Tensile Properties of Bamboo Fiber-reinforced Polypropylene Composites Modified by Impregnation with Calcium Carbonate Nanoparticles

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Tensile properties of bamboo fiber (BF)/polypropylene (PP) composites were investigated for different impregnation modification processes. The surface morphology of BF and the fracture morphology of the BF/PP composites were examined by scanning electron microscopy (SEM). Images showed that $CaCO_3$ nanoparticles (nano-$CaCO_3$) adhered to the fiber surfaces. Tension tests of individual bamboo fibers (IBFs) and the BF/PP composites were conducted. The results indicated that the tensile strength (TS), modulus of elasticity (MOE), and elongation at break (EAB) of IBFs increased by 16.0%, 22.2%, and 5.2%, respectively, while those of the BF/PP composites increased by 3.0%, 7.0%, and 15.8%, respectively, compared to the control samples. Various process parameters during the impregnation with nano-$CaCO_3$ had marked effects on the tensile properties of the composites. The optimal conditions for BF-reinforced PP composites by impregnation modification were determined to be a nano-$CaCO_3$ concentration of 1.0×10^{-2} g/mL, an ethylenediamine tetraacetic acid disodium salt (EDTA-2Na) additive concentration of 8.5×10^{-4} g/mL, and an impregnation time of 25 min.

Keywords: Calcium carbonate nanoparticles (nano-$CaCO_3$); Polypropylene (PP); Bamboo fiber (BF); Composites; Tensile properties

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INTRODUCTION

The use of natural fibers in place of synthetic fibers to reinforce polymer composites has attracted great attention among researchers in recent years because of the interest in developing environmentally friendly materials (Jindal 1986; Chattopadhyay *et al*. 2011). Natural fibers have many desirable characteristics, such as abundance, renewability, recyclability, and biodegradability, and they can provide the basis for a high-tech revolution (George *et al.* 2001). The advantages of natural fiber-reinforced polymer composites arise because of their sustainability, low cost and density, high dimensional stability, good processability, and favorable mechanical properties (Sain *et al.* 2005; Dominkovics *et al*. 2007; Banga *et al*. 2015). Various researchers have studied natural fibers containing polyolefins (Sain *et al.* 1993; Saxena and Gowri 2003; Ying *et al*. 2013). Natural fiber-reinforced polymer composites are widely used in the automotive industry, aircraft, interior decoration, and many other fields encountered in daily life (Davoodi *et al.* 2012).

Natural fiber-reinforced polymer composites may have problems related to fiber porosity and dispersion, affecting the interfacial compatibility between matrix and fiber (Bledzki *et al*. 2005). At present, there are three main methods to combat these issues: the modification of natural plant fibers or thermoplastic polymers or the addition of coupling agents to develop the interface compatibility. These strategies effectively work to improve the mechanical properties of composites (Albanoa *et al*. 2002; Lee *et al*. 2004; Plackett 2004).

Among the modification methods, nano-$CaCO_3$ modification has been developing considerable prospects because of the nano-scale, ultra-fine solid structure of $CaCO_3$, and the high economic value of the process. Nano-$CaCO_3$ plays an important role in the reinforcing and toughening of materials and enhances the processing and operational performance when used in plastic packing (Huang *et al*. 2011; Qiu and Zhou 2011; Zhang *et al.* 2011). Meanwhile, research has shown that nano-$CaCO_3$ on the surface of fibers has a strong electrostatic attraction, which can affect the interface performance of fibers and polymer (Lee *et al.* 2007). Shi *et al.* (2011b) prepared $CaCO_3$ particles on the surface of kenaf and conducted cell cavity *in situ* synthesis successfully, finding that nano-$CaCO_3$ particles could adhere to the surface of fibers, which enhanced the interface compatibility. Liang *et al.* (2014) reported that the mechanical properties of kenaf fibers and their composites were increased by impregnation with $CaCO_3$ particles. Gao (2012) found that the processing parameters of nano-$CaCO_3$ production by ion solution *in situ* synthesis had a significant impact on the tensile properties of bamboo fiber (BF) and its composites.

It is inconvenient to produce nano-$CaCO_3$ industrially because of its tedious manufacturing process and high price, although the nano-$CaCO_3$ *in situ* modification of BF induced an obvious improvement in the interface performance. In this study, nano-$CaCO_3$ impregnation modification of BF was carried out to study the feasibility of *in situ* deposition modification. The influence of the modification process on the tensile properties of the composites was explored, further revealing the functional mechanism of interfacial bonding performance in the inorganic nanoparticle-reinforced composites.

EXPERIMENTAL

Materials

Neosinocalamus affinis, aged 1 year, with a moisture content of 8% to 12% was collected from a bamboo plantation located in Sichuang Province, China. Nano-$CaCO_3$ with a particle size of 15 to 40 nm was supplied by Boyu Gaoke New Materials Co. Ltd. (China). Ethylenediamine tetraacetic acid disodium salt (EDTA-2Na), analytically pure, with a melting point of 25 °C, was purchased from Huabo Stand (Beijing, China). Polypropylene (PP) film with dimensions of 150 mm × 150 mm × 0.5 mm, a density of 0.905 g/cm^3, and a melting temperature of 165 to 170 °C was supplied by Beijing Gemstar Commerce Co. Ltd. (China).

Methods

Preparation of individual bamboo fiber (IBF)

According to the standard GB/T 15780 (1995), samples were prepared by cutting 1.5-m to 3.0-m samples of bamboo tube root, wiping off the bamboo green and tabasheer, and keeping the bamboo meat, which was then further split into bamboo sticks with an axial length of 1.5 to 2.0 cm, a radial length of 0.2 cm, and a tangential length of 0.2 cm. The sulfate process described by Chen (2011) was adopted to process the BF, resulting in IBFs with lengths of 2 to 4 mm and diameters of 8 to 22 μm.

Modification of BF

(1) Nano-$CaCO_3$ impregnation modification: At 25 °C, a certain content of BF was dissolved in distilled water for 30 min at 500 rpm through a magnetic stirring apparatus (ETS-D5 S25; IKA, Germany), then a varied amount of EDTA-2Na and nano-$CaCO_3$ were added and mixed for differing time periods. The process parameters for the nano-$CaCO_3$ impregnation are shown in Table 1.

(2) Nano-$CaCO_3$ *in situ* modification: At 25 °C, 0.5 g of BF was blended with 100 mL of a 0.3 mol/L $CaCl_2$ solution for 30 min at 500 rpm, then 0.5 g of EDTA-2Na and 0.03 mol of Na_2CO_3 solution, at a rate of 25 mL/min, were added and mixed for 25 min.

The suspensions were washed on a 200-mesh nylon net with distilled water and air dried. The modified bamboo fibers (MBFs) were obtained and preserved in a constant temperature and humidity box at 23 °C and 50% relative humidity (RH). An unmodified BF with 0.5 g of EDTA-2Na was used as the control.

Table 1. Process Parameters for Nano-$CaCO_3$ Impregnation

BF (g)	Distilled water Dosage (g)	$CaCO_3$ Dosage (g)	EDTA-2Na Dosage (g)	Impregnation Time (min)
2.0	800	0	2.0	25
2.0	800	2.0	2.0	25
2.0	800	4.0	2.0	25
2.0	800	6.0	2.0	25
2.0	800	8.0	2.0	25
2.0	800	12.0	2.0	25
2.0	800	2.0	0	25
2.0	800	2.0	0.5	25
2.0	800	2.0	0.68	25
2.0	800	2.0	1.0	25
2.0	800	8.0	2.0	10
2.0	800	8.0	2.0	40
2.0	800	8.0	2.0	60

Preparation of BF/PP composites

Circular BF papers with diameters of 100 mm, made from 0.2 g of MBF, were dried for 20 min at 103 °C in an oven until the moisture content was approximately 2%. A hot press (R-3221; Wuhan Qi'en Technology Co., Ltd., China) was used to form a sheet consisting of five BF papers with a mass of 1.0 g and six PP films with a mass of 2.0 g, layered in an alternating pattern; the sheet thickness was 1.0 mm, and three sheets were used for each specimen. The hot-pressing was conducted at a temperature of 180 °C, a pressure of 2 MPa, and a dwell time of 2 min.

Performance testing of BF

A trinocular microscope (LEICA DM LB2; Leica Microsystems, Germany) was used to test the length and diameter of the BF and to calculate the length-diameter (L-D) ratio. Three hundred testing samples were used. The contents of benzene-alcohol extractives, cellulose, holocellulose, and lignin were measured according to the standards GB/T 10741 (2008), GB/T 744 (1989), GB/T 2677.10 (1995), and GB/T 2677.8 (1994), respectively.

Mechanical properties

The tensile properties of IBF were measured using a high-precision short fiber mechanical properties tester (JSF08; Beijing YiKe Letter Technology Co., Ltd., China) to test the load and displacement of IBF; the tensile speed was set to 0.0008 mm/s, and the ball and socket system was used. The cross-sectional area of the IBF was measured by a laser scanning confocal microscope (CLSM; LSM 510 META; Zeiss, Oberkocken, Germany). The preparation of the sample and the testing process were carried out in accordance with the procedure described by Cao *et al.* (2010). Thirty effective samples were used for each group.

The tensile properties of the composites were measured according to the standard GB/T1040.3. (2006). Dumbbell-shaped specimens (type V), which were shorten to a half, were used for testing, and the tensile speed was set to 2 mm/min. Five samples were made from one sheet, and fifteen effective samples were used for each group.

Morphological observation

The instrument used for observing the surface morphology of BF and the BF/PP composites under various process parameters was an field emission environmental scanning electron microscope (SEM; XL30 ESEM-FEG; FEI Co., USA). A vacuum was pulled to less than 5×10^{-5} Pa in the sample chamber, while the scanning voltage ranged from 7 to 10 kV.

RESULTS AND DISCUSSION

Basic Properties of BF Produced by the Sulfate Process

As can be seen in Fig. 1, the surface of the BF had many small pores with a diameter of 0.7 to 1.5 μm, and folds formed on the surface of air-dry BF. This process may have occurred because the cell walls of the natural fiber have a mesoporous structure, and the sulfate process can remove the lignin and degrade parts of the hemicellulose through oxidation. These mesopores and folds could provide attachment sites for $CaCO_3$. The length, diameter, and L-D ratio of the BF made by the sulfate process were 1992.9 μm, 21.9 μm, and 93.7, respectively.

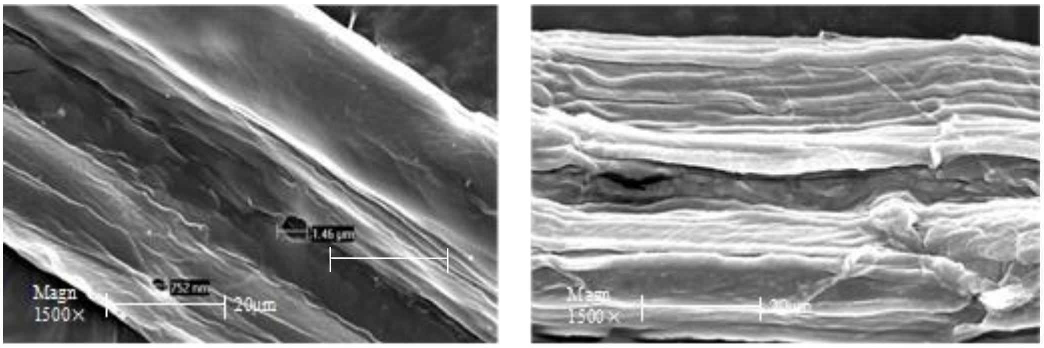

Fig. 1. Surface morphology of BF

Long-BF-reinforced composites have been shown to have superior mechanical properties compared with short-BF composites (Ochi 2014). Also, it is generally believed that, when the L-D ratio is less than 45, the value of pulping and papermaking is low (Pei *et al*. 2012). The number of winding times between fibers per unit area when forming the sheet increases, as does the strength of the resulting paper, as the L-D ratio increases. Therefore, the BF after the sulfate process could be used to make paper with a certain strength.

Table 2. Chemical Composition of BF

Benzyl Alcohol Extractives (%)	**Holocellulose** (%)	**Cellulose** (%)	**Hemicellulose** (%)	**Lignin** (%)
1.38	96.56	74.10	21.46	0.72

Table 2 shows that the lignin content of the BF was only 0.72%. The TS and EAB of the fiber cell wall increase with decreasing lignin content (Zhang 2011). The sulfate cooking liquor had a small effect on cellulose, with a content of 74.10%. Cellulose exists in the fiber cell walls in the state of a microfibril beam composed of orderly molecular chains and serves as the main provider of the cell walls' mechanical properties. However, the hydroxyl groups of the cellulose chain could have caused strong polarity on the surface of the BF, which would cause the interface compatibility of the two phases to weaken when the BF was combined with the thermoplastic polymer.

Tensile Properties of IBF and Its Composites under Various Modification Methods

As shown in Fig. 2, $CaCO_3$ particles were successfully deposited during the impregnation and *in situ* modification of BF, which was also reported in a previous study by the authors (Cheng *et al.* 2014a). This success was attributed to the ability of nano-$CaCO_3$ to fill the fissures or pores of the fibers and grow along with them (Bergert *et al.* 2005). The microporous and fold structures on the surface of the fiber cell walls could provide attachment locations for inorganic particles, whereas nano-$CaCO_3$ adhered to the cell walls could offer nucleation sites for initiating the crystalline formation of the semicrystalline PP matrix (Shi *et al*. 2011a). Nano-$CaCO_3$ with a particle size of 70 to 120 nm adhered to the surface of BF from the impregnation modification had the shape of an irregular tetrahedron, which dispersed very well without reunion. The amount of nano-$CaCO_3$ on the surface of BF from the *in situ* modification was noticeably greater, almost covering the whole fiber, but united the larger particles. Most of the nano-$CaCO_3$ particles were rice-shaped, and the particle size of $CaCO_3$, approximately 300 to 1280 nm, allowed it to distribute widely. Cementation of the nano-$CaCO_3$ appeared, especially in the concave parts of the fiber. This phenomenon might have occurred because the Ca^{2+} and CO_3^{2-} ions in the solution and the solid $CaCO_3$ were not in equilibrium. At the beginning of the reaction, Ca^{2+} would first combine with CO_3^{2-}, and $CaCO_3$ molecular clusters would start growing with a crystal nucleus until the concentration of the $CaCO_3$ molecular clusters in the solution reached a supersaturated state (Lin *et al.* 2001), which would lead to a gradual increase in the particle size of the $CaCO_3$ adhered to the surface of the BF.

Table 3 shows that the impregnation and *in situ* modifications had little impact on the cross-sectional area of the IBF, but it had a noticeable effect on tensile properties. Compared with the unmodified IBF, the breaking load, TS, MOE, and EAB increased. Some studies indicate that cell wall deficiencies, initially present in the fiber itself and generated by the preparation process, could affect the mechanical properties. The defective parts concentrated stress easily, which caused the deformation and rupture of the fiber (Mott *et al.* 1996). Modification could have allowed $CaCO_3$ to fill the defective parts such as pores and folds, improving the density of the fibers and also transmitting the load around the pores to the $CaCO_3$ particles. This load transfer would effectively prevent the fibers from fracturing because of defects, thus improving the tensile properties. The TS, MOE, and EAB of the unmodified IBF were 15.98%, 22.15%, and 5.21% lower than those of the IBF after impregnation modification, respectively, and showed a decrease of 18.00%, 26.19%, and 21.88%, respectively, compared to the IBF after *in situ* modification. The trend of TS correlated with the loading of $CaCO_3$. The specific surface area increased as the loading of inorganic nanoparticles increased, which would have a prominent effect on the properties of the BF (Xia *et al*. 2015).

Table 3. Mechanical Properties of IBF Produced by Various Modification Processes

Samples	Breaking Area (μm^2)	Breaking Load (mN)	TS (MPa)	MOE (GPa)	EAB (%)
Untreated	108.5 (16.77)	84.37 (11.27)	792.02 (208.02)	26.73 (3.20)	2.88 (0.18)
Impregnation Modification	105.13 (14.54)	95.12 (14.71)	918.62 (168.02)	32.65 (4.69)	3.03 (0.54)
In Situ Deposition	109.95 (17.68)	101.68 (19.54)	934.56 (162.27)	33.73 (5.11)	3.51 (0.77)
Note: The value in brackets is the standard deviation.					

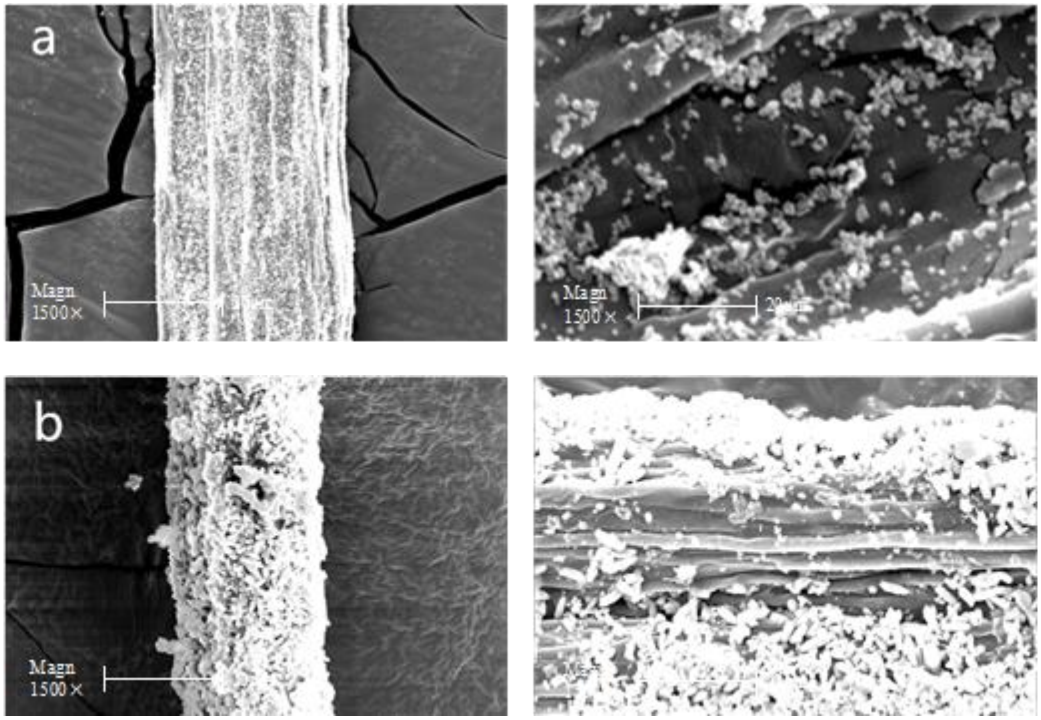

Fig. 2. Surface morphology of nano-$CaCO_3$-modified BF: (a) impregnation modification and (b) *in situ* modification

As shown in Table 4, the breaking load, TS, and MOE of unmodified BF/PP composites were 3.00%, 6.95%, and 15.80% lower than those of nano-$CaCO_3$ impregnation-modified BF/PP composites, respectively, and showed decreases of 5.28%, 18.68%, and 25.41%, respectively, compared to the nano-$CaCO_3$ *in situ*-modified BF/PP composites. These differences might have arisen because the $CaCO_3$-filled BF could develop compatibility between the BF and PP matrices and thus improve the TS of the composites (Cheng *et al.* 2014b). The fracture surfaces of the BF/PP composites produced by various modification treatments are shown in Fig. 3.

Table 4. Tensile Properties of BF/PP Composites Produced by Various Modification Processes

Samples	Breaking Load (mN)	TS (MPa)	MOE (GPa)
Untreated	85.97 (10.88)	33.75 (4.07)	0.98 (0.19)
Impregnation Modification	88.55 (6.95)	36.10 (2.51)	1.14 (0.18)
In-Situ Deposition	90.51 (10.98)	40.06 (4.84)	1.23 (0.15)
Note: The value in brackets is the standard deviation.			

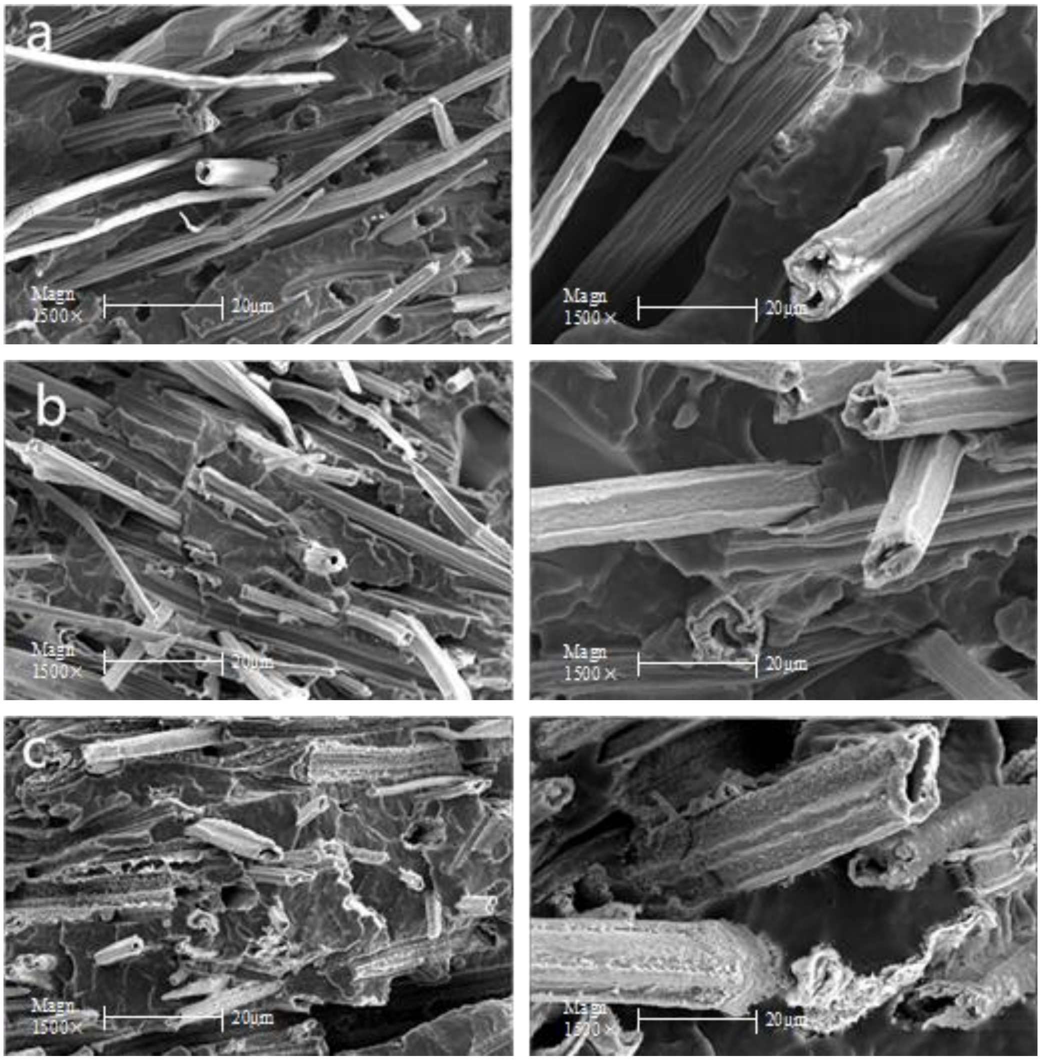

Fig. 3. Fracture surface of BF/PP composites after various pretreatments: (a) untreated, (b) impregnation modification, and (c) *in situ* modification

The surface of the unmodified BF was smooth. For the unmodified BF-filled composites, in addition to the crack propagation of PP (the main tensile failure form displayed in the slippage between BF and PP), a large gap existed at the junction of BF and PP. Fiber pullout was obviously shown at the fracture surface (Fig. 3a), which indicated that the interface adhesion between unmodified BF and PP was poor, further decreasing the mechanical properties. One probable reason for this result was that the main chemical components of BF are cellulose, hemicellulose, and lignin, which consist of hydrophilic groups such as hydroxyl and phenolic hydroxyl groups. The BF had a strong hydrophilicity and chemical polarity, while the thermoplastic polymer was a nonpolar and hydrophobic material. In addition, the voids and interfacial defects could have been caused by the mesoporous structure of BF in the processing of the composites, thus affecting the interfacial bonding strength. Figures 3b and 3c show that the interfacial adhesion between MBF and PP was strong, forming a dense interfacial bonding layer. Fiber pullout decreased at the junction of the composites, and the damage was mostly in the form of fiber breakage. The longitudinal TS of the IBF was far higher than that of the BF-reinforced thermoplastic polymer composites (Huang *et al*. 2009), so the breakage of the MBF needed more energy, which showed that the TS of the MBF/PP composites increased at a macroscopic level.

Tensile Properties Affected by Nano-$CaCO_3$ Treatment in the Impregnation Modification Process

$CaCO_3$ content

As Table 5 depicts, TS and MOE were increased as the nano-$CaCO_3$ dose rose from 0 to 8 g, achieving maximum performance (47.64 MPa and 2.85 GPa, respectively) at a $CaCO_3$-BF ratio of 4:1. Compared with the unmodified BF-reinforced PP composites, the TS and MOE values increased by 19.5% and 37.7%, respectively. This result might be due to the tendency of the $CaCO_3$ particles to fill the mesopores and grooves on the surface of fibers, decreasing the void defects in the process of forming the composites.

Table 5. Tensile Properties at Various Nano-$CaCO_3$ Contents

$CaCO_3$ (g)	**Breaking Load (mN)**	**TS (MPa)**	**MOE (GPa)**
0	155.73 (0.77)	39.88 (0.58)	2.07 (0.22)
2	175.41 (25.48)	40.87 (3.79)	2.31 (0.11)
4	168.39 (10.88)	41.88 (1.77)	2.33 (0.12)
6	183.18 (14.89)	44.72 (4.96)	2.47 (0.25)
8	187.85 (3.70)	47.64 (0.28)	2.85 (0.19)
12	198.85 (11.74)	45.29 (7.33)	2.73 (0.83)
Note: The value in brackets is the standard deviation.			

In addition, the surface effects of the nano-$CaCO_3$ helped the fibers and $CaCO_3$ particles to form strong chemical bonds or multiple hydrogen bonds, changed the surface orientation of the fibers, and decreased the hydrophilicity of the fibers, which all contributed to the increase in bonding strength. Moreover, nano-$CaCO_3$ particles could form hydrogen and covalent bonds by reacting with hydroxyl groups present at the interface between the fibers and PP (Zhou *et al*. 2009), which suggested improved interface performance. This increase in interfacial strength could effectively transfer the tensile stress from the matrix (PP) to the reinforcement phase (BF). Thus, modification treatment by nano-$CaCO_3$ improved the TS of composites. However, when nano-$CaCO_3$ content was greater than 8 g, the TS and MOE values of the composites began decreasing. This effect can be attributed to the uneven distribution of nano-$CaCO_3$ particles of larger size on the surface of the fibers and the production of air pockets between the MBF and PP, as shown in Fig. 4, weakening the interface compatibility.

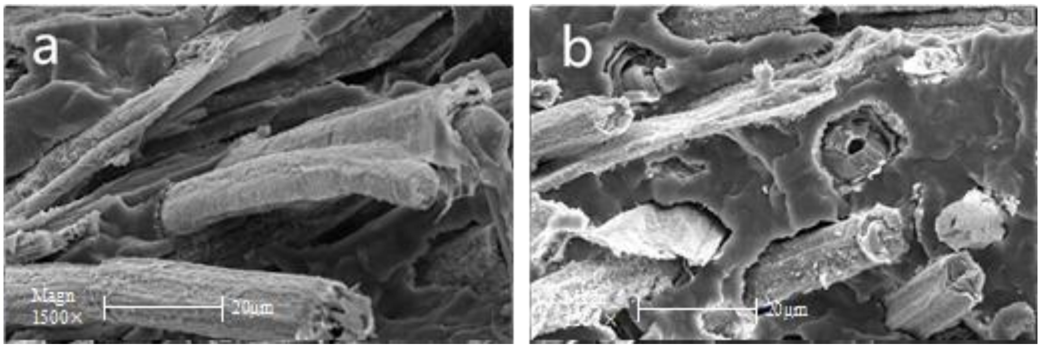

Fig. 4. Fracture morphology of composites at various $CaCO_3$ contents: (a) appropriate, and (b) excessive

EDTA-2Na content

Table 6 indicates that TS and MOE increased at EDTA-2Na contents ranging from 0 to 0.68 g, then decreased when the dose increased from 0.68 to 2 g, achieving maximum performance (47.20 MPa and 2.70 GPa, respectively) at an EDTA-2Na dosage of 0.68 g. Compared to the control group, the TS and MOE values increased by 37.8% and 20.0%, respectively. Along with the increase in EDTA-2Na, the particle size of nano-$CaCO_3$ adhered to the surface of the BF decreased, while specific surface area increased. Interfacial adhesion was enhanced because of the full adsorption of the EDTA-2Na and the resultant bonding achieved.

Table 6. Tensile Properties at Various EDTA-2Na Contents

EDTA-2Na (g)	Breaking Load (mN)	TS (MPa)	MOE (GPa)
0	151.21 (4.62)	34.26 (2.02)	2.25 (0.26)
0.5	196.27 (19.11)	46.23 (3.45)	2.28 (0.22)
0.68	195.86 (15.80)	47.20 (1.65)	2.70 (0.13)
1	170.12 (5.04)	42.72 (1.21)	2.35 (0.39)
2	175.41 (25.48)	40.87 (3.79)	2.31 (0.11)
Note: The value in brackets is the standard deviation.			

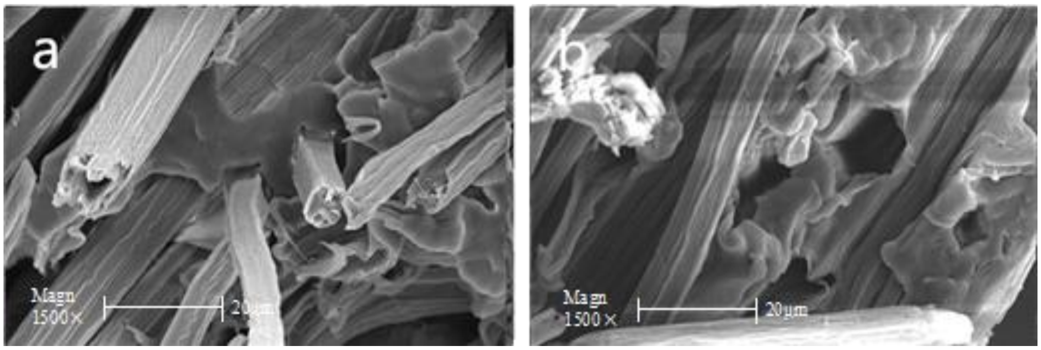

Fig. 5. Fracture morphology of composites at various EDTA-2Na contents: (a) appropriate, and (b) excessive

In addition, EDTA-2Na degraded the hemicellulose and lignin in the BF, which decreased the number of hydrophilic groups on the surface of the BF and hindered its hydrophilicity. Consequently, the interface was better developed and the mechanical properties of the composites increased. When the dosage of EDTA-2Na was excessive, the nano-$CaCO_3$ particles agglomerated, and the existence of large particles caused more void defects in the composites (Fig. 5), causing stresses to concentrate in the tensile direction and decreasing the tensile properties accordingly.

Impregnation time

As shown in Table 7, as impregnation time increased, the TS and MOE first increased and then decreased, achieving maximum performance (47.64 MPa and 2.85 GPa, respectively) at 25 min. More $CaCO_3$ adhered to the surface of the IBF with increasing impregnation time. However, an excessive amount of adhered $CaCO_3$ caused the particles to agglomerate, producing void defects at the interface. In the process of forming the composites, fracture primarily occurred because of fiber pullout and slippage between the BF and PP (Fig. 6), and the tensile properties therefore decreased.

Table 7. Tensile Properties at Various Impregnation Times

Impregnation Time (min)	Breaking Load (mN)	TS (MPa)	MOE (GPa)
10	160.24 (17.40)	41.90 (2.29)	2.25 (0.31)
25	187.85 (3.70)	47.64 (0.28)	2.85 (0.19)
40	204.71 (34.94)	47.46 (5.37)	2.41 (0.33)
60	187.72 (20.33)	39.97 (3.97)	2.16 (0.42)

Note: The value in brackets is the standard deviation.

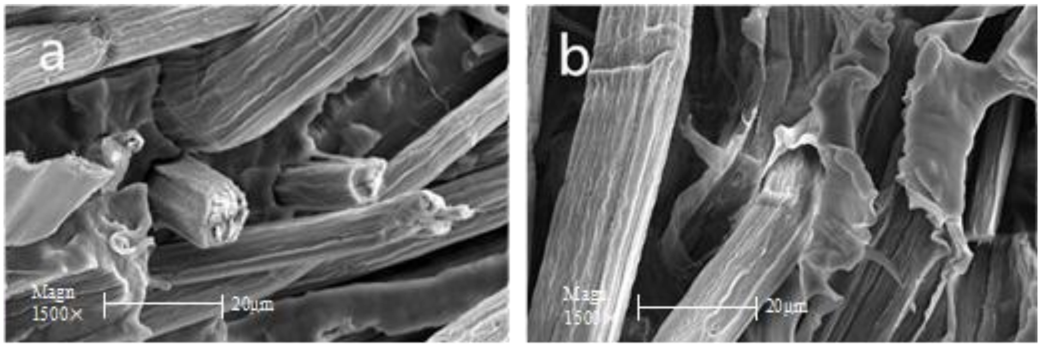

Fig. 6. Fracture morphology of composites at various impregnation times: (a) appropriate, and (b) excessive

CONCLUSIONS

1. Nano-$CaCO_3$ used in the impregnation modification of BF could make $CaCO_3$ particles attach to the surface of BF. This method played an active role in increasing the fiber density, filling the morphological voids and creases in the fiber, developing the interfacial compatibility of composites, and improving the tensile properties of the IBF and composites, which increased by 16.0% and 3.0% (TS), 22.2% and 7.0% (MOE), and 5.2% and 15.8% (EAB), respectively. Although the magnitude of these

increases was below that of *in situ* modified fiber, the nano-$CaCO_3$ impregnation modification method was easier to conduct and more noticeable.

2. Nano-$CaCO_3$ and BF had a synergistic effect when used to prepare bamboo-plastic composites. The level of nano-$CaCO_3$ treatment in the impregnation modification process had an obvious effect on the tensile properties of the composites. The TS and MOE first increased and then decreased with increases in $CaCO_3$ or EDTA-2Na dosage and impregnation time. The optimal nano-$CaCO_3$ conditions for the impregnation modification process of BF-reinforced PP composites were a temperature of 25 °C, a nano-$CaCO_3$ dosage of 1.0×10^{-2} g/mL, an EDTA-2Na dosage of 8.5×10^{-4} g/mL, and an impregnation time of 25 min.

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