Mechanical and Thermal Properties of Bamboo Pulp Fiber Reinforced Polyethylene Composites

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The purpose of this study was to investigate the mechanical and thermal properties of high-density polyethylene (HDPE) composites reinforced by bamboo pulp fibers (BPF). Using a twin-screw extruder, polymer composites were fabricated using BPF and bamboo flour (BF) as the reinforcement and HDPE as the matrix. Tensile and flexural tests of the HDPE composites were performed to determine the mechanical properties under different conditions. The thermal properties of HDPE composites were characterized by thermogravimetric analysis (TGA) and dynamic mechanical analysis (DMA). The results showed that BPF improved the mechanical and thermal properties of the polymer composites more than did BF. The tensile and flexural strength of composites with 30 wt% BPF were increased by 61.46% and 22.94%, respectively, while the tensile and flexural modulus were increased by 84.52% and 27.30%, respectively. Compared to composites with 50 wt% BF, the T_5% of composites with 50 wt% BPF increased by 20.18 °C. As the BPF content increased, the storage modulus (*E'*) and loss modulus (*E"*) initially increased, followed by a decrease. Compared to the BF/HDPE composites, BPF/HDPE composites reinforced at 30 wt% had a higher storage modulus (*E'*) and loss modulus (*E"*) and lower damping parameter (tanδ).

Keywords: Bamboo pulp fibers (BPF); Bamboo flour (BF); High density polyethylene (HDPE); Composites; Mechanical properties; Thermal properties

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INTRODUCTION

Because of concerns for the environment, the depletion of fossil fuels, and climate change, there has been a great interest in the replacement of synthetic fibers such as aramid and glass fibers in polymeric composites by natural plant fibers such as jute, coir, flax, bamboo, and wood fibers (Chattopadhyay *et al.* 2011). Natural fibers offer economical, functional, and environmental advantages and are gaining popularity, particularly in automotive, aircraft, and structural construction applications (Bao *et al.* 2011; Das and Chakraborty 2008). The promising benefits of natural fiber-reinforced polymer composites can be attributed to their low density, good thermal insulation, mechanical properties, low price, durability, sustainability, and biodegradability (Dominkovics *et al.* 2007; Sain 2005).

Among the natural plant fillers, bamboo flour (BF) is the most important one because of its small particle size and good dispersion (Han and Cheng 2010; Kim *et al.* 2008). Because of its small dimensions and incomplete structure, it is difficult to give full play to the advantages of the excellent properties of bamboo fibers as a filler to reinforce composites (Wang *et al.* 2011; Yu *et al.* 2011). The potential for bamboo fibers as natural reinforcement in polymer composites (Chen *et al.* 2009; Deshpande *et al.* 2000) has attracted considerable attention as an alternative to wood fibers due to their mechanical

properties, which are comparable to wood while growing to maturity in only 6 to 8 months. Bamboo fiber composites with different polymers have been reported, including polypropylene (Chen *et al.* 1998; Mi*etal.*1997), poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) (Jiang *et al.* 2008), and polylactide (Lee and Wang 2006).

Bamboo pulp fibers (BPF) have been used for papermaking as a substitute for wood pulp fibers because of their long fiber length and high length-to-diameter ratio; recently, they have been used in cellulose-reinforced concrete decorative panels (Guo 2005; Zhang 2009). Jiang *et al.* (2010) prepared BPF/PHBV composites by melt compounding and injection molding; the crystallization ability, tensile strength and modulus, flexural strength and modulus, and impact strength were found to be significantly increased by the addition of BPF. Awal *et al.* (2010) studied the thermal properties and spectral characterization of wood pulp reinforced bio-composite fibers and found the thermal stability of bio-composite fibers to be higher than that of pure wood pulp.

The long length and small diameter of bamboo pulp fibers, however, leads to less uniform dispersion in the matrix (Betterman *et al*. 1995); thus, studies of BPF-reinforced polymers are rare. In addition, a comparison of composites reinforced by BF and BPF has not been performed, and relevant experimental data are difficult to find. In this study, the mechanical and thermal properties of HDPE composites reinforced with BF and BPF at different contents were determined, and the differences in BF and BPF as reinforcement phase were investigated. The objective of this study was to optimize the proportion of BPF by evaluating the property improvement of the composites and to evaluate the possibility of BPF as reinforcement material in the composites.

MATERIALS AND METHODS

Materials

High-density polyethylene (DGDK-3364) with a density of 0.945 g/cm^3, a melt flow index (MFI) of 0.075 g/min (190 °C, 2.16 kg), and a tensile strength of 22.1 MPa was supplied by Zhang Mu Tou Plastic Co. Ltd. (Guangzhou, China). Maleic anhydride, in the form of maleated polyethylene (MAPE) coupling agent, was also supplied by Zhang Mu Tou Plastic Co. Ltd. The lubricating agent, polyethylene wax, was supplied by Yi-li Chemical Reagent Co. (Beijing, China). The BF (60-mesh) and BPF used as fillers in the composites were collected from the Gui Zhou Chi Tian Hua Paper Co. Ltd. (China).

Methods

Composite fabrication

BPF and BF were dried for 10 h at 103 °C in an oven until the moisture content (mass fraction) was less than 2%. A certain amount of BPF was added to a mixing machine (SHR-10A; China) with polyethylene wax, HDPE, and MAPE and mixed for 1 h at 1800 rpm/min. The composite was made by a two-step process. First, the mixtures which contained BPF, polyethylene wax, HDPE, and MAPE were placed in an extruder (SJZ45/90-YF110;China), subjected to melt mixing in the extruder, and then passed through the die of the extruder to form a composite. The composite was crushed with a crushing machine (ZJ300; China), and composite particles were obtained. Second, the particles were placed in the extruder (SJZ45/90-YF110) again, and subjected to melting and cooling to obtain the final composite materials. The different zones during composite preparation were 160, 165, 175, and 175 ºC at both the stages. The composition of the seven different composites are shown in Table 1.

Table 1. Composite Components

Material NO.	Components（%）				
	BPF	BF	HDPE	MAPE	Polyethylene wax
1	5	0	90	4	1
2	10	0	85	4	1
3	20	0	75	4	1
4	30	0	65	4	1
5	50	0	45	4	1
6	0	50	45	4	1
7	0	0	95	4	1

BPF characterization

The length, diameter, and aspect ratio of BPFs (200 fibers) were measured by use of an FC300FX optical microscope (Leica Microsystems, Germany). Tensile strength testing of an individual BPF was performed using a small commercial high resolution mechanical tester (Instron Microtester 5848, USA) according to the Yu's test (Yu *et al*. 2011). The capacity of the load cell was 5 N, with a gauge length of 10 mm and a crosshead speed of 0.048 mm/min. Tensile strength testing was carried out at 23 °C and 30% relative humidity (RH). The tensile strength and tensile modulus of bamboo pulp fibers were then calculated.

Mechanical properties of the composites

All samples originated from the extrusion sheet and were conditioned following ASTM D618 (2008) at 23 °C and 30% relative humidity (RH) for 88 h.

Flexural tests were conducted according to ASTM D790 (2010) on the Instron Microtester 5848 equipped with a load cell of maximum capacity 2 kN, at a crosshead speed of 17 mm/min. The samples were sawn from the composites sheets with 160×14×8 mm. Six specimens from each composite were tested.

Tensile tests were carried out according to the ASTM D638 (2010) specification on dumbbell-shaped specimens, type II. The tests were performed using the Instron 5800 device at a crosshead speed of 5 mm/min, with the strain measured in the mid-span of each specimen with an Instron extensometer (Dynamic Extensometer, 2620-601; USA). The tensile strength and modulus were calculated by the stress-strain curves. Five replicates from each sample were tested.

Thermal properties of the composites

Thermogravimetric analysis (TGA) was performed using a Q100 analyzer (TA Instruments; USA) with a heating rate of 10 °C/min. The samples were heated to 600 °C to determine the thermal degradation of BPF, HDPE, as wells as BPF/HDPE and BF/HDPE composites. Four replicates for each sample were tested. All tests were carried out in a nitrogen atmosphere using a flow rate of 50 mL/min.

The TA Instruments Q800 Dynamic Mechanical Analyzer (DMA) is a thermal analytical instrument used to test the mechanical properties of many different materials. It operates over a temperature range of –145 °C to 600 °C, using heating rates up to 20 °C/min. Changes in sample properties are determined, resulting from changes in seven experimental variables: temperature, time, frequency, stress, force, displacement, and strain. The device employs interchangeable clamps allowing the operator to measure many

properties, including: modulus, damping, creep, stress relaxation, glass transitions, softening points, temperature, time, frequency, stress, force, displacement, and strain. The DMA instrument characteristics were as follows:

Temperature Range –145 to 600 °C
Sample Length 50 mm (2 in.) maximum
Sample Width 15 mm (0.6 in) maximum
Sample Thickness up 5 to 10 mm (depending on clamp)
Displacement Range 25 mm (1.0 in.)
Loading 0.001 to 18 N
Atmosphere Controlled flow with inert gases or air.

The storage modulus, loss modulus, and damping parameter (*tan*δ) of HDPE as well as BPF/HDPE and BF/HDPE composites were determined using a Q800 analyzer (TA Instruments) with samples shaped into a strip of 60 mm×14 mm×3.2 mm. Before the analysis, the linear viscoelastic regions of the specimens were determined using a strain sweep test with the same instrument. The specimens were vibrated with a dual-cantilever fixture at a frequency of 1 Hz and heated from 35 to 120 °C at a heating rate of 1.5 °C/min with a strain amplitude of 30 μm.

RESULTS AND DISCUSSION

BPF Characterization

The fiber length affected the ultimate mechanical properties. The mean length, diameter, and ratio of the initial BPF were 1146.61 μm, 17.49 μm, and 63.1, respectively.

Table 2. Tensile Properties of BPF

	Area	Breaking load	Tensile strength	Tensile modulus	Elongation
BPF	μm^2	mN	MPa	GPa	%
	183.51(16.77)	84.96(11.27)	508.49(208.02)	6.73(3.20)	7.44(0.18)

Values in parentheses are the standard deviation for 30 BPFs

As shown in Table 2, the strength of BPF was 508.49 MPa, with an average elastic modulus and elongation of 6.73 GPa and 7.44%, respectively, which were lower than that previously reported for bamboo fiber (Cao 2010). This may be caused by the contents of cellulose, hemicelluloses, and lignin, the main constituents of bamboo cell walls, which provide the specific mechanical properties of the cell wall and ultimately affect the properties of the fiber and the composites (Zhang 2011).

Mechanical Properties of Composites

Figure 1 shows that as the BPF content was increased, both the flexural and tensile strengths of the composites increased initially, and then they decreased. The addition of BPF resulted in an overall improvement in the mechanical properties of the composites. The tensile strength and flexural strength of composites including 30 wt% BPF were 60% and 110% higher than those of HDPE, respectively, and also increased by 61.46% and 22.94% compared to BF/HDPE composites. On the other hand, the tensile modulus increased with increasing BPF content, while the values of flexural modulus reached a maximum at 30 wt% BPF content.

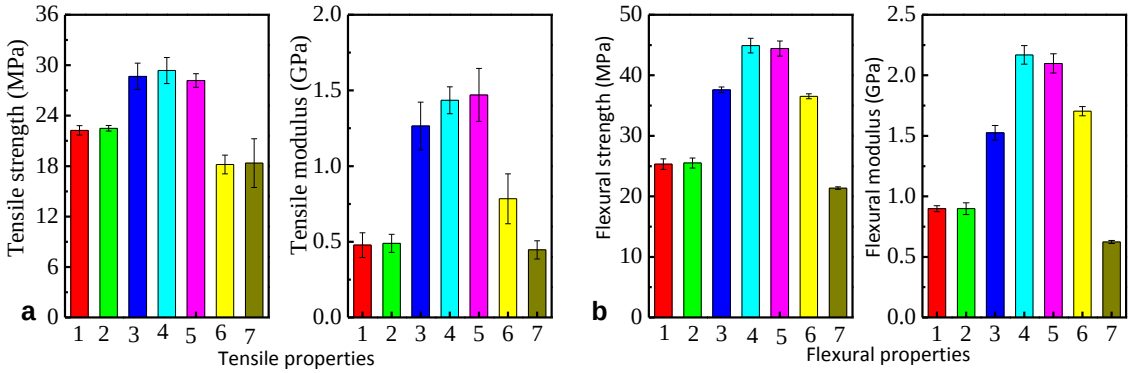

Fig. 1. Mechanical properties of HDPE and BPF/HDPE and BF/HDPE composites: (a) tensile properties and (b) flexural properties

The tensile modulus and flexural modulus of BPF/HDPE composites at 30 wt% were 221.27% and 586.79% higher than those of HDPE, respectively, and showed an increase of 84.52% and 27.30% compared to the BF/HDPE composites, respectively. while when the BPF contents went up to 50%, the tensile and flexural properties were decreased. That might be because an excess of bamboo pulp fibers leads to the agglomeration tendency of the filler, so that the contact area between fiber and matrix is reduced and some holes are present in the composites. So, the matrix cannot coat the fiber completely, which results in poor interfacial adhesion between the HDPE and the BPF. Besides, the polar material of BPF increased while the non-polar material of HDPE decreased. Therefore, only a small amount of stress could be transferred from the matrix to the filler. Thus, the mechanical properties had a tendency to decrease with a higher mass fraction (Mi *et al.* 1999). Similar results were published by Yam *et al.* (1990) and Raj and Kokta (1989). The mechanical properties of BF/HDPE composites were worse than those of the BPF/HDPE composites with 30 wt% BPF, which may be attributed to the ball-bearing effect of the BF particles with 250 μm. In other words, the BF particles facilitated the slippage of HDPE chains and enabled the molecular conformational changes, thus reducing the stiffness of the composites (Kamini 2013). The results in this study suggest that the mechanical properties of composites reinforced by BPF were superior to that reinforced by BF, probably because of the large aspect ratio and small diameter of BPF as a result of its chemical treatment in the paper making process. Fibers with small diameters have a larger specific surface area at the same fiber weight ratio (Jiang *et al.* 2008). Therefore, the smaller diameter of the BPF led to a larger interfacial area between the HDPE and the fibers, which increased the reinforcing effect.

Thermal Properties of Composites

It is well known that the degradation of natural fibers and the polymer plays an important role in the performance of a composite. The thermal degradation of fibers leads to poor mechanical performance and deterioration of color. Moreover, the surface chemistry changes of fibers may affect the interfacial adhesion between fibers and polymer. Thermogravimetric analysis can be used to study the reactions and physical changes in the specimens by detecting mass loss. Figure 2 shows the thermal stability of BPF, HDPE, BPF/HDPE composites, and BF/HDPE composites, as determined by TGA.

A summary of $T_{5\%}$, T_{p1} and T_{p2}, and carbon residue at 600 °C are shown in Table 3. High-density polyethylene was degraded in the range of 386.69 to 500 °C, and the

maximum degradation temperature was 469.84 °C. BPF degraded from 250 to 500 °C, and the degradation rate reached a maximum at 358.18 °C.

Table 3. TGA Data for BPF, HDPE, and BPF/HDPE and BF/HDPE Composites

Samples	$T_{5\%}$(°C)	T_{p1}(°C)	T_{p2}(°C)	Residue at 600 °C (%)
1	341.36	356.16	466.48	2.19
2	339.37	358.85	474.55	2.58
3	321.03	356.16	473.55	3.02
4	304.13	354.15	472.53	3.60
5	297.71	356.16	470.51	4.01
6	277.53	343.24	468.45	16.43
7	425.99	469.84	-	0.49
BPF	427.64	358.18	-	2.93

$T_{5\%}$ is the temperature of the materials at 5% loss weight, *i.e.*, the onset degradation temperature of the materials. T_{p1} and T_{p2} represent the first and second peak of DTA curves of BPF, HDPE, BPF/HDPE composites, and BF/HDPE composites.

The thermal stability of BPF/HDPE composites was between that of the HDPE matrix and that of the BPF. The low mass loss between 0 and 100 °C indicated that the hygroscopicity of the composites was low (Fig. 2a).

For the BPF-filled composites, the first shoulder peak in the temperature range of 300 to 400 °C suggested the degradation of cellulose, hemicelluloses, and lignin from the BPF, and the second shoulder in the range of 500 to 600 °C indicated HDPE degradation (Lee *et al.* 2009) (Fig. 2b).

As the BPF content increased, the initial decomposition temperature ($T_{5\%}$) of the BPF/HDPE composites decreased from 341.36 to 297.71 °C. A similar trend was noted by Lee *et al*. (2006) in their study of composites with natural bamboo fibers. However, the residues of the composites were all higher than that of pure HDPE and increased with increasing BPF content.

Compared to the composites with 50 wt% BF, the $T_{5\%}$ of composites with 50 wt% BPF was increased by 20.18 °C, which suggested that the thermal stability of the BPF/HDPE composites was better than that of the BF/HDPE composites.

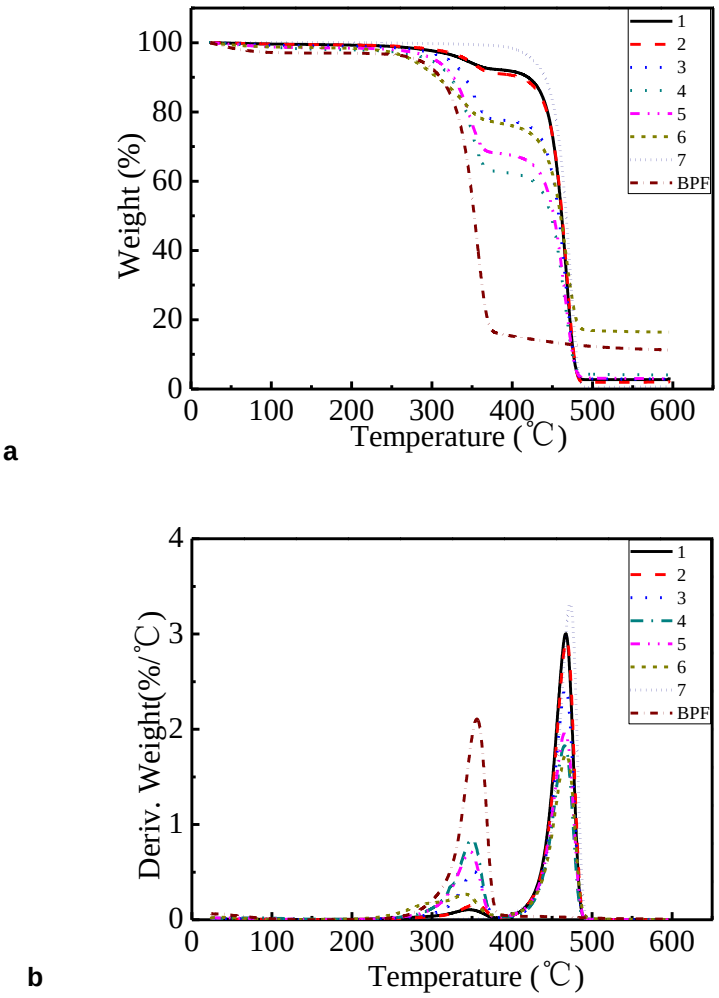

Fig. 2. The (a) TGA curves and (b) DTA curves of BPF, HDPE, BPF/HDPE composites, and BP/HDPE composites

The dynamic behavior of polymer composites is determined by both the polymer and discrete phases, *i.e.* the filler and the coupling agent. The concentrations of the discrete phase and the agglomeration play important roles in the properties of composites (Boyd 1985). Dynamic mechanical analysis is used to investigate the material properties of composites in the solid state under dynamic conditions of temperature or frequency.

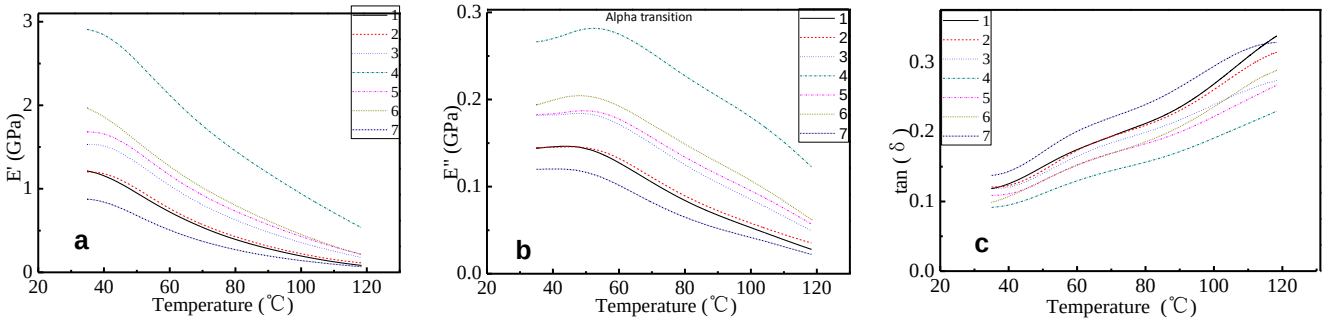

Fig. 3. Plots of (a) *E*', (b) *E*", and (c) *tan* δ *versus* temperature for the HDPE and BPF/HDPE and BF/HDPE composites

Changes in the storage modulus (*E'*) (Fig. 3a) in different formulations due to the increase in the temperature are presented along with the loss modulus (*E"*) (Fig. 3b) and damping parameter (*tan*δ) (Fig. 3c). Figure 3a shows that the *E*' value of BPF/HDPE composites decreased as the temperature was increased. When the temperature increased, the relaxation process of the molecular matrix began, and thermal expansion decreased the intermolecular forces (George *et al.* 1999). The *E'* values of composites were increased with the incorporation of BPF (Fig. 3a). This can be attributed to the reinforcing effect imparted by the BPF, which allowed a greater degree of stress transfer at the interface and led to an increase in the stiffness of the matrix (Jain *et al.* 1992). As shown in Fig. 3a, the *E'* value increased with increasing fiber content up to 30wt%, then tended to decrease at a 50 wt% fiber content. One probable reason for this result was the agglomeration of fillers that caused poor interfacial adhesion, which decreased the interfacial bonding strength (Mi *et al.* 1999). The *E'* values of the BPF/HDPE composites were higher than those of the BF/HDPE composites, which indicated that the stiffness of the composites reinforced by BPF was higher than that of the BF/HDPE composites.

Figure 3b shows that the *E"* value had the same tendency, decreasing as the temperature increased. The appropriate weight ratio of BPF to HDPE in the composite could enhance the area of the interface between the fibers and the matrix. Therefore more energy was needed to deform the composite. When the BPF content reached 50wt%, the agglomeration of BPF led to less contact area between BPF and HDPE, such that the matrix can't coat the fiber completely so that weak zones are present within the composites. As a result, less energy was needed to deform the composites. There was an α-relaxation peak in the range of 40 to 60 °C, which is related to the crystalline fraction (Tajvidi *et al.* 2006). The intensity and broadness of the peak increased with increasing BPF content up to 30wt%. This may be influenced by the conversion of composites to a leathery state or complete melting. The broadening of the relaxation spectra with an increase in BPF content can be attributed to both a decrease in the flexibility of the macromolecular chains in the surface layer and interaction between the matrix and BPF (Alberola and Mele 1997). The *E"* value of the BF/HDPE composites was lower than that of the BPF/HDPE composites (30wt% BPF).

The mechanical damping (*tan*δ) shows the amount of energy dissipated as heat during the deformation. The damping parameter is vital due to environmental concerns when concerning the industrial and structural applications. Using a material with high damping to a vibrating surface can convert the energy to heat, which is distributed within the material itself and is not radiated as airborne noise. Thus, a high damping is required in structural applications where sound or any other kind of vibration absorption is needed, which can decrease the effect of undesirable vibrations to safer limits. The variation of the damping parameter (*tan*δ) (*i.e.*, the ratio of E"/E') for HDPE and BPF/HDPE and BF/HDPE composites is shown in Fig. 3c. The *tan*δ values increased with increasing temperature in the range of 35 to 120 °C. When the BPF content reached 30 wt%, the *tan*δ values of the BPF/HDPE composites were the lowest; this indicated a more restrained molecular motion because of the strengthening interfacial action between the matrix and the fibers. The HDPE showed a higher *tan*δ and stayed on the top throughout the test temperature range. The lower *tan*δ value of composites demonstrated that these formulations displayed more elastic (spring-like) than viscous (dashpot-like) characteristics. Therefore, BPF considerably reduced damping, whereas the BPF/HDPE composite was more elastic at a higher fiber content (Tajvidi *et al.* 2010).

In conclusion, the dynamic mechanical properties of the polymer composite reinforced by 30 wt% BPF were better than those of the BF/HDPE composite, and the BPF/HDPE composite was not easily deformed under a dynamic load.

CONCLUSIONS

1. The mechanical properties of the polymer composites were improved by BPF as a reinforcing phase. When the BPF content reached 30 wt%, both the mechanical strength and modulus were optimal and better than the composites reinforced by 50 wt% BF.
2. The degree of composite degradation fell between those of BPF and HDPE and underwent two separate degradation steps. The thermal stability of the composites was reduced due to the incorporation of BPF, while the weight residue at 600 °C increased. The thermal stability of the BPF/HDPE composite was better than that of the BF/HDPE composite.
3. The composites with 30 wt% BPF had a higher storage modulus and loss modulus, and a lower tan delta than the other composites. The α-transition was observed in the range of 40 to 60 °C and was more distinct at 30 wt% BPF than other composites reinforced by BPF and BF. Compared to the BF/HDPE composite, the BPF/HDPE composite had better dynamic mechanical properties, *i.e.*, higher stiffness under a dynamic load. Thus, BPF can be an important alternative to BF as a filler in composites.

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