

## Characterization of Delignified Oil Palm Decanter Cake (OPDC) for Polymer Composite Development

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**Abstract**— For decades agricultural waste materials have been a subject of study for the production of sustainable bioproducts such as biodegradable composite. In Malaysia millions of tonnes of palm oil biomass are produced annually including Oil Palm Decanter Cake (OPDC). In this study, the objective was to characterize the delignified OPDC for potential biodegradable composite development. Chemical delignification is a process of removing the lignin from the plant biomass by using chemicals. Delignification process was performed by treating the raw OPDC with 10% NaOH (alkaline treatment) followed by 25% H<sub>2</sub>SO<sub>4</sub> (acid treatment) and finally 10% H<sub>2</sub>O<sub>2</sub> (bleaching). The result of the treated OPDC showed that cellulose content had increased from 29.4 to 87.6%, while hemicellulose had decreased from 11.2 to 3.1% and finally lignin had decreased from 25.3 to 9.3%. TGA, FTIR and XRD analysis of the raw and treated OPDC samples supported the findings as well. Through morphological analysis of the treated OPDC using FESEM, it showed that the chemical treatment had caused the raw OPDC fibre surface to break-up and open its structure. At the end of this study, the treated OPDC was also exposed to lauric acid for hydrophilic properties study and the result showed that the hydrophobic properties had been developed in the treated OPDC and thus made it suitable for biodegradable composite development.

**Keywords**— oil palm; biomass; oil palm decanter cake; delignification; cellulose; lauric acid; contact angle; natural fibre.

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### I. INTRODUCTION

For the past several decades, agricultural produce and wastes have been a subject of interest for the production of sustainable products such as biodegradable plastic [1], composite for automotive industry [2] and packaging films and coatings [3]. In any plant material, cellulose is the most important component to fabricate reinforce polymer composite material. Both cellulose and hemicellulose serve the same function in natural fiber structure; however, they have a different degree of strength because cellulose has a crystalline chain while hemicellulose has amorphous chain [4]. This difference gives cellulose higher strength compared to hemicellulose. The presence of the hydroxyl group (-OH) in cellulose and hemicellulose is the main reason why the

natural fiber is hydrophilic. Hydrophilic is the ability of any material to absorb water or moisture from the surrounding environment [5]. Hydrophilic properties can be identified using a contact angle measurement test or Fourier-transformed infrared spectrometer (FTIR). Generally, a fiber that has a contact angle of less than 45° is considered as a hydrophilic fiber [6],[7]. The problem with hydrophilic properties is that it could lead to lower dimensional stability, attract the fungal which then lead to deterioration of the fiber and resulting in unnon-uniform dispersion of natural fiber between the polymer [8], [9].

Unlike cellulose and hemicellulose, lignin is a natural adhesive in the natural fiber structure [4]. The structure of lignin is amorphous and has aromatic properties [10]. The previous study found out that lignin has a very low thermal degradation and thus could cause the material to change

color and produce not so pleasant odor [9]. In a plant structure, celluloses are bundled together and tied-up by the hemicelluloses. Lignin components are used to link and tied the hemicelluloses. In this cellulose, hemicellulose and lignin complex formation, the most critical components are cellulose because they provide strength to the whole structure formation. Some studies mentioned that cellulose has the highest strength and thus make it more favorable for biocomposite study and development compared to the rest.

Table 1 shows the percentage of cellulose, hemicellulose and lignin in oil palm shell (OPS), oil palm empty fruit bunches (OPEFB), oil palm mesocarp fiber (MF), corn stover (CS), pineapple leaf fiber, coir, bagasse, banana, wheat straw, rice straw, sorghum stalks and barley straw [11],[12]. Compared with the rest, the cellulose, hemicellulose and lignin contents of oil palm biomass such as OPS, EFB, and MF are in the range 25-30%, 19-21%, and 15-48%, respectively. Among the three, EFB has the highest cellulose content. EFB is currently heavily used as fuel materials for the steam boiler application in the palm oil mill. Due to this reason, its commercialization prospect for biocomposite material is not secured and promising. In addition to OPS, OPEFB, and MF, oil palm decanter cake (OPDC) is a new promising oil palm biomass material which had not been reported yet.

TABLE I  
TYPICAL CONTENTS OF CELLULOSE, HEMICELLULOSE, AND LIGNIN  
IN PLANT MATERIALS

No	Type	Cellulose (%)	Hemicellulose (%)	Lignin (%)
1	Oil Palm Shell	25	19	48
2	Oil Palm Empty Fruit Bunches	43	21	15
3	Oil Palm Mesocarp Fiber	30	21	24
4	Corn Stover	38-40	28	7-21
5	Pineapple leaf fiber	70-82	18	5-12
6	Coir	36-43	0.15-0.25	41-45
7	Bagasse	32-48	19-24	23-32
8	Banana	60-65	6-8	5-10
9	Straw from wheat	33-38	26-32	17-19
10	Straw from Rice	28-36	23-28	12-14
11	Stalks from Sorghum	27	25	11
12	Straw from Barley	31-45	27-38	14-19

To improve the properties of natural fiber, chemical treatment could be applied to reduce the hydrophilic properties and remove the lignin at the same time. In a study, the effects of alkaline treatment to remove the lignin was conducted [13] where it showed that the removal of lignin and impurities had provided a better interaction between the fibers and as a result the fiber surface areas had increased significantly [4]. This is consistent with our previous study which concluded that by increasing the surface area between the natural fiber and polymer, the bonding between them had also increased significantly [5]. However, if the alkali concentration is too high, it will damage the fiber, and as a

result, the properties of the fiber weakened badly as shown in the previous study [4].

Esterification is a method to reduce the hydrophilicity of fiber. It is a reaction between the carboxylic acids and alcohols [14]. In this study, OPDC was reacted with carboxylic acid (lauric acid) as proposed by an earlier study [15]. As mentioned by a previous study, OPDC contained 21.61% cellulose, 3.94% hemicellulose, and 30.66% lignin [16], and therefore requires chemical treatment to improve its physicochemical properties. Therefore this study was conducted to understand the physicochemical properties of OPDC after a chemical treatment.

## II. MATERIALS AND METHODS

### A. Materials

Fresh oil palm decanter cake (OPDC) sample was collected from a selected FELDA Palm Oil Mill in the state of Perak, Malaysia. The chemicals used in this study were hexane (R&M, USA), sodium hydroxide (SYSTEM, Malaysia), sulphuric acid (R&M, USA), hydrogen peroxide (R&M, USA), and lauric acid (R&M, USA).

### B. Methods

1) *OPDC preparation:* Once received, the OPDC sample was dried at 103°C in an oven (UNB-400 Memmert, USA). Then it was ground to fine powder and sieved to a size of < 250µm. To remove the remaining oil in OPDC, the OPDC was subjected to Soxhlet extraction process using hexane as an exchange solvent for 8 hours. Once completed, the OPDC was collected and washed with distilled water to remove the remaining hexane. Then the OPDC has dried again and ready for further use.

2) *Pre-Treatment of OPDC Fibre:* The removal of lignin and hemicellulose from OPDC was adopted and modified from the previous methods developed for extraction of cellulose from oil palm empty fruit bunches and isolation of cellulose from *Agave Angustifolia* [17], [18]. The removal of lignin was completed by treating the OPDC sample with 10% sodium hydroxide (NaOH) solution for 2 hours at temperatures between 70 to 80°C. The treatment to remove hemicellulose was completed by treating the OPDC sample with 25% sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) solution for 45 minutes at a temperature of 45°C. To make sure the dissolved lignin, hemicellulose, and impurities were completely separated from the fiber, hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) solution was used where 10% H<sub>2</sub>O<sub>2</sub> solution was mixed with 10% NaOH solution to increase the pH level to 11. Then OPDC sample was treated with the mixture for 2 hours at a temperature of 60°C. The sample was finally bleached with a 10% H<sub>2</sub>O<sub>2</sub> solution for 3 hours. The OPDC fiber then was subsequently dried in an oven until constant weight.

3) *Surface Modification:* For OPDC surface modification, the method was adopted from the previous study for oil palm leaves fibers [15]. The dried and pre-treated OPDC fiber was treated with 1.0 M lauric acid solution for 6 hours. The OPDC fiber was then filtered and rinsed with *n*-hexane several times. Then, the OPDC fiber was dried in an oven.

### C. Characterization

1) *OPDC Composition Analysis*: The analysis was carried out according to the standard procedure based on the analysis of acid detergent fiber (ADF), neutral detergent fiber (NDF) and acid detergent lignin (ADL). The ADF analysis determines the amount of cellulose and lignin; the NDF analysis determine the amount of cellulose, hemicellulose, and lignin. Meanwhile, ADL analysis determine the amount of lignin only. The cellulose content can be calculated by subtracting the value of ADF with ADL, and the hemicellulose can be calculated by subtracting NDF with ADF.

2) *Fourier Transmission Infra-Red Spectroscopy (FTIR)*: The FTIR was carried out using FTIR (Spectrum Perkin Elmer, USA). To identify the functional groups in the raw and chemical treated OPDC. The sample disc was prepared by mixing and compressing OPDC with KBr and the transmission wavelength range of 4000 to 450  $\text{cm}^{-1}$ .

3) *Thermogravimetric Analysis (TGA)*: The analysis was performed using thermogravimetric analyzer apparatus TGA/SDRA51e (Mettler Toledo, USA) to see the thermal degradation of the raw and treated OPDC. 10 mg of samples were used in a nitrogen environment at a temperature from 30°C to 1000°C.

4) *Contact Angle Test*: The contact angle was performed using OCA 15EC measuring instrument (Data Physic, Germany) to identify the hydrophilic and hydrophobic properties of the raw and treated OPDC. The OPDC fiber was initially formed into a flat and rigid surface before testing. One drop of ionized water was placed on the OPDC surface, and the contact angle image was captured and measured.

5) *Field Emission Scanning Electron Microscopy (FESEM)*: Morphological study of OPDC was carried out using field emission scanning electron (Zeiss Supra 55VP, Germany). The samples were coated with a thin layer of gold to prevent electrostatic charge during the imaging process.

## III. RESULTS AND DISCUSSION

### A. Characterization

1) *Chemical Composition*: The results for the chemical composition of the raw and treated OPDC samples are presented in Table 1. The contents of cellulose, hemicellulose, and lignin of the raw OPDC are consistent with the OPDC sample as reported in the earlier study [16]. OPDC has high percentages of cellulose and lignin which are 29.4% and 25.3%; respectively, meanwhile, the hemicellulose shows a much lower percentage at only 11.2%. The other 34.1% is categorized as ash and impurities. Cellulose and hemicellulose usually give strength to the natural fiber while lignin binds the cellulose and hemicellulose together [4]. Cellulose is the most important part required to reinforce the natural polymer composite (NPC). Lignin is amorphous and has aromatic properties [10]. The previous study found out that lignin has low thermal degradation which leads to color changes and odor

production [9]. Therefore, to improve the properties of OPDC and NPC, lignin needs to be removed.

Alkaline treatment using NaOH is one of the popular treatments for lignin, ash and impurities removal. NaOH breaks the lignin which covered the external surface of the fiber cell wall by dissolving it. Once the lignin has dissolved, ash and other impurities such as silica and sodium oxalate are easily separated from cellulose and hemicellulose [13]. A study showed that 10% of NaOH was enough for lignin removal (delignification) process and if NaOH concentration exceeded 10%, the excess delignification could occur, and as a result, the fiber would be damaged due to cellulose solubilization [4].

As shown in Table 2, the percentage of hemicellulose in the treated OPDC had decreased once treated with  $\text{H}_2\text{SO}_4$  solution. The reason is because acid treatment hydrolysed hemicelluloses bonds in the fibre and simultaneously removed hemicelluloses as well as reported in the previous study [19] The bleaching process was then conducted using hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) to remove the remaining traces of hemicellulose and lignin in OPDC fiber as proposed in the earlier studies [4], [10],[17].

TABLE II  
CHEMICAL COMPOSITION OF THE RAW AND TREATED OPDC

Composition \ Percentage (%)	Raw OPDC	Treated OPDC
Cellulose	29.4	87.6
Hemicellulose	11.2	3.1
Lignin	25.3	9.3

2) *Thermogravimetric Analysis (TGA)*: TGA was conducted to investigate the thermal degradation of the raw and treated OPDC samples. The thermal degradation profiles of the raw and treated OPDC samples are shown in Figure 1. The TGA curves show four stages of degradation, i.e. moisture evaporation, hemicellulose degradation, lignin degradation, and cellulose degradation. From the figure, the weight of OPDC had reduced as OPDC degraded once exposed to heat. As explained in the previous study, this is because of the percentage of fiber degradation highly related to the chemical compositions of the fiber [17].

The moisture started to evaporate at 30°C. At 160– 990°C, the degradation of hemicellulose, lignin, and cellulose occurred. The degradation in this phase shows the highest weight loss of more than 50%. Lignin started to decompose at a wider temperature range of 160-990°C because it has multiple functional groups as compared to cellulose and hemicellulose which allow the lignin to decompose and at the same time withstand higher temperature easily. In the temperature range between 180 and 320°C, hemicellulose started to degrade and followed by cellulose. The remaining traces at temperature >990°C were carbonaceous residue and ashes.

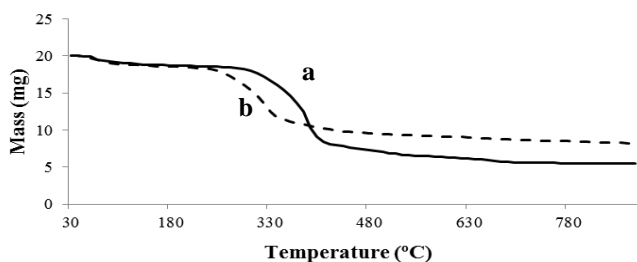


Fig. 1 TGA curves for (a) raw OPDC and (b) treated OPDC

### 3) Fourier Transmission Infra-Red Spectroscopy (FTIR):

Figure 2 illustrates the FTIR spectra for the raw and treated OPDC samples. The peak at  $3380\text{ cm}^{-1}$  shows the presence of cellulose hydroxyl group. The peak between  $3400\text{--}3300\text{ cm}^{-1}$  indicates the presence of free hydroxyl stretching vibration in cellulose molecules. The peak at  $2915\text{ cm}^{-1}$  attributes to the cellulose stretching vibration C-H group as reported in the previous study [17]. Meanwhile, the peaks at  $1430\text{ cm}^{-1}$ ,  $1375\text{ cm}^{-1}$ , and  $1320\text{ cm}^{-1}$  show the presence of  $\text{CH}_2$ , C-H and C-O groups bending vibration of cellulose molecules which is consistent with the previous study [18]. The peaks at  $1500\text{ cm}^{-1}$  and  $1245\text{ cm}^{-1}$  attribute to the aromatic asymmetric stretching and C-O-C of ether from OPDC lignin [18],[20]. The results clearly show that the peaks had disappeared after the removal of lignin and hemicellulose using NaOH and  $\text{H}_2\text{SO}_4$  as shown in line b.

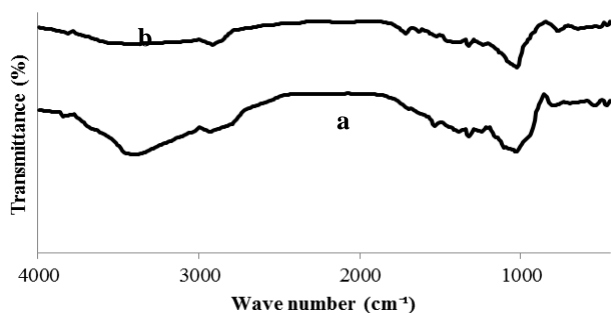


Fig. 2 FTIR spectra for (a) raw OPDC and (b) treated OPDC.

The previous study showed that the hydroxyl group absorbed was determined to be in the range  $1638\text{ cm}^{-1}$  to  $1627\text{ cm}^{-1}$ . [17]. The delignified OPDC (line b) which further treated with lauric acid shows no peak in that range due to the removal of the hydroxyl group in the fiber. The peak at  $1740\text{ cm}^{-1}$  attributes to the presence of C=O group stretching vibration of acetyl and uronic ester group which comes from ester linkage of the carboxylic group (COOH) and fiber [17]. The presence of ester peak proves that the hydroxyl group had been replaced with the carboxyl group from the lauric acid treatment process. The removal of the hydroxyl group had reduced the hydrophilic properties of the OPDC [4]. The intensity of the peak indicates the OPDC crystallinity [17]. The spectra change demonstrates the characteristic of cellulose characteristic and presence of this peak related to the cellulose, hemicellulose, and lignin in the OPDC [18].

4) X-Ray Diffraction (XRD): Figure 3 shows the X-ray diffraction pattern for the raw and treated OPDC. Through this analysis, the major components in OPDC which had

been determined were cellulose, hemicellulose, and lignin. Cellulose is a crystalline structure material while hemicellulose and lignin are of amorphous structures. The XRD analysis of raw OPDC indicates two major peaks which are  $16^\circ$  and  $25^\circ$ . The peak at  $16^\circ$  is identified as amorphous structure; meanwhile, the peak at  $22.5^\circ$  is identified as the crystalline structure.

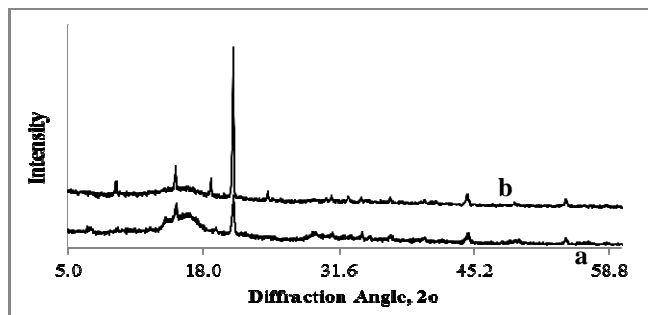


Fig. 3 XRD spectra for (a) raw OPDC and (b) treated OPDC.

The treated OPDC has a higher intensity peak at  $22.5^\circ$  as compared to the raw OPDC. This result suggests that the delignification and removal of hemicellulose using this treatment was successfully carried out. The change in intensity represents the change of crystallinity. The increase in the crystallinity index can be caused by two factors which are the removal of some of the amorphous part from the fiber and rearrangement of the crystalline regions into more order structure as proposed in the previous study [17]. The OPDC fiber exposure towards NaOH treatment also changed the orientation of highly packed crystalline cellulose order. The rearrangement dissolved the lignin which covers the OPDC fibers and removes the impurities such as silica and calcium oxalate as suggested in the previous studies [4],[18]. Acid treatment also had dissolved and removed hemicellulose which further increased the crystalline index of the OPDC as reported earlier [17]. As explained in the previous study, the intensity of the crystalline and amorphous may differ from the chemical composition analysis because a part of the broken cellulose structure may be recognized as the amorphous structure and thus some parts of the amorphous structure of lignin and hemicellulose are unable to identify [18].

5) Contact Angle Test: The hydrophobic and hydrophilic properties of OPDC before and after treatment was measured using a contact angle test. The contact angle of  $< 45^\circ$  indicates hydrophilic properties of OPDC [7]. High wettability of OPDC is not desirable because it can cause lower dimensional stability and deterioration of mechanical properties [6]. Figures 4 and 5 exhibits the contact angle of the raw and treated OPDC. The presence of a hydroxyl group in the OPDC fiber attracts the water molecules into the fibre. From the analysis, we found that the raw OPDC has a low contact angle of  $33^\circ$  which reflects the hydrophilic properties of OPDC.

The modification of the OPDC surface using lauric acid was able to remove the OPDC hydroxyl group of the sample. The chemical reaction between the OPDC and lauric acid has produced the ester functional group. During esterification, the carboxylic group penetrate through the

cellulose structure and substitute the hydroxyl group. As reported earlier, the removal of the hydroxyl group from the fiber decreases the hydrophilic properties of the OPDC [4]. The contact angle test for the lignin free OPDC which further treated with lauric acid shows the contact angle of  $66^\circ$  which was slight as compared to the raw OPDC result.

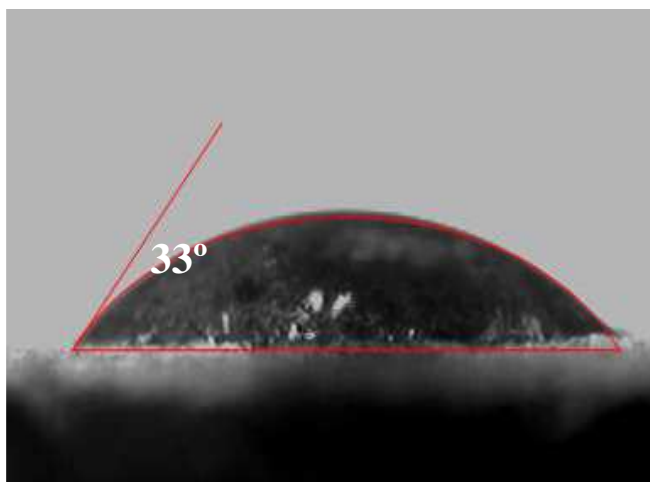


Fig. 4 Contact angle of the raw OPDC.

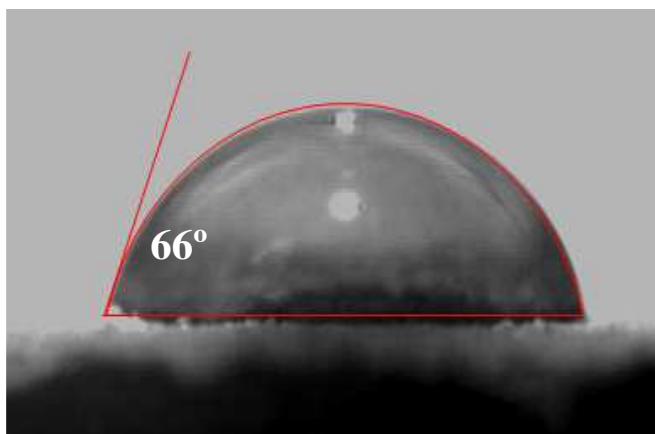


Fig. 5 Contact angle of the treated OPDC

6) *Morphological Study of Fibre:* Figures 6 and 7 show the OPDC before and after chemical treatment. The color of the OPDC changes from dark brown to light grey after alkali and acid treatments and bleaching process. After the treatment hemicellulose, lignin, pectin, and waxes have dissolved and leaving the cellulose component undisturbed.



Fig. 6 Physical state of the raw OPDC.



Fig. 7 The physical state of the treated OPDC.

Figures 8 and 9 show the FESEM images of the raw and chemical treated OPDC. The physical outlook of these images clearly show the changes in the raw OPDC dan treated OPDC samples. The changes in the fiber surface are important in this study as the treated OPDC will be used for biocomposite properties study. As shown in Figure 8, the surface of raw OPDC is covered with cement like component. The fiber is covered with impurities such as hemicellulose, lignin, pectin, and waxes.

Meanwhile, the treated OPDC in Figure 9 shows smoother fiber surface. Hemicellulose, lignin, pectin, and waxes have dissolved during the chemical treatment and bleaching processes. The removal of impurities increases the opening of the fiber which can further increase the bonding between the fibre and polymer in biocomposite study.

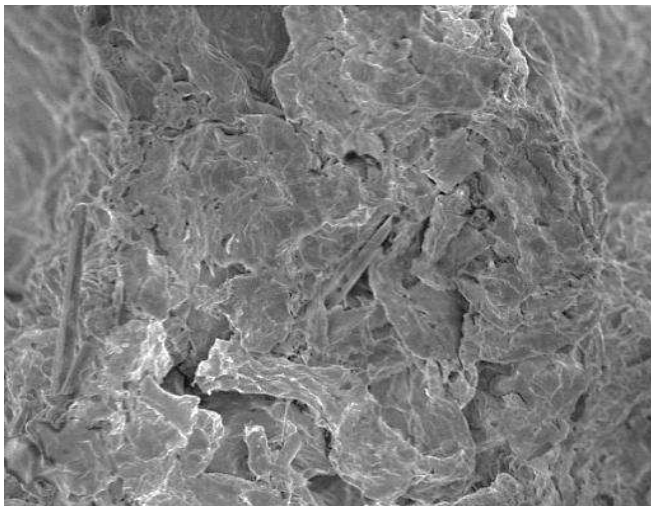


Fig. 8 FESEM micrograph of the raw OPDC

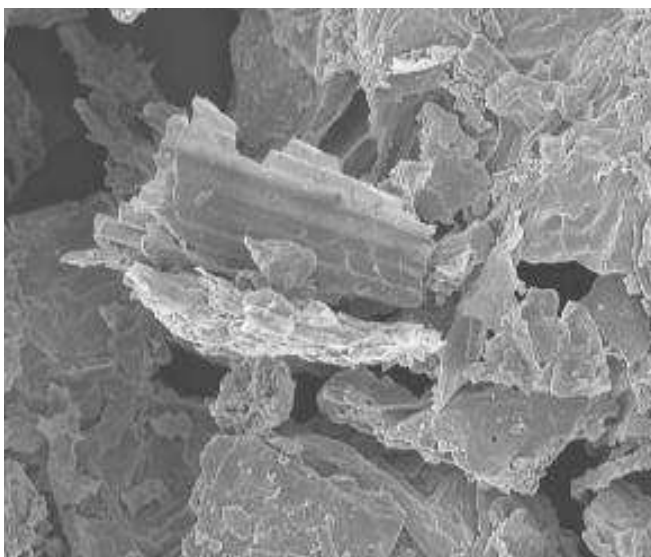


Fig. 9 FESEM micrograph of the treated OPDC.

#### IV. CONCLUSIONS

Cellulose was successfully extracted from OPDC using chemical treatment method. The treatment had caused the removal of hemicellulose and lignin from the OPDC. The cellulose content had increased from 30% to 88%. The morphological analysis using FESEM showed that the treatment had caused the OPDC fiber surface openings to increase. Treatment using lauric acid also reduced the hydrophilic properties of OPDC. This study has proved the successful chemical treatment method for OPDC modification which is more suitable for bio-composites development.

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#### REFERENCES

- [1] Tuates, A.M., and Caparino, O.A. (2016). Development of Biodegradable Plastics as Mango fruit Bag. *International Journal on Advanced Science Engineering and Information Technology*, 6(5), 799-803.
- [2] Nur Hanani, Z.A. and Abdullah, S. (2016). Development of Green Banana (*Musa paradisiaca*) as Potential Food Packaging Films and Coatings. *International Journal on Advanced Science Engineering and Information Technology*, 6(1), 88-90.
- [3] Hassan, F., Zulkefli, R., Ghazali, M.J., and Azhari, C.H. (2017). Kenaf Fiber Composite in Automotive Industry: An Overview. *International Journal on Advanced Science and Engineering Information Technology*, 7(1), 315-321.
- [4] Kabir, M.M., Wang, H., and Cardona, F. (2012). Chemical Treatments on Plant-Based Nature Fibre Reinforced Composite: An Overview. *Composite*, 43, 2883-2892.
- [5] Adam, M.A., Sulaiman A., Said, C.M.S., Som, A.M., Baharuddin A.S., and Mokhtar M.M. (2014). Preliminary Study of Oil Palm Decanter Cake Natural Polymer Composite (OPDC-NPC). *Advanced Materials Research*, 911, 40-44.
- [6] Adam, M.A., Sulaiman, A., Said, C.M.S., Som, A.M., and Tabatabaei, M. (2015). Enhanced Rigidity of natural Polymer Composite Development from Oil Palm Decanter Cake. *BioResources*, 10, 932-942.
- [7] Dodiuk, H., Rios, P.F., Dotan, A., and Kenig, S. (2007). Hydrophobic and self-Cleaning Coatings. *Polymer for Advanced Technologies*, 18, 746-750.
- [8] Nguong, C.W., Lee, S.N.B., and Sujan, D. (2013). A Review on Natural Fiber Reinforced Polymer Composites. *Engineering and Technology*, 73, 1123-1130.
- [9] Ashori, A. (2008). Wood Plastic Composite as Promising Green Composites for Automotive Industries. *Bioresource Technology*, 99, 4661-4667.
- [10] Saheb, D.N., and Jog, J.P. (1999). Natural Fiber Polymer Composites: A Review. *Advances in Polymer Technology*, 18, 351-363.
- [11] Reddy, N., and Yang, Y. (2005). Biofibers from Agricultural Byproducts for Industrial Application. *Trends in Biotechnology*, 23 (1), 21-27.
- [12] Garcia-Nunez, J.A., Ramirez-Contreras, N.E., Rodriguez, D.T., Silva-Lora, E., Stockle, C., and Garcia-Perez, M. (2016). Evolution of Palm Oil Mills into Bio-Refineries: Literature Review on Current and Potential Uses of Residual Biomass and Effluents. *Resources, Conservation and Recycling*, 110, 99-114.
- [13] Faruk, O., Bledzki, A.K., Fink, H.P., and Sain, M. (2012). Biocomposite Reinforced with Natural Fiber: 2000-2010. *Progress in Polymer Science*, 37, 1552-1596.
- [14] Barros, A., Lopez, C., Rico, V., Garcia, F., Gonzalez-Elipe, A.R., Richter, E., Battiston, G., Gerbasí, R., McSpornan, N., Sauthier, G., Gyorgy, E., and Figueras, A. (2007). Effect of Visible and UV Illumination on the Water Contact Angle of TiO<sub>2</sub> Thin Films with Incorporated Nitrogen. *Journal of Physical Chemistry C*, 111, 1801-1808.
- [15] Sidik, S.M., Jalil, A.A., Triwahyono, S., Adam, S.H., Satar, M.A.H., and Hameed, B.H. (2012). Modified Oil palm Leaves Adsorbent with Enhanced Hydrophobicity for Crude Oil Removal. *Chemical Engineering Journal*, 208, 9-18.
- [16] Rzak, M.N.A., Ibrahim, M.F., Yee, P.L., Hassan, M.A., and Aziz, S.A. (2012). Utilization of Oil Palm Decanter Cake for Cellulase and Polyoses Production. *Biotechnology and Bioprocess Engineering*, 17, 547-555.
- [17] Rosli, N.A., Ahmad, I., and Abdullah, I. (2013). Isolation and Characterisation of Cellulose Nanocrystals from *Agave Angustifolia* Fiber. *BioResources*, 8, 1893-1908.
- [18] Nazir, M.S., Wahjoedi, B.A., Yusoff, A.W., and Abdullah, M.A. (2013). Eco-Friendly Extraction and Characterisation of Cellulose from Oil Palm Empty Fruit Bunches. *BioResources*, 8, 2161-2171.
- [19] Jacobsen, S.E., and Wyman, C.E. (2000). Cellulose and Hemicellulose Hydrolysis Models for Application to Current and Novel Pretreatment Process. *Applied Biochemistry and Biotechnology*, 84, 81-96.
- [20] Chan, C.H., Chia, C.H., Zakaria, S., Ahmad, I., and Dufresne, A. (2013). Production and Characterisation of Cellulose and Nano-Crystalline from Kenaf Core wood. *BioResources*, 8, 785-794.