Size Effect of Oil Palm Fibers on Tensile Properties of Oil Palm Fiber-Reinforced Polypropylene Composites

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Abstract

In this study, the Malaysian oil palm fibers (OPF) were extracted from the empty fruit bunch (EFB) and pulverized before being used as a reinforcement of plastic composite materials. 10 wt.% and 25 wt.% of fibers were incorporated with polypropylene (PP) and maleated polypropylene (MAPP) to produce a composite material. For comparison purpose, a virgin PP named as Neat PP was also fabricated as a benchmarking sample. From the tensile test result, it was firstly confirmed that 25 wt.% of fiber loading brought higher tensile properties to the composites. This study secondly aims to investigate the effect of different fiber size on tensile properties of OPF reinforced PP composite materials. The pulverized OPF was mechanically classified into several size ranges using three different mesh sieves, 45μm, 90μm, and 180μm, respectively. The fibers were then mixed with PP and MAPP before molded into the desired shape via an injection molding machine. The tensile and impact tests were carried out for each type of sample in order to understand the influence of fiber size on the tensile and impact resistance behavior of the OPF reinforced PP composite materials.

1 INTRODUCTION

Ongoing research on composite materials has paid an extra attention to natural fiber based composites, reinforced with kenaf, flax, hemp, ramie and so on, as a high potential material to be further developed and applied. That is, the improvements on mechanical properties of the natural fiber based composites have often been reported in the literature in recent years [1,5-7]. Poor adhesion between fiber surface and polymer matrix, high moisture absorption due to the nature of the hydrophilic natural fibers, and variations of natural fiber's parameters, i.e. unstable properties due to different origin plant, water intake during growing process, harvesting time and so on, are among the issues related to using natural fiber as the reinforcement constituent in polymer composite. To some extent, current and ongoing research has established certain solutions on dealing with the issues mentioned above such as the use of compatibilizer or coupling agents as a *bridge*, i.e. an interactions between the anhydride groups of maleated coupling agents and the hydroxyl groups of natural fibers, to improve the incompatibility of natural fibers and polymeric resin. T.J Keener et al. [1] in their study on maleated coupling agents for natural fiber suggested that the optimization of 3wt.% of coupling agents in composite materials can greatly enhance the properties of natural fiber based polymer composites. Recently, the natural fibers that extracted from the plantation residues such as oil palm, bagasse, coconut/coir, pineapple leaf, rice husk and straw, and many others have attracted attention.

The production of palm oil is increasing by years due to high demand of diverse products using it as an organic raw ingredient. Palm oil has been used for various products including personal care products, cosmetics, cleaning liquids, and also as an ingredient in foods and beverages. In 2011, Indonesia and Malaysia accounted for approximately 86% of global palm oil production [2]. The increasing number of the palm oil production may also escalate the number of agricultural waste such as fronds and trunks, together with the palm oil residues, i.e. EFB, kernels and mesocarp fibers. In total, approximately 91 million metric tons of residues and wastes were produced every year and EFB correspond to about 20%

of this total [3]. Because of the arising aware of environmental problems due to the abundance of the agricultural waste, and to maximize the use of EFB, the fiber that extracted from the EFB, i.e. OPF, appear to be attractive enough to use as a reinforcement in fiber-filler polymer composite materials.
Azman Hassan et al. [4] in their review article on OPF-polymer composites mentioned several issues that are generally raised when it comes to natural fiber composites, i.e. the compatibility between OPF and matrix, and the moisture absorption. The optimization of OPF fiber and compatibilizer might resolve some of the related issues mentioned, that even lead to several consequences on the physical and mechanical properties of the composite material. On the other hand, fiber length or in certain literature are described as the aspect ratio of the fiber, is one of the parameter that significantly contribute to the performance of the composite materials. Amir Nourbakhsh et al. [5] suggested in their study on the effect of wood flour particle size on the mechanical properties of the particulate-filled polymer composite, that smaller particle size, approximately 0.25mm in size, bring excellent mechanical properties to the wood flour-PP composites. On the other hand, Sébastien Migneault et al. [6] studied the effect of fiber length on processing and properties of extruded wood fiber/HDPE (*high density polyethylene*) composites. The result implies that increasing fiber length improved both tensile and flexural properties of the wood plastic composites (WPC). As it known, WPCs are successfully used as the material for home furnishing such as kitchen cabinet, cladding, fence, flooring and decking, besides a competitive material for automotive interior parts and potentially for other non-load bearing application of wood. Thus, this study aims to investigate and identify the possibility of OPF to be utilized for structural material in the same way as wood in WPCs application. The mechanical properties of OPF filled PP composites, hereinafter referred as palm/PP composites, made with different loads and sizes of OPF using an extrusion and injection-molding process were investigated. Further analysis on fracture surface of the specimen was carried out using a field emission scanning electron microscopy (FE-SEM) and a laser microscope.

2 EXPERIMENTAL

2.1 Materials

The raw OPF was obtained in 5 to 10cm length was used as a filler in palm/PP composite material. As comparison, the average properties and compositions of OPF and other natural fibers are shown in **Table 1**. The OPF was treated with the stabilizer agent, malaeic anhydride polypropylene (MAPP) by Kayaku Akzo Corporation and mixed with J-107G type polypropylene produced by Prime Polymer Co. Ltd.

Table 1 The properties and compositions of several natural fibers [7-9].

Properties	OPF	Abaca	Bamboo	Sisal	Softwood
Tensile strength (MPa)	248	400	140-230	511-635	-
Density (g/cm^3)	0.7-1.55	1.5	0.6-1.1	1.5	-
Cellulose (%)	42.7-65	56-63	26-43	65	40-45
Hemicellulose (%)	17.1-33.5	20-25	30	12	7-14
Lignin (%)	13.2-25.31	7-9	21-31	9.9	26-34
Moisture (%)	-	1.5	8.9	11	-
Waxes (%)	4.5	3	-	2	-

2.2 Preparation of composites

Classification of OPF

Before mixing the OPF with the polymers granules, the fiber was classified into several ranges of size in order to investigate the influence of fiber size on the mechanical properties of the composites. For this purpose, the raw OPF was firstly cut into approximately 3cm long, then 2mm via a cutting machine, cutting mill P-15 (Fritsch Japan Co. Ltd.) and finally pulverized to 0.2mm using a cutter mill P-14

(Fritsch Japan Co. Ltd.). A medium size vibration sieve machine was employed to classify the pulverized OPF with the mesh opening size of 45μm, 90μm and 180μm, respectively. As the result, the fibers were classified into three sizes; less than 45μm, 45μm to 90μm and larger than 90μm, which are denoted as S, M and L, respectively.

Palm/PP composites specimen

The palm/PP composites specimen was fabricated by mixing the PP with the compatibilizer MAPP and each of the different class of OPF; S, M and L, in a twin screw internal mixer, Laboplasto Mill (Toyo Seiki Seisaku-sho Ltd.) at 30rpm and 190°C for 10 minutes. The compositions of reinforcement OPF filler and matrix are shown in **Table 2**. Mixed compound was then granulated and stored at 80°C for at least 4 hours. Using an injection molding machine BabyPlast 6/10P (Rambaldi Group), the granulated compound was then molded at 200°C into a 60mm×10mm×2mm dumbbell-shape and 60mm×10mm ×3mm rectangular shape of tensile and impact test specimen, respectively.

Table 2 Composition of Palm, PP and MAPP in Neat PP and composites specimens.

Specimens	Palm (wt.%)	PP (wt.%)	MAPP (wt.%)
Neat PP	0	100	0
P-10%	10	88.7	1.3
P-25%	25	74	1

2.3 Tensile Test

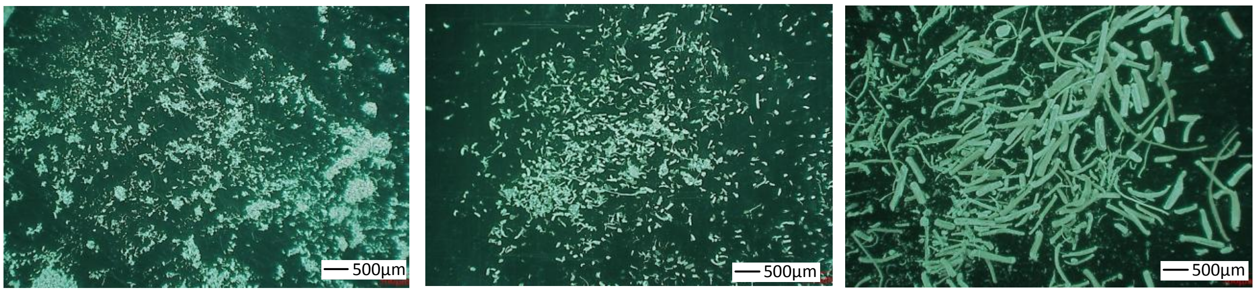
The tensile test was conducted using Servopulser hydraulic type EHF-FB-4LB (Shimadzu Corporation) at room temperature with three different cross-head speeds; 1.5 mm/min, 10 mm/min and 100 mm/min. Five specimens were tested for each type of the palm/PP composites and test conditions. Strain gauge (Kyowa Electronic Instruments Co., Ltd.) was installed at the middle of gauge length to measure the strain as the specimens were pulled.

2.4 Impact test

In this study, Charpy and Izod impact tests were carried out at room temperature for palm/PP composites using an Izod-Charpy impact test machine, CIT-25J-CI (A&D Company, Limited). The number of specimen was five for each type of composites. Both notched and un-notched specimens were tested for Charpy impact test while only un-notched specimen was tested for Izod impact test. The V-notch with radius of 0.25mm±0.05mm was made at the centre of the specimens using a PFA plastic forming machine (Yasuda Seiki Seisakusho Ltd.).

2.5 Analysis and observation

The fiber average size was technically measured for each classification class using a particle size distribution analyzer, LA-950V2 Partica laser scattering (Horiba Ltd.). **Figure 1(a)** to **1(c)** show the different of fiber dimension between each classification class; S, M and L respectively. Fracture surface of notched impact test specimen was observed using a field emission scanning electron microscopy, JAMP-9500F FE-SEM (JEOL, Ltd.) in order to understand the fracture mechanism of impact test specimens.



(a) **(b)** **(c)**

Fig. 1 OPF dimension between each of the classification; S, M and L, respectively.

3 RESULTS AND DISCUSSION

3.1 Tensile test results

Figure 2 shows the stress-strain diagram of representative curve of each type of palm/PP composite specimen at 10mm/min cross-head speed. As illustrated, up to 25wt.% of the OPF loading enhanced the stiffness and tensile strength properties of virgin PP. **Table 3** summarizes the average tensile properties of each type of palm/PP composite specimen at 10 mm/min cross-head speed. From **Table 3**, it is understood that tensile strength of the composites increases with the increasing of fiber size at any loading rate. Longer fiber length is conceivable to transfer the load more efficiently while smaller size of fiber has higher tendency to form a cluster, i.e. agglomeration of fiber increase the stress concentration that result in inhibition of stress transfer and accelerated the fracture behavior of the system.

Here, the effectiveness of classification of the fiber may be pointed out by the derivation of the coefficients of variation. The determination of this coefficient indicates the amount of variability in the production of palm/PP specimen, i.e. quality assurance of the palm/PP composites production. In this case, the coefficient of variation is calculated based on the tensile strength values of the palm/PP composites at different OPF sizes, which signify the main properties concern in structural application. It is calculated by dividing the standard deviation value of the maximum tensile strength with the average maximum tensile value for each classification range, as shown in equation 1.

$$\text{Coefficient of variation } CV = \frac{\text{standard deviation}}{\text{mean value}} \tag{1}$$

The calculation result is shown in **Table 3**. The data of Palm/PP composites contains 25wt.% of unclassified OPF fibers (P-25%-mix) was obtained from the previous study, showing that the coefficient of variation is approximately 1.93%, however no data collected for 10wt.% of OPF loading amount. Thus, coefficient values of P-25% specimens indicate that by classified the fiber into several ranges help to reduce the coefficient of variation and leads to the stabilization of composites properties with similar filler size contents.

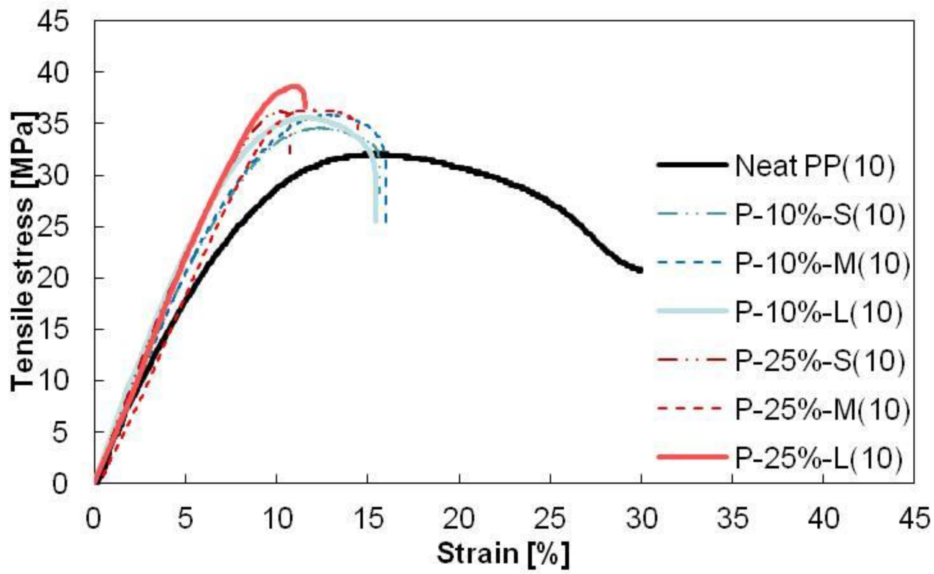


Fig. 2 Stress-strain diagram of representative curve of each type palm/PP composite specimen at 10mm/min of cross-head speed.

Table 3 Average tensile properties and coefficient of variation values of each type palm/PP composite specimen at 10 mm/min cross-head speed.

Specimen	Tensile strength (MPa)	Fracture strain (%)	Young's modulus (GPa)	Coefficient of variation (%)
Neat PP(10)	33.03	26.28	1.99	1.12
P-10%-S(10)	34.31	13.58	2.37	1.13
P-10%-M(10)	35.73	15.38	2.35	0.79
P-10%-L(10)	35.38	13.72	2.22	3.01
P-25%-S(10)	36.45	8.79	2.83	2.39
P-25%-M(10)	36.42	12.96	2.66	0.68
P-25%-L(10)	38.87	11.97	2.83	1.29
P-25%-mix(10)	38.36	9.98	0.204	1.93

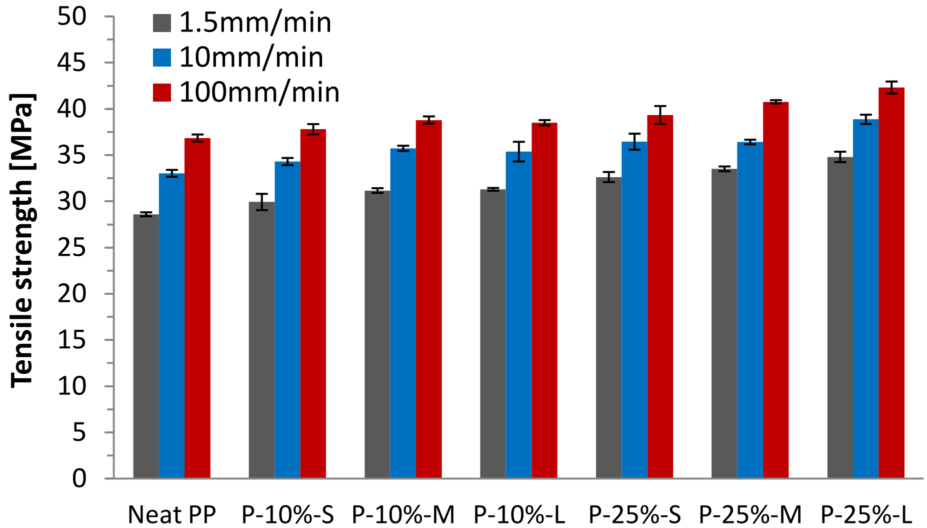
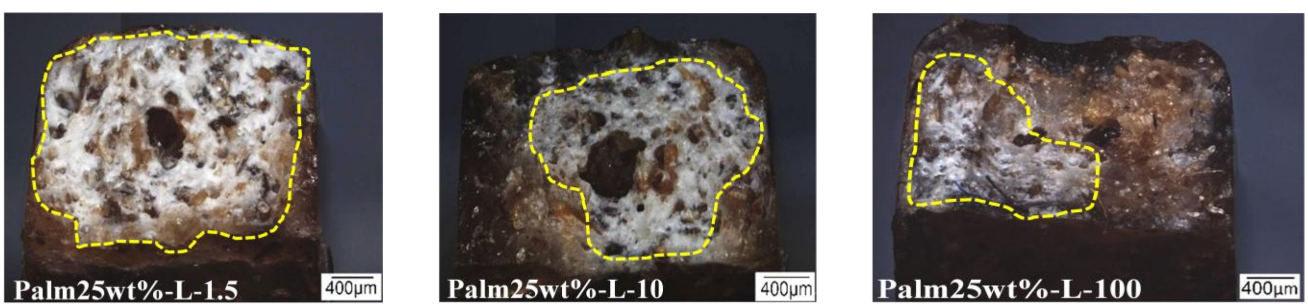


Fig. 3 The effect of OPF size at different cross-head speed on tensile strength of palm/PP composites.

In **figure 3**, the average values of tensile strength at different cross-head speed of each type of palm/PP composites specimens were presented. From the bar graph, it is understood that the tensile strength increases as the cross-head speed increases. This reflects the strain-rate dependency on the tensile properties of both virgin PP and composites specimens. As shown in **figure 4(a)** to **4(c)**, the whitened area at the fracture surface of the palm/PP composites specimen denotes the region where the crack growth along with the increasing of tensile load. It is confirmed that at higher cross-head speed of tensile test brings smaller crack growth region (**figure 4(c)**) as compared to the whitened area of the specimen tested at lower cross-head speed (**figure 4(a)** and **4(b)**). It is customarily to relate this phenomenon with the viscoelastic nature of PP, where it behaves as *hard* and *brittle* at higher tensile speed that results in lower fibrillation process (whitening), i.e. reduced the size of whitened area.



(a) **(b)** **(c)**

Fig. 4 Fracture surface observation on specimen tested at different tensile speed; (a) 1.5mm/min, (b) 10mm/min and (c) 100mm/min.

3.2 Impact test results

The effect of fiber size on impact properties of palm/PP composites was investigated. **Figure 5** illustrated the plotted data obtained from the notched specimen of Charpy impact test result. It is indicated that Neat PP has higher value of impact strength as compared to the composites material, however, at 25 wt.% of OPF amount in the palm/PP composites provides a nearly equal impact resistance performance to the virgin PP. This is expected that the fiber pulled-out mechanism with the increasing number of OPF loading amount, makes the energy absorption increase, i.e. the result from the Charpy impact test of notched specimens indicates that the higher loading amount of OPF might further increase the impact strength. **Figure 6** shows the scanning electron microscopy (SEM) images of fractured surface of notched Neat PP, 10wt.% and 25wt.% composites specimens. From the observation, holes from the fiber-pulled out fracture mechanism were identified and qualitatively confirmed that the number of hole rises as the amount of OPF increase, i.e. increase the energy absorption during the occurrence of specimen's failure. On the other hand, as for the notched specimens, there is no clear trend can be observed on the impact strength with the varying size of OPF.

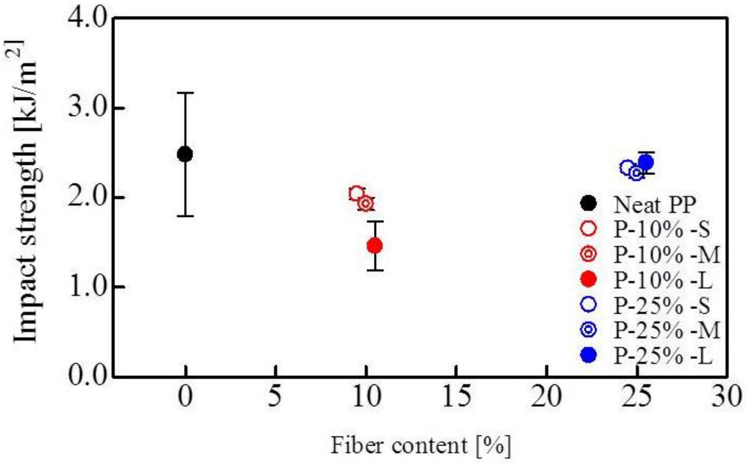


Fig. 5 Charpy impact test result of notched specimens.

Figure 7(a) shows the Charpy impact test result of un-notched specimens. The impact strength decreases with increasing OPF loading amount and Neat PP has the highest impact strength value. This trend agree with the previous literature [10] where the incorporation of fillers leads to decreasing of impact strength value due to the reduction of the material's ability to absorb energy during crack propagation. As plotted in **figure 7(a)**, the impact strength value of medium (-M) size fiber; P-10%-M and P-25%-M is slightly higher than that of smaller (-S) and larger (-L) size fiber. **Figure 7(b)** shows the Izod impact test result of un-notched specimens where the similar trends with Charpy un-notched specimen was confirmed. This implies that medium (-M) size fillers were well dispersed, and more surface area mechanism works during fiber pull-out. On the other hand, small (-S) size fillers tend to form secondary particles, i.e. agglomeration, and therefore the impact strength does not increase higher than the medium (-M) size fiber composites.

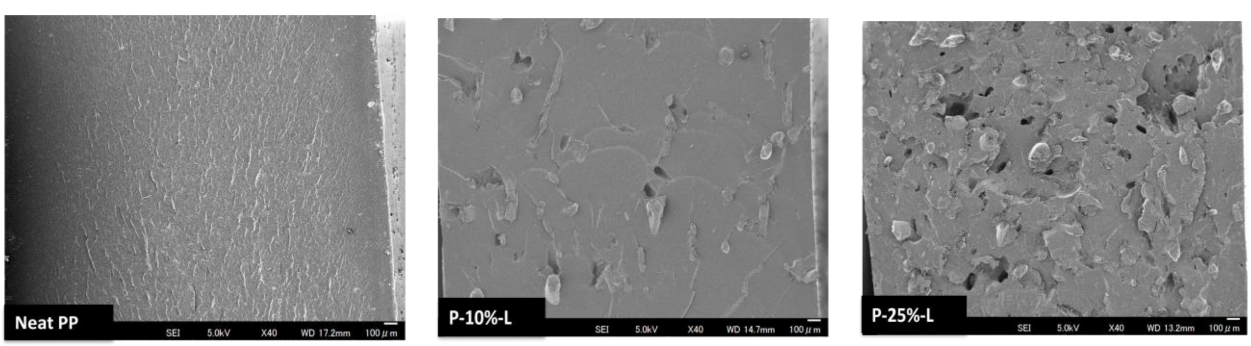
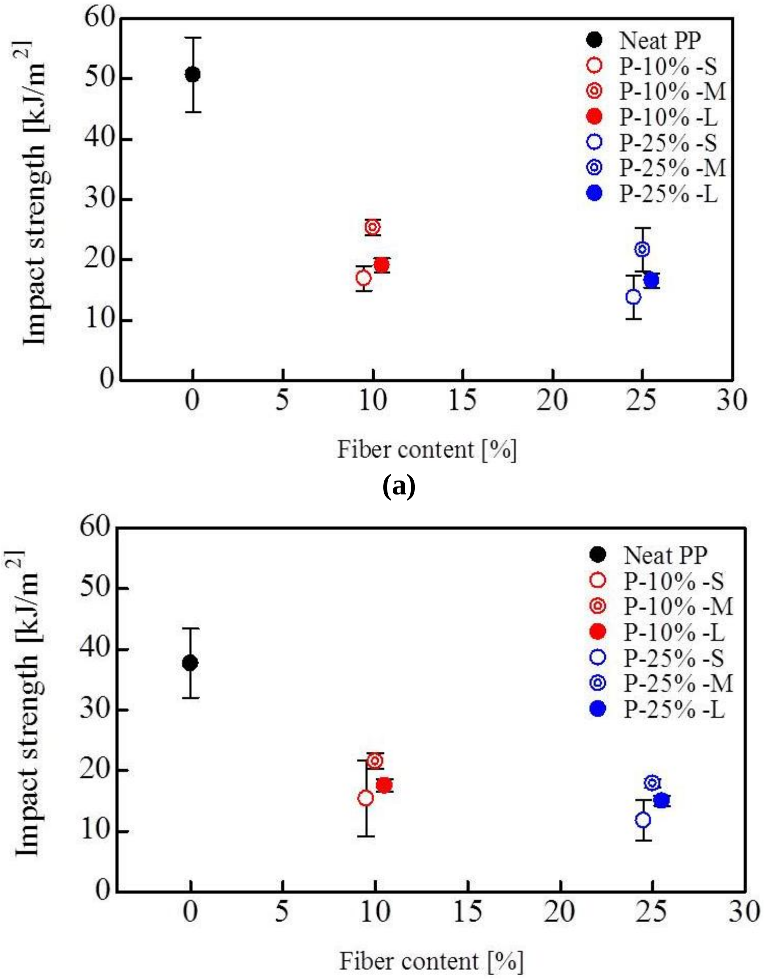


Fig. 6 Scanning electron microscopy (SEM) result of notched Charpy impact test specimen. Numbers of holes indicate fiber-pulled out at the fracture surface.



(a)

(b)

Fig. 7 (a) Charpy and (b) Izod impact test result of un-notched specimens.

4. Conclusion

In this study, Malaysian palm fiber was utilized as the reinforcement agent in PP matrix composite material. The fiber was classified into three different sizes in order to investigate the effect if fiber size on mechanical properties of the palm/PP composite materials. From the experimental result, it is understood that the tensile strength of the composites increase with increasing OPF amount. Larger size of OPF slightly increases the tensile properties of the palm/pp composites. Moreover, the incorporation of OPF into PP enhanced the tensile strength and stiffness properties of virgin PP specimens, however lead to decrement in the fracture strain value. Furthermore, the tensile strength value of each type specimen increases with increasing cross-head speed, i.e. signify the strain-rate dependency of Neat PP and composite specimens. From the impact test result of the notched specimens, there is no significant trend related with the different fiber size of the composite materials. It was observed from the un-notched specimens that, the impact resistance of the palm/PP composite material decreases with increasing OPF amount.

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