Properties of Oil Palm Empty Fruit Bunch-Filled Recycled Acrylonitrile Butadiene Styrene Composites: Effect of Shapes and Filler Loadings with Random Orientation

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Exploration of natural resources as composite fillers is still under intensive investigation. Previous works mention that nano-size natural fillers provide an alternative solution to improve certain composite properties compared with macro- and micro-size nanofibers. This work prepared biocomposites formulated from recycled acrylonitrile butadiene styrene (ABS) reinforced by nano powders or long fibers of oil palm empty fruit bunch (OPEFB). Composite properties in terms of density, Fourier transform infrared spectroscopy, surface morphology, melt flow rate, tensile strength, impact strength, and hardness were studied. The density of all composites generally increased with increasing filler loading. Composites with nano powder fillers had lesser voids than those composites with the long fiber. Melt flow rate of all composites fluctuated with filler loadings. Increasing filler loadings for composites with long fiber increased brittleness. By contrast, composites with nano powder fillers were more elastic at the higher filler loading. It was confirmed that impact and hardness properties of the composites with nano powder fillers increased with increasing filler loading. Moreover, composites with long fiber fillers decreased their impact and hardness properties with filler loadings increase.

Keywords: Biocomposites; Oil palm; Natural fillers; Mechanical properties

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INTRODUCTION

The manufacture of biocomposites in various industries has recently increased because of their superior properties, such as good thermal and mechanical behavior, biodegradability, low cost, low density, and renewability. In addition, environmental constraints have also pushed industries and researchers to exploit natural resources such as clay, coir, palm, banana, rattan, and kenaf to replace synthetic glass fillers for producing composites. Therefore, much work was conducted to exploit these natural fillers (Vilaseca *et al.* 2010; Faruk *et al.* 2012; Ramamoorthy *et al.* 2015). These studies established that the natural fillers were comparable with synthetic glass.

Composite fillers from natural resources are well known to have poor fiber/matrix interactions that directly affect their physical and mechanical properties, particularly with micro- or macro-size fillers. Zhou *et al.* (2010) found that the microfillers with very high loadings were required to reach the percolation threshold and obtain high thermal conductivity. To overcome this problem, they suggested that inorganic fillers with a nano

size, which require a relatively small amount of filler loading, can be used. Moreover, there is evidence that fillers with a nano size can be used to improve composite properties (Lin *et al.* 2015; Wang *et al.* 2016). Alternatively, Park and Jana (2003) found that the incorporation of clay with a micro size into polymethylmethacrylate (PMMA) decreased the composite strength. Conversely, PMMA with nanoclay shows improved mechanical strength. Exploration of other natural fillers with a nano size is still needed for confirming their benefits in improving certain properties of composites.

Acrylonitrile butadiene styrene (ABS), which has a chemical formula of $C_8H_8C_4H_6C_3H_3N$, is a copolymer that is commonly exploited in various industries because of its good mechanical properties, chemical resistance, and recyclability (Yang *et al.* 2004). Basically, it is made by polymerizing styrene and acrylonitrile in the presence of polybutadiene. This polymer has been used as a matrix for producing composites for various purposes using virgin (Meincke *et al.* 2004; Choi *et al.* 2005) or recycled ABS (Sun *et al.* 2015; Mao *et al.* 2016).

Considering the aforementioned necessity, this study set out to investigate the usefulness of oil palm empty fruit bunches (OPEFBs) with long fibers or nano powder shapes reinforced into recycled ABS for producing biocomposites. This paper only focuses on effects in their shapes and filler sizes to certain composites performances. The study provides an improvement of the understanding of OPEFBs with different shapes. It is essential to examine their properties for future research topics such as helmet materials, which is an ongoing subject of interest.

EXPERIMENTAL

Materials

In this work, OPEFBs were obtained from PTPN VIII Cikasungka, Bogor, Indonesia. Recycled ABS was supplied by PT MUB Jaya, Bogor, Indonesia. The additives used were as follows: maleic anhydride from Darmstadt, Germany, antioxidant primer from Mumbai, India, and acid scavenger from Mumbai, India. The composition of all composites is listed in Table 1.

Table 1. Composition of Composites with Various Filler Loadings

Designation	Composition			
	Filler (%)	Antioxidant primer (%)	Acid scavenger (%)	Maleic anhydride (%)
ABS/10PNP	10 % nano powder	1	0.3	2
ABS/15PNP	15 % nano powder	1	0.3	2
ABS/20PNP	20 % nano powder	1	0.3	2
ABS/10PLF	10 % long fiber	1	0.3	2
ABS/15PLF	15 % long fiber	1	0.3	2
ABS/20PLF	20 % long fiber	1	0.3	2

Preparation of Fillers

OPEFBs were cut into chip shapes of approximately 2 cm^2. Then they were dried in the sun for two days. This step was followed by oven drying at 100 °C for 1 h. OPEFB fibers were obtained from a milling process using a Fomec MDY 1000 g machine, Jakarta, Indonesia. It was operated at 25,000 rpm milling speed, a voltage of 220 V, and a power

of 2000 W. Long fibers 20 to 25 cm in length and 0.1 to 0.5 mm in diameter were collected from a 20-mesh sieve. To fabricate OPEFB nano powder, the method from a previous study was followed (Nikmatin *et al.* 2015). The nano powders used in this work had particle sizes in the range of 75 to 300 nm. Figure 1 shows the long fibers and the nano powder produced in this work.

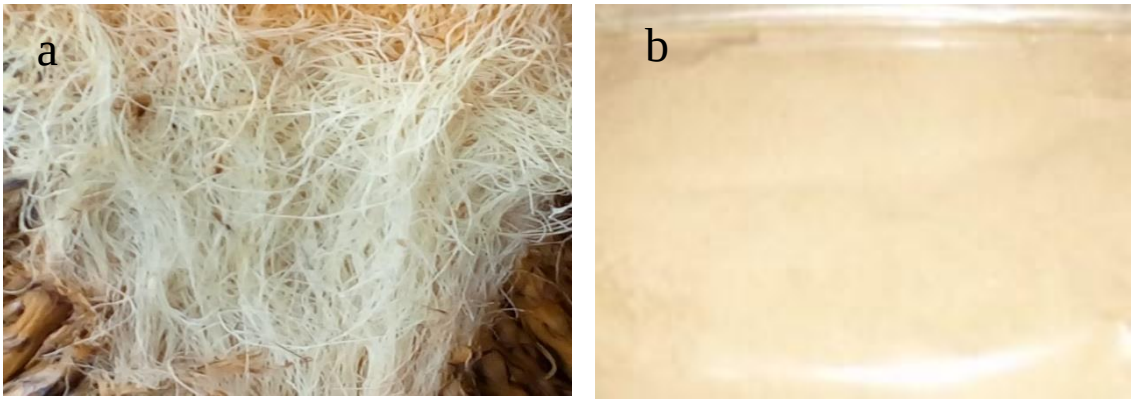

Fig. 1. Long fibers (a) and nano powders (b) produced in the present work

Preparation of Composites

Granular composites were manufactured using a single-screw extruder (Collin). These manufactures are conducted at PT MUB Jaya, Bogor, Indonesia. The composites were fabricated at consecutive temperatures of 195, 215, 220, 220, 220, 225, 225, and 225 °C at a mixing speed of 45 rpm for 15 min, then cooled at a temperature of 40 °C. To fabricate a test piece, granular composites obtained from the single-screw extruder were then processed by an injection molding machine (HC-series) from Ningbo, China. The composites were placed into the hopper at a temperature of 60 °C. They then entered the barrel and were processed at consecutive temperatures of 200, 220, 220, and 190 °C and injected into the molding. The cooling process was carried out at a temperature of 40 °C.

Density Measurement

In this study, the density of the biocomposites was determined using the liquid displacement method, based on Archimedes' principle, and carried out according to ASTM D792-91 (1991) with distilled water and a digital balance. This examination follows previous studies conducted by Sharba *et al.* (2016).

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectra were recorded using an ABB FTIR MB3000 model equipped with MB3000 software, Québec, Canada. This inspection was conducted using wavenumbers from 450 to 4000 cm^{-1} at a resolution of 4 cm^{-1}. In this sample preparation, pellets were produced by mixing biocomposites and KBr with a ratio of 1:100. The pellets were pressed using a hydraulic press at a pressure of 10 tons. Then, they were taken and placed in the FTIR holder.

Morphological Study

The surface morphology of the biocomposites was observed using scanning electron microscopy (SEM) with a JEOL JSM-6510LA model, Tokyo, Japan. An acceleration voltage of 20 kV with 1000x magnification was applied for this scanning. The

samples were sputter-coated with gold using BIO-RAD SEM coating system to prevent electrostatic charging and low image resolution.

Melt Flow Rate Assessment

Melt flow rate (MFR) assessments were carried out using a capillary plastometer (XNR-400D, LARYEE) from Ningbo, China. The capillary die was 8.001 mm in length and 2.096 mm in diameter. A pellet of approximately 20 g was entered into the plastometer and then compressed at a temperature of 230 °C for 300 s. Then, the sample was extruded through the die with a constant load of 2.06 kg. This preparation follows ASTM D1238-13 (2013).

The MFR is commonly defined as the extrudate weight in grams per 10 min. Furthermore, it can be expressed as follows,

$$MFR = \frac{600 A l \rho}{t} \quad (1)$$

where *MFR* is the melt flow rate (g 10 min^{-1}), *A* is the cross-sectional area of the reservoir (cm^2), *l* is the distance traveled by the piston (cm), ρ is the density of sample (g cm^{-3}), and *t* is the time (s).

Tensile Test

Tensile tests were conducted using a Jinan WDW-20 (serial number 20150539) model from Ningbo, China. The dimensions of the samples were 164.00 x 18.97 x 4.02 mm. The tests were prepared according to ASTM D638-14 (2014). In this work, Young's modulus was then calculated using the following equation,

$$E = \frac{F / A}{\Delta L / L} \quad (2)$$

where *E* is the Young's modulus (Pa), *F* is the force (N), *A* is the actual cross-sectional area (m^2), ΔL is the length changes (m), and *L* is the original length (m).

Impact Test

Impact tests were conducted using a cantilever beam impact machine from Ningbo, China using a sample with dimensions of 64.00 x 12.91 x 4.02 mm. The impact tests were conducted following ASTM D256-10 (2010).

Hardness Test

Hardness tests were carried out using a Jinan XHRD-50 model (serial number 20150541) from Ningbo, China. The sample dimensions used in this study were 49.61 x 7.02 mm. The hardness tests were conducted following ASTM D785-08 (2015). The tests were carried out at a room temperature of 23 °C.

RESULTS AND DISCUSSION

Density of the Composites

Several studies have concluded that the density of composites compromised their physical and mechanical properties. Studies have been conducted (Zhang *et al.* 2011;

Hosseini 2013) to evaluate the effects of density on composite properties. In this study, the effects of the shapes and filler loadings on the composite densities are shown in Fig. 2. It was found that increasing filler loadings with nano powder increased the density of the composites. A similar pattern was found in the composites with long fiber fillers. This is because the composites are denser and harder, thus improving the density of composites. Generally, the density values obtained in this work were in line with results obtained by Hill and Khalil (2000).

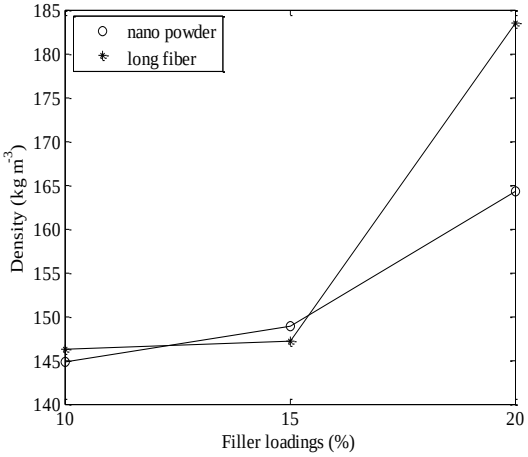

Fig. 2. Density of the composites with various filler loadings

FTIR Characteristics

To obtain an infrared spectrum of molecule absorption, FTIR is a reliable method that is commonly used. This method was used to analyze infrared spectra for various materials (Vijayakumar and Madhu Mohan 2009; Pongali Sathya Prabu *et al.* 2011). Figure 3 shows the FTIR spectra of ABS/10PNP, ABS/15PNP, and ABS/20PNP. FTIR spectra of the composites with long fiber fillers can also be seen in Fig. 3.

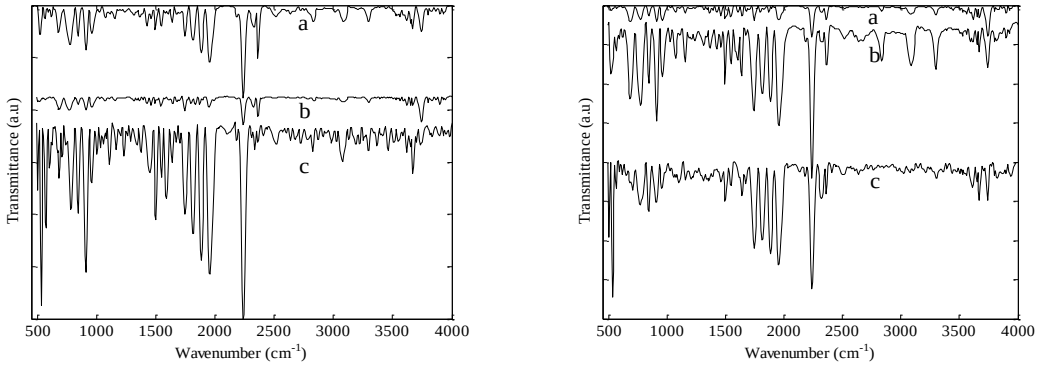

Fig. 3. FTIR spectra of left: (a) ABS/10PNP, (b) ABS/15PNP, and (c) ABS/20PNP; right: (a) ABS/10PLF, (b) ABS/15PLF, and (c) ABS/20PLF

Several studies were conducted to observe the FTIR spectra of OPEFB-filled matrix polymers (Rozman *et al.* 2003; Siyamak *et al.* 2012). In the present study, spectral bands between 650 and 1000 cm^{-1} were related to the =C-H or –NH group. In addition, spectral bands between 1300 to 1500 cm^{-1} were used to characterize a methyl group (Ibrahim *et al.* 2003). Moreover, spectral bands between 2000 to 2400 cm^{-1} are related to -C≡N or -C≡C- stretching. It is apparent that there were slightly different peak intensities for the

composites with increasing filler loadings for both nano powder and long fiber fillers. For composites with nano powder, starting from 10 to 15% in filler loadings, the intensity of the peaks initially decreased and continued to significantly increase with increased filler loadings. Otherwise, the composites with long fibers fabricated using 10 to 15% in filler loadings exhibited peak intensity that initially increased and then continued to slightly decrease with increased filler loadings. A possible explanation for this might be that changes in crystallinity can affect their peak intensities (Bloembergen *et al*. 1986).

Morphological Properties

The surface morphologies of the various filler loadings with nano powder of OPEFB are shown in Fig. 4. Increasing filler loadings from 10% to 20% of the nano powder of OPEFB led to decreased voids in the biocomposites. It is well known that the surface morphology of a composite is a crucial aspect that affects the composites properties. For comparison, Fig. 4 shows the surface morphologies of the composites produced with long fiber fillers. In these composites, many more voids appear compared with the composites with nano powder fillers. These results suggest that the nano powder of OPEFB is able to fill the biocomposite voids. Also, it is assumed that the composites with nano powder fillers had better bond between fillers and ABS matrix compared to the composites with long fiber fillers. It is evident that the composites with nano powder were superior in terms of the surface morphology compared with composites with long fibers.

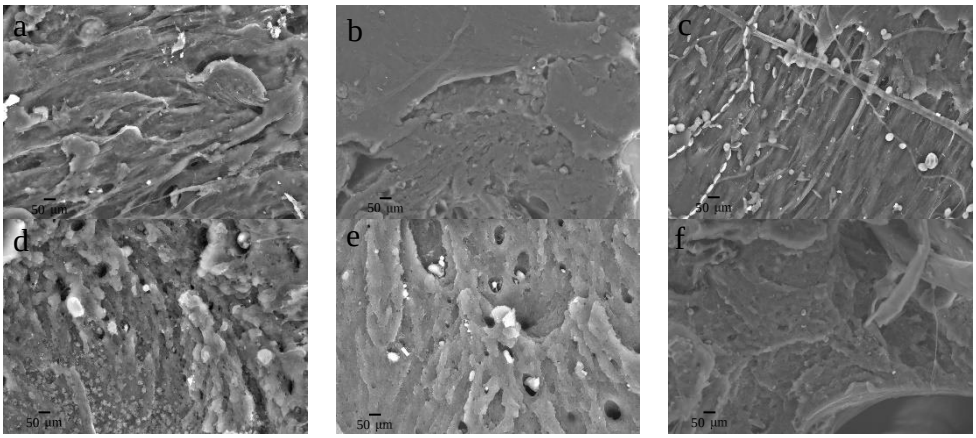

Fig. 4. Surface morphology of (a) ABS/10PNP, (b) ABS/15PNP, (c) ABS/20PNP, (d) ABS/10PLF, (e) ABS/15PLF, and (f) ABS/20PLF

Melt Flow Rate Characteristics

Melt flow rate can be defined as the melt flow of a thermoplastic polymer. This measurement can be used as an indirect measure of molecular weight, *i.e.*, a high melt flow rate corresponds to low molecular weight. Several studies reported that the melt flow rate decreased with increasing filler loading (Guerrica-Echevarría *et al.* 1998; Tang and Liang 2003). The melt flow rates of all composites in this study are presented in Table 2. The present work found that increasing filler loadings with nano powder from 10% to 15% led to a decrease in the melt flow rate. These trends are consistent with data obtained from a previous study (Tang and Liang 2003). When filler loadings increase, the hydrodynamic and mechanical interactions between fillers in the matrix are enhanced; as a result, the resistance between two melt layers increases (Guerrica-Echevarría *et al.* 1998; Tang and Liang 2003). Then, with an increase in filler loading from 15% to 20%, the melt flow rate of the biocomposites began to increase. For composites with long fibers, increasing filler

loadings from 10 to 15% slightly increased their melt flow rate. Furthermore, starting from 15 to 20% in filler loading, the melt flow rate started to decrease. Generally, the melt flow rate of all composites fluctuated with increased filler loadings.

Table 2. Melt Flow Rate of Composites

Designation	MFR (g 10 min^{-1})
ABS/10PNP	12.40 ± 2.73
ABS/15PNP	8.90 ± 3.39
ABS/20PNP	15.00 ± 3.62
ABS/10PLF	11.90 ± 0.34
ABS/15PLF	12.30 ± 3.49
ABS/20PLF	9.30 ± 3.87

Tensile Properties

Figure 5 shows tensile stress-strain relationships for composites produced by nano powder and long fiber fillers. For composites with nano powder fillers, ABS/20PNP exhibited more elasticity compared with ABS/10PNP and ABS/15PNP. These results were then confirmed by the Young's modulus values listed in Table 3. In general, the brittleness of the composites fluctuated with their filler loadings. For comparison, composites produced with long fibers became more brittle with increasing filler loadings from 10 to 20%. These findings are in line with results obtained by Yang *et al.* (2004). Another important finding was that the composites produced by nano powder fillers in this work mostly had higher Young's modulus than those with long fibers.

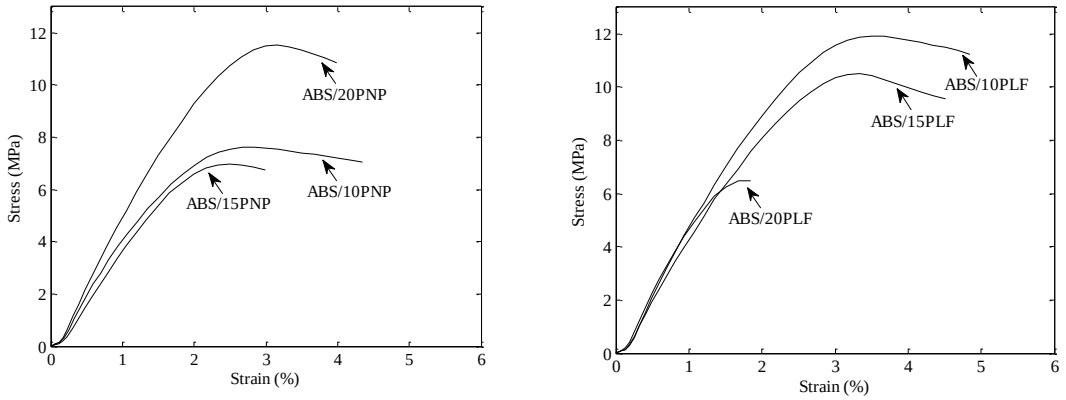

Fig. 5. Tensile stress-strain relationships for all composites

Table 3. Young's Modulus of Composites

Designation	Young's modulus (GPa)
ABS/10PNP	1.62 ± 1.62
ABS/15PNP	1.50 ± 1.58
ABS/20PNP	1.80 ± 1.45
ABS/10PLF	1.59 ± 2.28
ABS/15PLF	1.57 ± 3.47
ABS/20PLF	1.48 ± 3.12

Impact Properties

The impact strengths of ABS/10PNP, ABS/15PNP, and ABS/20PNP are listed in Table 4. The present work found that the impact strength increased with increasing filler loading, from 52 to 70.65 kJ m^{-1}. These findings suggest that OPEFB with nano size proved to improve a mechanical property in terms of the impact strength of the biocomposites compared with those with long fibers. These results are in agreement with those obtained by Khalid *et al.* (2008). The impact strength of the biocomposites is strongly influenced by the nature of the fiber, polymer, fiber–matrix interfacial bonding, and testing conditions (Joseph *et al.* 2003). For comparison, composites with long fibers showed a decreased impact strength with increasing filler loading. This result may be explained by the fact that long fiber fillers used in this work are in macro sizes, so that the bonds between filler and matrix are not as well established. By contrast, Khalid *et al*. (2008) prepared composites using micro sizes (less than or equal to 500 μm). Also, this is related to the fact that the composites composed of nano powder have fewer voids compared with those with long fibers, as confirmed by the surface morphology results.

Table 4. Impact Strength of Composites

Designation	Impact strength (kJ m^{-1})
ABS/10PNP	52.24 ± 0.00
ABS/15PNP	68.41 ± 4.31
ABS/20PNP	70.65 ± 6.24
ABS/10PLF	105.47 ± 8.90
ABS/15PLF	98.01 ± 3.34
ABS/20PLF	57.71 ± 6.44

As presented in Table 4, the impact strength of the biocomposites increased with increasing filler loading. These results suggest that the OPEFB with nano size were capable of absorbing energy because of the strong interfacial bonding between the filler and composite matrix (Haque *et al*. 2009). This is an advantage of the nano size use compared to micro or macro size for producing biocomposite. Accordingly, nanotechnology recently has become a popular approach in various areas of technology such as material construction, automotive, and other applications (Pacheco-Torgal and Jalali 2011; Pereira and Coelho 2015; Wen and Steinmetz 2016). Results from the current study show that incorporation of nano powder OPEFB into recycled ABS was superior in terms of the impact strength of the biocomposites, compared with long fibers.

Hardness Properties

Composite hardness is well defined as the measure of solid matter to resist its shape change when a compressive force is applied. Basically, the hardness of a material is strongly dependent on its rigidity or stiffness properties such as its bulk modulus, ductility, viscoelasticity, and microdurability. This study tested the hardness of materials according to the Rockwell hardness (HRB) measured on the B scale. The composite hardnesses of ABS/10PNP, ABS/15PNP, and ABS/20PNP are presented in Table 5. Their average hardness increased from 96 to 107.07 HRB as filler loadings were increased from 10% to 20% with the nano powder OPEFB. As mentioned in the above section, increasing filler loadings from 10 to 15% decreased their Young's modulus values. However, starting from 15 to 20% the Young's modulus started to increase. Results were inconsistent between

hardness and Young's modulus for composite with 20% in filler loading. This inconsistency may be due to the filler distribution phenomena such as agglomeration or aggregation, which commonly has been reported in nanoparticle studies. Increasing filler loadings with long fibers from 10 to 20% decreased the composite hardness. This is related to the fact that ABS with long fibers is more brittle with increasing filler loading, as confirmed by the tensile properties (Fig. 5). These results seem to be consistent with another study, which found that the composite hardness increases with increasing filler loading (Ismail *et al.* 2002).

Table 5. Hardness Properties of Composites

Designation	Hardness (HRB)
ABS/10PNP	96.60 ± 1.84
ABS/15PNP	97.50 ± 1.06
ABS/20PNP	107.07 ± 4.72
ABS/10PLF	107.22 ± 5.66
ABS/15PLF	106.33 ± 1.68
ABS/20PLF	106.11 ± 3.05

CONCLUSIONS

1. The density of all composites with nano powder and long fiber fillers increased as filler loadings increased.
2. Increased filler loadings slightly changed the peak intensities of FTIR spectra.
3. Surface morphology studies confirmed that the composites produced with OPEFB nano powder exhibited fewer voids compared with those with long fibers.
4. The melt flow rates of all composites were found to fluctuate as filler loading increased for both nano powder and long fibers.
5. Increasing filler loadings of the long fiber OPEFB led to a decrease in the Young's modulus of the composites. Otherwise, composites produced with nano powder fluctuated in terms of their Young's modulus with the maximum value at 20% in filler loading.
6. The present work confirmed that the impact strength of the composites with nano powder increased with increased filler loadings while the composites with long fiber decreased. The similar trend was also observed in their hardness properties.
7. The shapes and filler loadings are two factors affecting certain composite performances. Further studies need to be carried out in order to clearly validate filler distribution and possible effects of recycled ABS on their performances.

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