

Enhanced Rigidity of Natural Polymer Composite Developed from Oil Palm Decanter Cake

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A natural polymer composite (NPC) was developed from a palm decanter cake (OPDC), and its properties were determined. Oil palm decanter cake natural polymer composite (OPDC-NPC) samples were produced at different ratios of polypropylene (PP) and OPDC before being subjected to flexural, tensile, and water absorption tests. The results showed that by increasing the OPDC content from 5 to 15%, the rigidity as represented by the flexural strength and tensile strength increased from 29 to 43 MPa and 12 to 21 MPa, respectively. However, the flexural modulus decreased from 2230 to 1365 MPa and the water absorption rate increased from 0.16 to 0.44% because of the hydrophilic nature of the OPDC material. The rigidity of OPDC-NPC can thus be improved by increasing the OPDC content from 5 to 15%.

Keywords: Palm biomass; Oil palm decanter cake (OPDC); Polypropylene (PP); Natural polymer composite (NPC); Natural fiber content

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INTRODUCTION

Natural polymer composites (NPCs) were first introduced in 1970; since then, research on composites and related areas has grown and provided more insights into the development of new composite materials (Ashori 2008). To produce NPCs, natural fibers (NFs) such as agricultural waste can be mixed with various binders, such as polypropylene (PP), polyethylene (PE), polylactic acid (PLA), and polyvinyl alcohol (PVA). The material is then injected and molded under a suitable temperature (150 to 200 °C) and pressure (2 to 20 MPa).

Several advantages exist in utilizing NFs for NPC development, *e.g.*, low energy consumption, low density, low production cost, safe handling, renewability, and environmental friendliness (Ashori 2008; Singha and Thakur 2008; Mantia and Morreale 2011). In general, the primary constituents of NFs are cellulose, hemicellulose, lignin, and extractive components, and different types of NF contain different percentages of these components (Bledzki and Gassan 1999). However, the most important constituent affecting NPCs is cellulose, as it provides flexural strength and hardness to the NPC. There have been several studies conducted on NPC development using NFs such as flax, hemp, jute (Kabir *et al.* 2012) kenaf, sisal (Koronis *et al.* 2013), sisal, abaca (Nguong *et al.* 2013), empty fruit bunch (Khalid *et al.* 2008), and rice husk (Rosa *et al.* 2009). In NPC development, the purpose of using matrix binders such as PP, PE, and PLA is to hold the

NFs in position and to enhance the flexural modulus of the developed NPC (Kabir *et al.* 2012). Polymer-based matrix binders are easy to recycle using heat, and the properties of the recycled polymers are similar to those of virgin materials (AlMaadeed *et al.* 2012).

In the past few years, research on composites obtained from oil palm biomass has gained significant attention due to the increasing generation of oil palm biomass in tropical countries. In 2013, about 19.2 million tons of oil palm fresh fruit bunch (FFB) was processed to extract crude palm oil (CPO), resulting in large amounts of biomass such as oil palm frond (OPF), oil palm trunk (OPT), mesocarp fiber (MF), empty fruit bunch (EFB), palm oil mill effluent (POME), oil palm decanter cake (OPDC), and palm kernel shell (PKS) (Embrandiri *et al.* 2013). Among these types of biomass, OPDC is produced by the industry due to process improvements (*i.e.*, installation of the decanter machine) in the process of CPO production. The OPDC is produced from a decanter machine that is installed after the crude palm oil clarification process, the function of which is to improve the separation of the oil and the solid. Prior to using the decanter machine, the remaining oil from the sludge was separated by using a hydrocyclone machine only. Two types of decanter machines are currently used: a two-phase decanter and a three-phase decanter (Adam *et al.* 2014). The current uses of OPDC include composting, mulching, and animal feed production (Embrandiri *et al.* 2013). The chemical composition of OPDC is presented in Table 1. As can be seen in Table 1, the natural characteristics of OPDC material make it a promising candidate for NPC development.

Table 1. Chemical Composition of Oil Palm Decanter Cake

Composition	Reference	
	Ho <i>et al.</i> 1984	Razak <i>et al.</i> 2012
Cellulose (%)	24.87	21.61
Hemicellulose (%)	7.32	3.94
Lignin (%)	33.65	30.66

A previous study has shown that the OPDC (Adam *et al.* 2014) give promising results as compared to other established studies that used natural fiber such as jute/coir (Siddika *et al.* 2013), palm fiber (Goulart *et al.* 2011), EFB (Khalid *et al.* 2008), and rice husk (Prachayawarakorn and Yaembunying 2005) with the usage of polypropylene as a matrix. The primary objective of this study was to develop an oil palm decanter cake natural polymer composite (OPDC-NPC) by utilizing different percentages of OPDC. The mechanical and physical properties of OPDC-NPC, such as flexural modulus, flexural strength, tensile strength, and water absorption, were investigated to understand the effects of OPDC content on NPC strength.

EXPERIMENTAL

Materials

Fresh OPDC samples were obtained from FELDA Trolak Palm Oil Mill, Perak, Malaysia. The mill used a three-phase decanter system that separates solid, liquid, and oil. The solid fraction, known as OPDC, is produced in a solid form with high moisture content (about 68%). The samples were collected and stored in a freezer (-20 °C) before further

use. The purpose of freezing the OPDC was to maintain its fresh condition and to inhibit degradation. The polymer binder PP (Titan Petchem, Malaysia) was used in this study.

Methods

OPDC treatment

The OPDC was defrosted and dried at 103 °C in an oven (UNB-400 Memmert, USA) before treatment. The dried sample was ground to a fine powder and sieved to < 250 µm before further use. Because the OPDC material is highly lipophilic, the residual oil available in OPDC was first removed using the Soxhlet extraction process for 8 h. Hexane was used as the exchange solvent. The oil-free OPDC was washed several times with distilled water to remove the excess hexane. Then, the sample was quickly dried in an oven at 105 °C to remove the remaining moisture until it reached a constant weight.

NPC production

The dried and oil-free OPDC sample was compounded with PP at three different OPDC and PP concentrations (*i.e.*, 95% PP and 5% OPDC; 90% PP and 10% OPDC; and 85% PP and 15% OPDC). The OPDC and PP samples were mixed using a twin screw extruder (TSE-16 PRISM, USA) at 180 °C. Composite sheets of 3 mm thickness were produced using a compression hot press mould (Daylight Moulding, China) at 190 °C and 150 kg/m² pressure.

Flexural test

The flexural strength and flexural modulus of the OPDC-NPC were determined based on the standard method (ASTM D790 2010). Three samples at each percentage were prepared in a rectangular shape (3 mm thick, 12.7 mm wide, and 125 mm long). The three-point test was performed using a universal testing machine (Instron, UK). The rate of loading was 2 mm/min.

Tensile test

The tensile strength of the OPDC-NPC was determined using the standard method (ASTM D638 010). Three samples were prepared in a dumbbell shape (3 mm thick, 19 mm wide, and 165 mm long). The test was performed using a universal testing machine (Instron, UK). The load cell speed was 5 mm/min.

Water absorption test

The water absorption rate of OPDC-NPC was obtained based on the A standard method (ASTM D570 2010). Three samples at each percentage were prepared in a rectangular shape (3 mm thick, 25 mm wide, and 50 mm long). The test was conducted by soaking the sample for 24 h in water at room temperature.

Contact angle test

The contact angle measurement of OPDC was performed using OCA 15EC measuring instrument (DataPhysics, Germany) to determine its polarity. During the process, the oil-free OPDC was formed into a flat and rigid surface. One drop of water was placed on the OPDC surface, and the angle was obtained.

Field emission scanning electron microscopy

Morphological studies of OPDC-NPC were carried out with a field emission scanning electron microscope (Zeiss Supra 55VP, Germany) operating at an accelerating voltage of 5 kV. The samples were coated with gold using a sputtering method to avoid charging during the imaging process (Nazir *et al.* 2013).

Statistical analysis

The software SPSS (Version 19.0; IBM) was used to test for any significant differences in the results obtained using one-way analysis of variance (ANOVA). A 0.05 significance level was used. The mean values of flexural modulus, flexural strength, and water absorption of the samples produced were compared.

RESULTS AND DISCUSSION**Data Analysis**

ANOVA results are presented in Table 2. The statistical test revealed significant differences among the four types of materials investigated in terms of flexural strength, flexural modulus, tensile strength, and water absorption. The percentage of OPDC had a highly significant effect on the mechanical and water absorption properties of the OPDC-NPC ($p < 0.05$).

Table 2. ANOVA Table for Comparison of OPDC-NPC Properties among Different Fiber Percentages

Properties	OPDC Percentage	Mean value	Std Dev	F	p
Flexural Modulus	5%	2231	75	93.87	0.000
	10%	1601	111		
	15%	1441	115		
Flexural Strength	5%	30	3	9.27	0.006
	10%	37	3		
	15%	43	4		
Tensile Strength	5%	28	4	5.77	0.021
	10%	29	3		
	15%	33	3		
Water Absorption Rate	5%	0.16	0.04	47.24	0.000
	10%	0.20	0.02		
	15%	0.44	0.08		

Mechanical and Physical Properties

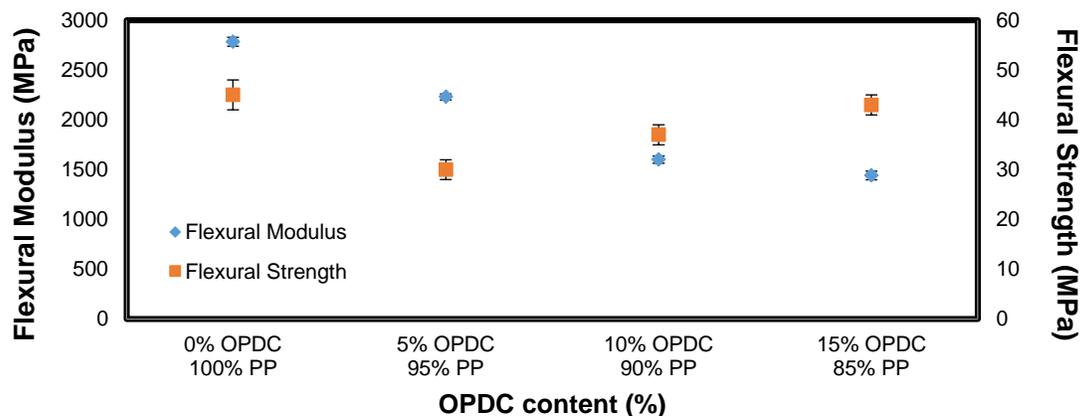
The effect of increasing OPDC percentage on OPDC-NPC in terms of flexural modulus, flexural strength, tensile strength, and water absorption can be seen in Table 2.

Table 3. Comparison of Mechanical Properties of Polypropylene

Flexural Modulus (MPa)	Flexural Strength (MPa)	Tensile Strength (MPa)	Water Absorption Rate (%)	References
2500	44	32	0.00	Haque <i>et al.</i> 2009
2000	40	35	0.00	Koronis <i>et al.</i> 2013
2785	45	38	0.00	This study

Flexural Modulus

The effect of increasing OPDC percentage on the flexural modulus of the OPDC-NPC is depicted in Table 2 and Fig. 1. As predicted, the control sample with 0% OPDC resulted in the highest flexural modulus mean value of 2785 MPa, which is consistent with previous studies (Haque *et al.* 2009; Koronis *et al.* 2013). As revealed in Fig. 1, increasing the OPDC percentage to 5, 10, and 15% decreased the flexural modulus values to 2231, 1601, and 1441 MPa, respectively. This was in agreement with a study previously conducted by Ibrahim *et al.* (2012), where the flexural modulus decreased with decreasing matrix content. The PP is known to exhibit good flexural modulus properties, which was also supported by the results obtained in the present investigation (Fig. 1), where 0% OPDC led to the highest flexural modulus properties. This was also supported by the findings of Bouafif *et al.* (2009), who indicated that the flexural strength of NPCs could be increased by decreasing the PP content.

**Fig. 1.** Flexural modulus and flexural strength of OPDC-NPC at different OPDC contents

Flexural strength

The effect of increasing OPDC percentage on the flexural strength of the OPDC-NPC is presented in Table 2 and Fig. 1. The control sample (0% OPDC) had the highest flexural strength value, 45 MPa; this finding is consistent with previous studies (Haque *et al.* 2009; Koronis *et al.* 2013). Adam *et al.* (2014) argued that the load applied to the OPDC-NPC was actually transferred from PP to OPDC through shear stress. This means that the flexural strength of OPDC-NPC is dependent on the ability of the composite to absorb the applied force. However, the findings of the present study (Fig. 2) show that the flexural strength of OPDC-NPC dropped when 5% OPDC was added. In fact, OPDC seemed to play an important role in enhancing the flexural strength of OPDC-NPC by absorbing the applied force (Khalid *et al.* 2008). This issue occurred because the presence

of only 5% OPDC failed to support the stresses transferred from the PP; as a result, the OPDC-NPC flexural strength deteriorated (Khalid *et al.* 2008). On the other hand, the OPDC-NPC flexural strength increased as the OPDC percentage increased. In a previous report, Bouafif *et al.* (2009) argued that a high natural fiber content in the composite caused the composite to become stiffer.

Figure 2 indicates that increasing the OPDC percentage from 5% to 10% and then to 15% caused the flexural strength values to increase from 30 to 37 MPa and then to 43 MPa. Lee *et al.* (2004) also stated that by adding additional fiber to the composite, it became more rigid and hardened. Overall, the composite with 15% OPDC content possessed the highest flexural strength value because of the increased interaction between the OPDC and the force applied. This demonstrated that OPDC reinforced the OPDC-NPC by absorbing the force energy before it ruptured. Therefore, it could be concluded that by adding a sufficient percentage of OPDC, the flexural strength properties of OPDC-NPC could be enhanced.

Tensile strength

The effect of increasing OPDC percentage on the tensile strength of the OPDC-NPC is presented in Table 2 and Fig. 2. The control sample, containing 0% OPDC, had the highest tensile strength value, 38 MPa. The results obtained were in agreement with those of previous studies (Haque *et al.* 2009; Koronis *et al.* 2013). According to Scott (2000), the tensile strength of the composite was dependent on the natural fiber content. As the OPDC content increased, the OPDC-NPC turned rigid, and the high rigidity of OPDC-NPC enabled it to withstand high tensile force (Bouafif *et al.* 2009). When the tensile load was applied, the load was actually transferred from the matrix (PP) to the natural filler (OPDC) by shear stress (Khalid *et al.* 2008). However, the tensile strength for 5% OPDC was lower than that of the 0% OPDC. The trend observed in Fig. 2 was similar to the trend of flexural strength as seen in Fig. 1. This could be ascribed to the fact that the flexural strength and tensile strength properties of the OPDC-NPC were influenced by the rigidity of the OPDC-NPC (Bouafif *et al.* 2009).

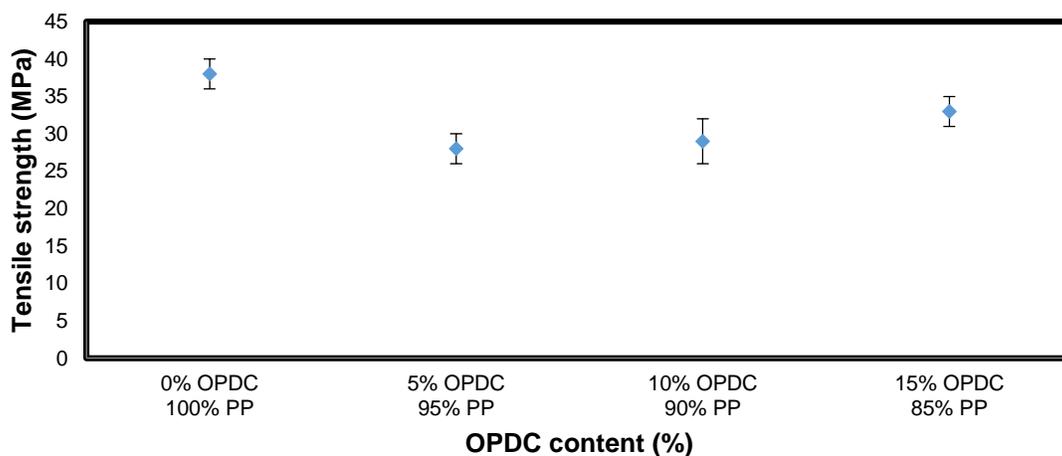


Fig. 2. Tensile strength of OPDC-NPC at different OPDC contents

As discussed earlier, adding a suitable amount of OPDC could enable the OPDC-NPC to withstand the applied force better. Increasing the OPDC percentage from 5% to

15% caused the tensile strength of the OPDC-NPC to increase from 28 to 33 MPa. As shown in Table 2, the OPDC-NPC with 15% OPDC content possessed the highest load transfer compared to the other OPDC-NPC composites.

Water Absorption Rate

The water absorption rate is defined as the amount of water absorbed by a composite when immersed in water for a certain period of time (ASTM D570 2010). The water absorption rate of OPDC-NPC using different percentages of OPDC is presented in Table 2 and Fig. 3. The control sample, 0% OPDC, resulted in no water absorption, which makes sense because it was produced from 100% hydrophobic PP (Southall *et al.* 2002; Deng *et al.* 2010). As shown in Fig. 3, by increasing the OPDC content from 5% to 10% and then to 15%, the water absorption rate increased from 0.16% to 0.20% and then to 0.44%. Given the trend observed in Fig. 3, it can be concluded that OPDC is a hydrophilic material. Generally, when the contact angle of a material is less than 90° , the material is hydrophilic (Dodiuk *et al.* 2007), and the contact angle results obtained for OPDC support this fact (Fig. 4). The hydrophilic properties of the OPDC are attributed to the presence of hydroxyl groups (OH), in the lignocellulosic material, *e.g.*, cellulose and hemicelluloses (Kabir *et al.* 2012), which causes water molecules to attach themselves to the composite through hydrogen bonds (Nachtigall *et al.* 2007; Hosseinaei *et al.* 2012). With increasing OPDC content, the hydrophilic properties of OPDC-NPC also increased. Higher water absorption rates for OPDC-NPC could lead to lower dimensional stability and deteriorated mechanical properties (Nguong *et al.* 2013).

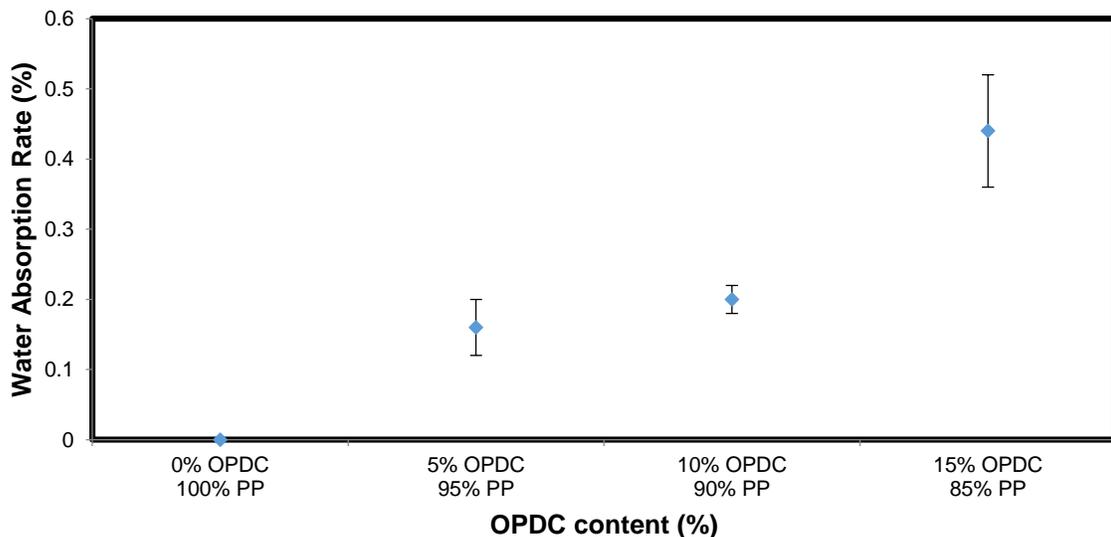


Fig. 3. Water absorption rate of OPDC-NPC at different OPDC content

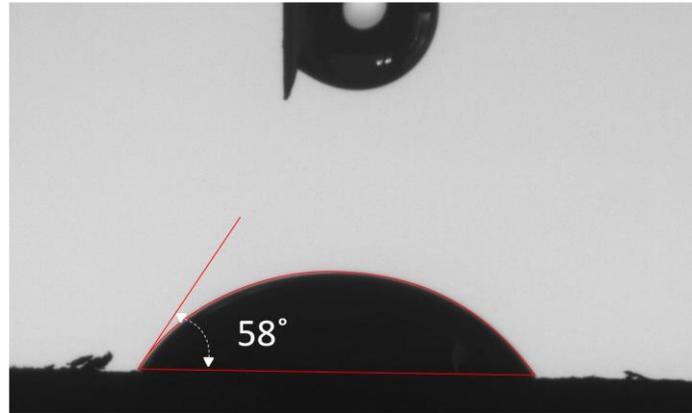


Fig. 4. Contact angle of OPDC surface

Morphological Study of Composites

Figure 5 displays FESEM images of the OPDC fiber and OPDC-NPC morphology. The morphology changed with OPDC content. The OPDC levels of 5% and 10% were not sufficient to reinforce the NPC, and the mechanical properties of NPC were not optimal. As discussed earlier, the strength of OPDC-NPC with 5% OPDC was not enough to absorb the force applied. The SEM image obtained for the highest OPDC content (Fig. 5d) clearly shows the presence of OPDC in the OPDC-NPC. As discussed previously, the OPDC fiber provides flexural strength to the composite by absorbing the load applied to the OPDC-NPC (Lee *et al.* 2004; Khalid *et al.* 2008; Bouafif *et al.* 2009). The energy was converted from the PP to OPDC fiber through shear stress (Adam *et al.* 2014), and hence the mechanical strength of OPDC-NPC made from 15% OPDC was higher than that of the OPDC-NPC made from 5% OPDC.

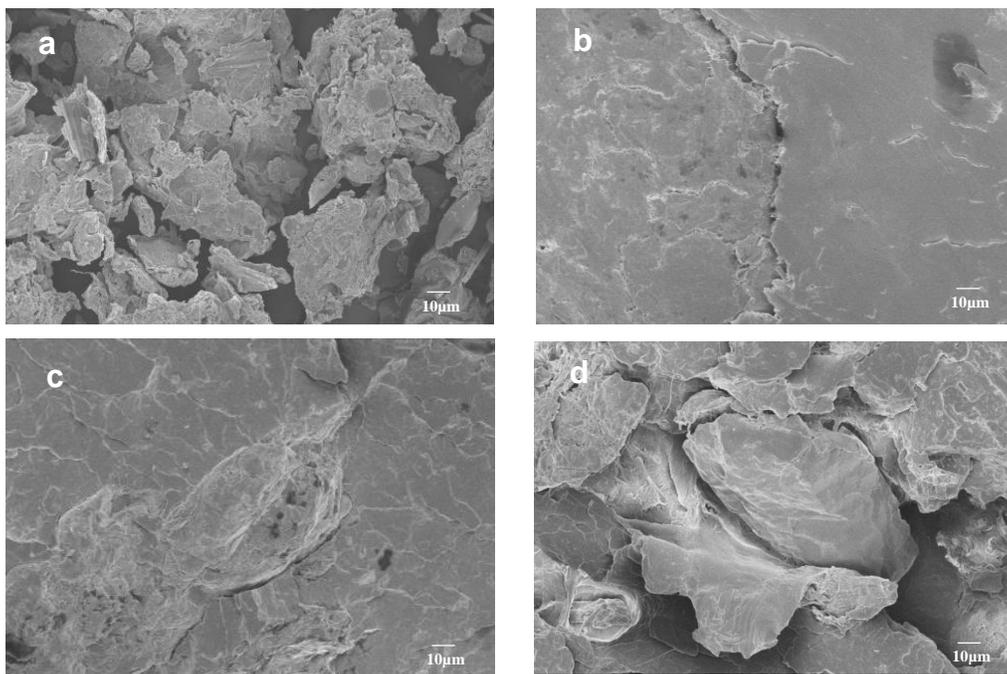


Fig. 5. SEM image of (a) OPDC fiber; (b, c, and d) OPDC-NPC with 5%, 10%, and 15% OPDC content, respectively

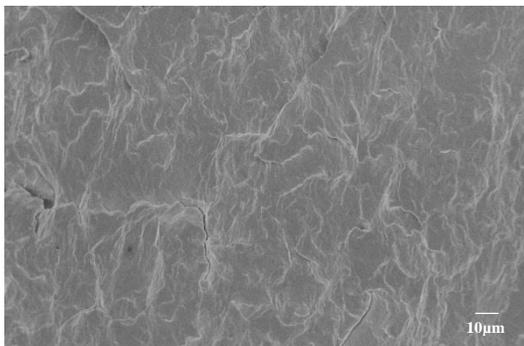


Fig. 6. SEM image of polypropylene

CONCLUSIONS

1. Mechanical and physical tests revealed the effects of different percentages of OPDC in OPDC-NPC. Variations in OPDC percentage changed the flexural modulus, flexural strength, tensile strength, and water absorption rate of OPDC-NPC. At the highest OPDC content investigated, *i.e.*, 15%, the OPDC-NPC displayed high flexural strength and tensile strength.
2. The initial hypothesis that the rigidity of OPDC-NPC can be increased by increasing the OPDC percentage was validated through this study. On the other hand, the flexural modulus of OPDC-NPC decreased when the OPDC percentage increased.
3. The flexural modulus of OPDC-NPC was inversely proportional to the OPDC percentage and flexural strength.
4. The ANOVA test showed that there were significant differences ($p < 0.05$) among OPDC-NPC composites with different OPDC percentages. It was shown that the percentage of OPDC affects the OPDC-NPC properties in a manner consistent with statistical analysis.

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