

Effect of Processing Temperature and Polymer Types on Mechanical Properties of Bamboo Fiber Composites

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Bamboo fiber was extracted after alkaline treatment, and the mechanical properties of fibers and polymer composites were measured. The results showed that the strength of bamboo fiber was higher when the diameter was smaller. Smaller diameter bamboo fibers were dense, while larger diameter ones were composed of vascular bundles, which contained inside voids and outside parts having insufficient lignification. Tensile tests were conducted on bamboo fibers after heating at constant temperatures, and a significant decrease in mechanical properties was observed at heating temperatures above 250 °C. Bamboo fibers were compounded with PE, PA12, ABS, PA6, and biobased PC (Durabio), and injection-molded to prepare the composite specimens for flexural testing. The composite of polyethylene with 30 wt% bamboo exhibited considerably high flexural modulus compared to pure PE. Nevertheless, a large plastic deformation, which was equivalent to that of pure PE was observed. In other polymer composites, those flexural moduli increased, and degree of plastic deformation decreased dramatically, leading to brittleness. For PA6, which was molded above 250 °C, the increment in flexural modulus by fibers was less than the other composites due to the thermal decomposition of the fibers.

DOI: 10.15376/biores.19.1.41-52

Keywords: Bamboo fiber; Natural fiber composites; Load and deformation; Flexural properties

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INTRODUCTION

The use of fiber-reinforced polymer composites, particularly glass and carbon fiber, have been ubiquitous in many engineering applications, as it offers significant advantageous properties over the traditional steel parts with reduced weight and comparable mechanical properties. However, growing environmental regulations in recent decades have led to exploitation of natural fibers as a viable alternative for synthetic fibers owing to their availability, low cost, biodegradability, and sustainability. Among known natural fibers (*e.g.*, banana, flax, sisal, jute, bagasse, hemp, and bamboo), bamboo is a prospective material for composite reinforcement owing to its rapid (Li *et al.* 2020) growth and abundance. The properties of bamboo fibers vary widely depending on various factors such as species and density (Li *et al.* 2020; Hartono *et al.* 2022), with tensile

strength and modulus up to 800 MPa and Young's modulus up to 43 GPa (Osorio *et al.* 2018).

Natural fiber thermoplastic polymer composites are made by compounding plant-derived natural fibers with thermoplastic polymer, typically polyolefins. Thermosetting polymers also can be compounded with natural fibers, and the composites have been found to have potential usage in automobile parts (Hassanin *et al.* 2016). However, plant-derived natural fibers consist of cellulose, hemicellulose, and lignin which contain hydroxyl functional groups. This could lead to a compromise in the composites' overall performances, as weak interfacial adhesion is formed from the hydrophilic fibers and hydrophobic matrix, limiting the effective stress transfer. Other limitations of natural fibers include moisture sensitivity and thermal stability, which could lead to dimensional instability. Processing temperatures of natural fibers are usually limited to 200 °C due to thermal degradation of fiber components such as hemicellulose and lignin (Mohanty *et al.* 2000; Araújo *et al.* 2008). Therefore, treatments to fibers are required prior to processing. Alkaline treatment using NaOH is widely used to alter the fibers surface and remove less thermally stable components such as hemicellulose and wax (Chen *et al.* 2018).

Investigations on alkaline treatment of natural fibers have been reported to have an effect on the crystallinity (Liu and Hu 2008; Keshk 2015) and interfacial adhesion of bamboo fibers to polymer matrix (Cai *et al.* 2016). Chen *et al.* (2017) studied the effect of NaOH concentration on the microstructure and mechanical properties of bamboo fiber. The study revealed that treatment at high NaOH concentration ($\geq 10\%w/v$) breaks down the cell wall, transforming the fiber to a more porous and ductile structure, which led to a decrease in the Young's modulus. At lower NaOH concentration ($< 10\%w/v$), only removal of hemicellulose occurs. Similar observations, which were in agreement, were made with other natural fibers at higher alkaline concentration (Chowdhury *et al.* 2013; Bartos *et al.* 2020; Hao *et al.* 2021; Noori *et al.* 2021). Besides polyolefins, blending natural fibers with a more polar thermoplastic such as polyamides could help improve the interfaces due to their ability to form hydrogen bonds (Haddou *et al.* 2017).

In this research, the focus was on the reinforcement of thermoplastic polymers (ABS, Durabio (biobased Poly carbonate Mitsubishi Chemical Co. Ltd. JAPAN), PE, PP, PA6, and PA12) with alkaline treated short bamboo fibers. These polymers were selected because they have processing temperatures in a suitable range of 200 to 270 °C degrees, which are close to the upper limit of natural fibers. The effect of difference in interfacial adhesion between bamboo fiber and those polymers on flexural modulus, strength, and impact strength was investigated. Moreover, the effect of thermal degradation and weight loss of treated bamboo fibers at different temperatures and holding time of 7 min on the mechanical properties in bamboo fibers were studied.

EXPERIMENTAL

Materials

Moso bamboo, which grows naturally in the western part of Tokushima prefecture in Japan, was used in this study. The bamboo had a height of approximately 15 meters, and the culm section ranging from 50 to 1000 mm from the base was utilized. The diameter of the bamboo culm was 110 mm. Excluding the node portions, the culm was cut into strips longitudinally of 70 mm lengths and circumferentially 10 mm widths. Subsequently, the outer skin was peeled off by a cutter. The strips in the flask were boiled with 3% NaOH at

120 °C for 2 h in an oil bath. After this alkali treatment, the strips were washed with pure water and dried in an electric oven for 24 h at 60 °C.

Polymers used in this study included polyethylene (HDPE SHA7260, Braskem, Brazil), polypropylene (PP, AZ864, Sumitomo Noblen, Japan), UMEX (UMEX1010, maleic acid-modified polypropylene, Sanyo Kasei, Japan), polyamide 12 (PA12 L1640, Polyplab Evonik, Japan), polyamide 6 (PA6 A1030BRL, Unitika, Japan), ABS (ABS150, Techno UMG, Japan), and isosorbide-based polycarbonate (Durabio D6350R, Mitsubishi Chemical, Japan). Table 1 shows the composition of composites used in this study.

Table 1. Composition of Composites Used in this Study (weight %)

	Polymer (%)	Bamboo Fiber (%)	UMEX (%)
PE	100	0	0
PE + Bamboo	70	30	0
PE + Bamboo + UMEX	56	30	14
PP	100	0	0
PP + Bamboo	70	30	0
PP + Bamboo + UMEX	56	30	14
PA12	100	0	0
PA12 + Bamboo	70	30	0
ABS	100	0	0
ABS + Bamboo	70	30	0
PA6	100	0	0
PA6 + Bamboo	70	30	0
Durabio	100	0	0
Durabio + Bamboo	70	30	0

The bamboo fiber obtained from both the outer and inner region in bamboo strips were each separated into their respective outer and inner portions. After that, each fiber was adhered to both ends of a cardboard in the shape shown in Fig. 1 using adhesive. This cardboard was mounted on the jig of a tensile tester (EZ-SX, Shimadzu, Japan), and a portion of the cardboard was cut just before the tensile test, which was then carried out. The diameter of the fiber was determined using the average from three different positions measured using a micrometer. When calculating the elliptical cross-sectional area based on the longest and shortest diameters measured at three points in a fiber, as well as when calculating the circular cross-sectional area based on the average diameter at the same three points, it was experimentally determined that the circular cross-sectional area was approximately 97.5% of the elliptical cross-sectional area. The total number of fibers measured was 200. Therefore, a circular cross-sectional shape was assumed for the fibers. Ten samples were tested for each condition. The gauge length was 30 mm and crosshead speed used was 1 mm/min. The above bamboo fibers were heated isothermally at different temperatures (210, 220, 230, 240, 250, 260 and 270 °C) for a constant time (7 min) in an electric oven. After the heat treatment, single bamboo fiber was isolated and both ends were attached on the cardboard with adhesive before subjecting to tensile testing as above.

Alkaline-treated bamboo fiber strips above were finely chopped by a grinder and sieved (ISO 3310-1, diameter size 560 micron meter) before characterization. The thermal stability and decomposition behavior of the bamboo fibers were investigated using the thermogravimetric analyzer (METTLER TOLEDO, TGA1, Switzerland) under isothermal run (210, 220, 230, 240, 250, 260, 270 °C) for 7 min twice to simulate the processing conditions of the bamboo fiber composites during pelletizing and injection molding process. For the dynamic thermogravimetric analysis of alkaline treated bamboo with

moisture removed beforehand, the experiment was conducted from 25 to 800 °C under a heating rate of 20 °C/min and N₂ gas flow rate of 50 mL/min.

Mixtures of composites pellets (ABS, PE, PP, PA6, Durabio and PA12) with bamboo fibers were processed by melt blending with a single extruder. All the fiber content used in this study were 30 wt%. The obtained strand compounds were then cut into pellets with an average length of approximately 5 mm. The pellets were molded into specimens using a 10-ton injection-molding machine (type PS10E1ASE, Nissei Co. Ltd., Japan). The injection was carried out at a nozzle temperature 165 to 260 °C, as shown in Table 2, holding pressure of 11 MPa, mold temperature of 60 °C, cooling time of 7 s, screw rotation speed 50 rev./min, and an injection speed 0.5 sec/shot. The prepared composites were cylindrical with 5.8 mm diameter and 60 mm length.

Three-points flexural tests were conducted in accordance with ISO 178 specifications with at least five cylindrical specimens using a universal testing machine (EZ-SX, Shimadzu JAPAN). The cross-head speed was 1 mm/min. The flexural modulus was determined by the initial derivative of the load-displacement curve. The derivative part was from 0 to 15% of maximum load. The flexural strength was calculated by maximum load.

SEM micrographs were taken from the fracture surface of the flexural test specimens using a scanning electron microscope (JEOL 6510A, Japan). The specimens were gold sputtered before observation. The accelerating voltage used was 5 kV.

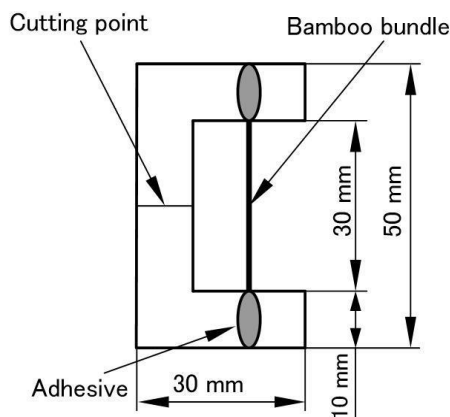


Fig. 1. Configurations of bamboo bundle in tensile test

RESULTS AND DISCUSSION

Figure 2 displays an optical micrograph of a typical cross-section of a bamboo culm specimen. The bamboo tissue structure consists of isolated vascular bundles in an island-like configuration between parenchyma cells. The vascular bundles are surrounded by rigid fiber cells called bast fiber with heavily thickened secondary cell wall made from cellulose, hemicelluloses, and lignin (Palombini *et al.* 2016). As the outer part of the bamboo culm is comprised mostly of vascular bundles, the density of bast fibers is higher, which plays a role of a more mechanically tough structure. In this study, the parenchyma cells were removed via alkaline treatment to obtain only the bast fibers bundle. The fiber obtained from both the inner and outer part of the bamboo strips within 5 mm of their respective side were mechanically extracted by hand, using a tweezer for tensile testing.

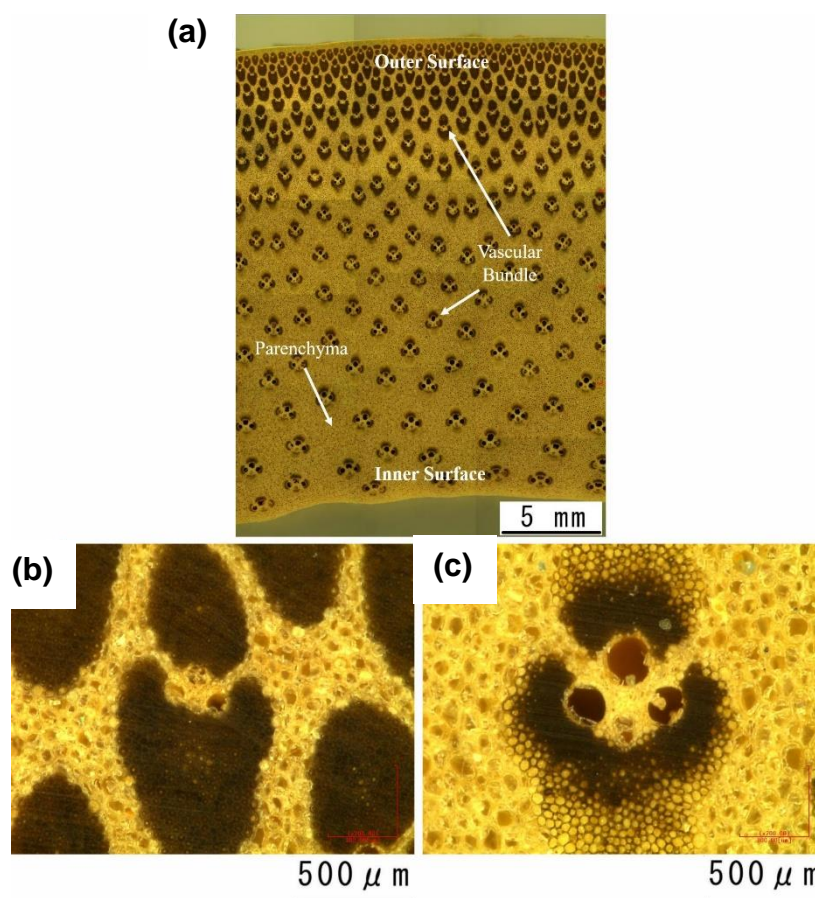


Fig. 2. Optical micrograph of (a) bamboo culm cross-section, (b) magnified vascular bundle at the outer surface, and (c) the inner surface

Figure 3 displays a frequency distribution graph of bamboo fiber diameters. As can be seen in the figure, in the outer region of bamboo stems, there was a relatively large number of fibers with larger diameters, whereas in the inner region, there were many fibers with smaller diameters. This observation is consistent with the appearance of the photographs in Fig. 2(a). Figure 4 shows the relationship between fiber diameter and tensile strength. The fibers obtained from the outer region possess higher tensile strength value (av. 580 MPa) than the inner region fibers (av. 464 MPa). Moreover, fibers with smaller diameter in both outer and inner region were observed to exhibit higher tensile strength. As can be seen from Fig. 4, the strong fibers tend to have smaller diameters of around 250 μm , while the weak fibers tend to have larger diameters of around 500 μm . Typical large-diameter fiber has a shape where the entire vascular bundles have shrunk, whereas the small-diameter fiber can be seen to be dense fiber bundles. This is due to the overall higher density of defects present in larger diameter fibers (Defoirdt *et al.* 2010). SEM micrographs of the fibers in the encircled region (green and red) are shown in Fig. 5(a) and (b), respectively. Compared to the smaller diameter fiber in Fig. 5(a), microcavities near the center of the fiber could be found in the case of larger diameter fiber in Fig. 5(b). The aforementioned factors contributed to the lowered mechanical properties in large diameter fibers. Thus, the bamboo fibers obtained in the outer region were used for subsequent experiments.

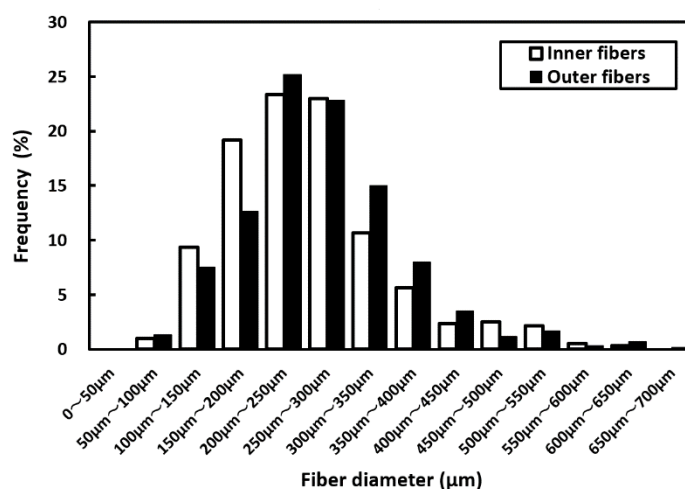


Fig. 3. Relationship between frequency and a fiber diameter distribution in outer and inner parts of bamboo

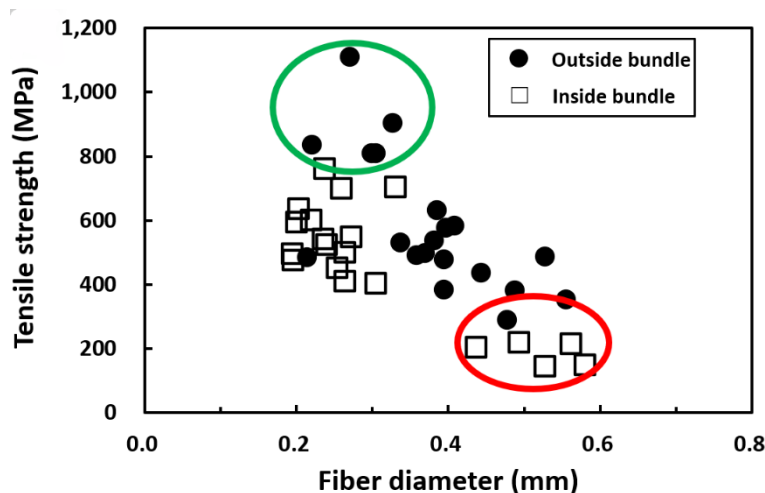


Fig. 4. Relationship between fiber diameter and tensile strength in single fiber tensile test

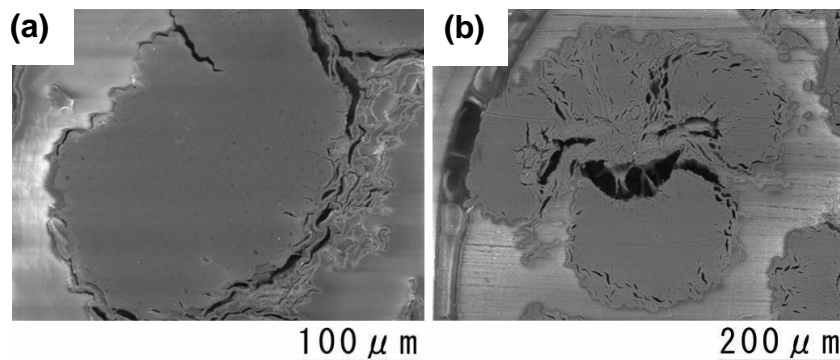


Fig. 5. SEM micrographs of (a) small diameter fiber bundle (b) large diameter fiber bundle

Figure 6(a) shows the dynamic TGA conducted from 25 to 800 °C. As can be seen in the figure, the apparent major thermal decomposition occurred in the range of 200 to

580 °C. This weight decrease in the figure is a result of hemicellulose, lignin, and cellulose decomposition. The weight of bamboo fiber decreased rapidly between 200 and 270 °C. This may occur even at short heating such as compounding and injection process (4 to 8 min). Therefore, the relationship between the weight loss by isothermal heating loss and tensile strength was investigated.

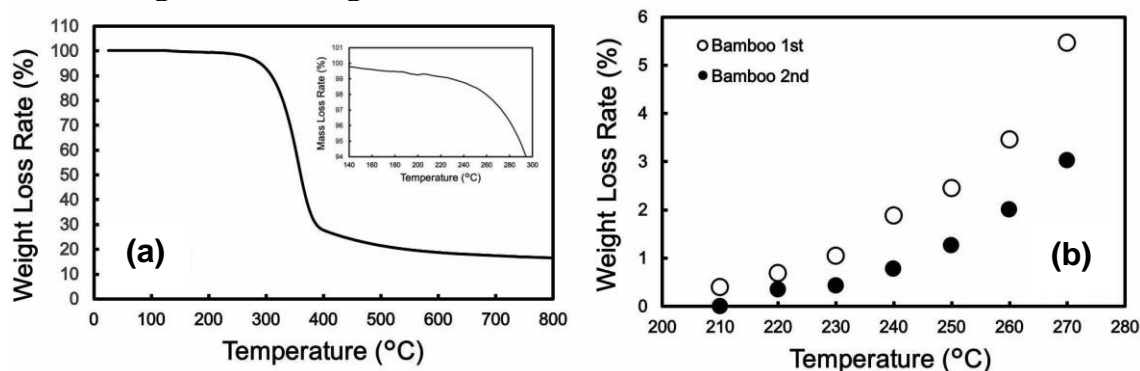


Fig. 6. Relationship between temperature and mass loss percentage in bamboo fiber (a), relationship between mass loss and temperature on isothermal thermogravimetric analysis (b)

To simulate the decomposition of fibers in composites, bamboo fibers were subjected to two rounds of isothermal heating, first for the pelletizing extrusion process and second for the composite injection molding process. Figure 6(b) shows the fiber mass loss as a function of holding temperature from the isothermal thermogravimetric analysis. The mass loss increases with increasing temperature with significant mass loss observed at holding temperature above 250 °C. The high temperature processing resulted in the thermal degradation of fiber structure, loosening of the cell wall, and generation of pores (Cui *et al.* 2023). Furthermore, tensile tests were performed to investigate the changes in mechanical properties of the bamboo fiber, with results shown in Fig. 7(a). The tensile strength of the fiber was found to be significantly decreased at temperatures above 250 °C. The decrease was pronounced even at a low mass loss of 2%, where about 50% of the fiber original strength was retained, as displayed in Fig. 7(b).

Figures 8(a-f) show the load-displacement curves of polymer composites, with the properties summarized in Table 2. Figure 8(a) presents the curve in the flexural testing of PE composites. The flexural modulus and flexural strength in PE-bamboo composites increased compared to those in PE. In the case of PE-bamboo composites, the degree of plastic deformation was almost equivalent to that of PE. This is a very interesting result that contradicts the rule of mixtures. This is because despite adding rigid fibers to soft and highly plastically deformable PE, large plastic deformation, which is equivalent to pure PE was observed in the testing curve. On the other hand, it is known that the inherent hydrophilic nature of fiber does not form good interfacial adhesion with hydrophobic polyethylene. Therefore, it is considered that although the flexural modulus was increased at the initial stage, as the test progressed further, interfacial separation of fiber and matrix occurred inside the specimen, resulting in large plastic deformation. This will be discussed in more detail later. To improve the interfacial adhesion, a commercial maleic acid-modified PP coupling agent (UMEX) was added at 14 wt%. The grafted maleic anhydride could chemically react with the hydroxyl group of fiber to form stable ester linkages. This is reflected in the further improved modulus and strength, but lowered plastic deformation, as shown in Fig. 8(a). Figure 8(b) presents the flexural properties of PP composites. Similar

with the results observed for PE composites. However, the degree of plastic deformation decreased compared to the result of PE-bamboo.

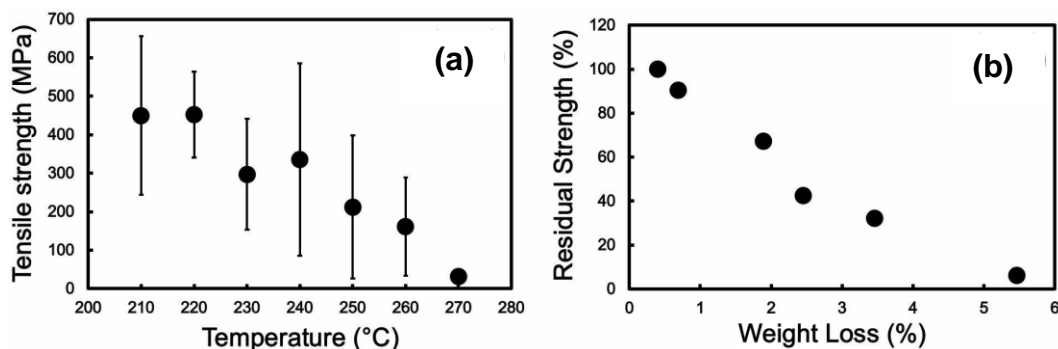


Fig. 7. Relationship between temperature and tensile strength in single bamboo fiber after isothermally heated for 7 min (a), relationship between mass loss and residual strength in fiber (b)

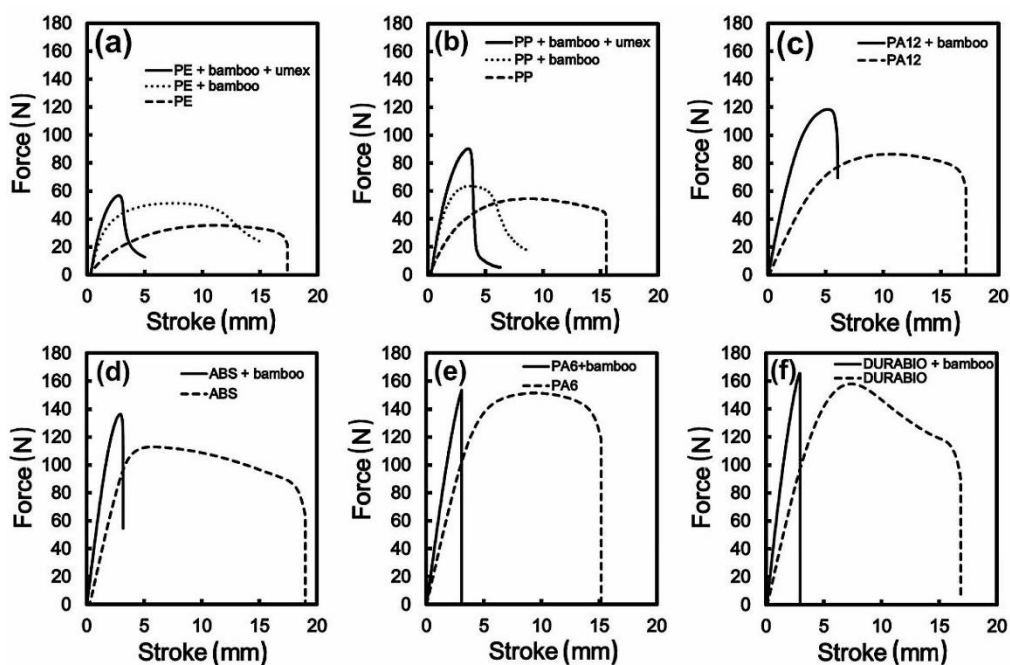


Fig. 8. Flexural Load-Displacement curves of fiber-reinforced composites at 30 wt% fiber and polymer composites: (a) PE, (b) PP, (c) PA12, (d) ABS, (e) PA6 and (f) Durabio

Figures 8(c-f) present the load-displacement curves of PA12, ABS, PA6, and Durabio composites, respectively. For all polymer composites except PA6, flexural modulus and strength increased with incorporation of bamboo fiber, and the level of plastic deformation decreased dramatically. The increments in moduli were in the order of Durabio > ABS > PA12 > PA6, which were approximately 2750, 2,570, and 2,110 times the corresponding values for each base material, respectively, as shown in Table 2. This is thought to be due to the good adhesion between high modulus fibers and matrix polymers. However, in the case of PA6, the strength decreased, and the increase in modulus was less, at 1,500 MPa. The high processing temperature (250 °C) required for PA6 causes the fibers to suffer degradation, releasing decomposition gas, which can generate microcavities in the molded specimen, as shown in Fig. 9(e).

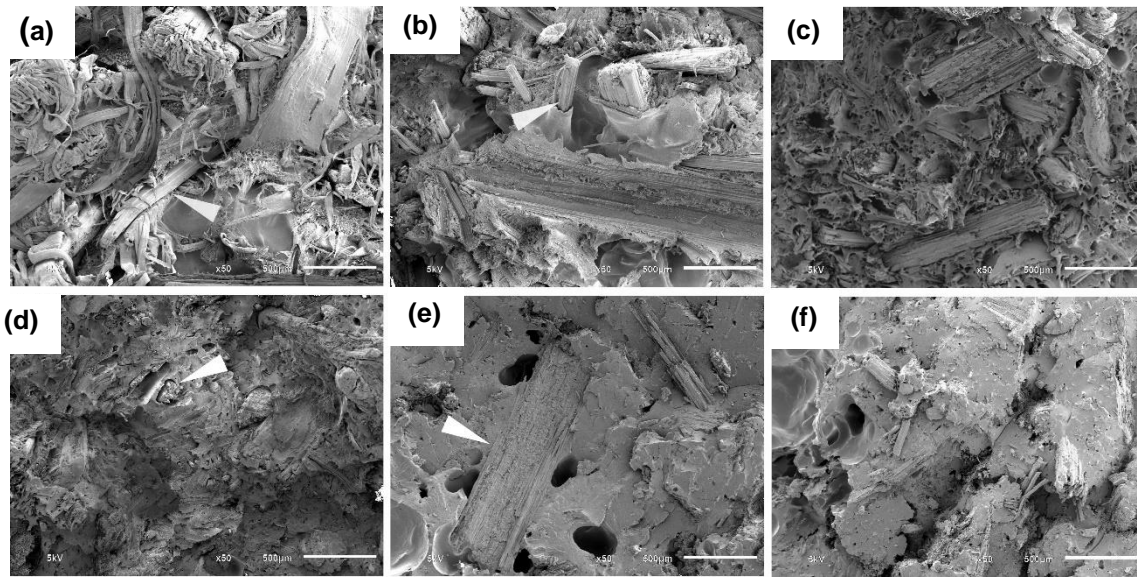


Fig. 9. SEM micrographs of fractured composite surfaces: (a) PE, (b) PP, (c) PA12, (d) ABS, (e) PA6 and (f) DURABIO compounded with 30 wt.% bamboo fiber

Table 2. Properties of Composites Used in this Study

	Injection Molding Temperature (°C)	Impact Strength (mJ)	Flexural Strength (MPa)	Flexural Modulus (MPa)	Increment in Flexural Modulus (MPa)	T _c	T _m
PE	170	491	26	800	-	115.7	133.2
PE + BF(30wt%)	170	620 (126%)	37	3017	2217	116.4	134.6
PP	170	694	40	1307	-	131.0	165.0
PP + BF(30wt%)	170	298 (43%)	46	3695	2388	126.1	167.8
PA12	200	1195	60	1312	-	156.9	181.5
PA12 + BF(30wt%)	215	301 (25%)	81	3424	2112	155.9	181.1
ABS	225	1774	76	2669	-	-	-
ABS + BF(30wt%)	225	186 (10%)	89	5241	2572	-	-
Durabio	230	2022	105	3057	-	-	-
Durabio + BF(30wt%)	230	277 (14%)	106	5808	2751	-	-
PA6	240	1882	106	3152	-	186.9	225.4
PA6 + BF(30wt%)	250	258 (14%)	91	4686	1534	187.9	224.4

The impact absorption energy is generally defined as the integral of elastic and plastic deformation and force during an impact test. However, in this study, absorption energy was estimated by the integral values of displacement and load in the load-displacement curve in each flexural test. The calculated values are shown in Table 2. The impact absorption energy was in the PE bamboo composite, 126% higher than that in the base pure PE. In comparison to PE composites, other polymer composites showed significant decrease in their absorption energy due to the good adhesion between the brittle fiber and soft matrix resulted in a stiffer composite. These experimental results are very important. This is because the toughness in a material is an essential factor, especially for automobile external components.

Figures 10(a-f) show the SEM micrographs of fracture surface of the bamboo fiber-polymer composites at 30 wt% fiber. Matrix-fiber debonding indicated by white arrows and fiber agglomeration were observed in the case of Fig. 10(a) and (b) due to poor adhesion. Furthermore, observation of Figs. 10(a) and (b) reveals no signs of matrix adhering to the surface of fibers, which is consistent with the discussion above. Composites in Figs. 10(c-f) show better matrix-fiber adhesion, perhaps because high processing temperature could loosen the fiber and that hydrogen bonds could form between the fiber hydroxyl functional groups and the nitrile groups (ABS), ester groups (PC), or amides (PA6, PA12) of the matrix. Micro voids could be observed because of fiber pull-out. Figure 10(e), PA6 shows that the surface was flat, including both fibers and polymer caused by brittle fracture. The micropores in the vicinity of the arrow might be due to thermal degradation gas that was released due to the high processing temperature of 250 °C.

CONCLUSIONS

1. Bamboo fiber was extracted after alkaline treatment and subjected to tensile testing. The results showed that fiber bundles located in the bamboo outer surface possessed higher tensile strength and elastic modulus than those of the inner part. However, regardless of the site of the fiber, the entire vascular bundle, which is the large diameter fiber, had higher porosity. Consequently, the mechanical strength was lowered due to the defects.
2. The tensile strength of bamboo fibers showed a significant decrease after isothermal heating above 250 °C, coinciding with the weight loss from the fiber decomposition. The tensile strength of the fibers heated at 250 °C for 7 min was approximately 50% of that heated at 210 °C.
3. The bamboo fibers were compounded with various polymers (PE, PP, PA12, ABS, PA6 and Durabio) using a single extruder and injection-molded into cylindrical shape specimens for flexural testing. In the case of PE bamboo composites, results contrary to mixture of law were obtained. Specifically, in the initial stages of the test, the flexural modulus considerably increased. Nevertheless, as the test progressed, interfacial separation between the fiber and matrix occurred within the specimen, resulting in large plastic deformation of the specimen. PE composites can be considered as potential applications which need high impact strength. In contrast, in the case of the good adhesion polymers with bamboo such as PA12, ABS, PA6, and Durabio, those flexural modulus and strength increased while those degree of plastic deformation dramatically decreased compared to their pure polymers.

ACKNOWLEDGEMENTS

The authors would like to thank Prof. Sanong Ekgasit at the Department of Chemistry, Faculty of Science, Chulalongkorn University for SEM experiment, and National Research Council of Thailand (NRCT) and Chulalongkorn University (N42A660910) for the funding used for the liq N₂ and DSC pans.

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Article submitted: September 15, 2023; Peer review completed: October 14, 2023;
Revised version received and accepted: October 22, 2023; Published: November 3, 2023.
DOI: 10.15376/biores.19.1.41-52