Physicochemical Characterization of Oil Palm Decanter Cake (OPDC) for Residual Oil Recovery

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A characterization study on oil palm decanter cakes (OPDC) was performed to gain an in-depth understanding of the material's characteristics to aid in potential residual oil recovery. The OPDC was characterized by a high moisture content, high biodegradability, high organic content, and a nutrient-rich composition. Microscopic observation showed that the oil attachments in OPDC, and a vast majority of the droplets, were less than 50 µm in size. Furthermore, contact angle measurement revealed the hydrophilic and oleophilic characteristics of OPDC. Specifically, the contact angles of water and crude palm oil (CPO) with OPDC were both less than 45° with absorption rates of 0.0265 ± 0.003 µL/s and 0.1042 ± 0.05 µL/s, respectively. The OPDC is a fibrous material, and the surface area and pore size measured were 7.103 m^2/g and 481.7 Å, respectively. Fourier transform infrared spectroscopy (FTIR) and thermogravimetric (TG) analysis results showed the functional groups and degradation properties of OPDC, respectively.

Keywords: Oil palm decanter cake; Physicochemical properties; Contact angle; Microscopic observation; Lignocellulosic materials

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INTRODUCTION

Palm oil is one of the major agricultural commodities in the world, making it one of the largest agricultural industries. Malaysia is a leading producer in the industry. As of 2009, there are about 416 palm oil mills operating in Malaysia (Price 2012). From a typical palm oil mill, the estimated generated waste for every ton of fresh fruit bunch (FFB) range from 0.6 to 0.8 m^3 of palm oil mill effluent (POME), with an accumulation of 22 to 23% of empty fruit bunches (EFB), 3.5% of oil palm decanter cake (OPDC), and 13.5% palm mesocarp fiber (PMF) (Ooi and Kumar 2008; Ng *et al.* 2011). The biomass waste production was quite high, and it negatively affected the total oil extraction rate (OER) of the palm oil industry due to the oil losses in the wastes. The OER indicates the actual amount of oils extracted from FFB, as well as the overall efficiency of the palm oil mills. Additionally, the generation of abundant biomass waste also raises concern in regards to the environmental effect and sustainability of the palm oil industry. Hence, any strategy to minimize the waste by converting it into valuable by-products is highly supported by both the palm oil industry and the government.

Many researchers have studied the suitability of OPDC as animal feed (Chavalparit *et al.* 2006), fertilizer, and composting material, due to its high nutrient content (Ramli *et al.* 2012; Razak *et al.* 2012), its ability to be used as a source of biogas production (Kaosol and Sohgrathok 2012), and the potential use of pyrolyzed OPDC as an adsorbent for the removal of metal ions from wastewater (Dewayanto *et al*. 2010). Currently, most of the mills in Malaysia use OPDC as animal feed and composting materials for the plantation. However, its economic value is low and not attractive. In the milling process, decanter is used to treat the underflow of the clarification tank. It separates the remaining crude palm oil (CPO) from the sludge before feeding into the purification tank. The sludge is then discharged from the decanter as OPDC. However, this lignocellulosic material still absorbs and retains oils in its fibre, regardless of the advanced technology being implemented (*e.g.*, two and three-phase decanters). Hence, by referring to the annual OPDC generated in a mill, the residual oils content in the OPDC is considerably high.

A surface study has emerged as one of the important counterparts of any research in order to gain understanding of a given material, physically, chemically, or how it reacts with the environment. The surface characteristics (*e.g.*, surface roughness, surface area, contact angle, and wetting behavior) play a significant role in the study to fully utilize the material in many applications. Most studies have investigated the surface characteristics of a given material before and after a modification. In these studies, the researchers improved the surface characteristics of the materials according to their particular process. The most common applications used by researchers are related to coating (Kalin and Polajnar 2013), lubricating (Santos *et al.* 2006), and sorbent materials (Lim and Huang 2007). Carmody *et al.* (2007) characterized and determined the key properties of adsorption and absorption behaviors of selected sorbent materials based on the porosity, pore data, surface area, and contact angle measurement. Adam *et al.* (2014) studied the suitability of OPDC as a natural polymer composite by molding 5% of OPDC with 95% polypropylene (PP). This study showed that in terms of elasticity, stiffness, tensile strength, and water absorption rate, OPDC was slightly better than the other types of fibers.

To date, no study has been conducted that reported the surface characteristics or the characteristics of OPDC *e.g.* contact angle and microscopic observation that contribute directly to oil loss. Therefore, the objectives of this study were to characterize and understand the physicochemical properties of OPDC for residual oil recovery.

EXPERIMENTAL

Materials

Sample preparation

The OPDC used in this study was obtained from a three-phase decanter, located in the FELDA Trolak Palm Oil Mill, Perak, Malaysia. The fresh sample was stored in a chiller (4 °C) prior to the analysis. Ten grams of dried OPDC was extracted using 300 mL of n-hexane (Merck, Germany) for 8 h in a Soxhlet extractor. The extracted oil was concentrated in a vacuum rotary evaporator and dried in an oven, with temperatures ranging from 103 to 105 °C, until a constant weight was obtained.

$$Oil\ (\%\ dry\ basis) = \frac{Weight\ of\ extracted\ oil\ (g)}{Initial\ weight\ of\ dry\ sample\ (g)} \times 100\% \quad (1)$$

Proximate, ultimate, and chemical composition determinations of OPDC

The proximate analysis was carried out in accordance to the standard method (ASTM D 5142-02a 2003), using a thermobalance apparatus TGA/SDRA51e (Mettler Toledo, USA). The ultimate analysis was performed using a Thermo Finnigan Flashed 1112 analyzer (USA) in accordance with ASTM D 5373-02 (2007). For the chemical composition, chemical oxygen demand (COD) and biological oxygen demand (BOD) determinations were analysed using the methods of analysis of AOAC International (Horwitz 1994) and Eaton *et al.* (2005).

Microscopic observation on OPDC

A Leica DMLP optical microscope (Germany), at a magnification of 800x, was used to observe the attachment of oil onto OPDC. Fresh OPDC was mixed with 2 mL of distilled water and placed on a microscope slide; a few drops of Sudan (III) dye solution (Merck, Germany) were added to increase contrast of the preparation. The surface morphology of the dried OPDC was examined with a Supra 40VP field emission scanning electron microscope (FESEM; Carl Zeiss, Germany) operating at an accelerating voltage of 5 kV and a magnification of 1700x. The OPDC was mounted on an aluminium stub with tape and then coated with a layer of gold to minimize the charging effects.

BET analysis and contact angle measurement

Surface area, pore size, and volume were characterized using an Autosorb-1 (Quantachrome; USA), based on the Brunauer, Emmett, and Teller (BET) method. Nitrogen adsorption at 77 K was applied and full adsorption isotherms were collected. The porosity data (pore size and volume) were calculated based on the Dollimore Heal (DH) (Dollimore and Heal 1964) and Barrett-Joyner-Halenda (BJH) (Barrett *et al.* 1951) methods. A OCA15ES Contact Angle Goniometer (Data Physics, Germany) was used to determine the contact angle measurement. During this process, the oil in the OPDC was removed and the dry OPDC formed into a flat, rigid surface in order to obtain the intrinsic contact angle. One microliter of fluid (*i.e.*, CPO or water) was injected onto the solid OPDC surface and the measurement calculated automatically. The oil absorption rate in regards to OPDC was calculated using the following formula,

$$Adsorption\ rate = \frac{Dosing\ volume\ (\mu L)}{\Delta t\ (S)} \quad (2)$$

where $\boldsymbol{\Delta t}$ is the time for oil droplet to reach an equilibrium state.

Fourier transform infrared spectroscopy (FTIR) and thermogravimetric (TG) analysis

A spectrum one Fourier transform infrared spectrometer (Perkin Elmer, USA), equipped with attenuated total reflection (ATR) at a wavelength of 400 to 4000 cm^{-1}, was used to identify the presence of functional groups in OPDC. A TGA/SDTA851^e thermogravimetric analyzer (Mettler Toledo, USA) was used to determine the mass loss and degradation properties of OPDC. An OPDC sample that was dried and crushed was subjected to a heating program from 25 °C to 950 °C, at a heating rate of 20 °C/min under a constant nitrogen flow rate of 100 mL/min. The sample was introduced to the combustion process under purified air (flow rate 100 mL/min) with the temperature raised to 950 °C at 20 °C/min.

RESULTS AND DISCUSSION

Physicochemical Properties of OPDC

Table 1 presents the physicochemical properties of OPDC from this study compared to the work of other investigators. The data reveals that the OPDC from the FELDA Trolak palm oil mill had a high moisture content, a high biodegradability, and a nutrient rich composition, which was in agreement with the other studies (Haron *et al.* 2008; Paepatung *et al.* 2009; Yahya *et al.* 2010; Razak *et al.* 2012). In this study, a substantial amount of residual oil in OPDC was found (13.60 ± 3.33% dry basis). The residual oils were fully recovered, as no more oils were observed in the OPDC after the second extraction was performed on the same sample. The oil content measured in the OPDC was in agreement with the results as reported by the FELDA palm oil mill (from 11 to 16%, dry basis) (personal communication), as well as in line with the data of Kandiah and Batumalai (2013) (12.25%, dry basis). The oil content seems low but when referring to the annual FFB processed in a mill, it generates abundance of OPDC biomass that eventually contains high residual oils in total.

Table 1. Comparison of OPDC Characteristics

Parameter	Current Study	Yahya *et al.* 2010	Haron *et al.* 2008	Paepatung *et al.* 2009	Razak *et al.* 2012	Kandiah and Batumalai (2013)
pH	5.03 ± 0.04	-	4.8	-	4.08	-
Moisture content (%)	78.20 ± 1.27	76.38	78	76.7	76.46	-
COD (g/kg)	136.916 (wet)	-	-	880 (dry)	316.927 (wet)	-
BOD (g/kg)	46.538 (wet)	-	-	470 (dry)	41.813 (wet)	-
Oil content (%)*	13.60 ± 3.33	-	-	-	-	12.25
Proximate analysis*						
Volatile matter (%)	76.15	-	-	83.4	-	-
Ash (%)	19.72	-	-	-	22.5	-
Fixed carbon (%)	4.09	-	-	-	-	-
Ultimate analysis*						
Carbon (%)	43.73 ± 0.09	51.7	-	43.6	55.17	-
Hydrogen (%)	5.93 ± 0.13	-	-	5.79	-	-
Nitrogen (%)	2.33 ± 0.06	2.38	2.42	2.21	2.8	-
Sulfur (%)	-	0.39	-	0.15	0.3	-
Oxygen (%)	-	-	-	31.7	-	-
Chemical composition*						
MgO (%w/w)	0.62 ± 0.03	0.80	0.54	-	-	-
P_2O_5 (%w/w)	0.41 ± 0.01	0.39	0.51	-	-	-
K_2O (%w/w)	2.73 ± 0.06	2.39	1.24	-	-	-
CaO (%w/w)	2.10 ± 0.00	1.02	1.68	-	-	-
Silica content (%w/w)	5.83 ± 0.42	0.61	-	-	-	-

*dry basis
- Data not available
Variance reported as ± Standard Deviation

The chemical composition of the OPDC, particularly K_2O and CaO, had a slightly higher content than found in previous work (Haron *et al.* 2008; Yahya *et al.* 2010). The composition therefore enhanced the properties of the OPDC as composting material. The OPDC is an oil palm mill waste that has a low utilization rate, and composting is considered a sustainable way to minimize the waste created in the palm oil mill industry. Recent studies have demonstrated the suitability of OPDC as adsorbent and natural polymer composite (Dewayanto *et al.* 2010; Adam *et al*. 2014). Additionally, the COD value of the OPDC was lower than that reported by Razak *et al.* (2010) and Paepatung *et al.* (2009). The silica content for the OPDC in this study was surprisingly higher than the previous study by Yahya *et al.* (2010), *i.e.*, 5.83% w/w compared to 0.61% w/w. This may be due, in part, to the presence of crystalline silica in the OPDC which may reduce the solvent efficiency during the oil recovery process, as it may hinder the solvent to penetrate deeper into the matrix of the cake. The FFB for the mill was supplied by different suppliers, some of which may not have been well trained to carefully handle the fruits, hence resulting in poor handling and harvesting from the plantation to the grading stage at the mill. This may have exposed the fruits to minimal amounts of the dirt and sand.

Microscopic Observation of OPDC

Figure 1 shows the attachment of oils and the surface of OPDC. On the surface, the sample appeared rough but intact. It was also observed that some of the oils were attached on the fibers, and some were in the free form.

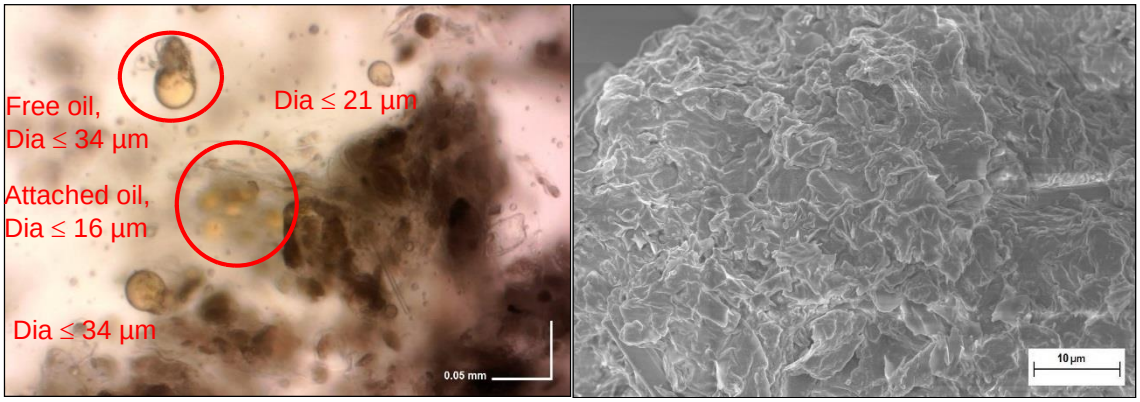

Fig. 1. (a) Oil attachment in OPDC (Light Microscope); (b) Surface of OPDC (FESEM)

It is interesting to note that the size of almost all of the oil droplets was less than 50 µm. As described in previous work, oil droplets in an oil-water mixture, with sizes ranging from 20 to 150 µm, are classified as a dispersed oil mixture (Abass *et al.* 2011). According to Chow and Ho (2002), it is possible that these small droplets could have originated from the ripe and unripe oil cell. However, they have concluded that the majority of the droplets are formed due to the turbulent pumping throughout mill processing and also highlighted that the natural surface active materials, namely monoglycerides, phospholipids, and glycolipids, are released from the ruptured cellular membranes of palm fruits. Hence, they suggest that throughout the milling process, these natural surface active materials are solubilized into a crude oil slurry which contributes to the stability of the oil droplets.

Meanwhile, the use of a centrifuge to enhance the oil and sludge separation is likely to increase the oil dispersion into smaller droplets (Floury *et al.* 2000). In wastewater treatment, oil droplets less than 50 μm have been removed by packed bed filters and dissolved air flotation (DAF) (Rubio *et al.* 2002); however, this method is not applicable for removing oils from OPDC. A decanter is used to treat the underflow of the clarification tank in order to reduce the use of dilution water as well as minimize oil loss in the OPDC. During the clarification process, small droplets take an extremely long time to rise to the surface. Without an efficient method to coalesce the droplets, they remain subsurface and lead to more oil being trapped and absorbed by the fibrous materials of the solid during further processing.

Contact Angle Measurement

Water and CPO were used to identify the wetting properties of OPDC. Table 2 presents the equilibrium contact angles and absorption rate for both fluids in OPDC. In Fig. 2, the contact angles of water and CPO with OPDC were less than 45°. A contact angle of less than 45° indicates both high wettability and the spreading of fluid over the surface. It corresponds to the amphiphilic properties of OPDC, meaning that it exhibits both hydrophilic and oleophilic properties. Hence, during the mill processing, it has the ability to absorb water and CPO, simultaneously. However, OPDC is slightly oleophilic, as the oil contact angle was lower than the water contact angle. Therefore, it might tend to capture CPO before water, which parallels the absorption rate of both CPO and water with OPDC. The CPO absorption rate was significantly higher than water absorption rate in regards to the OPDC, 0.1042 ± 0.05 μL/s and 0.0265 ± 0.05 μL/s, respectively.

Table 2. Contact Angles of Water and CPO with OPDC

Parameter	Experimental Result
CPO contact angle (°)	26.87 ± 1.72
CPO absorption rate (μL/s)	0.1042 ± 0.05
Water contact angle (°)	33.57 ± 3.24
Water absorption rate (μL/s)	0.0265 ± 0.003

Variance reported as ± Standard Deviation

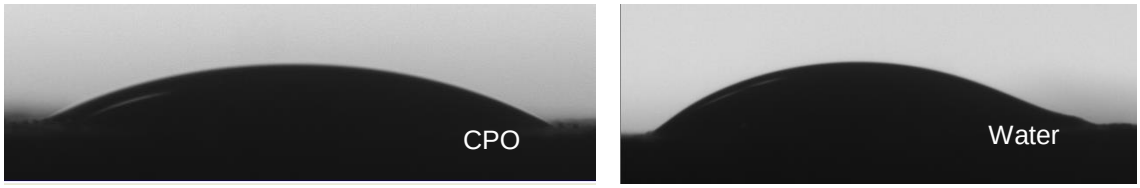

Fig. 2. Contact angles of CPO and water with OPDC at equilibrium

The OPDC is a fibrous material which basically has two main adsorption mechanisms, surface adsorption (initial contact angles) followed by capillary adsorption (Huang *et al.* 2010). Carmody *et al.* (2007) studied the adsorption mechanism of oil on raw cotton fiber, and highlighted that surface adsorption occurred when the oil coated the fiber and the uptake of oil was conducted by the capillaries. The presence of nonpolar groups on the surface of OPDC (particularly silica), may induce lower surface energy, thus increasing the attraction of the oil molecules. The same mechanism was applied for water with fiber. The presence of a hydroxyl group in the OPDC also attracts water through the hydrogen

bonds. However, during the milling process, the presence of RCPO in the OPDC may hinder the ability of OPDC to further absorb water since the OPDC was already saturated with oil. Furthermore, capillary adsorption was influenced by the porosity of the surface. Higher porosity induces lower contact angle, and low contact angles indicate high sorption of liquid into solid. According to Tan *et al.* (2008), the high adsorption capacity of the adsorbent could be due to its relatively high surface area and its mesoporous structure. This finding is in agreement with the porous structure and high surface area of OPDC. Therefore, the oils not only attach on the cake surface, but also penetrate and then are trapped in its matrix, thereby increasing the oil loss in OPDC.

Surface Area and Pore Size

Table 3 provides the BET analysis results for OPDC. The surface area and pore diameter of the sample were measured at 7.103 m^2/g and 481.7 Å, respectively. The OPDC surface area was not much different than a previous study on cotton fiber (8.2 m^2/g) and organoclay (8.7 m^2/g) (Carmody *et al.* 2007). The OPDC naturally has a fine fiber size due to mechanical pressing during the milling process. Therefore, it has a higher surface area than other types of natural fibers such as EFB and PMF. A larger surface area enhances the internal bonding of OPDC with other material, particularly in making composites. Furthermore, a larger surface area creates a better load transfer from PP to OPDC and gives the sample better flexural strength (Adam *et al.* 2014). Surface area plays a major role in the adsorption capacity of any material. A higher surface area increases the adsorption capacity and a higher adsorption capacity means high wettability and lower contact angles.

Table 3. BET Analysis of OPDC

Parameter	OPDC
Surface area (m^2/g)	7.103
Average pore diameter (Å)	481.7

FTIR Spectral Analysis

Figure 3 illustrates the FTIR spectra of OPDC. Peaks are shown at wave numbers of 3284 cm^{-1}, 2917 cm^{-1}, 2851 cm^{-1}, 1737 cm^{-1}, 1625 cm-1, and 1032 cm^{-1}. Peaks in these regions are commonly found in EFB, rice husk, coconut husk, and other biomass (Sim *et al.* 2012; Zakaria *et al.* 2014). For the lignocellulosic material, a broad band at 3600 to 3200 cm^{-1} normally corresponds to the O-H group. In the OPDC, the band appeared at a peak of 3284 cm^{-1}.

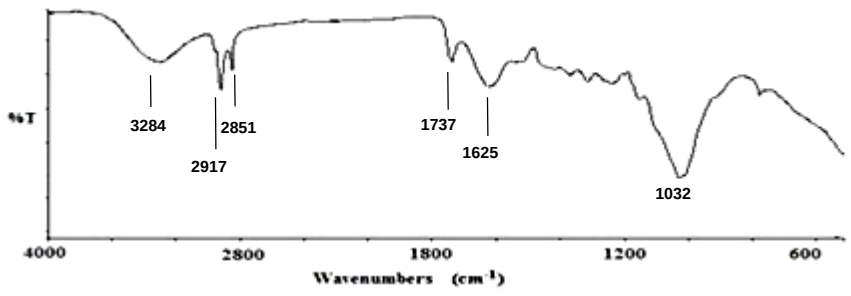

Fig. 3. FTIR spectra of OPDC

Meanwhile, absorption bands at 2917 cm^{-1} and 2851 cm^{-1} and 1737 cm^{-1} and 1625 cm^{-1} correspond to the stretching region of CH_2 and CH_3, and C=0 ester, respectively. The bands of 1000 to 1200 cm^{-1} represent the hydroxyl and phosphate groups. These bands are regarded as characteristic for lipids and a fingerprint region for phospholipids (Laurens *et al.* 2011), thus indicating the presence of residual oils in the OPDC. Furthermore, these peaks are also associated with hemicelluloses, cellulose, and lignin components in the biomass (Tandy *et al.* 2010).

Thermogravimetric (TG) Analysis

Figure 4 shows the decomposition of OPDC during the combustion process. In general, three major decomposition phases occurred during the combustion. Phase 1 occurred at temperatures below 100 °C, where the weight loss was due to the release of moisture in OPDC. Phase 2 took place when the temperature ranged from 200 to 400 °C. The decomposition was the highest at this region (*i.e.*, 50% weight loss), and it corresponded to the decomposition of hemicelluloses, cellulose and also lignin. Lastly, phase 3 occurred when the temperature was above 400 °C, with a maximum of 1000 °C. This phase may be due to the decomposition of lignin and other mineral matter, and contributed to 20% of the weight loss. The remaining components were carbonaceous residue and ash.

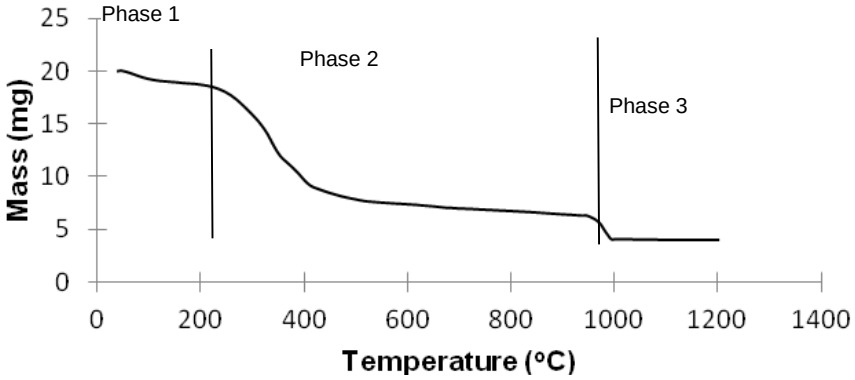

Fig. 4. TG curve of OPDC

The results showed trends similar to the findings of previous studies, where at a temperature range of 200 to 315 °C, the decomposition of hemicelluloses, followed by cellulose and lignin, occurred. The degradation of lignin may occur at temperatures ranging from 160 to 900 °C (Yang *et al.* 2007; Md. Yunos *et al.* 2012).

Hemicelluloses and cellulose were the easiest component to remove. At low temperature, the compounds are easily volatile due to their amorphous structures, *i.e.*, rich in branches and random organization of various saccharides (Wang *et al.* 2009). Due to their volatility, the compounds not only enhanced the ignition characteristics of OPDC, but also facilitated the decomposition of lignin during the combustion process.

Lignin is a complex structure of aromatic polymer that has C-C bond linked with phenyl propane. Therefore, it has the lowest decomposition rate in comparison to the other components (Spinace *et al.* 2009).

CONCLUSIONS

1. The OPDC was characterised by several key properties, such as high moisture content, high biodegradability, and nutrient-rich contents. The majority of the residual oil droplets were less than 50 μm. During typical palm oil mill processing, these small oil droplets are difficult to remove via mechanical extraction. Using solvent extraction, the residual oils content (13.6±3.33 %, dry basis) in the OPDC were fully recovered. Therefore, it seems that the sole method to recover the oil loss in the OPDC is *via* solvent extraction.
2. The results also demonstrate that the OPDC exhibits amphiphilic properties, as it is both oleophilic and hydrophilic in nature *i.e*, low contact angle and high affinity to oil and water). The contact angles of water and CPO with OPDC were influenced by the surface area and porosity.
3. Every application requires certain surface characteristics. In future work, the OPDC surface can be modified according to the desired material properties for the selected applications.

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