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EFFECT OF PROCESS PARAMETERS ON REMOVAL OF OIL FROM POME USING POLYPROPYLENE MICRO/NANOFIBER

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Abstract

In the present study, the effectiveness of the micro/nanofiber to adsorb oil was evaluated by varying the process parameters such as oil concentration, time, weight of micro/nanofiber and temperature. The reusability of the micro/nanofiber was also studied. The results showed that the oil adsorption capacity of the micro/nanofiber using refined palm oil was $29.4g\ of\ oil/g\ of\ MNF$. The oil adsorption process found that increasing on oil concentration, weight of micro/nanofiber and contact time showed increasing in oil removal (%), but increasing in temperature showed reduction in oil removal (%). The performance of micro/nanofiber in oil removal after pressing technique were decreased. However, solvent washing helps to regenerate the micro/nanofiber and increased the oil removal performance. Therefore, it was concluded that

the polypropylene micro/nanofiber had been demonstrated to be effective in removing oil from POME.

Keywords

Oil Removal, Micro/Nanofiber, POME, Polypropylene, Oil Recovery

1. Introduction

In palm oil milling process, about 2.5 tons of palm oil mill effluent (POME) are produced from every tons of oil extracted (Putri Primandari, Yaakob, Mohammad, & Mohamad, 2013). Typically, raw POME contents about 1 wt. % of oil and grease losses from the milling process (Bello & Abdul Raman, 2017). In facts, the oil losses in palm oil wastewater causes serious environmental pollution due to elevation of BOD and COD values. Besides, it is also very toxic for aquatic organisms, hence delay the treatment process (Madaki & Seng, 2013). Other than that, it is a great challenge dealing with this oily wastewater due to the present in emulsion form. Methods like solvent extraction and adsorption using several types of adsorbent are often times shows very good results in dealing with emulsified oil (Ahmad, Bhatia, Ibrahim, & Sumathi, 2005; Ahmad, Sumathi, & Hameed, 2005; Hameed, Ahmad, & Ng, 2012). However, solvent extraction is always questioned in terms of health and environment, due to toxicity of solvent itself even some solvent claimed as green solvent (Sahad, Md. Som, & Sulaiman, 2014). Adsorption process using adsorbent is much favorably in removal of emulsified oil from POME due to its effectiveness, reusability and safety.

Rubber powder and chitosan could be used as an effective adsorbent in removal of oil in POME (Ahmad, Bhatia, et al., 2005; Sethupathi, 2004). However, the reusability of these adsorbent might requires regeneration every time usage. Schatzberg (1971) reported that reusable sorbent could reduce the managing cost, as well as pollutant when comes into disposal. Another study on exfoliated graphite sorbent, Linh et al. (2016) stated that the utilization of this sorbent has disadvantages in terms of separation, where it was found very difficult to gather after adsorption process. Therefore, the technology that good in adsorbing emulsified oil in water, better in reusability and easy to gather in once piece after adsorption process is very favorably in this works.

Nanofiber is an emerging technology in oil removal. The application is widely range in oil spill control and clean-up in sub-marine industry, Other than that, the ability to adsorb oil in water shows the potential in adsorbing oil from POME (Avila et al., 2014). However, there are limited study have been reported in removal of oil from POME. Therefore, the polypropylene micro/nanofiber have been prepared in this work and studied the potential as oil sorbent in palm oil industry. Subsequently, study the reusability of the used nanofiber.

2. Materials and Methods

2.1 Preparation of Polypropylene Micro/Nanofiber

In this work, polypropylene-based micro/nanofiber was used as oil adsorbent in palm oil mill effluent (POME). This micro/nanofiber was generated using a melt-blowing system which consist of four major components, such as extruder, die assembly, web formation and winding (Rosalam, Chiam, Widyaparamitha, Chang, & Lee, 2016). The polypropylene resin (PP, Sun-Allomer) was fed to extruder from hopper, then melted at 300°C. The melted resin was carried by rotating screw to the die assembly at 10 Hz. After that, the melted resin was sprayed through spinneret (0.8 of diameter) to the web formation, at a distance of 2.4 m and a maximum flow rate of hot air was supplied.

2.2 Sample of Refined Palm Oil and Palm Oil Mill Effluents (POME)

The refined palm oil (Vesawit Cooking Oil) that used in this work, was bought from a local market. Whilst, the POME sample was retrieved at Sawit Kinabalu, Langkon Palm Oil Mill, Kota Marudu, Sabah, Malaysia. The point of raw material was collected, is located at the position before the wastewater flows into the cooling pond, which is called sludge pit. The POME sample was kept in 8 gallons containers and stored in room temperature for further experiment.

2.3 Determination of Oil Adsorption Capacity

The determination of oil adsorption capacity of micro/nanofiber was evaluated by using refined palm oil. A 100 g of refined palm oil was prepared into beaker and 1 g of micro/nanofiber was weighed (W_0). Then, the micro/nanofiber was gravitationally immersed in the oil to allow a complete adsorption and coalescence process for 10 minutes. The micro/nanofiber was removed and suspended for 10 minutes, to remove the loosely attached of oil on the micro/nanofiber. The micro/nanofiber with oil adsorbed was weighed (W_1). The oil adsorption capacity was calculated by gravimetric basis, which quoted as in Equation (1):

$$Oil\ adsorption\ capacity \left({}^{g\ of\ oil}/_{g\ of\ NF} \right) = \frac{W_1 - W_0}{W_0} \quad (1)$$

Next, the oil desorption process was done physically by hand pressing technique. A maximum human energy was used to press the micro/nanofiber. The oil was collected and weighed ($W_{oil,\ pressed}$), as well as the pressed micro/nanofiber (W_2). Subsequently, the micro/nanofiber was reused and recycled for 8 times of adsorption and pressing process in a fresh refined palm oil.

2.4 Effect of Temperature

The effect of temperature was studied at 30, 50, 70, 80, and 90°C. 5 g of micro/nanofiber (W_3) was used to remove oil from a beaker with 700 g of refined palm oil ($W_{oil,\ overall}$). The adsorption process was carried out for 10 min. Subsequently, the micro/nanofiber with oil adsorbed was removed and suspended for 10 min. Then, the micro/nanofiber was weighed (W_4). The percentage of oil removal was calculated from weight of oil adsorbed and weight of overall oil, which quoted as in Equation (2):

$$Oil\ removal\ (\%) = \frac{W_4 - W_3}{Woil,overall}\ x\ 100 \quad (2)$$

2.5 Effect of Oil Concentration

The adsorption of refined palm oil was carried out in a STEM-Reactor, which consist of 10 vials. The reactor was operated at 800 rpm and 30°C of temperature. The effect of oil concentration was studied at 0.2, 0.4, 0.6, 0.8 and 1.0 % of oil/water ratio ($W_{oil,\ overall}$) by using 0.04 g of micro/nanofiber (W_3). The micro/nanofiber was immersed into vial and the adsorption process was carried out for 10 min. Then, the micro/nanofiber was removed and put inside oven at 104-106℃, to remove water from micro/nanofiber. The dryness of micro/nanofiber (W_4) was determined by calculating the difference weight of micro/nanofiber every hours. The difference of weight micro/nanofiber should lower than 4%. The percentage of oil removal was determined by using Equation (2).

2.6 Effect of Time

The experiment on effect of time was carried out in the open tank setup, shown in Figure 1. The 1 g of micro/nanofiber was put inside the tank and let it gravitationally immersed in the POME sample. The oil adsorption on the micro/nanofiber was implied by monitoring the concentration of oil in the POME sample. The oil concentration was determined every 6 hours using the Standard Method of Oil and Grease Determination (U.S. Environmental Protection

Agency, 1999). The initial and final oil concentration were recorded and the oil content in the system at 0 hour (W_4 and $W_{oil,\ overall}$) and 18 hour (W_3) were determined by multiply with volume of the system. The percentage of oil removal was determined by Equation (2).

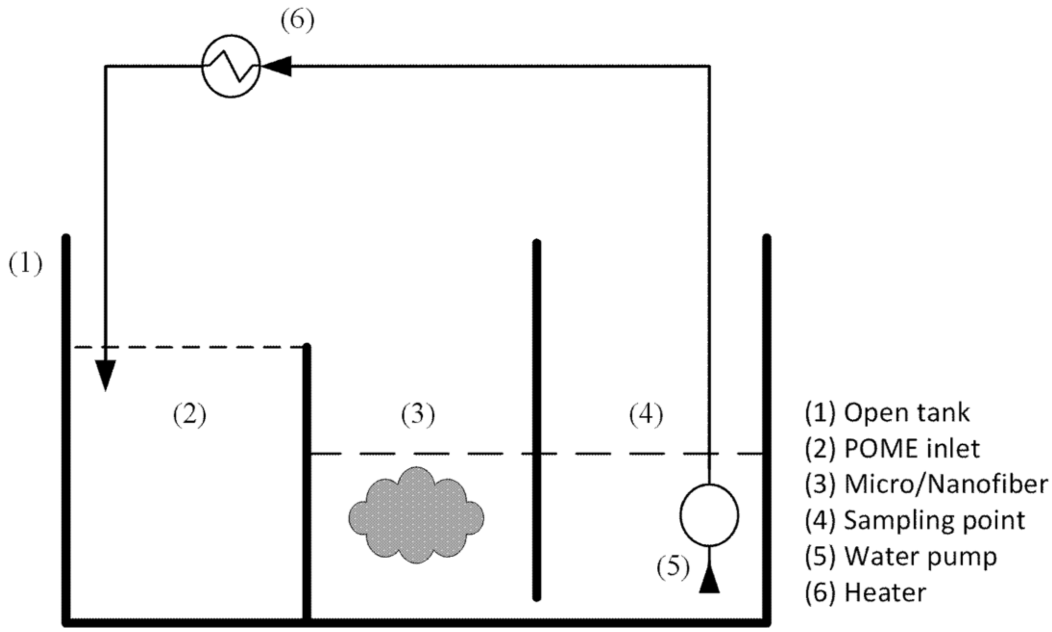

Figure 1: *The schematic diagram of oil adsorption process from POME in (1) open tank with three compartments; POME inlet (2), adsorption process using micro/nanofiber (3) and sampling point (4). The tank was consisted with water pump (5) with flowrate of 600 L/hour and heated using heater (6) at 60°C.*

2.7 Effect of Micro/Nanofiber Weight

The experiment process were repeated as in Section 2.6 by substitute the weight of micro/nanofiber to 2 g and 3 g for 12 hours of adsorption process.

2.8 Reusability and Regeneration of Micro/Nanofiber

The reusability of the micro/nanofiber was evaluated by recycling the pressed micro/nanofiber. 1 g of fresh micro/nanofiber (W_{R0}) was used in the oil removal experiment using POME. Subsequently, the oil recovery by desorbing oil from micro/nanofiber has been done via hand pressing technique. Then, the oil was collected and weighed ($W_{oil,\ R1}$). The pressed micro/nanofiber was weighed (W_{R1}) and reused in the second cycle of adsorption process for 12 hours. Afterwards, the micro/nanofiber was removed and suspended for 10 minutes. Next, the micro/nanofiber was pressed manually to recover oil and the oil collected was weighed ($W_{oil,\ R2}$).

After the oil desorption from second pressing technique, the pressed micro/nanofiber was continued into chemically desorption via solvent washing (Velu, Watanabe, Ma, & Song, 2003).

A 100 ml of solvent (n-hexane, 99%) was mixed into a beaker of the pressed micro/nanofiber and gently stirred for 10 minutes. The washed micro/nanofiber was then removed from the beaker and was dried in oven at 70°C. The solvent solution was filtered using vacuum pump and was distilled to extract the oil and to recover the solvent. The extracted oil was weighed *($W_{oil, R3}$)*, as well as the washed micro/nanofiber (W_{R3}). Then, the washed micro/nanofiber was reused in the third cycle of adsorption process and the oil was recovered via hand pressing technique, followed by solvent washing. The oil recovered from pressing ($W_{oil, R4}$) and solvent washing ($W_{oil, R5}$) were weighed. The pressed micro/nanofiber (W_{R4}) and washed micro/nanofiber (W_{R5}) were also weighed.

The percentage of oil removal in this works was determined using Equation (2). Whilst, the oil recovery yield from pressing and solvent washing were determined by dividing the weight of oil recovered and the weight overall oil adsorbed, which is quoted in Equation (3) and (4), respectively:

$$Pressing\ yield\ (\%) = \frac{W_{oil,Rn}}{W_{oil,overall}}\ x\ 100; n = 1, 2, 4 \qquad (3)$$

$$Solvent\ yield\ (\%) = \frac{W_{oil,R3} - W_{oil,R2}}{W_{oil,overall}}\ x\ 100 \qquad (4)$$

3. Results and Discussion

3.1 Oil Adsorption Capacity

The removal of oil from POME using polypropylene micro/nanofiber was initiated by determining the amount of oil adsorbed on micro/nanofiber. Figure 2 shows the oil adsorption capacity of 1 g micro/nanofiber using refined palm oil. The oil adsorption capacity from refined palm oil indicates that the fresh micro/nanofiber can adsorbed up to 29.42$g\ of\ oil/g\ of\ MNF$. The adsorption process occurred very fast about 8 to 10 s., due to oleophilic properties of the micro/nanofiber. However, the adsorption capacity were decreased as the micro/nanofiber was recycled after 8 cycles. The hand pressing technique may vary from the first cycle to the last cycle. Due to manual pressing by hands, different energy and pressure was given to desorb the oil from micro/nanofiber. So, a mechanical pressing process is recommended to get a uniform pressure to the micro/nanofiber.

Graphically, Figure 2 shows that micro/nanofiber has the potential of reusability. Such results show the evidence of the importance of regenerate the used micro/nanofiber to improve the adsorption performance, as well as reusability. Hence, the reusability and the regeneration of used micro/nanofiber have been studied in *Section 3.6*.

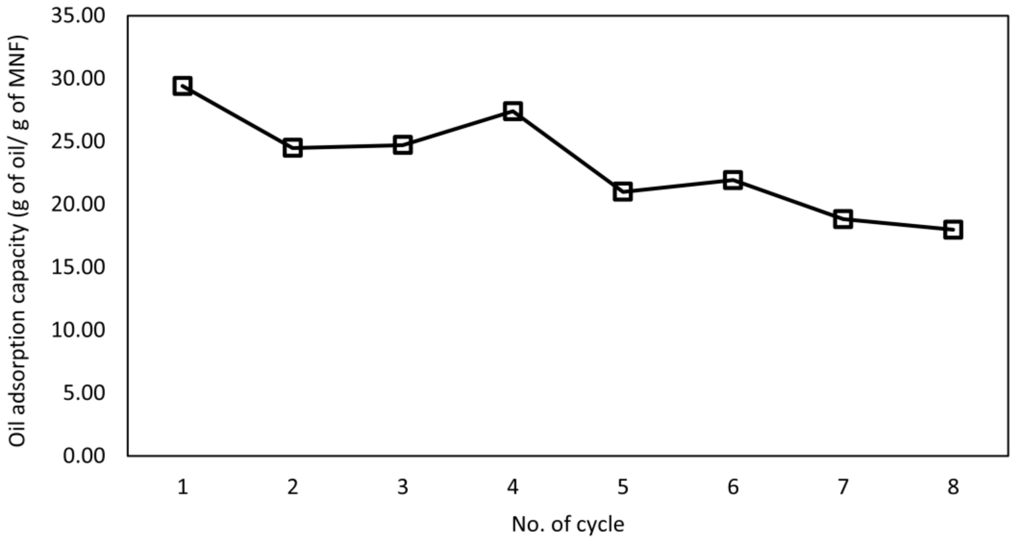

Figure 2: *Capacity of oil adsorbed in 1 g of micro/nanofiber from refined palm oil*

According to the Sarbatly and co-workers (2016), the oil removal efficiency has been influenced by different factors including oil viscosity and sorption selectivity between oil and water. Meanwhile, Wahi *et. al* (2013) reported that the fibers have been affecting by factors, such as sorbent dosage, contact time and initial concentration. Hence, different temperature and oil concentration of refined palm oil were studied, as well as contact time and weight of micro/nanofiber to evaluate the potential of micro/nanofiber on oil removal from POME.

3.2 Effect of Temperature

The oil viscosity is affected by the temperature. High temperature causes low of oil viscosity, thus increase the oil adsorption – desorption on the fiber (Cao, Yang, Wei, & Han, 2011). Figure 3 shows the effect of temperature on the oil removal from refined palm oil. The percentage of oil removal decreased as the temperature of oil increases. This indicates that low oil viscosity results the oil desorbed easily from the micro/nanofiber.

However, the palm oil milling process use steam at high temperature (80-90℃) to sterilize their full fruit bunch and other usage. Most of these steam end-up as POME in the pit with temperature range in 60-70℃. Subsequently, the POME flows into cooling ponds (wastewater pre-treatment pond) at 30-40℃ (Igwe & Onyegbado, 2007). Hence, it is difficult to control temperature in POME stream. But, the utilization of micro/nanofiber still can be deployed in oil removal from POME stream, either in pit or cooling pond where the temperature of POME has been cooling down from 60°C to ambient temperature.

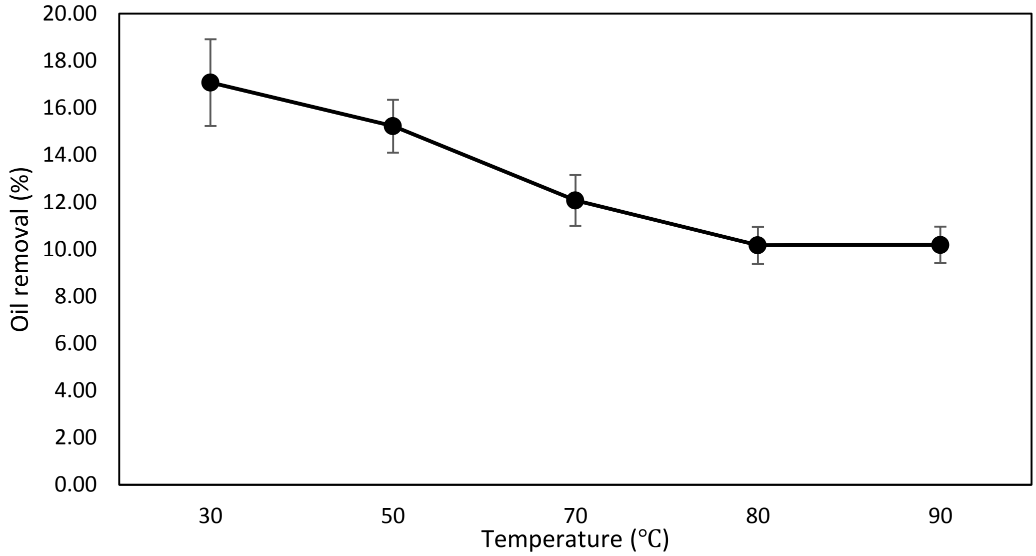

Figure 3: *The effect of temperature on the oil removal from refined palm oil with 5 g of micro/nanofiber*

3.3 Effect of Oil Concentration

The effect of the initial oil concentration was studied under fixed conditions (stirring time 10 min. at 800 rpm, temperature at 30°C and 0.04 g of micro/nanofiber). As POME contains of 1 wt. % of residual oil (Putri Primandari et al., 2013), hence the simulated POME was prepared by oil and water mixture with 0.2, 0.4, 0.6, 0.8 and 1.0 wt. %. The decreases of oil/water ratio were studied for the evaluation of micro/nanofiber adsorption in lower oil concentration, as well as its oil and water selectivity.

Figure 4 shows the effect of initial oil concentration in oil/water mixture. It is clearly shows that the micro/nanofiber able to adsorbed oil in the water. However, the error bars shows very high at 0.2 wt. % of ratio compared to 1.0 wt. %. This might be cause by the selectivity of oil and water to the sorbent surface (Deschamps, Caruel, Borredon, Bonnin, & Vignoles, 2003). The increasing of oil/water ratio indicates that the micro/nanofiber initially adsorbed the oil in the water and after that, adsorbed water or other impurities in the mixture.

In the physical appearance of POME, it is consist of oil, water and suspended solid. The suspended solid was found coated with oil, due to its oleophilic properties (Alrawi, Ab Rahman, Ahmad, Ismail, & Mohd Omar, 2013). This tells that the oil removal from POME using micro/nanofiber will adsorbed not only oil, but suspended solid and also water.

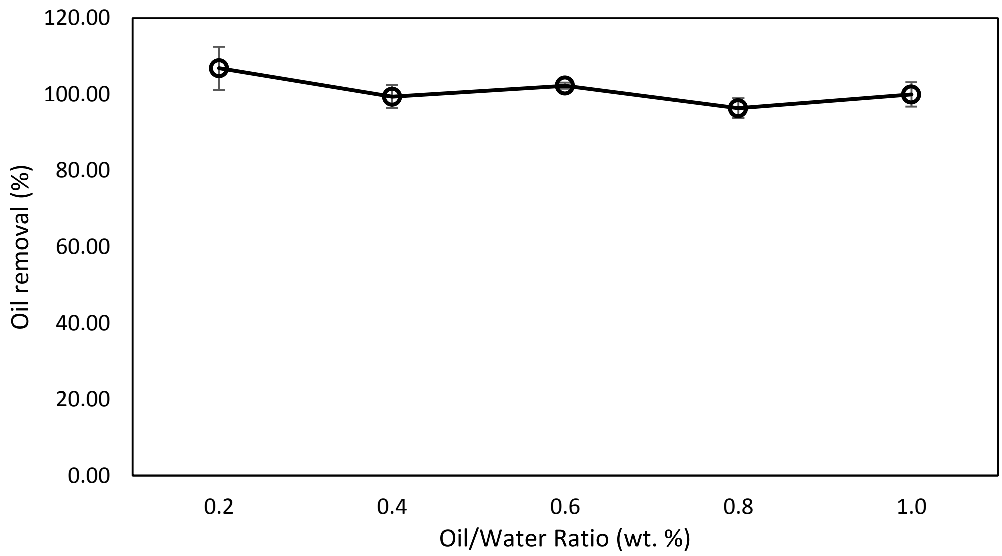

Figure 4: *The Effect of Oil Concentration on the Oil Removal in Oil/Water Mixture with 0.04 g of Micro/Nanofiber*

3.4 Effect of time

In this study, the experiment was done in the tank setup that shown in Figure 1 using actual POME sample. The effect of time was studied for determined the saturation time of the micro/nanofiber. According to Ahmad *et al.* (2005), the adsorption of oil will increased when the contact time also increase.

Figure 5 illustrates the relationship of oil removal (%) and time in adsorption process. The graph shows clearly that the percentage of oil removal from POME increased with increasing time of adsorption process. The rate of oil removed is rapid at the beginning and within 12 hours (68.76%), however slow down reaching to 18 hours (72.59%). Similar findings with Agarwal et al. (2017), the removal (%) was high at the beginning but slow down and almost constant after 3 hours contact. This work results indicated that the micro/nanofiber was almost saturated and has limited contact area between oil and micro/nanofiber, after 12 hours adsorption. Hence, this work concluded that 12 hours was considered as a sufficient time for the adsorption oil, as well as oil removal from POME using 1 g of micro/nanofiber. This is due to the other impurities were observed stuck on the micro/nanofiber and reduced the adsorption ability.

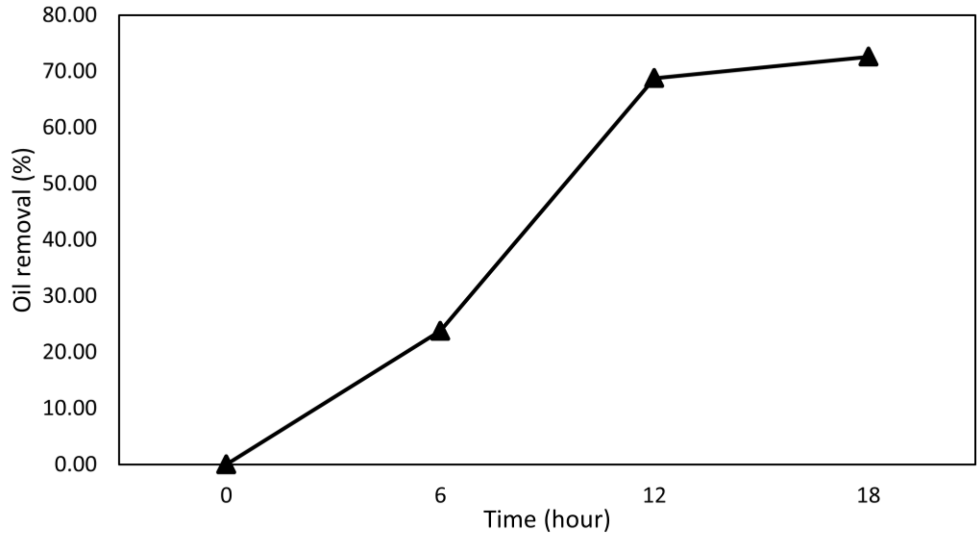

Figure 5: *The effect of time on the oil removal from POME with 1 g of Micro/Nanofiber*

3.5 Effect of Micro/Nanofiber Weight

The increasing of adsorbent dosage led to increases of oil removal (%) (Ahmad, Bhatia, et al., 2005). Figure 6 shows the effect of micro/nanofiber weight under fixed conditions in the tank setup (Figure 1). Three quantities of micro/nanofiber weight were studied on 1g, 2g and 3g for the removal of oil from POME.

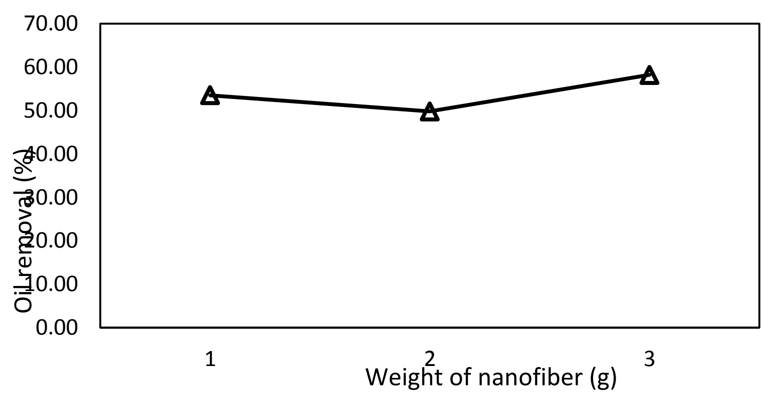

Figure 6: *The effect of micro/nanofiber weight on the oil removal from POME at a fixed temperature on heater (60℃) and flow rate (600 L/hr.)*

At 2 g of micro/nanofiber (Figure 6), the graph shows slight decreased in the oil removal (%). This could be explained by the initial oil concentration, where its concentration was 2 times higher than the oil concentration for 1 g of micro/nanofiber and almost similar concentration on 3 g of micro/nanofiber. However, the graph shows that the percentage of oil removal increased as the weight of micro/nanofiber increased. This pattern is associated that more available surface area for oil binding on the micro/nanofiber (Hamid, Malek, Mokhtar, Mazlan, & Tajuddin, 2016).

3.6 Reusability and Regeneration of Micro/Nanofiber

The reusability of micro/nanofiber has been studied by using 1 g of micro/nanofiber in the tank setup. Pressing technique has been deployed in desorbing the oil from micro/nanofiber. Subsequently, solvent washing has been carried out to extract oil that remained on the micro/nanofiber.

Figure 7 shows the performance of used micro/nanofiber over 3 cycles in removing oil from POME. The fresh micro/nanofiber as the first cycle in oil removal show that up to 30% of oil removed from POME. Then, the second cycle of used micro/nanofiber was reduced approximately to 20% of oil. At the third cycle, the percentage of oil removal was increased (38%) and higher than the first cycle. The result reduced at second cycle due to oil remained on the micro/nanofiber after pressing, which indicates of limited surface area for oil binding sites. At the third cycle, the micro/nanofiber has been washed with solvent before undergone the adsorption process. The results indicates that the oil has separated from the micro/nanofiber and homogenous with the solvent due to non-polar to non-polar behavior (Nwabueze & Okocha, 2008). Hence, the micro/nanofiber has more oil binding surface that available.

Pressing technique is considered as economical technique to recover oil from sorbent (Schatzberg, 1971). However, Sarbatly *et. al* (2016) reviewed the study on reusability and found that compression could breakdown the fiber results in decreasing in oil sorption capacity. Therefore, study on mechanical strength and maximum compression could reduce the breakdown of fiber. Thus, in this experiment found that micro/nanofiber has its retention quantity that indicates the maximum compression to recover the oil from micro/nanofiber. Figure 7 shows the pressing yield that indicates the oil recovery by pressing. The reusability of used micro/nanofiber by pressing have been evaluated and shows decreasing in the second cycle due to limited surface area for oil binding. Whilst, solvent washing helps to recover the remaining oil from micro/nanofiber. At the same time, the solvent washing also helps to regenerate the micro/nanofiber, which indicated by the increasing of oil removal (%) (Velu et al., 2003). The reusability at the third cycle shows increased almost twice from its oil removal after washed with solvent. Therefore, this results indicates the micro/nanofiber shows very good in reusability, either after pressing or washing.

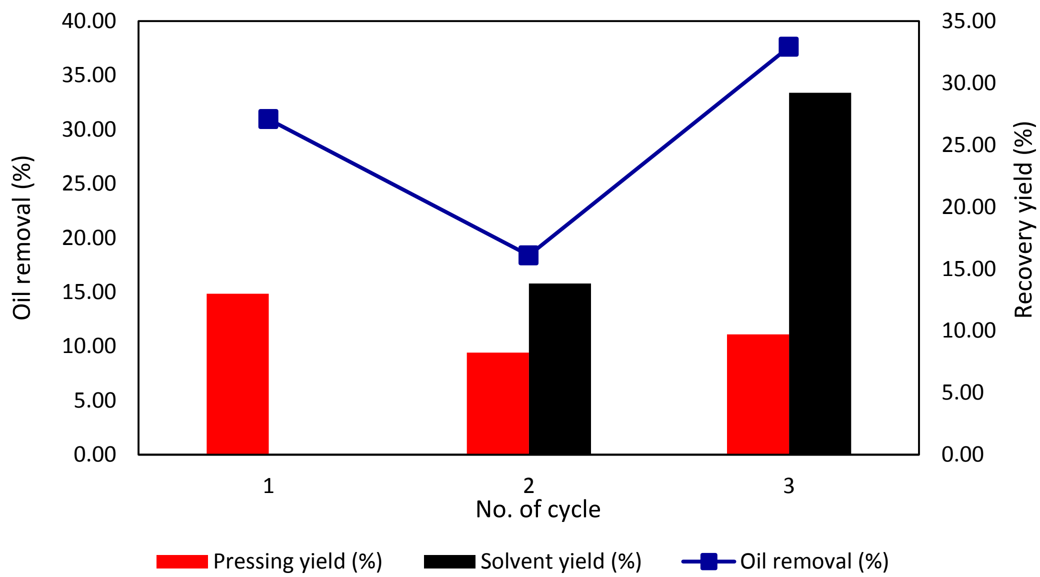

Figure 7: *Performances of used micro/nanofiber over 3 cycles in the oil removal (closed rectangular symbols) from POME and the yield on oil recovery using pressing (red bars) and solvent washing (dark bars) techniques.*

4. Conclusion

In this study, the oil adsorption capacity and the effect of process parameters in oil removal from POME using polypropylene micro/nanofiber have been studied. The fresh micro/nanofiber found that the oil adsorption capacity of micro/nanofiber was up to 29.42$g\ of\ oil/g\ of\ MNF$ using refined palm oil. However, the performance of micro/nanofiber in oil removal from POME was affected by factors, such as temperature, oil concentration, contact time and weight of micro/nanofiber. Increasing of oil concentration, weight of micro/nanofiber and time shows on increasing of oil removal (%), but increasing in temperature shows reduction in oil removal (%). Though the increasing of temperature reduced the chances of removing oil in POME, the micro/nanofiber is adsorbed oil due to its hydrophobic and oleophilic properties. Moreover, the micro/nanofiber founds a good result in reusability with and without regeneration. In conclusion, the polypropylene micro/nanofiber shows very good potential in oil removal from POME.

5. Acknowledgement

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