Presence of Residual Oil in Relation to Solid Particle Distribution in Palm Oil Mill Effluent

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The production of palm oil requires a large amount of water, which subsequently turns into wastewater known as palm oil mill effluent (POME). Because of its high organic content, there has been debate over how to utilize POME for oil recovery. POME is usually mainly comprised of water (95 to 96%), total solids (4 to 5%), suspended solids (2 to 4%), and oil (0.6 to 0.7%). The lignocellulosic particles in POME are highly oleophilic and capable of absorbing oil. Therefore, the objective of this study was to understand the presence of residual oil and try to relate with the oil loss in POME and to identify the solid particles in POME and their correlations. Microscopic observations showed that most of the oil droplets available in POME were less than 100 µm in size. If given the opportunity to settle, the highest quantity of oil droplets and solid particles was in the bottom layer, followed by the middle layer, and lastly the upper layer. In cases where the contact angle of water was less than 45° on POME solids, the absorption rate was 0.11 \pm 0.03 µL/s and 0.09 \pm 0.01 µL/s, respectively. This study concluded that the oil losses in POME were partly due to the absorption of oil by the fibers.

Keywords: Palm oil mill effluent (POME); Residual oil; Oil loss; Settling velocity; Stokes law; Solid particles

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INTRODUCTION

Malaysia is currently the second largest palm oil producer and exporter in the world, after Indonesia (Rupani *et al.* 2010). The production of crude palm oil (CPO) in Malaysia increased from 1.3 million tonnes in 1975 to 18.6 million tonnes in 2010 (Hassan *et al.* 2013). Oil palm is scientifically known as *Elaeis guineensis* Jacq. This industry is very important to Malaysia and it has contributed significantly to the country's gross domestic product (GDP) (Sulaiman *et al.* 2010). Regardless of the good economic return to the country, the industry also generates a large amount of waste in the form of empty fruit bunch (EFB), oil palm frond (OPF), mesocarp fibre, palm kernel shell (PKS), palm oil mill effluent (POME), and sludge from pond and anaerobic tanks (Sulaiman *et al.* 2010). Among all, liquid waste known as palm oil mill effluent (POME) is considered as the most harmful waste if untreated (Sulaiman *et al.* 2009). In fact, the demand for palm oil is still

increasing, and so is the generation of POME. POME is regarded as a major contributor to worldwide industrial pollution (Rajagopal *et al.* 2013). Malaysia presently accounts for 39% of world palm oil production and 44% of world exports (Rajagopal *et al.* 2013). About 3.5 tonnes of POME is generated per ton of CPO (Madaki and Seng 2013). In 2013, approximately 48 million tonnes of POME was generated in Malaysia. POME is mainly composed of water (95 to 96%), total solids (4 to 5%), suspended solids (2 to 4%), and oil (0.6 to 0.7%) (Ahmad *et al.* 2005).

Currently, the palm oil industry is very much concerned over factors that limit the oil extraction rate (OER), and among those are the oil losses from POME. Although the oil loss from POME is less than 1.0%, the accumulated oil loss from POME over a year is considerably high and worth potentially recovering. To date, no study has been conducted to evaluate the recovery of residual oil in POME. The recovery of oil in POME will reveal a new economic opportunity to the palm oil industry. The success in oil recovery from POME will eventually increase the overall OER and contribute to the total revenue for mills. Moreover, there is an economic advantage for the use of waste oil feedstocks, such as recovered oil, for potential biodiesel production (Aladetuyi *et al.* 2014).

On the other hand, it is also important to note that the palm oil industry is facing a tremendous challenge in meeting stringent environmental regulations (Najafpour *et al.* 2006). As mentioned earlier, POME contains about 4000 to 6000 mg/L (0.4 to 0.6%) of oil and grease, which is above the allowable discharge limits; therefore, its direct disposal into the waterways is prohibited (Ahmad *et al.* 2005). Besides, the triacylglycerols and degradatives present in POME could hinder the degradation processes involved in POME treatment (Salmiati *et al.* 2007). For these reasons, the removal of residual oil in POME is critical before addressing its treatment and disposal.

Although the economic benefits of residual oil POME removal and recovery are huge, such implementations have been restricted due to the lack of sufficient understanding regarding the presence and behaviour of oil droplets in POME. So far, little information is known about residual oil and its relationship with the solid particles. Limited studies were conducted to understand the characteristics of solids in POME, such as the particle morphology, size, and distribution (Ahmad *et al.* 2003; Alrawi *et al.* 2013). Moreover, no research has been conducted to actually report on the characteristics of POME derived from different sources in mills, nor on the microscopic properties which might provide insights into the oil loss phenomenon from different sources in palm oil mills. Thus, the main objective of this study was to characterize POME, understand the presence of the residual oil and solid particles in POME, and use correlation to determine the potential usefulness of residual oil recovery.

EXPERIMENTAL

Materials

Palm oil mill effluent (POME)

The palm oil mill effluent (POME) samples were collected from Landang Felda Besout Palm Oil Mill, Sungkai, Perak. For the characterization study, POME samples were collected from four different sources in the mill, as shown in Fig. 1. For the oil extraction study, POME samples were collected at the entrance of the mixing pond, representing the consolidated samples in the mill. Once collected, the samples were brought to the laboratory, cooled, and stored at -4 °C until further use.



Fig. 1. Schematic diagram of palm oil milling and sources of palm oil mill effluent (POME)

Methods

Chemical analyses

Standard methods of the American Public Health Association were followed. Chemical analyses, *i.e.*, for moisture content, total solids (TS), total suspended solids (TSS), pH, and oil and grease (O&G) were obtained (APHA 2005).

Visualization of oil droplets and solid particles

Light microscopy analysis was used to investigate the presence of residual oil droplets and solid particles in POME. Briefly, the sample was left for 1 h in a 50 mL beaker to settle in order to understand the oil loss phenomenon in POME. Then, 2 mL of the sample from the upper, middle, and bottom layers of POME were transferred into different glass tubes, 0, 30, and 60 min after settling.

A few drops of Sudan (III) dye (Merck Co., Germany) was added to the glass tubes. Finally, 1 mL of each sample was transferred onto clean glass slides, and a light microscope (Leica, Germany) was used to observe the oil droplets and solid particles. The experiment was carried out in triplicate.

Evaluation of residual oil percentage in POME by Soxhlet extraction

The Soxhlet extraction technique was performed to determine the residual oil percentage in POME samples. The method was based on EPA 3540C (Munaretto *et al.* 2012) using an analytical grade n-hexane (Merck Co., Germany). The dried POME samples were weighed, placed into filter paper extraction thimbles, and inserted into 500 mL reflux flasks. The extraction was carried out using 300 mL of n-hexane as a solvent at 150 °C. The extraction was terminated after 8 h, and the n-hexane was removed using an oven at 80 °C. After completion, the oil percentages were calculated based on the weight of the extracted oil.

Contact angle measurements

Contact angle, θ , is a quantitative measure of wetting of a solid by a liquid. It is defined geometrically as the angle formed by a liquid at the three phase boundary where a liquid, gas, and solid intersect (Anderson 1986). The purpose of the contact angle test is to determine the interfacial tension between the solids and oil/water. In this study, this measurement was used to determine the wetting properties of the solids particles in POME whether it is hydrophilic or hydrophobic and oleophilic or oleophobic.

Contact angle measurements were carried out for the POME samples using an OCA15ES Contact Angle Goniometer (DataPhysics Instruments, Germany). The residual oil in POME was removed during this process and subsequently the dry, oil-free POME was formed on a flat and rigid surface. One μ L of crude palm oil (CPO) was injected into the solid surface of POME, and the measurements were taken automatically. The oil absorption rate was calculated using the following equation (Eq. 1),

$$\text{Oil absorption rate} = \frac{DV}{\Delta t}$$
(1)

where Δt is the time for the oil droplets to reach equilibrium (sec), and *DV* is the dosing volume (μ L).

Settling velocity

Settling velocity of sediment particles, also called the terminal or fall velocity, is one of the key variables in the study of sediment transport and is important in understanding suspension, deposition, mixing and exchange processes (Song *et al.* 2008). Settling velocity of the particle in this fluid, v, where the particle has reached steady state. The purpose of settling velocity test in this study is to determine the settling time of solid particles in the sludge pit tank in the mills in order to relate with the efficiency of the current sludge pit tank design in the mills.

The settling velocity (v) and the settling time in sec of the solid particles in POME were calculated using Stoke's Law, as shown in the equation below (Eq. 2) (Ferguson and Church 2004),

$$v = g d^2 \frac{(\rho_p - \rho_m)}{18\mu}$$
(2)

where Vt is the settling velocity (m/s²), g is the acceleration of gravity constant (m/s²), d is the particle diameter (μ m), ρ_p is the density of the particle (g/mL), ρ_m is the density of medium (g/mL), and μ is the viscosity of water (Cp). The constants used are listed in Table 1.

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Constants	Values
Acceleration of gravity (g; m/s ²)	9.81
Particle Density (Pp; g/mL)	1.0410*
Density of the medium (water at 90°C; Pm; g/mL)	0.9653
Viscosity of water (µ;Cp)	1
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 Table 1. Constants used for Generating Settling Velocity

*Alrawi *et al*. 2013

RESULTS AND DISCUSSION

Characteristics of POME

The three major processing operations in the mill responsible for generating POME are sterilization, clarification, and hydrocyclone, contributing to approximately 36, 60, and 4% of the total POME generated, respectively (Ahmad *et al.* 2004). In this study, POME samples from all the major sources were collected for chemical analysis and labelled POME 1, POME 2, and POME 3, respectively. An additional source, labelled as POME 4, represented a consolidated sample collected before POME entered the treatment system. Table 2 shows the characteristics of POME samples taken from different sources in the palm oil mill.

	POME S	ource 1	POME	Source 2	POME	Source 3	POME	Source 4
Parameters	Range	Average	Range	Average	Range	Average	Range	Average
Water Contents (WC; %)	97.0 -97.5	97.25	92.9-93	92.95	98.1- 99.0	98.55	95.0- 95.9	95.45
Total Solids (TS; mg/L)	24,000- 24,600	24,300	64,200- 71,300	67,750	19,000- 19,300	19,150	40,600- 46,900	43,750
Total Suspended Solids (TSS; mg/L)	4,950- 6,290	5,620	35,700- 49,300	42,500	15,000- 16,400	15,700	19,800- 30,700	25,250
Oil and Grease (O&G mg/L)	749-768	758.5	200- 550	375	253- 385	319	294- 452	373
рН	4.2-5	4.6	4.5-6.9	5.7	4.8-5.0	4.9	4.1-4.5	4.3

Table 2. Comparison of Basic Characteristics of POME Collected from Different

 Sources

In palm oil mills, POME 1 originated from the sterilization process, which is the first contributor to the accumulation of POME. During this process, fresh fruit bunches (FFBs) were subjected to three cycles of extreme heat and pressure for 90 min (Rupani *et al.* 2010). The results showed that the average value of solid residues contained in POME 1 was the second lowest, at 24,300 g/mL. This was attributed to the sterilization process, which does not involve any harsh processing because the FFBs are not crushed at this stage. The main purpose of sterilization is to soften and loosen the fruits from the bunch and deactivate the enzymes responsible for the build-up of free fatty acids (Rupani *et al.* 2010). The total suspended solids in POME 1 were the lowest, at 5620 g/mL, compared to POME 2 and POME 3. Moreover, the oil and grease (O&G) content of POME 1 (758.5 mg/L) was the highest on average compared to POME 2 and POME 3. The residual oil may have come from the steam water flowing through the FFB during this process. Obviously, this was the first stage in palm oil processing, and the oil was not fully extracted yet. Therefore, the residual oil content was high.

POME 2 was generated after the oil clarification and purification processes. This was the stage where dirt and impurities are removed from the oil by centrifugation (Rupani *et al.* 2006). The moisture content of POME 2 was the lowest, at 92.95% compared to other sources. This occurred because it was subjected to higher temperatures in order to reduce the moisture content. Compared to POME 1, POME 2 exhibited higher levels of solid

residues, at 67,750 mg/L. This is because before the clarification process, the palm fruits were subjected to several harsh mechanical processes such as bunch stripping, digestion, and pressing (Ujang et al. 2010). In fact, the CPO was deposited in the mesocarp of palm fruits, meaning that the fruits needed to be crushed and pressed for maximum oil extraction. Due to these harsh processes, the mesocarp was transformed into solid residues, which were later mixed with the wastewater, *i.e.*, POME 2. The residual oil contained in the POME 2 (375 mg/L) was either attached to the surface or adsorbed in the matrix of the fibrous solid residues. There are two main adsorption mechanisms for which surface adsorption and capillary adsorption may have occurred (Huang et al. 2010). The fibrous solid residues, owing to the presence of non-polar groups on their surface, attract oil molecules to their surfaces by the surface adsorption mechanism and adsorb the oils into their matrixes by capillary adsorption (Huang et al. 2010; Subramaniam et al. 2010). On the other hand, the presence of hydroxyl groups in the solid residues attracts water through hydrogen bonding (Sahad et al. 2014). The residual oil may also be the small-sized free oil formed during the harsh mechanical processing before the clarification stage. The harsh forces would create turbulent pumping and break apart the oil layer, forming oil droplets in a water emulsion. The plant cells, including oil droplets, may be ruptured during the pressing stage, creating smaller oil droplets. These small oil droplets, covered by natural surface-active agents such as monoglycerides, phospholipids, and glycolipids, may resist the motion, causing oil loss in POME 2 (Chow and Ho 2002).

POME 3 was generated during the hydrocyclone washing and cleaning stage. This process involved the separation of cracked kernel mixtures from the shells in a winnowing column using upward suction (hydrocyclone) or a clay bath (Rupani *et al.* 2010). Therefore, POME 3 was extracted from the washwater of the hydrocyclone. The results obtained showed that POME 3 contained the lowest amount of solid residues, at 19,150 mg/L on average. During the processing, the only sources of solid residues were from the remnant fibers on the shells. The residual oil in POME 3 may have originated from the oils attached to the remnant fibers on the shells and from the cracked kernels within the nuts known as Crude Palm Kernel Oil (CPKO) (Subramaniam *et al.* 2010).

POME 4 was the consolidated POME or the mixture of the POME 1, POME 2, and POME 3 sources. POME 4 contained the last stages of oil recovery before the POME was pumped into the treatment plant. At this stage, POME was collected in a sludge pit and retained for 24 to 48 h to allow the residual oil to rise to the surface and be skimmed off. The purpose of this stage was to minimize the amount of oil getting into the treatment plant in order to avoid the inefficiency of treatment. The results revealed that even though POME 4 was the most consolidated POME, the measured total solids of POME 4 were lower than that of the POME 2 (Table 1). This may have occurred due to the settling of solid residues that were trapped along the drain before reaching the sludge pit tank. The residual oil content in POME 4 was measured as the second highest after POME 1, at 373 mg/L. This finding demonstrated that the oil was not fully extracted at the earlier stages of palm oil mill processing, even though all the extraction processes in the palm oil mill were completed. The residual oil in POME 4 originated from POME 1, POME 2, and POME 3, and was composed of extremely stable, small-sized oil droplets that were easily absorbed into mesocarp fibers.

Meanwhile, the pH value of POME 1, 2, 3, and 4 ranged from 4.2 to 5. The pH value from different sources of POME obtained in this study was close to the findings of previous studies, which reported the pH range of POME to be: 4.7 (Ahmad *et al.* 2003); 4.8 (Zinatizadeh *et al.* 2006); 4.5 (Zhang *et al.* 2008); and 3.6 (Ibrahim *et al.* 2013).

However, the values of pH recorded in this study were lower than IFC (2007) guideline values (pH 6 to 9) for the effluents from vegetable oil processing. The lower value of the pH indicates that the effluents is acidic (Ohimain *et.al.* 2012). The acidic characteristics of POME might be caused by the organic acids originated from palm fruits (Ohimain *et al.* 2007). According to Ohimain *et al.* (2007), the acidity of the POME can affect the nutrients of the plants if it is discharged into the soil, because the optimum pH for plan is range from 6.5 to 7.5.

Visualization of Residual Oil and Solid Particles in POME

Figure 2 compares the microscopic images of the upper, middle, and bottom layers of POME after settling for 60 min. As shown in Fig. 2, the number of residual oil droplets and solid particles in the upper layer were the lowest followed by the middle layer and bottom layer. According to Alade *et al.* (2011), the types of residual oil in POME are classified based on the droplet size, as shown in Table 3.

Sizes (µm)	Classification	
5-20	Soluble oil mixture	
20-150	Dispersed oil mixture	
>150	Free oil	

Table 3. Classification of Residual Oil in POME Based on the Droplet Sizes

Most of the oil droplets in POME were less than 100 μ m in size. Hence, the residual oil in POME include: free oil, dispersed oil, and the soluble oil mixtures. According to Chow and Ho (2002), the presence of small-sized oil droplets in POME may result from the turbulent pumping during palm oil processing. The oil cells from the palm fruits were ruptured during the harsh milling processes and an oil-in-water emulsion was created leading to oil losses as POME (Chow and Ho 2002). Once created, the small oil droplets were very stable because of natural surface-active reagents (surfactant), such as monoglycerides, phospholipid, and glycolipids that were released from the ruptured cellular membrane of palm fruit surrounding the oil droplets.



Fig. 2. Light microscope images of the (a) top layer, (b) middle layer, and (c) bottom layer of POME samples at 400X magnification

The results obtained in this study also revealed that most of the fibrous particles were generally bigger than the oil droplets and tended to settle to the bottom faster. One explanation is that the oil droplets may have been trapped inside the fibrous solid particles. Palm Oil Mill Effluent (POME) contains a high amount of organic substances that are mainly lignocellulosic materials (Sulaiman *et al.* 2009). As mentioned by Ahmad *et al.*

(2005), the solid fraction was mostly composed of fibrous debris that is oleophilic in nature and easily absorbs oil. Thus, during the settlement process, the solid particles blocked the oil droplets and settled to the bottom, carrying along the oil droplets that were trapped inside their fibrous matrix. Therefore, the residual oils were not able to rise to the surface, contributing to the loss of oil.

Dry Matter and Oil Yield Correlation

Figure 3 presents the results for dry matter (DM) and oil yield (OY). As mentioned earlier, the DM is actually the lignocellulosic debris material. This result showed that the oil contained in the palm oil fruit mesocarp was not fully extracted in the mill and turned into the residual oil, thus supporting the results obtained in the visualization of residual oil by light microscopy.

In the present experiment, before the drying process and Soxhlet extraction of oil by n-hexane, the settled sample formed three different layers of POME. The layers were classified as top, middle, and bottom layers. The time taken for settlement of the samples was 0, 30, and 60 min, respectively. The main purpose of sedimentation was to understand the behaviour of submerged residual oil in POME. The results showed that as the DM content of the top layer of POME decreased from 2.0 to 1.5%, the OY (%) from the top layer also decreased from 0.15 to 0.05 % after 60 min. Moreover, as the dry matter content of the bottom layer of POME increased from 4.0 to 6.8 %, the residual oil content increased from 0.25 to 0.43% after 60 min.

The data presented in Figure 3 shows the dry matter and oil yield obtained from top, middle and bottom layer of POME. This result indicate that the residual oil in the POME composed of the adsorbed oil that attached or adsorbed onto the solid particles in the POME. This is because the solid particles in POME are mainly oil-bearing cellulosic materials that have natural oleophilic properties (Madaki and Seng 2013). Thus, the residual oil droplets might be attracted to or adsorbed by the solid particles and were unable to coalesce and rise to the surface. This finding is supported by previous research, suggesting that some residual oil droplets are suspended in the solid particles (Ibrahim *et al.* 2013).



Fig. 3. Average dry matter and oil yield percentages. T: Top; M: Middle; B: Bottom

Contact Angle Measurement

To demonstrate the oleophilicity of the fibers and the CPO absorption, wettability studies were conducted. Wettability studies usually involve the measurement of contact angles as the primary data, in which the degree of wetting is related to solid and liquid intercalation (Yuan and Lee 2013). In this study, CPO and water (H₂O) were used to identify the wettability properties of dried POME. Table 2 shows the contact angle measurements and absorption results for both CPO and H₂O. Figure 4 shows that the contact angles of CPO and H₂O were both less than 45°. The contact angle results for both CPO and H₂O revealed that wetting of the surface was favourable, meaning that both fluids covered a large area on the surface of POME (Yuan and Lee 2013).

According to Sahad *et al.* (2014), a contact angle less than 45° indicates that the wettability properties of solid particles corresponds to their amphiphilic properties; this means that the material exhibits both hydrophilic and oleophilic properties. Thus, the solid particles in the POME were capable of absorbing both CPO and H₂O. However, based on the results obtained, POME was slightly oleophilic because the CPO contact angle was lower than the H₂O contact angle. Additionally, the absorption rate for CPO was higher than that of H₂O, 0.1103 ± 0.03 µL/s and 0.09 ± 0.005 µL/s, respectively.

Table 4. Contact Angle	Measurements for CPC	2 and H ₂ O with POME
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Parameter	Values
CPO contact angle (°)	19.35 ± 0.74
CPO absorption rate (µl/s)	0.11 ± 0.03
H ₂ O contact angle (⁰)	34.20 ± 1.80
H ₂ O absorption rate (µl/s)	0.090 ± 0.01

Values are expressed as mean ± SD



Fig. 4. Contact angles of CPO and H₂O with POME at equilibrium

The solid particles in POME are actually lignocellulosic fibers originating from FFB. These lignocellulosic fibres are three-dimensional, polymeric composites made primarily of cellulose, hemicelluloses, and lignin. These materials are responsible for most of the physical and chemical properties of POME, such as the natural oleophilicity and hydrophilicity (Hassan et al. 2013; Teli and Valia 2013). The high oil wetting properties or oleophilicty of the solid particles in POME result from the presence of the nonpolar groups on the surface of solid particles, which may induce the lower surface energy and increase the attraction of oil molecules (Shinoj et al. 2011; Sidik et al. 2012; Sahad et al. 2014). Owing to the natural oil sorption capacities of solid particles contained in POME, the oil droplets were absorbed into the solid particles and settled when the sample was left for 60 min. Moreover, the surface adhesion of solid particles in POME sludge was also very strong due to the high surface area and mesoporous structure of the fibrous materials (Tan et al. 2008). Hence, the residual oil found in POME was contributed by the absorption of oil in the fibrous solid particles. Furthermore, the hydrophilic properties, brought about by the presence of hydroxyl group of the solid particles, were enhanced by hydrogen bonding.

Estimation of Settling Velocity and Time of the Fiber Particles

Figure 5 shows the settling velocity (m/s) and time for the fiber particles to settle (3 meters depth) for the normal sludge pit design in the mill. Figure 5 shows that as particle size increased, the settling velocity increased, and therefore the time until settling decreased. This phenomenon might be affected by the properties of solid particles in POME. Allen (2003) argued that the particle's settling behaviour was dependent on the particle density, morphology, size, and distribution. According to Senthilkumar (2008), particle density is an important factor that affects the settling velocity of the particles. Senthilkumar (2008) concluded that when the density of the particles was high, the settling rate increased. In the present study, the particle density used for calculating the settling velocity of solid particles was obtained from Alrawi *et al.* (2013).



Fig. 5. A) The settling velocity (m/s) and B) settling time (sec) of solid particles in POME

In palm oil mills, the conventional practice is to re-collect the residual oil using a sludge pit retention system. More specifically, the consolidated POME from various sources was collected in a 3 meter high pit and retained for 8 to 24 h to allow the residual oil to rise to the surface and be skimmed off. Based on the results presented in Fig. 5, it was concluded that the present retention time was sufficient for the fiber particles to settle and for absorption of the oil droplets at the bottom of the sludge pit to take place. Therefore, a deeper study into the feasibility and efficiency of the sludge pit retention system is needed.

CONCLUSIONS

- 1. The characterization study showed that POME components and characteristics, such as total solids, suspended solids, oil and grease, and pH value vary, possibly because of the processing operations at the mill during the sterilization, clarification, and hydrocyclone stages.
- 2. The microscopic images showed that the highest quantity of oil droplets and solid particles was contained in the bottom layer, followed by middle and upper layer of POME. This indicated that fiber particles impeded the movement of oil droplets to the surface, since they were involved in the absorption of the POME oil, leading to oil

losses. Moreover, most of the oil droplets that presence in the POME as residual oil were the small sizes oil droplets

- 3. The solid particles in POME exhibited amphiphilic properties due to the low contact angle and high affinity for oil and water. Therefore, besides free oil, dispersed oil, and soluble oil, the residual oil in the POME also contributed to the adsorbed oil because it was attached to or adsorbed by the solid particles. These amphiphilic properties contribute to the high affinity the solid particles and the oil.
- 4. This study concludes that small sizes oil droplets and the oil that absorbed to the fiber particles are the main causes of the residual oil presence, which later results in the oil loss in the POME.

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