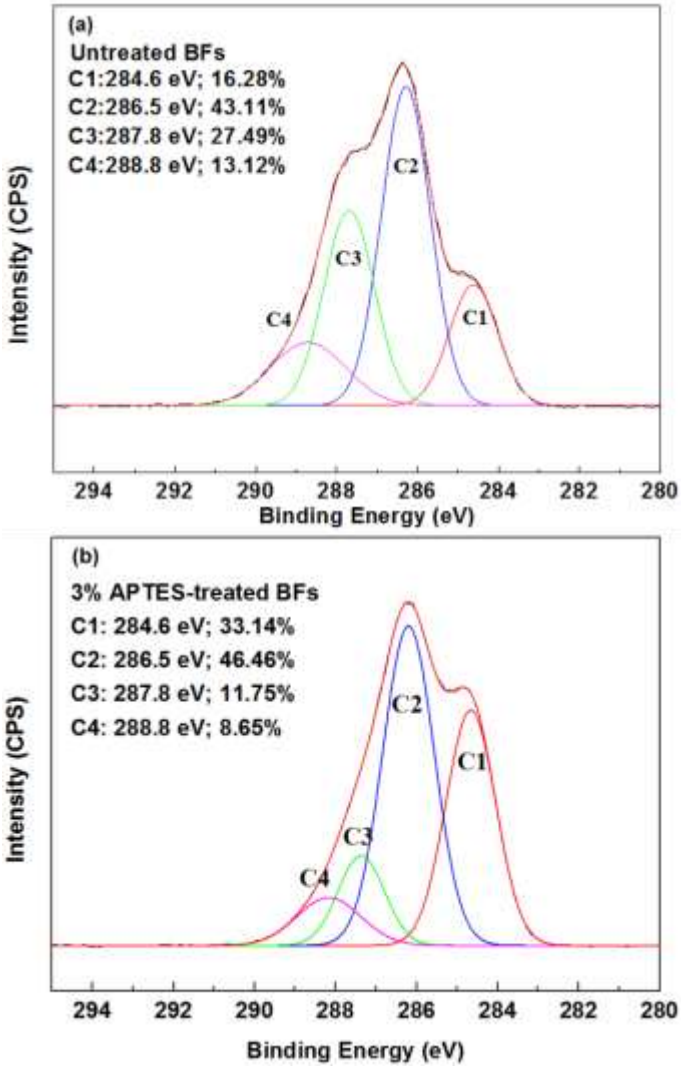

Fig. 5. High-resolution XPS C1 spectra of (a) untreated BFs and (b) BFs treated with 3 wt% APTES

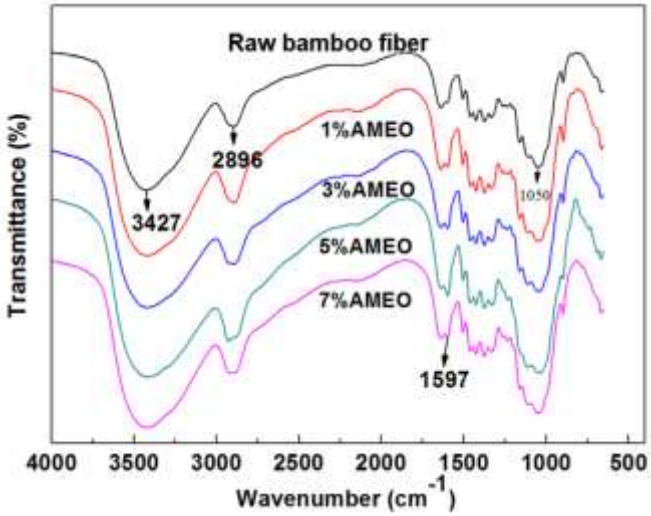

Fig. 6. FTIR spectra of raw BFs and treated BFs

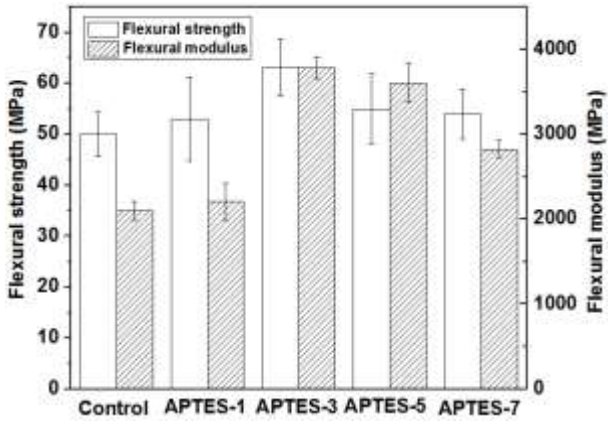

Fig. 7. Effects of fiber surface grafting on the flexural properties

However, the 7 wt% APTES resulted in a mild reduction in the flexural strength and flexural modulus of the composites. It was possible that the coupling agents on the interface were excessive when APTES concentration was 7 wt%. The coupling agents accumulated into the weak boundary layer as their concentration increased. When the composites are subjected to an external force, the weak boundary layer becomes the fracture area and its mechanical properties are reduced (Xie *et al*. 2010).

SEM Analysis

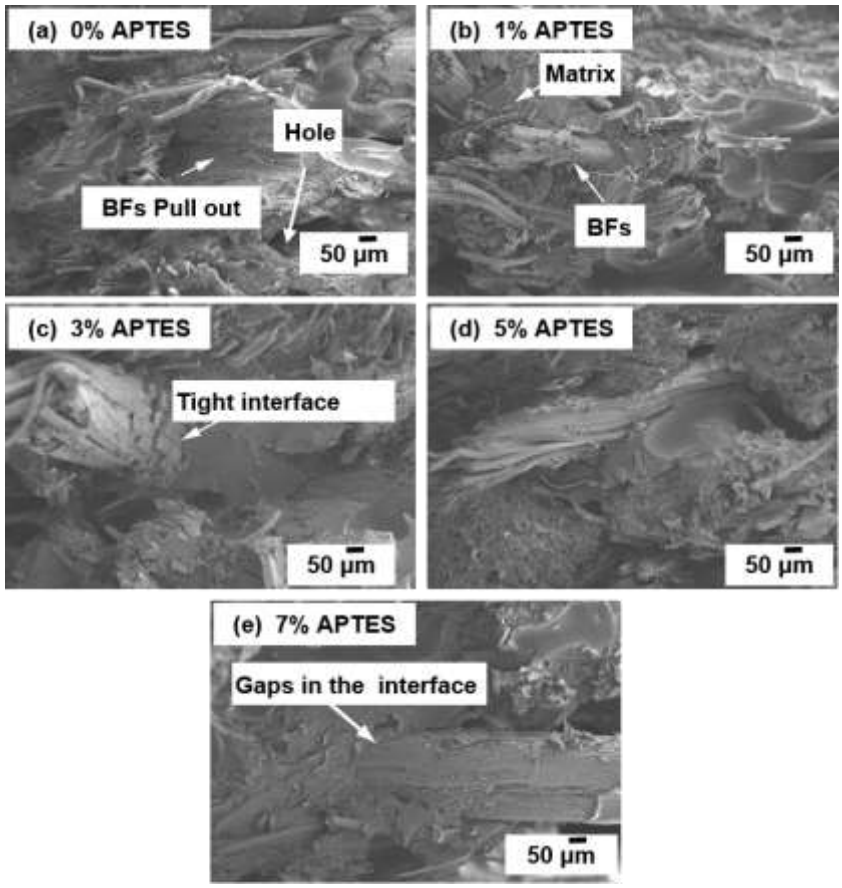

Fig. 8. SEM of fractured surface of BF/PP composites

The morphology of the composites' fractured surfaces was analyzed through SEM (Fig. 8). Holes were generated from the pull-out of fibers on the fractured surface of the untreated-BFs/PP composites (Fig. 8a), which led to a poor interfacial adhesion between the BFs and PP. In contrast, in APTES-3-modified composites, the BFs were combined with PP closely, and the composites destruction mode was brittle failure (Fig. 8c). The 7 wt% APTES decreased the adhesion between the BFs and PP (Fig. 8e). Wang *et al*. (2012) indicated that there is a critical ratio that is equal to 3 mass% for 100 mass% in wood fibers. This ratio is consistent with the current study's ratio.

Thermal Stability Analysis

Thermal behavior also has an important role for the practical applications of composites. Pure PP and BF/PP composites were heated from 50 to 250 °C based on the melting point of pure PP of 173.2 °C. The DSC curves and the thermal data are shown in Fig. 9 and Table 1. After adding BFs to PP, the melting point decreased. It was possible that the molecular chains of PP could not arrange in an orderly manner under restriction from the BF, which resulted in a lower melting point (Ying *et al*. 2013). When the concentration of APTES is changed, the melting points of the BF/PP composites have negligible changes, which may be due to the experimental error. Therefore, it can be concluded that APTES has no obvious effects the melting point of composites. Similarly, the variation tendency of melting enthalpy was approximately similar with the melting point.

Table 1. Melting-Point and Melting Enthalpy of Pure PP and BF/PP Composites

	Pure PP	Control	1% APTES	3% APTES	5% APTES	7% APTES
Melting Point (°C)	173.2	167.8	166.8	166.2	167.6	167.2
Melting Enthalpy (J/g)	96.9	20.6	19.0	19.1	26.0	19.4

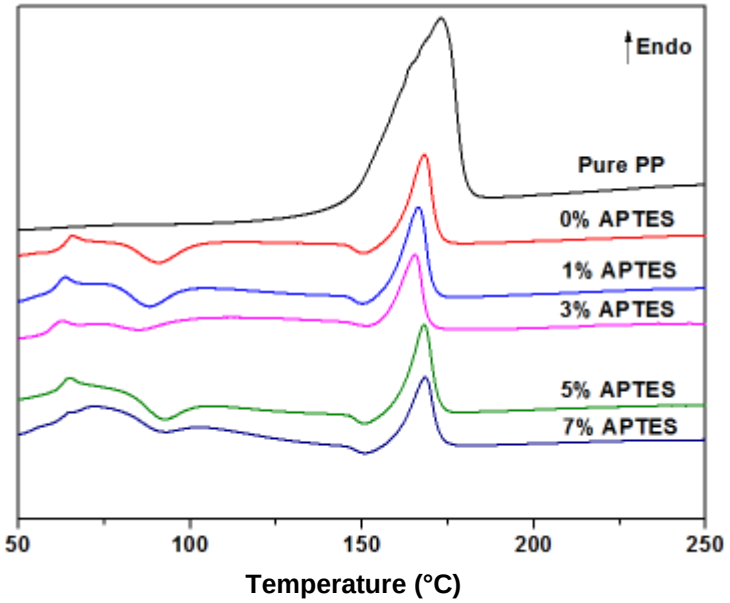

Fig. 9. DSC curves of pure PP and BF/PP composites

Water Resistance of Composites

Figure 10 shows the *WA* and *TS* curves of BF/PP composites. The percentage of *WA* and *TS* increased in all BF/PP composites with soaking time but gradually reached a plateau. As shown in Fig. 10, untreated BF/PP composites showed a high *WA* and *TS*. The WA values of APTES-3 treated BF/PP composites were approximately 0.4 to 3.2%, which was lower than that after 162 h of water immersion. The rapid increases of *WA* and *TS* were observed during immersion of before 20 h for all of the composites, and then the curves of *WA* and *TS* reached a plateau. This is to say the *WA* and *TS* gradually increased. As is already known, the water uptake of BF/PP composites is closely related to the hydrophilicity of the BFs, which is closely associated with the surface chemical composition of the BFs and the interfacial chemistry of the composites. When the concentration of APTES was between 0 and 3 wt%, the substitution of –OH groups of BFs with APTES reduced the number of available –OH groups on the fiber surface, which reduced the hydrophilicity of the fibers. Moreover, the improved adhesion to the polymer matrix in composites may also reduce water penetration into the cellulose fibers. As the concentration of APTES increased, the *WA* and TS increased, possibly because a weak boundary layer weakened the bonding between the fiber and matrix.

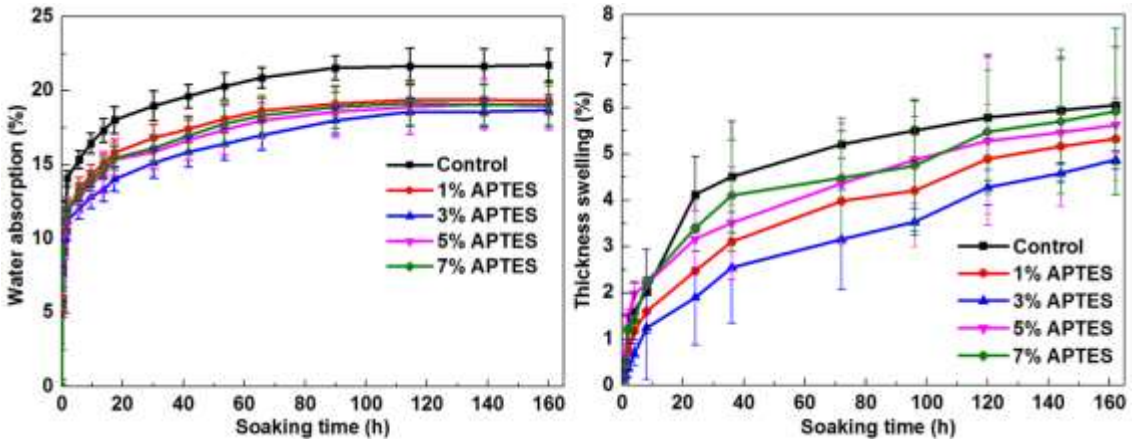

Fig. 10. Effects of fiber surface grafting on the water absorption properties of BF/PP composites

CONCLUSIONS

1. Through the analysis of X-ray photoelectron spectroscopy (XPS) spectra of untreated bamboo fibers and 3 wt% 3-aminpropyltriethoxysilane (APTES)-modified bamboo fibers, it was found that the O/C ratio of modified bamboo fibers decreased, and the absorption peaks of silicon and nitrogen appeared. The contents of C1 and C2 increased, while the contents of C3 and C4 decreased. These phenomena indicated that APTES reacted with bamboo fibers and achieved molecular grafting onto the surface of bamboo fibers.
2. The Fourier transform infrared (FTIR) analysis showed that the stretching vibration peaks of Si-O-Si and Si-O-C increased after modification, and the bending vibration peaks of N-H appeared, which further explained the chemical reaction between APTES and bamboo fibers.

3. Through improved interfacial adhesion, the water resistance and static mechanical properties of bamboo/PP fiber composites were remarkably improved. The properties of the composites were optimal at a concentration of 3 wt% of APTES.

ACKNOWLEDGMENTS

This study was financially supported by the Special Fund of Chinese Central Government for Basic Scientific Research Operations in Commonweal Research Institutes (Grant No. CAFYBB2016ZD006) and the National Key Point Research and Invention Program of the Thirteenth (Grant No. 2017YFD0600800)

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Article submitted: Nov. 6, 2019; Peer review completed: Jan. 23, 2020; Revised version received and accepted: April 23, 2020; Published: July 1, 2020.
DOI: 10.15376/biores.15.3.6230-6243