Effect of Hot-Compressed Water Treatment of Bamboo Fiber on the Properties of Polypropylene/Bamboo Fiber Composite

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A water-soluble fraction of bamboo ranging from 9.2 to 23.5 wt% was obtained by hot-compressed water (HCW) treatment as the temperature increased from 140 to 180 °C. Both untreated and HCW-treated bamboo fibers (BF) were then compounded with polypropylene (PP), either with or without the addition of 5 phr maleic anhydride-grafted PP (MAPP), to net a total BF content of 50, 30, or 10%. It was found that both the HCW treatment and the MAPP addition effectively improved the tensile properties of the composite. Furthermore, the re-compounding of 70/30 and 90/10 composites from a 50/50 master batch proved to be more effective in improving the tensile properties than the direct compounding of PP and non- or HCW-treated BF to the same composition.

Keywords: Bamboo composite; Hot-compressed water; Tensile strength; Thermal property

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INTRODUCTION

Bamboo is one of the most abundant natural resources in many countries, particularly those located in Asia, a region that accounts for over 80% of the worldwide availability of bamboo (Han *et al*. 2008; Khalil *et al*. 2012). The growth rate of bamboo is typically quite rapid, with six to eight months required to reach mature size, which is only 5% of the time required for most timbers. Because of bamboo's excellent mechanical performance, good renewability, and economic efficiency, a large number of studies pertaining to the use of bamboo fiber (BF) as natural organic filler in polymer composites have been conducted (Mi *et al*. 1997; Chen *et al.* 1998; Okubo *et al.* 2004; 2005; Chattopadhyay *et al.* 2011; Ying *et al.* 2013). For example, Chattopadhyay *et al*. (2011) investigated the mechanical, thermal, and morphological properties of short BF-reinforced polypropylene (PP) composite with various loadings of chemically modified BFs. Chen *et al.* (1998) also reported the mechanical properties of BF-reinforced PP composite. In their research, maleic anhydride-grafted PP (MAPP) was used to enhance the adhesion between BF and PP matrix, resulting in the improvement of mechanical properties. Also, BF-reinforced PP composite showed lighter, water-resistant, and higher tensile properties, compared to commercial wood pulp-based board materials. Lee and Wang (2006) found that the use of lysine-based diisocyanate (LDI) as a bio-based coupling agent improves the tensile properties, water resistance, and interfacial adhesion of biodegradable poly-lactic acid (PLA) and poly-butylene succinate (PBS)-based

composites with BF. Ying *et al.* (2013) investigated the effect of heat treatment of BF-reinforced PP composite on the crystallinity of PP matrix and how much tensile property is improved. Heat treatment resulted in enhancing the crystallinity of the composite, but a longer treatment time decreased tensile strength.

Recently, a combination of hot-compressed water (HCW) treatment and mechanical grinding was used to obtain micro/nano-fibrillation of BFs for use in nanocomposites (Chang *et al*. 2012). In this research, the extraction of partial hemicellulose and lignin by HCW treatment was found to be effective in loosening the cell wall structure, thus leading to greater fibrillation by mechanical grinding and a more effective reinforcement of the polymer matrix. During HCW treatment, both the acetyl groups of hemicellulose and water itself are utilized as acids to dissolve the hemicellulose and lignin of lignocellulosic materials; however, the water-soluble fraction contains very little dissolved lignin when compared to the amount of hemicellulose (Mok and Antal 1992; Lee *et al*. 2010). This preferential extraction of hemicelluose has recently attracted the interest of wood-based material researchers, especially those involved in the wood plastic composite and board industries (Hosseinaei *et al*. 2011; 2012a,b). For instance, Hosseinaei *et al.* (2012a) reported the effects of HCW-extraction of hemicellulose from southern yellow pine at different temperatures on the properties of the resulting wood flour and on wood-plastic composites formed by combining this flour with PP in an extruder, either with or without a coupling agent. The thermal stability of the wood flour increased after extraction, and the tensile strength and water resistance of the composites improved. Pelaez-Samaniego *et al*. (2013) reported the effects of applying hot water extraction to wood fibers used in the production of wood plastic composites by monitoring the change in the chemical composition of ponderosa pine chips. Furthermore, they found that approximately 20% of the mass is reduced by hot water treatment and that the extracted product was mostly composed of the degradation products of hemicellulose. After extruding the treated product with high-density polyethylene (HDPE) and PP, it was discovered that the fibers treated with hot water had a reduction in the thickness swelling of the composite and the mechanical properties improved in both flexure and tension modes. Andrusyk *et al*. (2008) also reported that the aspect ratio of the wood flour was not changed after the extraction, and hence, the mechanical properties of the composite were enhanced.

Based on the premise that the extraction of hemicellulose under optimal conditions should generate substantial amounts of material while still leaving the woody substrate structurally intact for use in the fabrication of traditional board products, Sattler *et al*. (2008) investigated the effects of hot water extraction on the physical and chemical characteristics of wood flakes used for oriented strand board (OSB). Their results suggest that the composite manufacturers may find benefit in extracting hemicellulose from the raw materials used in board manufacturing.

Taib *et al*. (2012) investigated the effect of using hydrothermally treated kenaf on the flexural properties of biocomposites. In this research, high-density fiber boards were manufactured using kenaf fibers treated for 30 min in a laboratory autoclave at temperatures of 100, 130, and 150 °C. The result seen was that higher treatment temperatures increased the bending strength of the panel, with the use of kenaf fibers treated at 150 °C producing a sufficiently high MOE value, which suggests a potential use as a building material in applications requiring high elasticity.

The goal of this study is to improve the distribution and fibrillation of BFs during extrusion by selectively extracting hemicellulose through HCW treatment. The effects of

the HCW treatment and the addition of maleic anhydride-grafted PP (MAPP) on the properties of PP/BF composites will be then investigated. A comparison will be also made between the direct compounding of PP and BF and the re-compounding of a pre-compounded master batch composite.

EXPERIMENTAL

Materials

Moso bamboo (*Phyllostachys pubescens*) fiber (BF) from Hyogo Prefecture in Japan was prepared by cutter-milling until it could pass through a 3-mm sieve. Polypropylene (PP, Novatec® MA3) with an isotactic pentad fraction of 93.7% and containing 0.15 wt% of an antioxidant compound was purchased from the Japan Polypropylene Corporation; its molecular weight (Mw) and melt flow rate were rated as 260 kg/mol and 11 g/10 min, respectively. Maleic anhydride-grafted PP (MAPP, Umex1010), with a maleic acid content of 52 mg KOH/g, was purchased from Sanyo Chemical Industries, Ltd. (Kyoto, Japan).

Methods

Chemical composition analysis

The chemical composition of the BF was determined using the following methods. First, BF was extracted in a Soxhlet extractor with ethanol:toluene solution (1:2 in volume) for 6 h, and dried at 40 °C for 24 h in vacuumed drier. The holocellulose content was determined from a $NaClO_2$-delignified residue using the Wise method (Wise *et al*. 1946), in which a 0.5-g sample of BF was treated with 0.2 g of $NaClO_2$ in 30 mL of 7.5% acetic acid solution for 1 h at 80 °C; the process was repeated four times to ensure full delignification. The holocellulose obtained was then used to determine the α-cellulose content from the amount of insoluble residue in a 17.5% NaOH solution. The hemicellulose content was calculated by simply subtracting this α-cellulose content from the mass of holocellulose. The Klason lignin content of BF after removing extractives was determined as the amount of insoluble residue in a 72% sulfuric acid solution. Ash content was obtained by heating BF to 600 °C by following ASTM D1102-84. Starch quantification was conducted as follows; BF (0.1 g) was dispersed in 80% ethanol/waster solution (0.2 mL), and α-amylase (MOPS) buffer solution (3.0 mL) was added to the solution. The solution was incubated for 6 min. Sodium acetate buffer (4.0 mL, pH 4.5) and amylo-glucosidase (0.1 mL) were added and stirred at 50 °C for 30 min. After centrifugation at a speed of 3000 rpm for 30 min, glucose oxidase (GOPOD, Megazyme D-glucose, 3.0 mL) was added and incubated at 50 °C for 20 min. Glucose content from starch was quantified by measurement of the absorbance at 505 nm using the JASCO UV/VIS spectrophotometer (Model UV 510).

Hot-compressed water treatment

Samples were treated at temperatures ranging from 140 to 180 °C for 30 min (not including a period of warm-up) following the method described in our previous study (Chang *et al.* 2012). The initial pressure was 1 MPa, and the ratio of water to starting material was adjusted to 5:1. After purging with nitrogen gas, the reactor was placed inside a mantle heater and heated to the desired temperature at a rate of 10 °C/min. Following treatment, the reactor was immediately soaked in cold water; then, the treated

product was filtered to remove the water-soluble fraction. The obtained water-soluble fraction was oven-dried at 100 °C to reach constant weight and calculated in the basis of initial BF weight.

Composite preparation

The water-insoluble residue remaining after HCW treatment was extruded at 180 °C using a twin-screw extruder (2D15W; Toyo Seiki, Tokyo, Japan), with the screws co-rotating at a speed of 45 rpm. Composites with PP/BF compositions of 50/50, 70/30, and 90/10 were prepared, both with and without MAPP, from HCW-treated BF and raw BF. A re-compounding process was also applied in the preparation of 70/30 and 90/10 composites, wherein a 50/50 master compound was first prepared by mixing PP and BF and was then re-compounded with additional neat PP to give the desired composition. The extruded samples were compression molded into sheets for tensile testing and other measurements by applying 15 MPa of pressure at 180 °C for 2 min and then quenched by cold pressing at 5 MPa at room temperature.

Tensile test

Ten dog-bone-shaped samples (5 mm × 0.4 mm × 50 mm) cut from the compression-molded sheets were tensile tested using an Autograph AG-1 (5kN) (Shimadzu Co., Kyoto, Japan) at a crosshead speed of 10 mm/min. The morphology of the fracture surface produced by testing was observed by scanning electron microscopy (SEM, S-4800, Hitachi Co. Tokyo, Japan) at accelerating voltages from 5.0 to 10.0 kV.

RESULTS AND DISCUSSION

As can be seen in Table 1, the chemical composition of the BF used in this study was primarily carbohydrates such as cellulose (40.1 wt%), hemicellulose (32.9 wt%), and starch (7.6 wt%). It is already well known that HCW treatment at 140 to 180 °C allows for the selective removal of sugars, especially hemicellulose, from lignocellulosic materials (Sakaki *et al.* 1996; Amidon *et al.* 2008; Pelaez-Samaniego *et al*. 2012). HCW treatment can also readily degrade starch (Wells and Payne 1980). The water-soluble fraction obtained by HCW is relatively low (9.2 wt%) at 140 °C, but increases drastically to 23.3 wt% at 170 °C and remains fairly constant at 180 °C (Table 2). However, considering that the initial concentrations of hemicellulose and starch were 32.9 and 7.6 wt%, respectively (Table 1), the amount extracted at 170 and 180 °C was still smaller than expected if the carbohydrates were fully removed. Gonzalez *et al*. (2011) have also conducted HCW treatment of bamboo (produced in North-West of Spain) at 180 to 230 °C and utilized the residual fibrous product as the reinforcement for polylactic acid-based biocomposite. According to their chemical composition analysis, xylan and arabinan are the main hemicellulosic components, and their contents in raw bamboo were found to be 20.8 and 1.5 wt% in the basis of oven-dried weight, respectively. Klason lignin was 30.9 wt%. Furthermore, they have also analyzed water-soluble fraction and found that it mainly contains hemicellulose-derived oligo- and mono-saccharides. Under selected condition, 62.6% of the initial hemicelluloses in the raw material were converted into oligosaccharides (Gonzalez *et al*. 2011). These results are well matched with our results, even though initial hemicellulose content is smaller than that of the bamboo used in the present study.

Table 1. Chemical Composition of BF

Components	α-Cellulose	Hemicellulose	Klason lignin	Ash	Starch	Extractives
Composition	40.1	32.9	26.6	1.2	7.6	3.1
All values represent percentages of the original weight (wt%) of the BF						

Table 2. Water-soluble Fraction Obtained by HCW Treatment at 140 to 180 °C

HCW temperature (°C)	Water-soluble fraction (wt%)
140	9.2
150	15.4
160	17.6
170	23.5
180	23.9

Figure 1 shows the effect of varying the temperature at which the BF was treated by HCW on the tensile strength, elastic modulus, and elongation at break of PP/non- and HCW-treated BF (50/50) composites. Note that both the tensile strength and elastic modulus of the composite reinforced by HCW-treated BF were notably higher than those of the composite reinforced by non-treated BF, yet there was little difference between the HCW temperatures. A small improvement was evident in the elongation at break by HCW treatment, but all values were less than 4%. Furthermore, even though the water soluble fraction at 140 °C was only 9.2 wt%, the tensile properties of the resulting composite were nevertheless comparable to those produced using BF treated at higher temperatures. This confirms that even just a small amount of hemicellulose removal was effective in improving the tensile properties of a composite. This finding is in agreement with the findings of Hosseinaei *et al*. (2012), who found that the effective extraction of hemicellulose components from southern yellow pine flour through HCW treatment improves the tensile properties of PP composites in which it is used. This improvement was attributed to a decrease in the total surface energy of the wood filler and an increase in its hydrophobic character, both of which improve the compatibility between it and the polymer matrix (Hosseinaei *et al*. 2012a,b).

In Fig. 2, the effects of HCW treatment and BF content on the tensile strength and elastic modulus of the composites can be seen. Two different compounding methods, *i.e.*, direct compounding of PP and BF and re-compounding of a pre-compounded PP/BF (50/50) master composite with neat PP, were compared. It is evident from this that with increasing BF, the tensile strength and elastic modulus of both composites with non- and HCW-treated BFs decreased and increased, respectively. However, those composites with HCW-treated BF display a higher tensile strength and modulus than comparable non-treated BF composites. As discussed above and described by Hosseinaei *et al*. (2012a,b), this improvement is believed to be a result of BF having a lower polarity after HCW treatment, which in turn results in better compatibility with the PP matrix. Generally speaking, an increase in compatibility can improve filler dispersion. The improved fibrillation produced during extrusion due to the loosened cell wall structure of the HCW-treated BF could provide another possible reason for its enhanced dispersion in the PP matrix. This notion is based on the results of a previous study, in which the authors reported that the cell wall structure of wood and BF can be effectively loosened by HCW treatment, thereby resulting in an improvement in fibrillation by mechanical shearing (Lee *et al*. 2010; Chang *et al*. 2012).

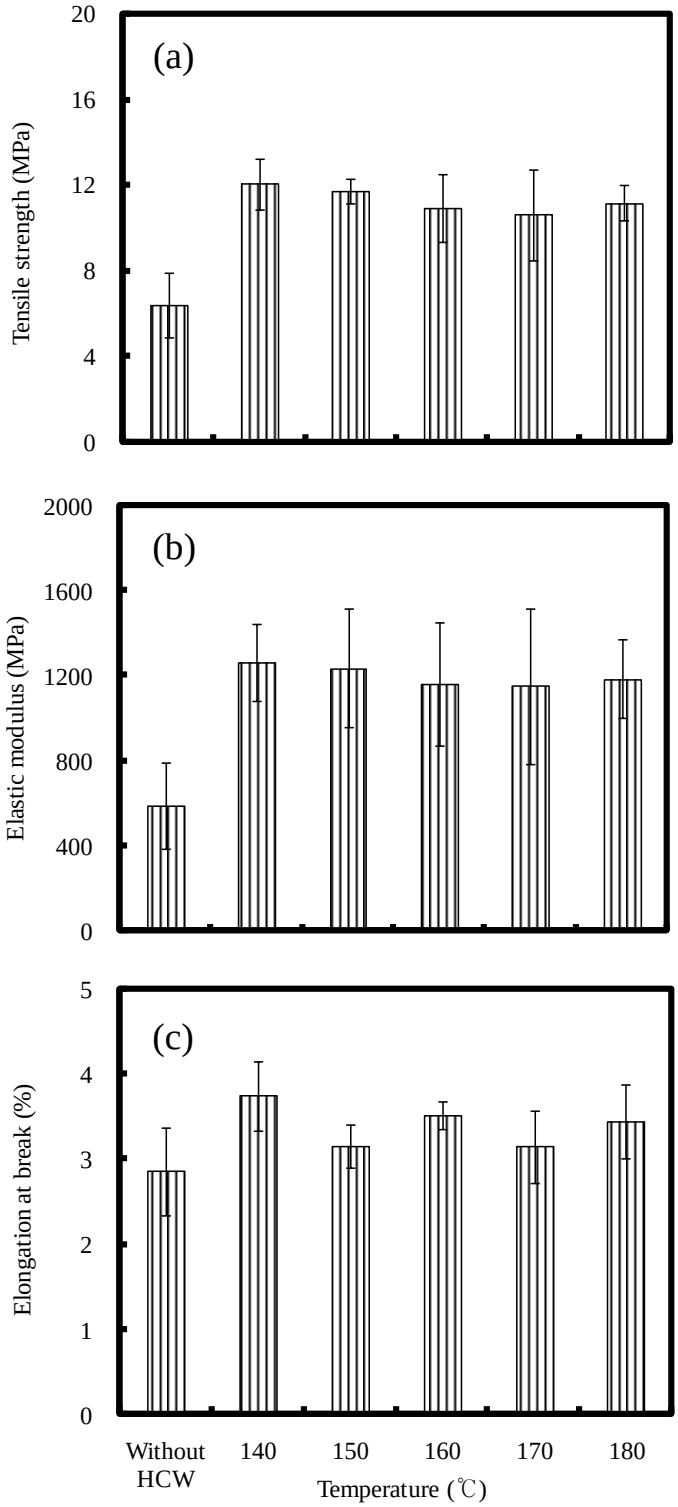

Fig. 1. Effect of varying the temperature at which BF is treated by HCW on the tensile strength (a), elastic modulus (b), and elongation at break (c) of a PP/BF (50/50) composite. Data provided as the mean ± standard deviation

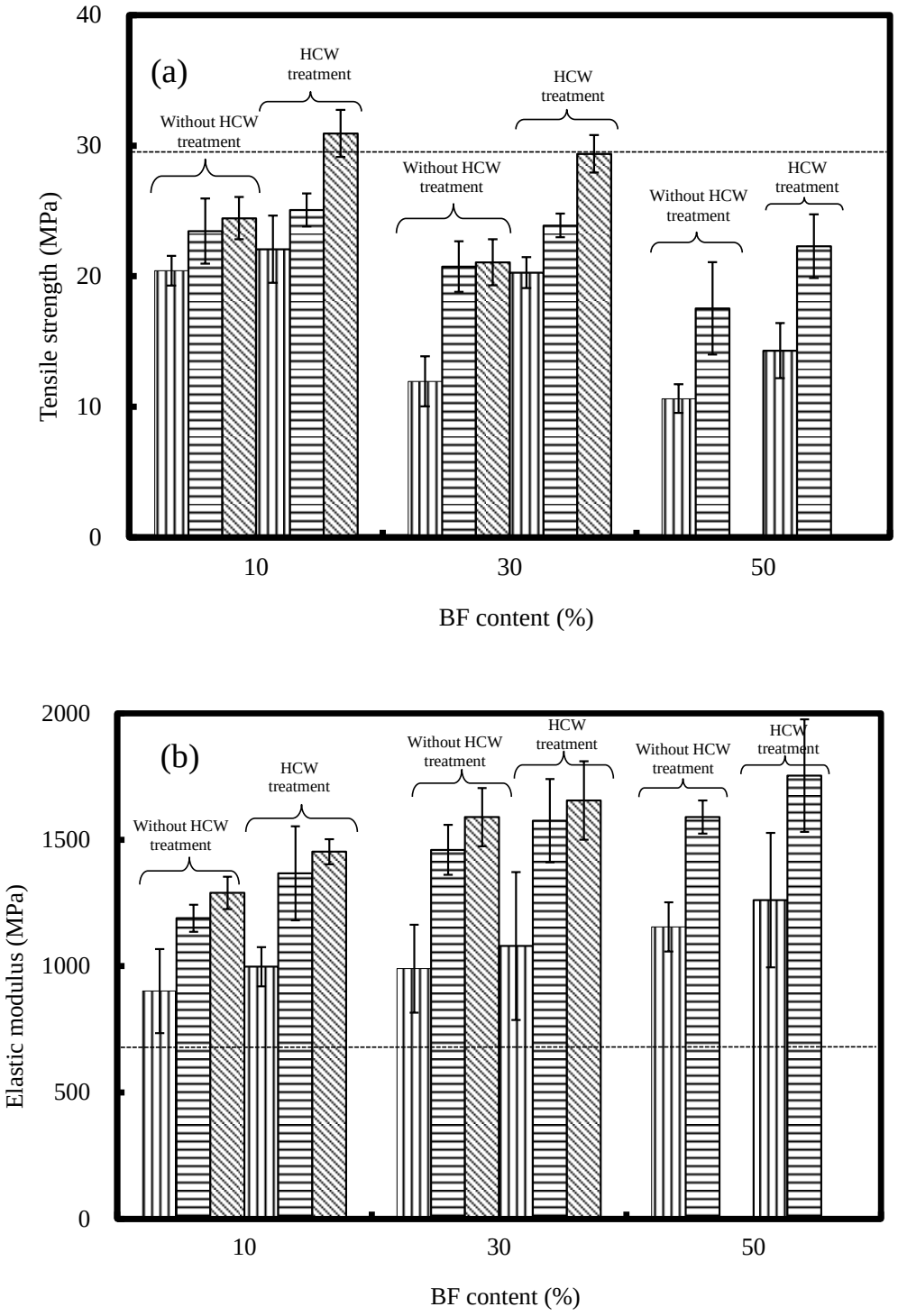

Fig. 2. Effect of HCW treatment at 170 °C and BF content on the tensile strength (a) and elastic modulus (b) of composites prepared by two different compounding processes: direct compounding (▥) without MAPP or (▥) with MAPP of 5 phr, and re-compounding (▧) with MAPP of 5 phr, of a PP/BF (50/50) master batch compound (without MAPP) with additional neat PP into 70/30 and 90/10 composites. Dash line in figure is tensile strength (29.8±1.2 MPa) of neat PP. Data provided as the mean ± standard deviation

A comparison of the two compounding methods reveals that the 90/10 and 70/30 composites prepared by re-compounding a 50/50 master composite with PP exhibited a higher tensile strength and elastic modulus than comparable composites produced by direct compounding. Moreover, the tensile strength and modulus of those composites reinforced by HCW-treated BF were higher than those with non-treated BF. As the second re-compounding induces more shearing force during extrusion, it was expected to improve the dispersion of filler in both composites; however, its greater effectiveness in the case of HCW-treated BF can be explained by the aforementioned fact that the loosened cell wall structure produced by HCW is more readily fibrillated and dispersed in a PP matrix.

The addition of MAPP was also found to improve the tensile strength and elastic modulus in both composites, which was attributed to the enhanced interfacial adhesion. It is generally known that MAPP can enhance interfacial bonding between a natural fiber filler and PP matrix due to ester linkages generated between the hydroxyl groups of the former and anhydride groups of the latter (Felix and Gatenholm 1991; Kazayawoko *et al*. 1999; Okubo *et al*. 2005; Chang *et al*. 2012). Greater dispersion and enhanced interfacial adhesion will therefore clearly contribute to improving the mechanical properties of a composite through better stress transfer from the matrix to the filler. Figure 3 clearly shows the effect of MAPP content on the tensile strength and modulus of PP/HCW-treated BF (70/30) composites prepared by re-compounding from a 50/50 master composite. It is evident that 3 phr (parts per hundred) of MAPP addition is sufficient to notably improve the tensile properties of the composite.

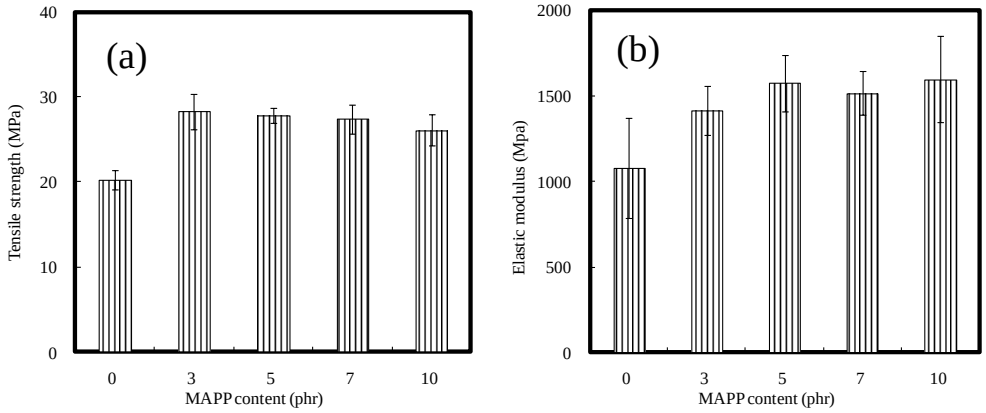

Fig. 3. Effect of MAPP content on the tensile strength (a) and elastic modulus (b) of a PP/HCW-treated (170 °C) BF (70/30) composite prepared from a master batch compound (PP/BF = 50/50). Data provided as the mean ± standard deviation

The SEM images in Fig. 4 show the fractured surface of the composites after tensile testing, with the morphology of this surface providing a good indicator of the filler dispersion and interfacial adhesion between the filler and polymer matrix. It is clearly evident that some voids were present between the filler and matrix in both the PP/non- and HCW-treated BF (50/50) composite without MAPP (Figs. 4a and 4c), suggesting that the interfacial adhesion in these was very poor; this is a characteristic feature of incompatible composite materials. Moreover, a greater number of fine, fibrillated fibers were seen in the PP/HCW-treated BF composite (arrows in Fig. 4c and 4d), but aggregated fiber bundles were seen in the PP/non-treated BF composite (Figs. 4a and 4b).

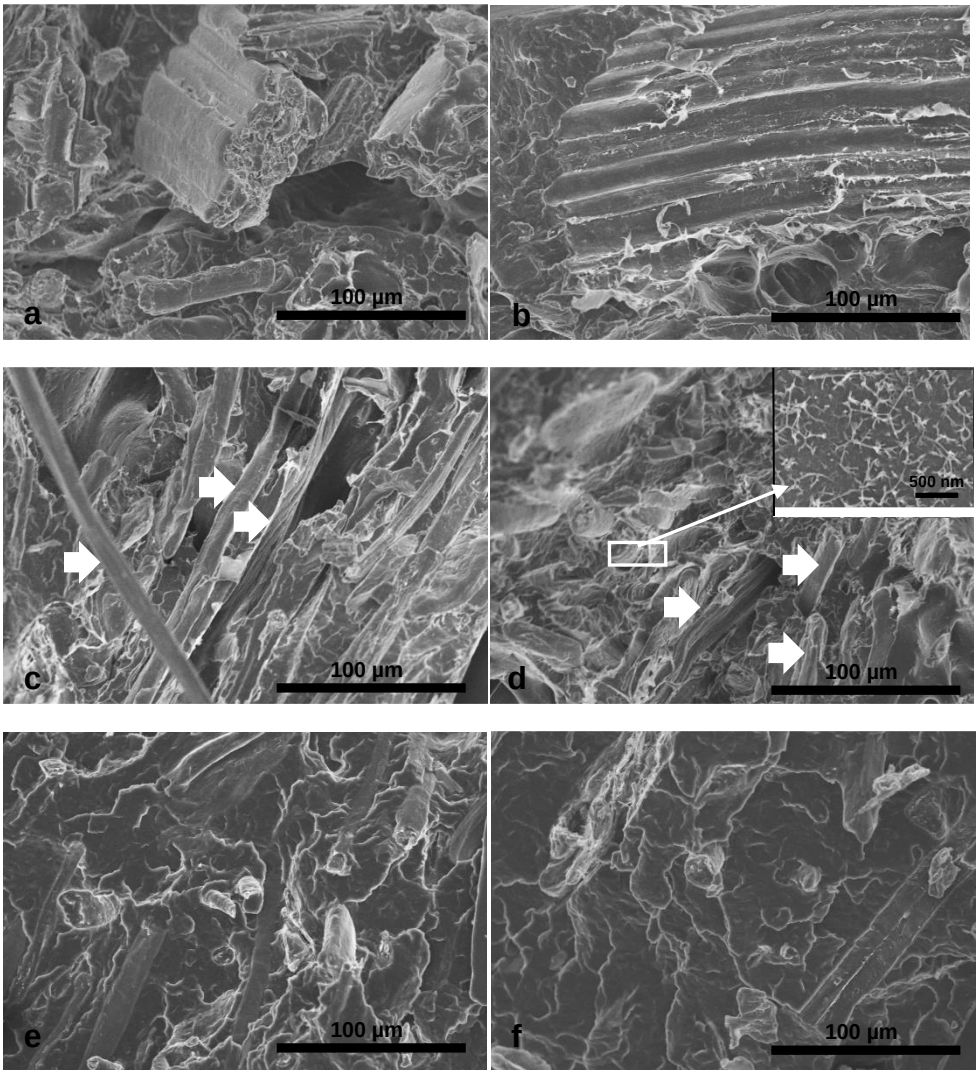

Fig. 4. Morphology of fracture surfaces produced by tensile testing composites of: (a) PP/BF (50/50); (b) PP/BF (50/50) with 5 phr MAPP; (c) PP/HCW-treated BF (50/50); (d) PP/HCW-treated BF (50/50) with 5% MAPP; (e) PP/HCW-treated BF (70/30) with 5 phr MAPP; and (f) PP/HCW-treated BF (90/10) with 5 phr MAPP. Note that both (e) and (f) are samples that were prepared by re-compounding from a 50/50 master batch composite

As discussed earlier, the observed phenomenon may be due to the weakened BF structure produced by HCW treatment, which improves fibrillation during extrusion and should result in improved dispersion of the filler in the matrix. On the other hand, MAPP addition clearly improved the interfacial adhesion, as shown in Figs. 4b and 4d. Interestingly, the magnified image in Fig. 4d shows that the "fuzzy" morphology of the BF surface was produced at a nanoscopic scale. This suggests that the strong interaction between MAPP and BF pulled cellulose microfibrils out of the HCW-treated BF surface during mechanical fracture. Conversely, in the PP/HCW-treated BF (70/30 and 90/10) composites with MAPP that were re-compounded from a 50/50 composite (Figs. 4d and 4e), the majority of the BFs remained embedded in the PP matrix. These morphological features are in full agreement with the observed improvement in the tensile properties.

CONCLUSIONS

1. Hot-compressed water treatment at temperatures of 140 to 180 °C were found to extract from bamboo fiber (BF) a water soluble fraction of 9.2 to 23.9 wt%.
2. When BF residue is used to fabricate polypropylene composite, the tensile strength and modulus of elasticity improved over comparable composites using untreated BF. These same properties were also found to be improved by the addition of maleic anhydride-grafted PP.
3. A comparison of two different preparation methods identified that re-compounding from a master composite using neat PP was more effective in improving the tensile properties of a PP/BF composite than direct compounding.
4. This study provides a novel idea to utilize the hemicellulose components extracted from bamboo by HCW treatment for biorefinery industry and to apply the residual product for composite materials.

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