

International Symposium on Robotics and Intelligent Sensors 2012 (IRIS 2012)

## Effect of OPEFB Size on the Mechanical Properties and Water Absorption Behaviour of OPEFB/PPnanoclay/PP Hybrid Composites

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### Abstract

In recent years, lignocellulosic fibres are increasingly used as reinforcements in many thermoset and thermoplastic matrices for the production of low-cost and lightweight materials to be used as building materials, automotive components and consumer goods due to the environmental concern. In this study, polymer composites were prepared using oil palm empty fruit bunch (OPEFB) fibre at four different sizes (180  $\mu\text{m}$ , 250  $\mu\text{m}$ , 300  $\mu\text{m}$ , and 355  $\mu\text{m}$ ) as the secondary filler that was added in the clay and Polypropylene mixture as their matrix. XRD was employed to investigate the presence of clay structure in composites. Meanwhile Flexural and impact tests (both notched and un-notched) were performed according to ASTM D790 and D256 to assess their mechanical performances. Results show that the increase of OPEFB fibre size has increased its flexural strength and modulus at smaller OPEFB fibre size. However a decrease of both flexural strength and flexural modulus was observed at higher OPEFB fibre size, which is believed due to the poor interfacial bonding between OPEFB and matrix. Meanwhile the impact strength for notched samples has slightly increased with the increase of OPEFB size and no increase was observed for the impact strength of un-notched composites. As expected the increase of OPEFB fibre size has increased the water absorption capability of composites mainly due to the hydrophilic nature of OPEFB fibres. Finally, observation on the fracture surface of samples using scanning electron microscope indicates that the OPEFB fibre treatment has improved the interfacial bonding between OPEFB fibre and matrix.

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*Keywords:* Oil palm empty fruit bunch (OPEFB) fibre, Polymer composites, Mechanical properties, Alkali Treatment.

### 1. Introduction

Composite materials consist of two constituents namely a binder or matrix and reinforcement. The reinforcement normally is stronger and stiffer as compared to the matrix, while the matrix keeps the reinforcement in a set place. The binder also protects the reinforcement from environment. Generally, composite materials have excellent compressibility combined with good mechanical properties, making them versatile in a wide range of situations. The attractive features of composite material have been their high strength to weight ratio (low density and high tensile strength), high creep resistance, high toughness and low density [1]. In recent years, the use of composites has increased many folds in every sphere of life. This increase can be attributed to the versatile properties which these materials offer. The large scale and increasing use of composite material in the field of construction is not limited to just houses and other buildings but extend to automobiles, aeroplanes and boats. Recently, with rising oil prices and environmental considerations, there has been revival of the use of natural fibres replacement of synthetic fibres such as carbon and glass fibre in composite material. Clearly, natural fibres are the most popular materials of reinforcement's now days, as they satisfy the desired conditions and transfer

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strength to the matrix constituent, influencing and enhancing their properties as desired [2]. Natural fibres are renewable, completely recyclable, and cheap. Furthermore, they are fully biodegradable and do not leave residues that harmful for environment [4]. Besides, natural fibres as fillers have advantages over mineral fillers, as they are non-abrasive, require less energy for processing and reduce the density of furnished product [3].

Oil palm empty fruit bunch (OPEFB) fibre was extracted from empty fruit bunch (EFB) which is abundantly found as one of the natural fibres that form an interesting alternative for the most widely applied fibre in the composite technology due to its well known availability [5]. Increased utilization of abundant and renewable resources is strategically viable as it can contribute to the country's sustainability of energy supply while minimizing the negative impacts on the environment concern [6]. It can also solve the agriculture disposal problem in an environmental friendly manner while recovering energy and higher value of plastics for commercial applications. Currently there are several researches have been done which found that OPEFB fibre filled polymer composites are comparable to other natural fibre composites in term of its mechanical performance and environment advantage [7 - 9].

Therefore, the purpose of this work is to study and evaluate the mechanical properties of OPEFB/PPnanoclay/PP hybrid composites by varying the OPEFB fibre size and performed alkaline (NaOH) treatment on the OPEFB fibres to improve the interfacial bonding of OPFFB fibres and matrix.

## 2. Material and Experimental Procedure

### 2.1. Material

Pulverised oil palm empty fruit bunch fibre as shown in Fig. 1 (a) was supplied by Poly Region Sdn. Bhd. Polypropylene copolymer (PP) was purchased from Tree Q Communication Sdn.Bhd. Polypropylene nanoclay (PPnanoclay) pellets with 50% clay content were supplied by Nanocor Inc. in the form of master batch which ready for injection moulding. Sodium hydroxide (NaOH) was procured from MERCK Sdn.Bhd. Maleic anhydride grafted polypropylene (MAPP) manufactured by Sigma Aldrich was also used as the coupling agent.

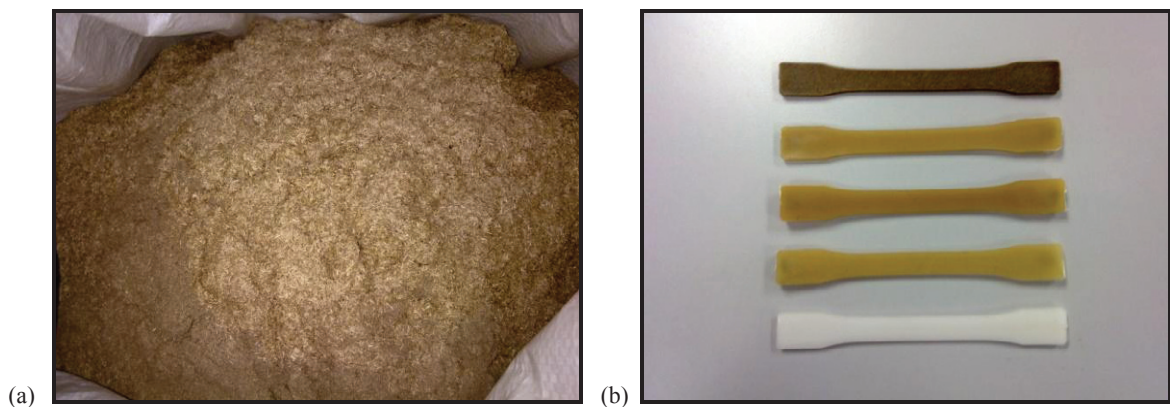


Fig. 1: Illustration for (a) pulverised OPEFB fibres and (b) injected dog bone samples.

### 2.2. Alkali treatment of Oil Palm Empty Fruit Bunch Fibre

Fibre treatment using Sodium Hydroxide (NaOH) was performed on the 355  $\mu\text{m}$  OPEFB fibres. Alkali treatment on OPEFB fibre leads to the reversible mercerization effect, which increases the amount of amorphous cellulose. It was also increases the adhesive nature of the fibre surface by removing natural and artificial impurities [10]. The OPEFB fibres were soaked in 1% NaOH solution at room temperature for 24 hours. After 24 hours, the OPEFB fibres were taken out and washed with distilled water for 7 times to remove excessive alkaline solution. The fibres were then dried in an oven at 80°C for 8 hours. Sodium hydroxide was applied in this project to remove the hydrogen bonding in the structure of the fibres cellulose, thereby increasing fibres surface roughness.

### 2.3. Preparation of Composites

The formulation of 6.7 phr PPnanoclay/PP composite with 78 wt% will be added to 20 wt% of OPEFB powder and 2 wt% of MAPP. The size of OPEFB powder was varied at four different sizes which are 180  $\mu\text{m}$ , 250  $\mu\text{m}$ , 300  $\mu\text{m}$  and 355  $\mu\text{m}$  and were dried in an oven at 80  $^{\circ}\text{C}$  for 8 hours. Polypropylene, PPnanoclay and MAPP pellets were dried mixed to ensure the material well mixed before compounding process. The compounding process was performed in a dispersion mixer at 180  $^{\circ}\text{C}$  follows the standard of thermoplastic temperature with rotating speed of 50 rpm and OPEFB powder was poured into the mixer after 20 min. Compounding process continued at same speed for 10 to 15 min, to ensure the OPEFB powders mixed thoroughly with the matrix before the compound was poured and cool at room temperature. After injection moulding, the test specimens were conditioned at room temperature,  $23 \pm 2$   $^{\circ}\text{C}$  for three days before test. The composite were fabricated using injection moulding technique into dumbbell-shaped with mould size 165 mm x 13 mm x 4 mm (length x width x thickness).

### 2.4. Mechanical Testing

Flexural and impact tests of the specimens were carried out. For each test and type of composites, ten specimens were tested and the average value is tabulated. The flexural properties were measured using a three-point bending test method on Instron 8032 Digital Control Testing Machine. The tests were carried out at room temperature at a crosshead speed 5 mm/min followed the standard of ASTM D790. Rectangular composites sample of 65 mm x 13 mm x 4 mm (length x width x thickness) were tested using a span length of 60 mm. The calculation for flexural modulus and flexural strength are given below:

$$\text{Flexural strength} = \frac{3WL}{2bd^2} \quad (1)$$

$$\text{Flexural modulus} = \frac{L^3\Delta W}{4bd^3\Delta S} \quad (2)$$

Where; W is ultimate failure load (N), L is the span between centres of supports (mm), b is the mean width of sample (mm), d is the mean thickness of sample (mm),  $\Delta W$  is the increment in load (N), and  $\Delta S$  is the increment in deflection (mm).

Izod impact test for notched and un-notched specimens was conducted according to ASTM D256 using a Universal Pendulum Impact Tester having dimension approximately 60 mm x 13 mm x 4 mm (length x width x thickness). The sample were rigidly mounted on the horizontal position and struck by a pendulum with a force of 5.5 J at the centre of the samples.

### 2.5. Water Absorption Test

Rectangular specimens having dimensions of 50 mm x 13 mm x 4 mm were prepared in order to measure the water absorption characteristics of the composites. An analytical balance was used for weight measurements. The specimens were dried and weighed prior to immerse in distilled water according to ASTM D 570-98. After immersion, the excess water on the surface of the specimens was wiped up using a soft cloth and the final weights of the specimens were then taken. Several measurements were done at regular interval time. The increase in the weight of the specimens was calculated by using the following equation:

$$\text{Water absorption (\%)} = \frac{\text{Final weight} - \text{Original weight}}{\text{Original weight}} \times 100 \quad (3)$$

### 2.6. X-ray Diffraction Analysis (XRD)

X-ray diffraction (XRD) analysis is mainly used in crystalline materials for structural identification. The diffractograms were scanned in range 2 theta ( $2\theta$ ) from  $5^{\circ}$  to  $35^{\circ}$  with the steps of  $0.02^{\circ}$  at room temperature that was performed using Rigaku X-ray diffractometer.

## 2.7. Scanning Electron Microscope (SEM)

The fracture surface morphology of composites was investigated using LEO 1455 VP SEM analyzer. Fracture surfaces of the specimens obtained from testing were sputter coated with gold prior to morphological observation. This test was carried out to determine dispersion of fibre in the matrix, adhesion between fibre and matrix and to detect the presence of any micro defect in the composites.

## 3. Results and Discussion

### 3.1. XRD analysis

X-ray diffraction (XRD) analysis was carried out for PPnanoclay/PP matrix and OPEFB/PPnanoclay/PP hybrid composites as shown in Fig. 2. The addition of OPEFB fibre in PPnanoclay/PP composites did not affect their crystallinity, which indicated by the same trend of XRD pattern. The peaks of interest were found to be from  $2\theta = 6^\circ$  to  $2\theta = 25^\circ$ . The observed diffraction curves of clay and polypropylene crystal structure at about  $6.7^\circ$ ,  $14^\circ$ ,  $16.8^\circ$ ,  $18.4^\circ$ ,  $21.8^\circ$ , and  $25.4^\circ$  of  $2\theta$  corresponding to the (000), (110), (040), (130), (111), and (150) planes. The shifting of the peak (000) for PPnanoclay/PP and OPEFB/PPnanoclay/PP composites to lower angle as compared with the peak of clay indicated an increase in interlayer spacing of silicate layers. The reduction in peak intensity as well as shifting of peak to lower angle suggested an increased disorder of the nanoclay layers in the nanocomposites [11]. The silicates nanolayers might be partially exfoliated and partially intercalated.

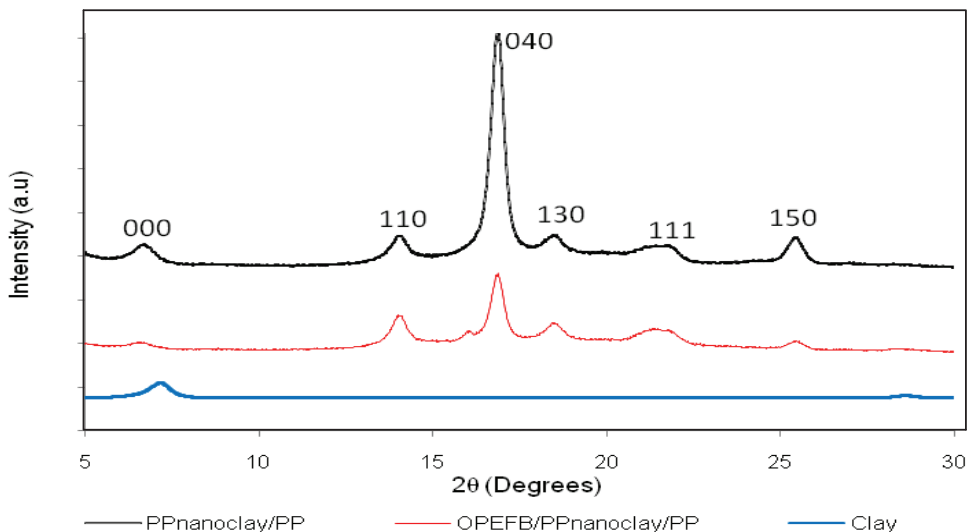


Fig. 2: XRD result of PPnanoclay/PP and OPEFB/PPnanoclay/PP composites.

### 3.2. Flexural Strength and Flexural Modulus

Flexural properties of OPEFB/PPnanoclay/PP composites are given in Fig. 3 (a), and (b). Both flexural strength and flexural modulus show the same trend, increased with the addition of OPEFB fibre at smaller size ( $180 - 250 \mu\text{m}$ ). The incorporation of  $180 \mu\text{m}$  OPEFB fibres in PPnanoclay/PP composites has increased the flexural strength to 38 MPa, 8% higher than the flexural strength of PPnanoclay/PP composite meanwhile 29% increase is observed for flexural modulus. However further increase of OPEFB size ( $300 - 355 \mu\text{m}$ ) has decreased their flexural strength and flexural modulus. This trend may be due to the poor bonding between OPEFB fibre and matrix hence reduces the ability of the OPEFB to absorb stress transfer. As highlighted by previous researcher [9], the degree of compatibility and interfacial bonding is dependent on a number of factors, such as the nature of lignocellulosic and thermoplastics as well as their composition, the aspect ratio of the fibre, the method of incorporating the lignocellulosic into the resin, the processing condition and on the treatment of the fibre with various chemicals such as compatibilizers or coupling agent. Larger OPEFB fibre size has more contact surface

with matrix as compared with small OPEFB fibre size, hence need more coupling agent to enhance the interface bonding. However the increase of coupling agent content may induce brittleness on the matrix that lead to lower mechanical properties. Thereby alkali treatment was performed on the 355  $\mu\text{m}$  OPEFB fibres as an attempt to improve interface bonding between OPEFB fibre and matrix. The result indicates slight increase of flexural strength and flexural modulus suggesting an interfacial bonding improvement. However more research needs to be done to determine the optimum processing parameter during fibre treatment such as soaking time and alkali concentration.

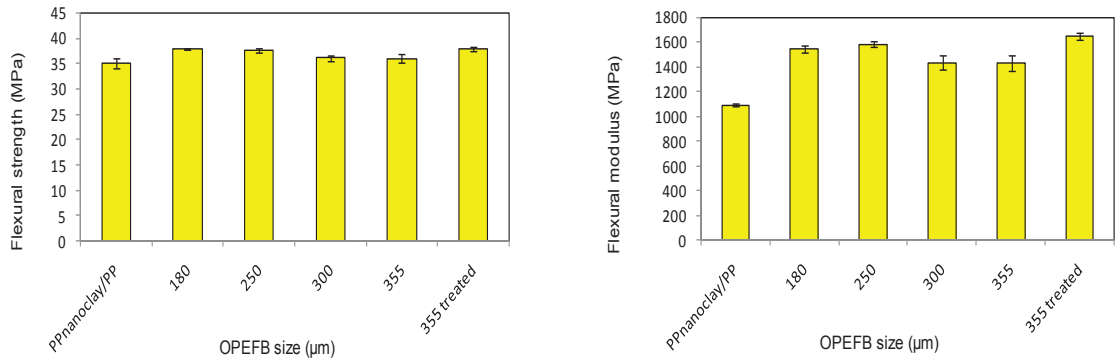


Fig. 3: Comparison of (a) flexural strength and (b) flexural modulus at different size of OPEFB fibres.

### 3.3. Impact Strength Notched and Un-notched

The impact strength of notched and un-notched OPEFB/PPnanoclay/PP composites at various OPEFB size is presented in Fig. 4 (a), and (b). Generally, the addition of OPEFB fibre in PPnanoclay/PP composites has drastically decreased the impact strength of OPEFB/PPnanoclay/PP hybrid composites. However for notched specimens the impact strength has increased with the increasing of OPEFB size, meanwhile no significant contribution from the OPEFB in OPEFB/PPnanoclay/PP composites indicated by the unchanged of impact strength for un-notched specimens. This is believed due to the low OPEFB loading (20 wt %) used in this study and it was agreed by [9, 12]. There are many other factors that contribute to the impact strength namely impact fracture mechanism which consists of filler debonding, filler fracture, matrix shearing and filler pull-out [12].

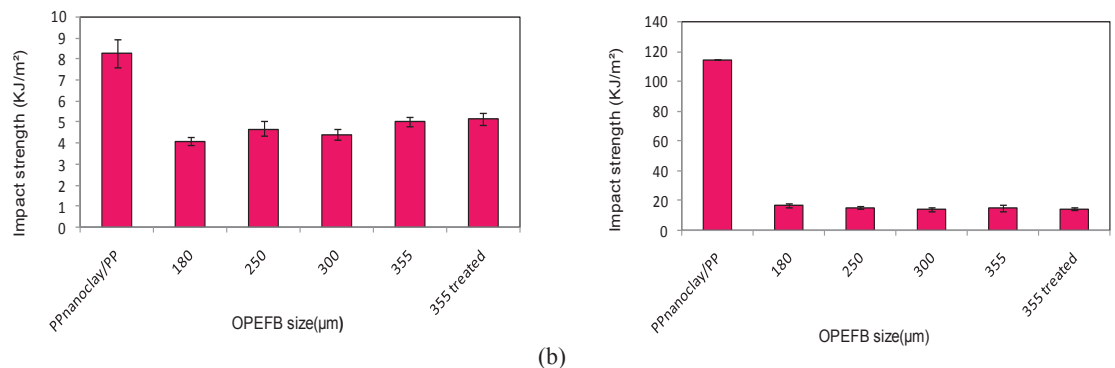


Fig. 4: Comparison of (a) notched and (b) un-notched testing at different size of OPEFB fibre on Izod impact test.

The un-notched Izod impact strength of PPnanoclay/PP composite showed significantly high impact strength, which was drastically decreased with the addition of 20 wt % of OPEFB fibre. The notched tip, which is the stress concentrating point, causes relatively low impact strength in the notched samples, thereby allowing the crack to propagate easily. Un-notched Izod impact energies were considerably larger than notched Izod impact energies. This is due to the different fracture process for notched and un-notched samples. The un-notched impact behaviour is controlled by fracture initiation processes

that, in turn, are controlled by stress concentrations at defects in the system. In other words, un-notched Izod impact energies are not only a measure of crack propagation but also of crack initiation [13]. Meanwhile notched impact behaviour is controlled by factors affecting the propagation of fracture initiated at the predominating stress concentration at the notched tip [13].

### 3.4. Water absorption characteristics

Water absorption behaviours of the composites in water immersion against the days are shown in Fig. 5. The incorporation of OPEFB fibres has significantly increased the water absorption behaviour of hybrid composites. This is well understood and agreed by most researchers is due to the hydrophilic nature of OPEFB fibre or other natural fibres. The highly hydrophilic nature of the OPEFB fibre owing to the free hydroxyl group present in the cellulose and lignin structures is the main contributor of absorbed water [4]. These hydroxyl groups can hold the water molecules, via hydrogen bonding, within the fibre cell wall [4]. Water absorption (%) increases with the increase of OPEFB size due to the poor interfacial bonding as was evident in the flexural strength and flexural modulus. This trend is also expected due to the pore structures of OPEFB fibre itself, hence has higher ability to retain water. Another region which can hold water is micro gaps or voids in the interface between the fibre and matrix phases as can be seen in Fig. 6 (b). Voids due to water vapour generation during hot melt processing can also contribute to the subsequent water absorption of the materials due to increased porosity [14]. Chemically treated OPEFB fibre reinforced PPnanoclay/PP composites showed lower water absorption compared to untreated one which may due to encapsulation of the OPEFB fibre into the matrix. The reduction of water uptake after 160 days is about 28% for the treated OPEFB fibres compared to the untreated OPEFB fibres. As also found by [4] this may due to the hydrophilic nature of OPEFB fibre that has substantially decreased upon its chemical treatment with Sodium hydroxide (NaOH).

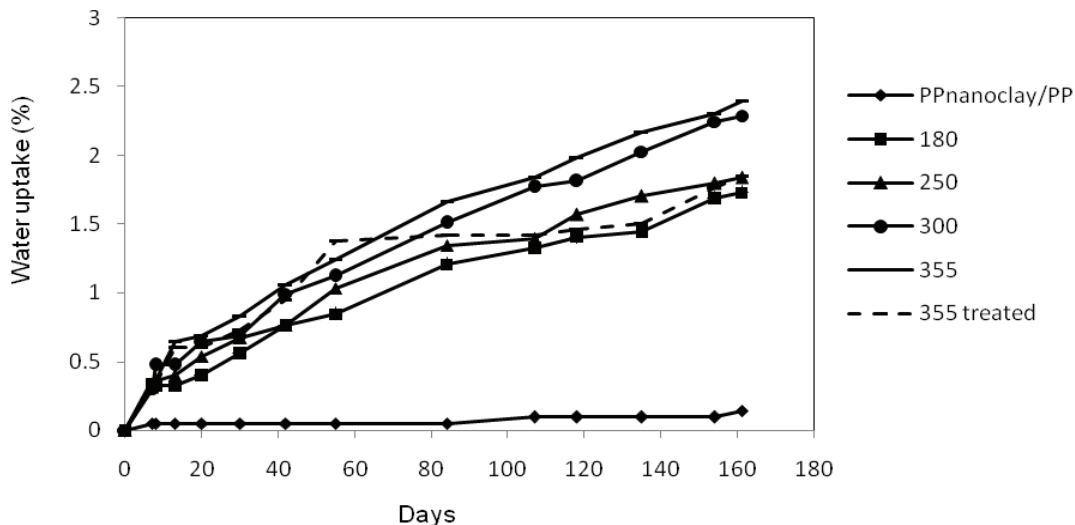


Fig. 5: The effect of OPEFB size on the water absorption behaviour for OPEFB/PPnanoclay/PP composites.

### 3.5. Fracture Surface Morphology

Fracture surface morphology of izod impact test specimens for treated and untreated OPEFB fibres are shown in Fig. 6. It has been observed that surface morphology of treated OPEFB/PPnanoclay/PP composites is smoother compared to the untreated OPEFB/PPnanoclay/PP composites. Several fibres are also seen encapsulated with matrix indicated by black arrows, meanwhile the black circle indicates fibre fracture. Those fracture surface characteristic implying good interfacial bonding between fibre and matrix. Untreated OPEFB/PPnanoclay/PP composites shows rougher fracture surface due to the fibre pull out shown by white circle indicating poor interfacial bonding. Closer observation on the fibre also can see gaps between OPEFB fibre and matrix, indicated by white arrows, this feature explains why the untreated OPEFB/PPnanoclay/PP composites has higher water absorption behaviour compared to the treated OPEFB/PPnanoclay/PP composites that is also agreed by other researchers [15].

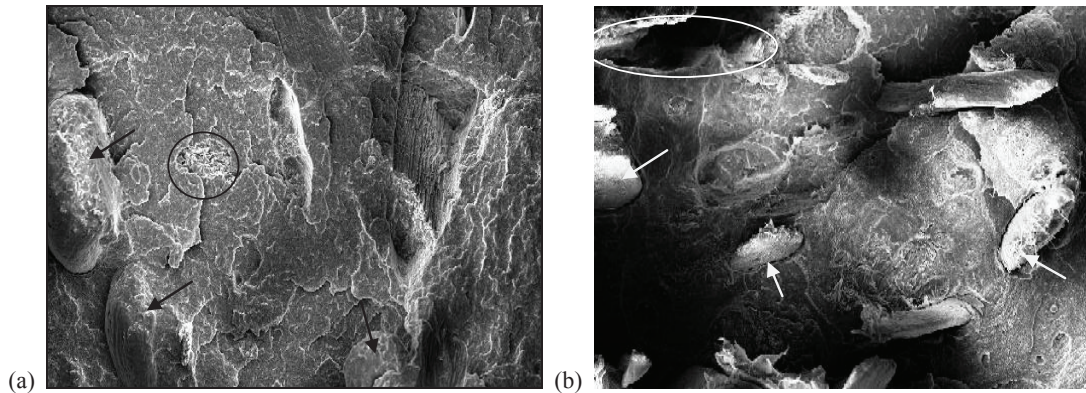


Fig. 6: SEM morphology of the notched Izod impact fracture surface for (a) treated OPEFB, and (b) untreated OPEFB at magnification of 100 x.

#### 4.0 Conclusion

The combination of oil palm empty fruit bunch fibres with PPnanoclay/PP matrix produced hybrid composite material that is competitive to synthetic composites. Flexural properties of OPEFB/PPnanoclay/PP composites increase with the increasing of OPEFB fibre size especially at smaller OPEFB size. Decrease of both flexural strength and flexural modulus at higher OPEFB fibre size is due to the poor interfacial bonding between OPEFB fibre and matrix. The impact strength for notched samples has slightly increased with the increase of OPEFB size and no increase was observed for the impact strength of un-notched composites. The increase of OPEFB fibre size has increased the water absorption capability of composites mainly due to the hydrophilic nature of OPEFB fibres.

#### Acknowledgements

The authors wish to thank Kementerian Pengajian Tinggi, for providing the research grant to UiTM under FRGS that has resulted in this article.

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