

# Effects of Fiber Size and Fiber Content on Mechanical and Physical Properties of Mengkuang Reinforced Thermoplastic Natural Rubber Composites

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Thermoplastic mengkuang composites are an alternative material to solve environmental pollution issues associated with synthetic polymers. Mengkuang, or *Pandanus atrocarpus*, raw fiber was cut, dried, ground, and sieved to the required size. The fiber was filled into the matrix of natural rubber (NR) and high-density polyethylene (HDPE) by melt blending *via* internal mixer. The blend of HDPE/NR at 60/40 ratio with fiber sizes of 125  $\mu\text{m}$ , 250  $\mu\text{m}$ , and 500  $\mu\text{m}$  were prepared at fiber contents of 10%, 20%, and 30%. The effects of fiber size and fiber content on the thermoplastic composite were investigated using tensile test, impact test, water absorption, and field emission scanning electron microscopy (FESEM). The maximum tensile strength and tensile modulus were obtained at 20% fiber content of 250  $\mu\text{m}$  fiber size. Impact strength gradually decreased with the increased percentage of fiber content at fiber size, 125  $\mu\text{m}$  and 250  $\mu\text{m}$ . The highest tensile strain at break and lowest water absorption was observed at 10% fiber content for all sizes being studied. The effects of fiber size on water absorption, and percentage of fiber content on impact strength and tensile strain at break were statistically significant ( $p < 0.05$ ).

*Keywords:* Fiber size; Fiber content; Natural fiber; Thermoplastic composites

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## INTRODUCTION

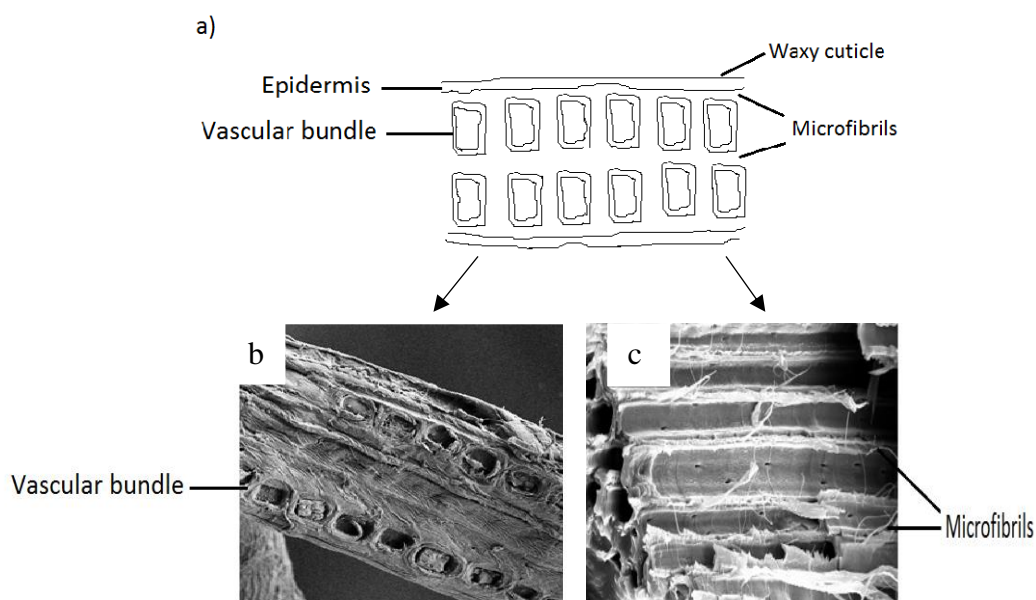
Natural fiber based polymer composites have been produced in various applications in the global market. There is increasing research on natural fiber polymer composites (NFPC) to explore their potential to fulfill the market demand in the automobile, packaging, construction, sport, leisure, and other industries (Bledzki and Gassan 1999; Wambua *et al.* 2003; Ku *et al.* 2011; La Mantia and Morreale 2011; Prasanna and Subbaiah 2013). Global awareness towards products that are more eco-friendly and with lower energy consumption in processing helps to reduce the global warming effect (Luckachan and Pillai 2011; La Mantia and Morreale 2011). Natural fiber polymer composites have a wide range of acceptable strengths depending on the origin of the fiber (Alves Fidelis *et al.* 2013; Verma *et al.* 2013; Sanjay *et al.* 2016). Natural fiber based products that are biodegradable, light weight, and low cost are able to draw interest in the market (Malhotra *et al.* 2012; Morais *et al.* 2013; Poletto 2016).

There are many factors that influence the effectiveness of fiber in the processing and mechanical properties of natural fiber polymer composites. Knowledge of factors such as aspect ratio size, particle size, particle shape, particle size distribution, particle surface area, fiber orientation, volume of fiber, and chemical composition of fiber are important as a basic understanding for further research (Pickering *et al.* 2016).

Many researchers have investigated the effect of size and fiber content on mechanical properties and morphology (Ismail *et al.* 2002; Koubaa *et al.* 2008; Migneault *et al.* 2009; Basiji *et al.* 2010; El-Shekeil *et al.* 2014; Barbhuiya and Ismail 2016; Nasution *et al.* 2016; Sharba *et al.* 2016; Subramonian *et al.* 2016). Bouafif *et al.* (2009) investigated the effect of fiber variability, size, and content on mechanical and physical properties of wood plastic composites. They reported that a range of 24-, 42-, and 65-mesh size showed an increase of fiber size, produced higher tensile strength and modulus of elasticity, while energy to break and elongation were decreased. The increasing of fiber content increased the strength, stiffness, and water uptake.

Yao *et al.* (2008) examined the effect of fiber length/ aspect ratio and fiber content of rice straw fiber. They found that the increasing fiber content increased the tensile modulus but decreased the tensile strength and impact strength. Moreover, a study on bark fiber found that increasing fiber content decreased mechanical properties due to poor fiber dispersion in the plastic matrix and poor bark-plastic adhesion (Yemele *et al.* 2010). However, increasing fiber length improved tensile strength and modulus of elasticity but decreased toughness and tensile strain at failure.

The chemical composition of natural fibers are varies depending of fiber types (Fortea-Verdejo *et al.* 2017). The composition also may differ for the same species because of growth condition factors (Mohammed *et al.* 2015). Mengkuang leaves were reported to contain  $37.3 \pm 0.6\%$  cellulose,  $34.4 \pm 0.2\%$  hemicellulose,  $15.7 \pm 0.5\%$  pentosans,  $24 \pm 0.8\%$  lignin and ash, and  $2.5 \pm 0.02\%$  extractives (Sheltami *et al.* 2012). Aspects of the morphology of mengkuang structure are shown in Fig.1.



**Fig. 1** a) Illustration of mengkuang leaves cross section. b) Cross section image of mengkuang fiber. c) Longitude cross section image of mengkuang fiber

Different types of fiber exhibit different mechanical strength due to their different cellulose content (Liu *et al.* 2013). Mengkuang fiber is expected to be among other natural fibers like sisal, coir, rice straw, pineapple, jute, hemp, and kenaf that work as reinforcement filler in enhancing the strength of polymer composites (Ghasemi and Farsi 2010; Chauhan and Chauhan 2012; Salleh *et al.* 2014).

The aim of the study was to investigate the influences of fiber size and fiber content of untreated raw mengkuang fiber on the mechanical and physical properties as well as morphology of mengkuang fiber in the matrices. The understanding of those behaviour of mengkuang fiber are important relative to the promotion of research directions in the mengkuang composites area and natural fiber polymer composites (NFPC) as well.

## EXPERIMENTAL

### Materials

High density polyethylene (HDPE) with a density of 0.95 g/cm<sup>3</sup>, melt flow rate (MFR) of 1.1 g/10 min, and  $T_m$  at 135 °C was purchased from Polyethylene Malaysia Sdn Bhd (Malaysia). Natural Rubber (NR) Standard Malaysia Rubber grade SMR-L was received from Felda Rubber Industries Sdn Bhd (Malaysia). The mengkuang leaves consumed as a part of this research were harvested in Bangi (Selangor, Malaysia) and prepared in the laboratory of Universiti Kebangsaan Malaysia.

### Fiber Preparation

The dried mengkuang leaves were ground with a universal cutting mill (Model-Pulverisette 19, Fritsch) (Germany). The fibers were immersed in tap water for another 3 days with the water changed frequently. Next, the fibers were dried at room temperature for 3 days and then oven-dried at 80 °C for 24 h to remove moisture by using Universal Oven (Memmert, Model UFB400)(Germany). The fibers were sieved into 3 sizes using a sieve (Retsch Test Sieve, Model ZM200): 125 µm, 250 µm, and 500 µm. The length and diameter were obtained using a Zeiss Optical Microscope (Germany) with the aspect ratio (L/D) shown in Table 1.

**Table 1.** Experimental Design and Detail Dimensions of Raw Mengkuang Fiber

% Weight Fiber Content	Data Label	Mengkuang (MK) Fiber size (µm)				
		Range Fiber Length (µm)	Mesh Size	Average Length, L (µm)	Average Diameter, D (µm)	Average Aspect Ratio L/D (µm)
10	125 µm	38-125	400-120	55.20 ± 2	8.01 ± 4	6.89
20						
30						
10	250 µm	125-250	120-60	158.59 ± 5	20.22 ± 3	7.84
20						
30						
10	500 µm	250-500	60-35	292.34 ± 3	32.56 ± 2	8.98
20						
30						

## Composite Preparation

The composite HDPE/NR at ratio 60/40 was prepared by melt blending *via* internal mixer (Model-Haake Rheomix R600) (Germany). The processing parameters were 135 °C, 50 rpm, and 15 min total time of mixing. Before blending, the fiber was oven-dried for 2 h at 105 °C to eliminate dampness. After blending, the first fiber was added for 3 min, followed by NR, and HDPE was added after 5 min. The samples were compressed for 10 min at 145 °C with 8 GPa pressure, using a hot press to produce 1-mm and 3-mm thick sheet for tensile and impact testing respectively.

## Mechanical Characterization

Ten specimens for tensile test were cut with dumbbell cutter JIS K-6251-6 (Model DMK-1000-D) (Japan). Tensile properties were measured by universal testing machine (Instron, Model-5566) (USA) with a 1 kN load cell at 50.0 mm/min crosshead speed, in accordance with ASTM 412-98a type B (2004). Notched impact strength was measured using a pendulum digital universal fractoscope machine (Tinius Olsen, Model - IT 504), (United Kingdom), according to ASTM D 256-04 (2004). Five specimens were cut at dimensions of 65 × 12.7 × 3.0 (length × width × thickness) mm<sup>3</sup> with 0.25 mm depth notch for each specimen. The impact test energy was performed at 2 joules. The impact specimens were drenched in liquid nitrogen for 1 min before performing the test in such a manner as to completely break up the specimens.

## Water Absorption

Water absorption tests were run in accordance with ASTM 570-98 (2010). Five specimens were cut into 76.2 × 25.4 × 3.2 (length × width × thickness) mm<sup>3</sup>. Before immersion, specimens were oven-dried for 24 h at 80 °C to remove moisture. Specimens were weighed using an analytical balance (Mettler Toledo, Model TLE 204E) ( $W_0$ ) and then immersed in distilled water at room temperature. After 24 h of immersion, specimens were removed from the water. All remaining water on the surface was wiped off with a dry cloth, and the specimen was weighed again ( $W_t$ ). The rate of water absorption ( $W_a$ ) was calculated by Eq. 1,

$$W_a (\%) = (W_t - W_0) / W_0 \times 100 \quad (1)$$

## Morphology

The morphology of the impact fracture surfaces of specimen was examined and analyzed using a SUPRA 55VP Model field emission scanning electron microscope (Germany). Cross sections of fractured surfaces of specimens were coated with platinum to prevent electric charging during investigation. Specimens were observed at 250X magnification.

## Statistical Analysis

Statistical analysis was performed using one-way analysis of variance (ANOVA) to evaluate the significance of differences observed among fiber sizes (125 μm, 250 μm, and 500 μm) and fiber contents (10%, 20%, and 30%) with control sample (HDPE/NR 60/40 blend without fiber) set up in the study. The software IBM SPSS Statistics Data Editor 20 was used to perform the calculation.

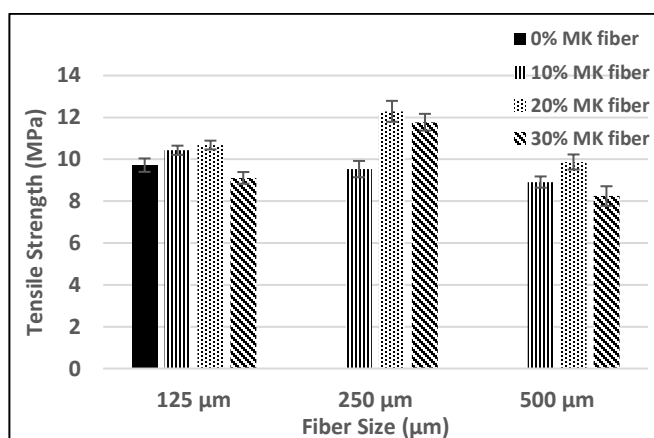
## RESULTS AND DISCUSSION

### Tensile Properties

The tensile strength for fiber sizes of 125  $\mu\text{m}$ , 250  $\mu\text{m}$ , and 500  $\mu\text{m}$  are shown in Fig. 2. Fiber contents for each size of fiber were varied at 10%, 20%, and 30%. The tensile strength at 10% of fiber content for a size range of 125  $\mu\text{m}$ , 250  $\mu\text{m}$ , and 500  $\mu\text{m}$  showed a 5%, 22.5%, and 17.5% lower tensile strength, respectively, than at 20% of fiber content. These results were attributed to volume take up by the presence of fiber at a low fiber content. A low tensile strength were attained as fiber were not enough to restrain the matrix. A decrease of tensile strength at 5-10% of fiber content was also reported by Ahmad *et al.* (2006)

The tensile strength for every size of fiber was optimized at 20% of fiber content. Similar reports by El-Shekeil *et al.* (2014) and Prasad *et al.* (2015) also achieved the maximum tensile strength at 20% fiber content. Good fiber distribution and dispersion also facilitated the strength of the composites. As mentioned by Ozturk (2010), uniform stress distribution, which depends on volume of fibers, produces good mechanical properties in composites. As fiber content increased up to 20% for fiber size 250  $\mu\text{m}$ , the thermoplastic composite exhibited the maximum tensile strength. The stronger surface interaction between the fiber and matrix contributed to less fiber agglomeration when observed at that size and fiber content. Fibers were also highly dispersed in the matrix. Tensile strength for fiber size 500  $\mu\text{m}$  was also optimized at 20% fiber content as less agglomeration of fiber occurred at this loading of fiber.

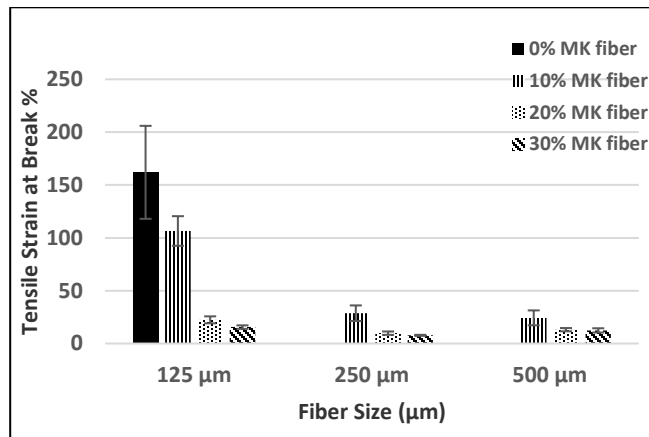
The addition of fiber content up to 30% for a size range of 125  $\mu\text{m}$ , 250  $\mu\text{m}$ , and 500  $\mu\text{m}$  showed a 14.7%, 4%, and 16.5% drop in tensile strength. At 30% of fiber content, an additional increase of fiber content caused more fiber agglomeration and poor fiber dispersion. Hashim *et al.* (2016) reported that higher fiber content increases the fiber pull out in composites, causing a reduction in tensile strength. Furthermore, as more fiber was added, more fiber ends were produced. This led to a reduction of tensile strength, which agreed with previous studies (Ahmad *et al.* 2013; Prasad *et al.* 2015).



**Fig. 2.** Effect of fiber size and fiber content (%) on tensile strength of Mengkuang reinforced 60/40 HDPE/NR composites

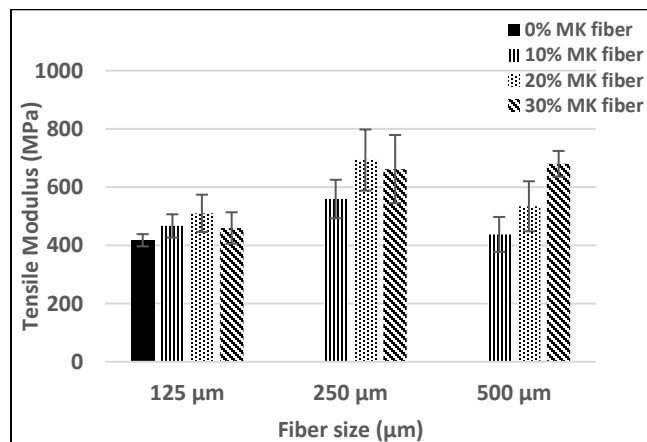
Figure 3 shows the tensile strain at break. Results showed that the tensile strain at break for 10% fiber content was decreased simultaneously as the fiber size was increased. This was attributed to weak internal interaction of larger fiber size, and an inability of a

larger fiber size to withstand the load transferred from the matrix. Thus, the increase of fiber content up to 20% and 30% for all sizes lowered the tensile strain at break. As MK fiber incorporated in the matrices, the rigidity of the composites restricted the flexibility of matrices HDPE and NR. According to Prasad *et al.* (2015), incorporation of the brittleness of natural fiber created an obstacle to polymer chains mobility in the matrix. The composites became stiffer as the ratio of elasticity matrix phases were replaced by increasing of fiber content.



**Fig. 3.** Effect of fiber size and fiber content (%) on tensile strain at break of mengkuang reinforced 60/40 HDPE/NR composites

As shown in Fig. 4, the tensile modulus at fiber sizes 125 μm, 250 μm, and 500 μm at different fiber content from 10% to 30% were observed. Tensile modulus explains the stiffness of the composite that contributes from the amount of fiber content and the filler aspect ratio. Stark and Rowlands (2003) reported that aspect ratio has a largest effect on wood polypropylene composites.



**Fig. 4.** Effect of fiber size and fiber content (%) on tensile modulus of mengkuang reinforced 60/40 HDPE/NR composites

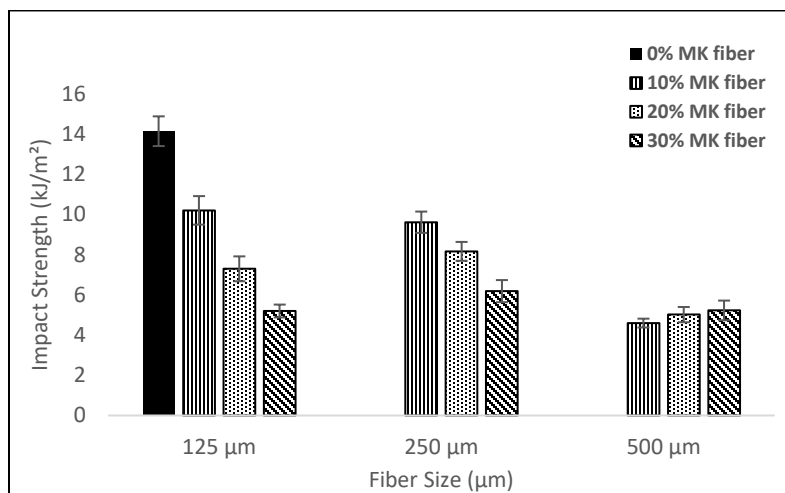
The optimum tensile modulus at 125 μm and 250 μm was observed at 20% fiber content, whereas the highest tensile modulus in the system occurred at 250 μm and 20% fiber content. The increased fiber interaction with the matrix with adequate fiber content

increased the tensile modulus. Prasad *et al.* (2015) on coir fiber/ LDPE also reported the optimum tensile modulus and tensile strength were at 20% of fiber content. A slight decrease to 9.9% was observed as fiber content increased from 20% to 30% at fiber size 125  $\mu\text{m}$ , this might be due to the increasing of fiber agglomeration at particular condition.

Tensile modulus slowly increased when the fiber size increased from 125  $\mu\text{m}$  to 250  $\mu\text{m}$  and 500  $\mu\text{m}$  at 30% fiber content. The same result occurred at 500  $\mu\text{m}$  of fiber size; tensile modulus increased with the increase in fiber content. The result was dominated by the volume fiber factor. Increasing fiber content meant reducing the matrix content, whereas reducing the ductility meant the composite would become stiffer with more fiber. Furthermore, there was less fiber-matrix interaction at this size of fiber. A similar result was also reported by Zahari *et al.* (2015).

### Impact Strength

The influence of mengkuang fiber content and fiber size on the impact strength of the composite is shown in Fig. 5. At 10% fiber content the impact strength decreased with increased fiber size. The weak adhesion of bigger fiber size hindered the efficiency of stress transferred. As the fibers were added to the matrix, the volume taken up by the fiber caused a deformation to the composite, and it interrupted the local motion of the polymer chains in matrix. A study of oil palm empty fruit bunch fiber reinforced polymer composite materials by Hassan *et al.* (2008) revealed that impact strength were decreased with increasing of fiber size.



**Fig. 5.** Effect of fiber size and fiber content (%) on impact strength of mengkuang reinforced 60/40 HDPE/NR composites

At 20% fiber content the highest impact strength at 250  $\mu\text{m}$  was shown, followed by 125  $\mu\text{m}$  and 500  $\mu\text{m}$ . This was probably due to fiber-matrix surface interactions efficient in stress-absorbing capacity. At 30% of fiber loading, for all sizes of fiber, showed almost constant impact strength, due to the weak ability of the higher volume of fiber to absorb the energy transferred. The increased fiber content lowered the matrix content to absorb energy given. This result is consistent with Rozman *et al.* (1998) and Jamil *et al.* (2006), which showed that impact strength decreased with increasing of fiber content.

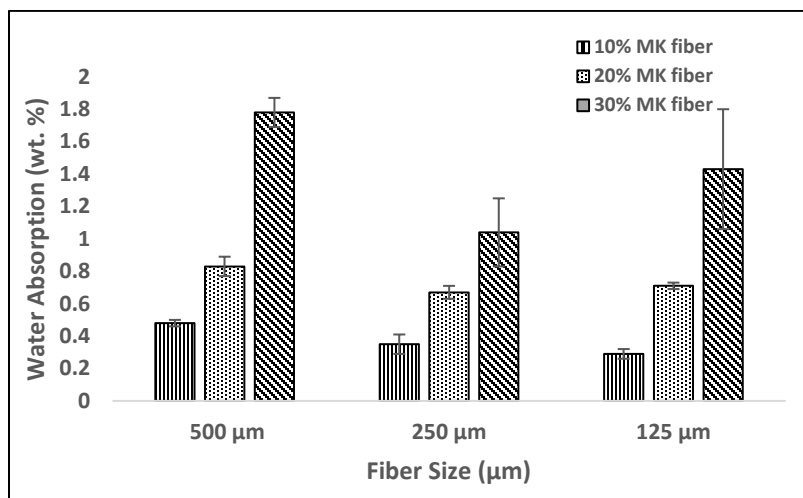
At a 250  $\mu\text{m}$  fiber size, the impact strength dropped steadily with the increase of fiber content. It was due to the efficiency of the stress absorbing capacity once the fiber

was entered into the matrix. As fiber content increased in the matrix, the fibers tended to interact with each other into bundles and interrupted the fiber dispersion in the composites.

At a 500  $\mu\text{m}$  fiber size, the lowest impact strength was shown due to weak surface interaction. There was less energy needed to overcome the inter particle interaction that exhibited a lower impact strength. The weak surface adhesion between fiber and the matrix initiated the crack upon the energy transferred to the composite. As reported by Syafri *et al.* (2011), impact strength was decreased due to poor interfacial bonding between fiber and matrix.

### Water Absorption

All lignocellulosic fibers have low resistance to water absorption due to the many O-H groups in the lignocellulosic fiber chemical structure. After 24 h immersion in distilled water, there was a noticeable effect of fiber content on water absorption test results, as shown in Fig. 6.



**Fig. 6.** Effect of fiber size and fiber content (%) on water absorption of mengkuang reinforced 60/40 HDPE/NR composites

The lowest water absorption rate was achieved at 10% fiber content. There was only a slight reduction in the rate of water absorption when the size of fiber was reduced from 500  $\mu\text{m}$ , 250  $\mu\text{m}$ , and 125  $\mu\text{m}$ . When the volume of the fiber doubled to 20% fiber content, the rate of water absorption was increased to 73%, 91%, and 145% for the size of 500  $\mu\text{m}$ , 250  $\mu\text{m}$ , and 125  $\mu\text{m}$ , respectively. When the fiber content was doubled to 30%, the rate of water absorption showed a dramatic increase up to 270%, 197%, and 343% for each fiber size of 500  $\mu\text{m}$ , 250  $\mu\text{m}$ , and 125  $\mu\text{m}$ , respectively. As more lignocellulosic fibers were added in composite, more hydrogen bonds formed between molecules of water and O-H groups of the lignocellulosic fibers. Huner (2015), Zahari *et al.* (2015), and Chen *et al.* (2016) also found that the rate of water absorption increased with increased fiber content.

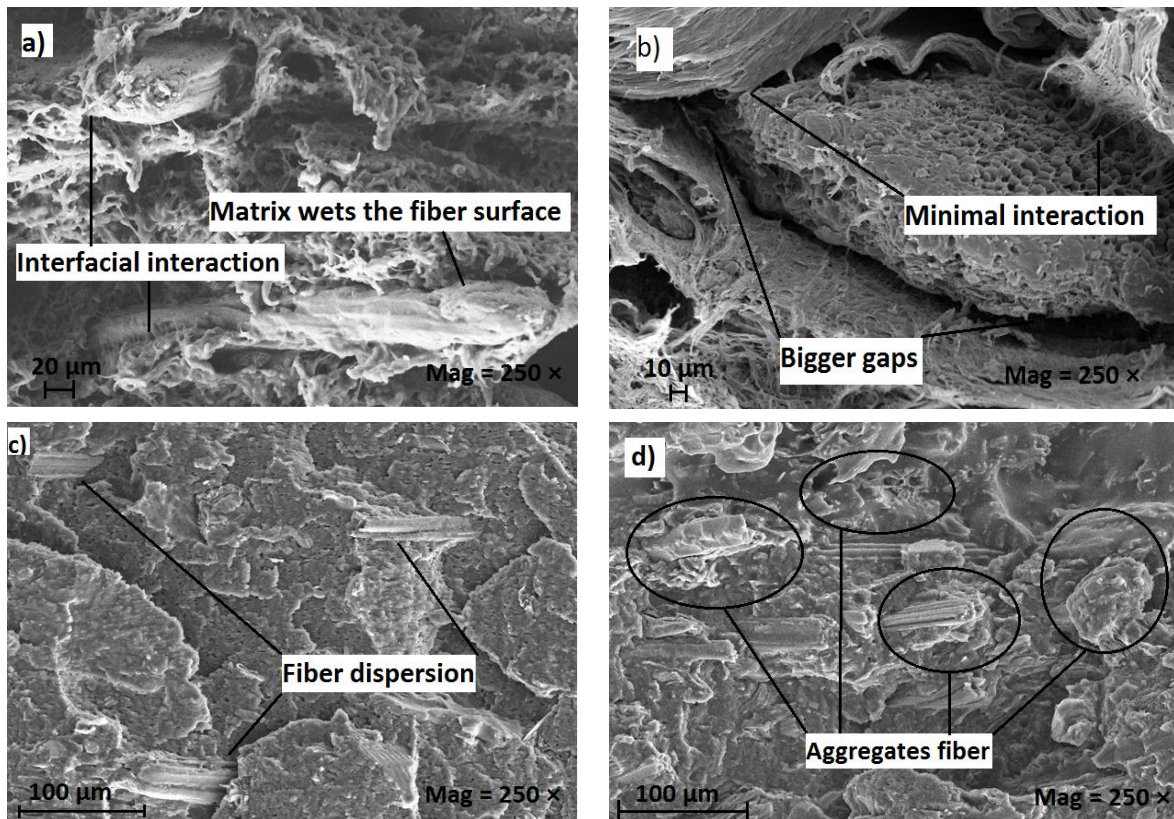
The overall result according to the size showed the highest water absorption demonstrated by the fiber size of 500  $\mu\text{m}$ , followed by 125  $\mu\text{m}$  in size, and the lowest at fiber size of 250  $\mu\text{m}$  for all fiber content at 30%, 20%, and 10%. This was due to the mengkuang composites that were blended using a large size of 500  $\mu\text{m}$ , thus forming less surface interaction between the matrix and fiber. These led to the formation of voids and micro cracks, which meant that more water diffused into the composites (Dhakal *et al.*



2006; Gerald Arul Selvan and Athijayamani 2016). Meanwhile, a 250  $\mu\text{m}$  fiber showed a lower water absorption rate due to stronger surface interaction which formed less void and micro cracks between matrix and fibers compared to fiber size at 500  $\mu\text{m}$ . FESEM imaging showed a good fiber distribution, and interaction increased at 250  $\mu\text{m}$  size. Mohebbi *et al.* (2010) reported voids and micro cracks at interphase which caused more water to penetrate and subsequently thickness swelling. However, the composite at a fiber size of 125  $\mu\text{m}$  absorbed more water than the composite with a fiber size of 250  $\mu\text{m}$ . A bigger surface area of the fiber exposed more hydroxyl groups to form hydrogen bonds with water molecules. The fibers also tended to bond with each other to form fiber bundles that caused the interaction between fiber-matrix phase to be reduced. This situation resulted in large micro cracks, which absorbed more water in the composite.

### Morphology

Field emission scanning electron microscope (FESEM) micrographs of fractured surfaces from the tensile and impact test for thermoplastic mengkuang composites were carried out to observe the morphology of the thermoplastic mengkuang composites. The fractured surfaces of thermoplastic composites are shown in Fig. 7. Figure 7(a) shows the tensile fracture surface for fiber size range of 125 to 250  $\mu\text{m}$  at 20% fiber content ( $\times 250$ ). Wetting of fiber by the matrix and the availability of surface interaction between fiber and the matrix were observed in the micrograph.



**Fig. 7.** Field Emission Scanning Electron Microscopy (FESEM) micrograph thermoplastic mengkuang composite. a) Tensile fracture surface for fiber size range of 125-250  $\mu\text{m}$  at 20% fiber content ( $\times 250$ ). b) Tensile fracture surface for fiber size range of 250-500  $\mu\text{m}$  at 20% fiber content ( $\times 250$ ). c) Impact fracture surface for fiber size less than 125  $\mu\text{m}$  at 10% fiber content ( $\times 250$ ). d) Impact fracture surface fiber size range less than 125 at 30% fiber content ( $\times 250$ ).

Wetting of fiber by the matrix and the availability of surface interaction between fiber and the matrix were observed in the micrograph. The improvement of wettability of fiber either by physical or chemical treatment further will improve the interfacial strength (Pickering *et al.* 2016). Figure 7(b) shows tensile fracture surface for fiber size range of 250 to 500  $\mu\text{m}$  at 20% fiber content ( $\times 250$ ). It shows bigger gaps with a minimal interfacial interaction of fiber and matrix. Figure 7(c) shows impact fracture surface for fiber size less than 125  $\mu\text{m}$  at 10% fiber content ( $\times 250$ ) with fiber dispersion in the matrix. Figure 7 (d) shows impact fracture surface fiber size range less than 125 at 30% fiber content ( $\times 250$ ). It shows the increase of fiber agglomeration and poor fiber dispersion in the matrix with increased fiber content.

### Statistical Analysis

A summary of statistical analysis using ANOVA is shown in Table 2. A one-way analysis of variance ANOVA was conducted to compare the effect of fiber size and fiber loading on tensile strength, tensile strain at break, tensile modulus, and impact strength, as well as water absorption. Out of ten analyses carried out, only three properties were significantly different by fiber size and wt. % fiber content. There were significant differences among wt. % of fiber content on tensile strain at break,  $p = 0.024$  and impact strength,  $p = 0.023$ , respectively. A significant difference was also found for the effect of water absorption and fiber size,  $p = 0.004$ .

**Table 2.** Summary of ANOVA Table

Mechanical/ Physical properties	Factor Variable	Sum of Squares	Degree of freedom (df)	Mean Square	F- ratio	P- value
Tensile Strength (MPa)	Fiber Size ( $\mu\text{m}$ )	3.296	2	1.648	0.901	0.455
	wt. % fiber Content	7.320	3	2.440	2.072	0.205
Tensile Strain at Break (%)	Fiber Size ( $\mu\text{m}$ )	3158.147	2	1579.074	2.157	0.197
	wt. % fiber Content	18534.624	3	6178.208	6.761	0.024*
Tensile Modulus (MPa)	Fiber Size ( $\mu\text{m}$ )	19471.886	2	9735.943	1.268	0.347
	wt.% fiber Content	55385.73	3	18461.910	2.692	0.140
Impact Strength (kJ/ m <sup>2</sup> )	Fiber Size ( $\mu\text{m}$ )	10.114	2	5.057	1.218	0.360
	wt. % fiber Content	64.427	3	21.476	6.856	0.023*
Water Absorption (wt. %)	Fiber Size ( $\mu\text{m}$ )	1.683	2	0.841	16.457	0.004*
	wt. % fiber Content	0.181	2	0.091	0.301	0.751

\*  $P < 0.05$  = significant difference

## CONCLUSIONS

1. Fiber size and percentage of fiber content both influenced the mechanical and physical properties, as well as morphology of mengkuang reinforced thermoplastic natural rubber composites.
2. The optimum tensile strength and tensile modulus were obtained at a fiber size range of 125 to 250  $\mu\text{m}$  at 20% fiber loading.
3. The highest tensile strain and lowest water absorption measurements were obtained at 10% fiber content for all sizes range of 125 to 500  $\mu\text{m}$ .
4. Impact strength decreased with increasing percentage of fiber content at fiber size less than 125  $\mu\text{m}$  and 125 to 250  $\mu\text{m}$ .
5. There were statistically significant differences as a result of different fiber size on water absorption,  $p = 0.004$  and wt. % fiber content on impact strength,  $p = 0.023$  and tensile strain at break,  $p = 0.024$ .

## ACKNOWLEDGMENTS

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