

The Effect of Operating Parameters on Ultrafiltration and Reverse Osmosis of Palm Oil Mill Effluent for Reclamation and Reuse of Water

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An attempt was made to reclaim and recover palm oil mill effluent (POME) for water reuse using tubular ultrafiltration (UF) and reverse osmosis (RO) membranes. The reclaimed water was compared with the final discharged water of the local mill. The raw POME was first subjected to a physical pre-treatment process to remove the content of organic matter and suspended solids. The pre-treatment process was coupled with membrane technology (UF and RO) to reclaim the clean water from POME. From the combined techniques of UF (5 bar) and RO (30 bar) the results showed that the turbidity and BOD₅ were reduced by 99% and 98.9%, respectively. Compared to the final discharged POME, this suggested method gives a significant difference in BOD₅ and turbidity. The final permeate of RO was found to comply with the standards for water reuse. Therefore, the combined UF and RO method is a viable alternative and has a great potential for use in the palm oil industry.

Keywords: Palm oil mill effluent; Tubular ultrafiltration; Reverse osmosis; Water reuse

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INTRODUCTION

Oil palm is an important crop in Malaysia, accounting for 39% of the world's palm oil production and 44% of the world's exports (MPOC 2010). About 4.49 million hectares of land in Malaysia are under oil palm cultivation and are producing 17.73 million tons of palm oil (MPOC 2010). Currently, due to the demand for palm oil and oleo chemical industries, its production is expected to increase (Ahmad *et al.* 2011). Given the huge capacity of this sector, it is critical to treat palm oil mill effluent (POME) to an acceptable level before discharging it.

By definition, POME is a thick brownish viscous liquid waste with an unpleasant odor that is high in colloidal suspended matter (Ahmad *et al.* 2009). Raw POME is a combination of 95 to 96% water, 0.6 to 0.7% oil, and 4 to 5% total solids (Ma 2000; Ahmad *et al.* 2003). Moreover, POME also has a high organic content, namely oil and fatty acids (Baharuddin *et al.* 2010). If POME is discharged without being treated properly, it is certain to pollute waterways due to its high biological oxygen demand (25,000 mg/L), chemical oxygen demand (53,630 mg/L), oil and grease content (8,370 mg/L), and suspended solid content (19,020 mg/L) (Ma 1995; Wu *et al.* 2007). For this reason, the palm oil industry has an enormous responsibility to face the problem in terms of environmental protection, economical viability, and sustainable development. It is important to find a way to treat the POME that addresses all of these concerns.

There are several innovative methods that have been developed and applied by palm oil mills for the treatment of POME. Currently, conventional biological treatments involving anaerobic or facultative digestion are the most popular (Quah *et al.* 1982; Ahmad *et al.* 2003). However, the biological treatment system requires proper maintenance and monitoring, as the bacteria are very sensitive to the changes in the environment. Thus, environmental conditions must be maintained that are conducive to the survival of the microorganisms. Attention from skilled operators and commitment from the management are also required. Moreover, biological treatment also generates vast amounts of biogas, which is corrosive and odorous (Ahmad *et al.* 2003). Because the effluent treatment is often viewed as the most significant burden, these issues have normally been ignored and given the lowest priority by mill owners. Due to these problems with the biological treatment method, it is believed that membrane separation technology will be able to treat POME in a more beneficial way. The membrane separation technology is normally coupled with chemical and physical treatment as a pre-treatment. Pre-treatment is necessary later to avoid high fouling affect toward the membrane. Commonly, fouling occurs due to the high content of sludge, colloidal matter, and suspended solids in POME (Ahmad *et al.* 2006; Wu *et al.* 2007). In the previous studies done by Ahmad *et al.* (2003), Ahmad *et al.* (2006), and Wu *et al.* (2007), most of the difficulties in membrane technology were to overcome the fouling membrane due to the POME characteristics. POME is rich in suspended solids, sludge, and colloidal particles which need to be degraded first. In this study, the membrane technology was coupled with pre-treatment to overcome the problem by using alum and lime. The combination of this coagulant and flocculant gave a huge good impact in water reuse recovery, making it possible to avoid the usage of acid or base to adjust the pH later in order to comply with the standard.

There are several advantages to use membrane separation technology. It can be applied across a wide range of industries; the quality of the treated water is more consistent regardless of the influent variations; it can be used in a process to allow the recycling of selected waste streams within a plant; highly skilled operators would not be required when the plant can be fully automated (Cheryan and Rajagopalan 1998); and the water reclaimed from this treatment could be reused in the mill. Thus, the primary advantages lie in the reduction of the cost for the water supply and its further treatment as well as in the effective elimination of the pollutant from the POME. Furthermore, if the methods of treatment are easy to operate, this method will then reduce the cost of operation (Wang *et al.* 2012).

There were several stages involved in the pre-treatment, such as pH adjustment, coagulation, flocculation, and sedimentation. The coagulant used in this study was aluminum sulphate hydrate (alum), which was reacted to destabilize the colloids in order to cause the formation of floc during the subsequent flocculation process. However, the pH adjustment process was eliminated from this pre-treatment because of the alkalinity of the flocculation agent used, which was calcium hydroxide (lime). The flocculation agent was used in the transport step, during which there are collisions between destabilized colloidal particles. In this way the colloidal particles form larger particles than those obtained in the coagulation process, whereupon they can be removed by settling and then filtration (Tchobanoglous *et al.* 2003).

The ultrafiltration (UF) and reverse osmosis (RO) membrane techniques were used to recover crystal clear water from the POME. The UF membrane has a finely porous surface layer or skin corroborated on a much more open microporous substrate.

That finely porous surface layer is what actually performs the separation for which the microporous substrate provides mechanical strength. Meanwhile, the UF retains the bacteria and viruses, which allows for its use as a water disinfectant (Arnal *et al.* 2004; Ahmad 2006). However, the UF permits the metal ions, aqueous salts, and non-protein nitrogen-containing compounds to pass through the membrane into the permeate (Perry *et al.* 1997; Ahmad 2006). In order to eliminate all the solute that the UF fails to reject, it is important to further treat the POME using an RO membrane. Reverse Osmosis (RO) is a process of desalinating water by means of a membrane that is permeable to water but impermeable to salt (Baker 2004). Pressurized water containing dissolved salt engages with the feed side of the membrane; water that contains less salt is withdrawn as low-pressure permeates. Generally, RO is a dense membrane through which liquid can permeate by a solution-diffusion mechanism. Selectivity depends on the solubility ratio and diffusivity ratio between the RO membrane and the liquid feed. It is believed that crystal clear water can be obtained after this membrane treatment and that the water can be reused again by the mill. Therefore, this study was conducted to investigate the effects of applied pressure on tubular UF and RO membranes with regards to the potential of these membranes for the treatment of palm oil mill effluent for water reuse.

EXPERIMENTAL

Materials

Samples of wastewater were taken from a local palm oil mill in Bahau, Negeri Sembilan. Raw palm oil mill effluent (POME) was taken from a sludge pit that had a temperature of around 80 °C to 90 °C, and the final discharge sample was collected from the anaerobic pond. All of the samples were stored at 4°C to avoid biodegradation due to microbial action. For the analysis and experimental purpose, the temperature of the sample was allowed to reach room temperature.

Pilot Plant Experimental Set-up

The raw POME was added with chemical coagulant alum at a dosage of 2000 mg/L without any pH adjustment. The mixing speed was set at 80 rpm for 1 minute to allow the formation of microflocs. A 2500 mg/L dosage of the flocculant, lime, was added during the slow mixing (30 rpm) over the course of about 30 minutes. The mixture was settled for about 30 minutes and filtered; a vacuum pump was used to filter the flocs. The 3.5 L of pre-treated POME was then prepared for the membrane treatment.

The model of the reverse osmosis and ultrafiltration unit that was used was FD-01, fabricated by Solution Engineering Sdn. Bhd (Malaysia). The process flow diagram for this unit is shown in Fig. 1. This unit was especially designed to accommodate UF and RO membranes. The membranes supplied with the FD-01 unit were classified as the tubular type with turbulent flow conditions. The system was in a cross flow configuration, in which the feed solution was pumped parallel to the membrane. The membrane housing is made up of 316 pieces of stainless steel with nitrile rubber seals. The effective area of the membrane used for the filtration was 0.024 m². The pre-treated POME was filled into the tank and pumped into the FP 200 tubular module PVDF UF membrane (MWCO of 200kDA) unit. The operating pressure used was varied at 1, 2, 3, 4, and 5 bar. The UF membrane can be operated using pressure of 1 to 10 bar. The cross

flow velocity used during UF membrane experiment was 22.92 m/h. The optimum UF permeate was then further purified using the RO membrane.

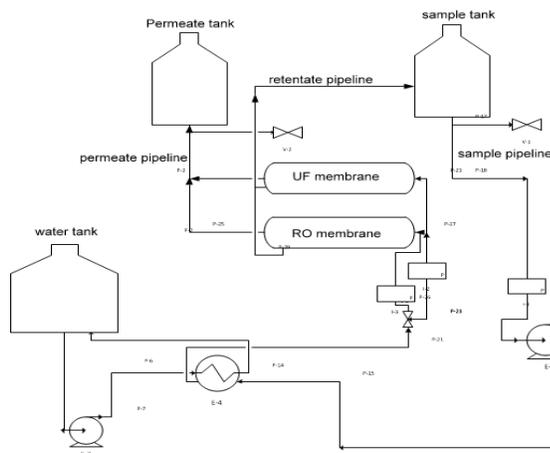


Fig. 1. Process flow diagram of experimental set up

The type of RO membrane used was polyamide film, AF99, which could remove 99% of NaCl at the pressures of 20, 25, 30, and 35 bar. A higher range of pressure was used in the RO process to overcome the feed side osmotic pressure (Fritzmann *et al.* 2006). Therefore, when the differential pressure was larger than the osmotic pressure, the directional flow was reversed, and water flowed from the concentrate to the dilute side. The RO membrane can be used up to 100 bar. The temperature was maintained at 25°C using a plate heat exchanger.

Analysis

The Biological Oxygen Demand (BOD₅) was measured using the Azide modification of the iodometric method. Meanwhile, the Chemical Oxygen Demand (COD) analysis was conducted using a dichromate reactor digestion method. The suspended solid analysis was carried out by evaporating the filtrate of the sample to dryness in a weight crucible and then drying it to a constant weight at 105°C. The addition in the weight indicated the presence of suspended solids. Turbidity was measured using a turbidity meter (Eutech, brand model TN 100). The pH was measured by a pH meter (Eutech instrument). The details of all the analytical methods conducted were based on procedures given in the APHA, Standard Method for the Examination of Water and Wastewater (Hammer *et al.* 2005). Each analysis was done twice. Membrane fouling can be observed by the reduction of flux through a membrane (WEF, 2006). It is caused by the buildup of contaminants at the surface or inside (pore) the membrane.

Jar Test

500 mL of raw POME together with solutions of aluminium sulphate hydrate (alum) at different dosages (1000 to 3000 mg/L) were added into each of the six beakers. Each sample was simultaneously mixed by the “ChemixFloc test”, which is one of the Jar test apparatuses. The mixing was conducted at 80 rpm for one minute, followed by slow mixing at 30 rpm for approximately 15 minutes. Then, the samples were allowed to settle for 30 minutes. To determine the optimum flocculant dosage, about 50 mL was pipetted from each beaker for analysis of turbidity.

While the optimum coagulant obtained earlier was maintained, the flocculant dosages were varied from beaker to beaker (1000 to 3000 mg/L). The flocculant used in this study was calcium hydroxide, $\text{Ca}(\text{OH})_2$ which also known as slaked lime. The flocculant was added during the slow mixing. 50 mL was pipetted from each beaker. The pH and turbidity were analyzed for each sample. Then, 3.5 L of sample was prepared based on the optimum dosages of flocculant and coagulant.

Permeate Flux Analysis

In accordance with another study, the permeate flux, J , was calculated by equation (1) (Tchobanoglous *et al.* 2003):

$$J = (1/A_m) dV/dT \quad (1)$$

where J is the permeate flux of solution ($\text{L}/\text{m}^2\cdot\text{h}$), A_m is the membrane area, dV is the amount of permeate collected, and dT is the time taken for the permeate flow to be collected.

Membrane Cleaning Procedure

The UF and RO membranes were cleaned after each experiment using a chemical cleaning method with the application of sodium chloride (NaCl). For the preparation of 3 L of NaCl solution, 45 g of NaCl was weighed. Then, the solid was immersed in 3 L of distilled water. The solution was stirred until the NaCl had dissolved completely. The same method was used to clean both membranes. First, the membrane was circulated with the clean water for about 10 minutes to flush out the POME. Then, the NaCl solution was circulated for about 20 minutes. For further cleaning, the membrane was circulated again with clean water for about 10 minutes.

RESULTS AND DISCUSSION

Quality of Pretreated POME

The effects of coagulant dosage were determined by analyzing the characteristics of the supernatant of the Jar test. Figure 2 shows that the turbidity decreased when the dosage was increased from 1000 to 2000 mg/L. However, the turbidity started to increase when the dosage of alum was higher than 2500 mg/L. The reason for this was that insufficient dosages of alum (<2000 mg/L) failed to neutralize the particle charges. On the other hand, when the dosage of alum was increased (>2000 mg/L), the turbidity started to increase again. It was found that when the alum dosage exceeded its optimum (saturation) for charge neutralization, the over-dosage caused the charges of the particles to reverse and later caused destabilization. The reversal of the charges hindered the completions of the coagulation process where the remaining particles in the supernatant increased in cloudiness (turbidity). This trend was also observed in the previous study by Ahmad *et al.* (2003). The optimum dosage was found to be 2000 mg/L, with a turbidity of 183 NTU.

Five different flocculant (alum) dosages were examined using the optimum coagulant dosage that was obtained. Alum was added during the slow mixing portion of the process. The flocculants agent used is an alkaline material, and thus further pH adjustment was not required. After the settling, sedimentation of flocs which comprise of

colloidal particles was observed at the bottom layer of the sample. As a result, the cloudiness of the supernatant was reduced.

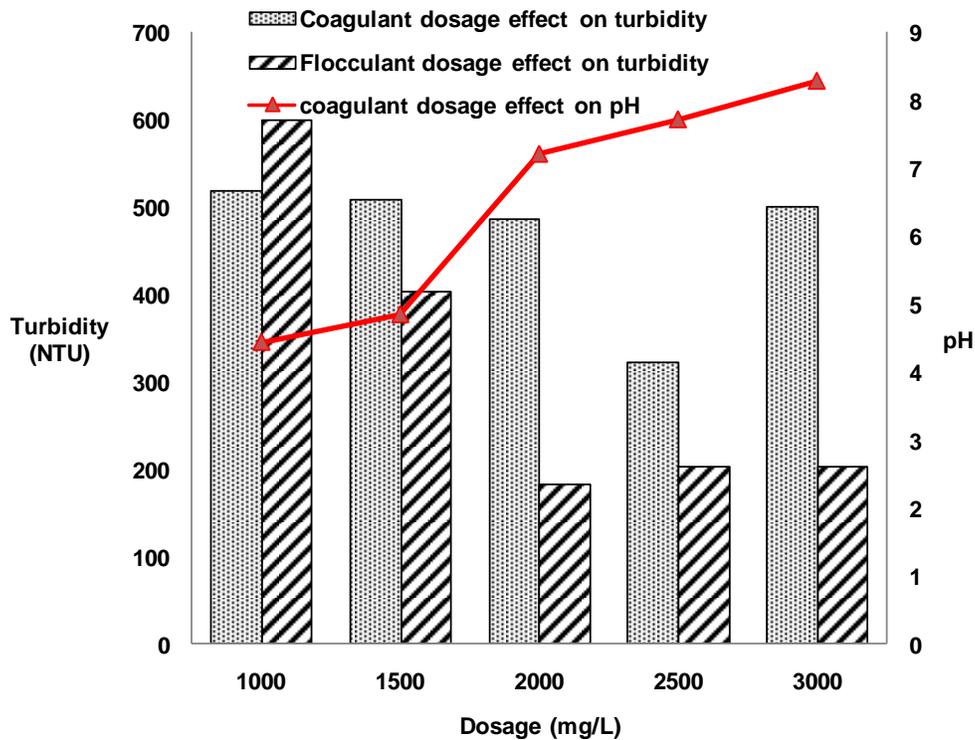


Fig. 2. Effect of coagulant (Alum) dosage on turbidity and effect of flocculant dosage (optimum Alum dosage + varied Lime dosage) on turbidity and pH

As seen in Fig. 2, the pH value was found to increase when the dosage of the Lime was increased. The results in Fig. 2 show that the turbidity decreased when the Lime dosage was less than 2500 mg/L. However, when the dosage was higher than 2500 mg/L, the turbidity increased. The increase in turbidity was due to the inability of flocculants to improve the settle-ability of the small particles. The optimum dosage for the flocculant was 2500 mg/L, with pH 7.72 and turbidity 321 NTU.

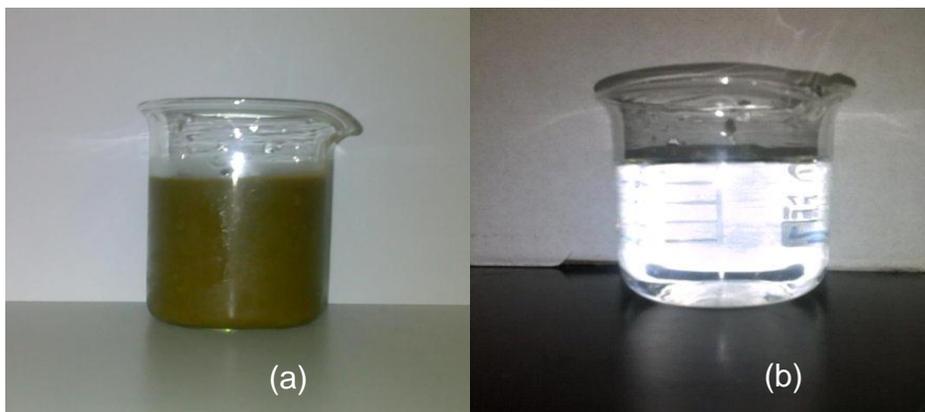
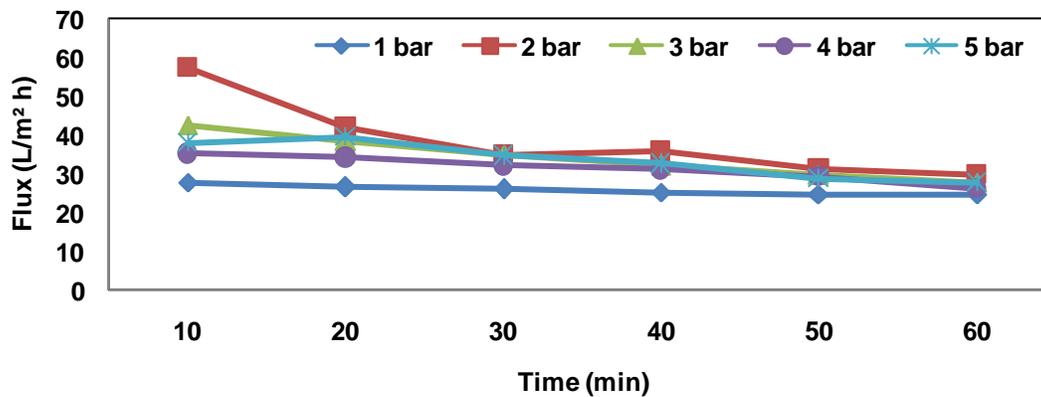


Fig. 3. Quality of water after membrane treatment: (a) Raw POME; (b) Permeate of reverse osmosis

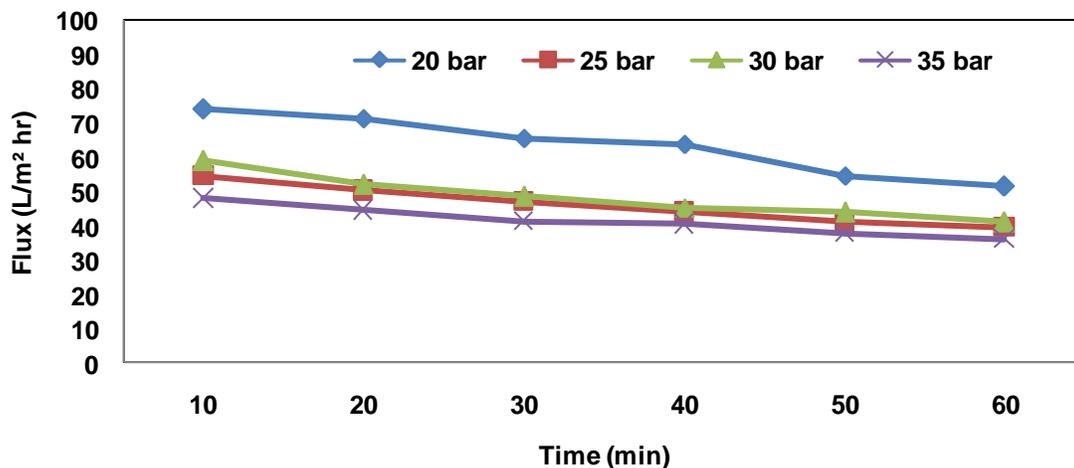
Permeate Quality and Effect of Applied Pressure

The visual and sensory observation showed that the quality of the reclaimed water had improved in terms of color and odor, as shown in Fig. 3. At the final stage of the treatment, the RO produced clear, odor-free water. The percentage removal of BOD and turbidity were 98.9% and 99%, respectively.

Figures 4(a) and 4(b) show that for the range of pressure between 1 and 5 bar for UF membrane and 20 to 25 bar for RO membrane, both the permeate flux decreased over time. These data were in agreement with the findings of Wu *et al.* (2007), who reported a decrease in permeate flux. This phenomenon could be explained in term of fouling and resistance of the membrane. The fouling and resistance of the membrane generally occur due to pore blocking, gel layer formation, or concentration polarization. The pore blocking occurs due to the structure of the pore membrane, which is unsymmetrical.



(a)



(b)

Fig. 4. Permeate flux. (a) Ultrafiltration with difference transmembrane pressure; (b) reverse osmosis with difference transmembrane pressure

There were some particles or carbohydrate or protein in the pre-treated POME that are not able to pass through the membrane but were trapped in the pores of the UF membrane (Wu *et al.* 2007). Hence, the pore was blocked by those particles. The concentration polarization occurs when the concentration of particles is higher near the membrane surface than it is in the bulk stream. Wu *et al.* (2007) also reported that carbohydrate and protein in POME can also form layer at the surface of the membrane which will caused fouling. When the membrane is used to separate colloidal matter, the flux is reduced (Baker 2005). RO membrane is a dense membrane that has high selectivity characteristics. Therefore, only substances that can dissolve into the RO membrane and diffuse through the segments of the macromolecules chains can pass through as permeate. Unfortunately, there might be some finer particles or other contaminant in POME that barely passes through the UF pore membrane that will dissolve in RO media but cannot diffuse. This will lead to the blocking in the RO membrane.

Table 1. Effect of Applied Pressure on pH, Turbidity, and Suspended Solids of the Permeate

Pressure (bar)	pH	Turbidity (NTU)
UF – 1 bar	6.70	26.7
UF – 2 bar	6.67	16.9
UF – 3 bar	6.72	7.01
UF – 4 bar	6.72	1.58
UF – 5 bar	7.12	1.08
RO – 20 bar	6.82	0.53
RO – 25 bar	6.78	0.08
RO – 30 bar	6.67	0.05
RO – 35 bar	6.56	0.07

In the UF membrane treatment, the pH value was different from one pressure to another. Table 1 shows that the highest pH was at 5 bar and the lowest at 2 bar. For the RO membrane treatment, the results showed different pH readings at different pressures. The RO membrane separation system was used to desalinate the treated POME. As a result, there was a change in pH from the initial feed pH (7.12). The trace amount of minerals and heavy metals that were retained increased with pressure due to concentration polarization. Consequently, the pH of the RO permeate was found to decrease with the pressure (5 bar) from the initial pH (7.12) of UF-treated permeate.

Turbidity indicates the cloudiness of the sample. The raw POME, which was a thick brownish liquid, had a turbidity of 8124 NTU. As seen in Table 1, the turbidity decreased when the applied pressure was increased. The UF permeate at 5 bar was less cloudy. The value of the turbidity was reduced by 43%, from 183 NTU (pre-treated POME) to 105 NTU (after UF treatment). Furthermore, the color of the raw POME was found to change from dark brown to clear yellow. This change in color reflected the reduction in turbidity value. A different trend was observed in the turbidity analysis for the RO membrane treatment. The turbidity decreased between 20 and 30 bar but increased again at 35 bar. When the pressure was too high, there might have been other retentate molecules that diffused through the membrane and mixed with the permeate.

For the UF membrane treatment, the best permeate quality was observed at a pressure of 5 bar and with a flux value of 33.38 L/m²h. The best permeate quality was found at 5 bar of pressure, because the pH value for the sample at 5 bar was 7.12 (neutral), and the turbidity was 1.08 NTU, which was the lowest. The pressure for the UF membrane was limited to 5 bar, because beyond this pressure, the permeate flux was insignificant (Wu *et al.* 2007). A similar trend could be observed for the RO membrane process. For the RO membrane treatment, the best quality RO permeate was found at 30 bar of pressure and with the flux value of 49.08 L/m²h. The turbidity and BOD₅ for the RO permeate were 0.05 NTU and 30mg/L, respectively, and the pH was 6.67.

Overall, it was proved that this membrane technology could completely remove and reduce the color, turbidity, organic matter, and BOD at the pH value of 6.67.

Quality Comparison of Final Discharged POME with the RO Permeate

This study showed that the variables of pH, turbidity, suspended solids, and BOD were improved at each stage of treatment. The turbidity of the raw POME was significantly reduced in the pre-treatment, from 8124 NTU to 321 NTU, and further reduced by the UF and RO treatments with the turbidity values of 1.08 NTU and 0.05 NTU. The decline in the turbidity could be correlated with the reduction in suspended solids through each treatment. This indicated that when the suspended solids were removed in the course of each treatment, the turbidity became lower. BOD₅ also decreased from 2700 mg/L to 30 mg/L after RO treatment because of the filtration of the macromolecules during UF and because of desalination during RO treatment. The BOD is reduced when the organic matter is removed from the POME.

As can be seen in Table 2, there were significant differences in each parameter between the RO permeate and the final discharge POME that was formerly treated using the pond system. When these two samples were compared in terms of pH, the RO permeate was slightly lower than pH 7, whereas the final discharged POME was slightly higher than pH 7. The turbidity was also lower for the RO permeate than it was for the final discharged POME, as the cloudiness of pre-treated POME was reduced during UF and RO treatments. The reduction is related to the removal of solid content during the pre-treatment stage and UF treatment. Furthermore, the value of BOD for membrane treatment (30 mg/L) was significantly lower than the value of BOD for the final discharged POME (630 mg/L).

Table 2. Comparison in Difference Analysis

Parameter	Raw POME	UF permeate (5 bar)	RO permeate (30 bar)	Final discharge POME	DOE standard discharge limit (Ahmad <i>et al.</i> 2003)	WHO water reuse standard (Vigneswaran <i>et al.</i> 2004; Saleem <i>et al.</i> 2011)
pH	3.93	7.12	6.67	7.78	5.0 to 9.0	6 to 9
Turbidity (NTU)	8124	1.08	0.05	105	-	-
Suspended Solid (mg/L)	5709	505.74	198.17	856.10	400	150
BOD ₅ (mg/L)	2700	-	30	630	100	200

The method suggested in this study is believed to reduce the operational time of the conventional method for the completion of one cycle to only two hours. Thus, the cost of the treatment and the pollutant in the POME can be reduced simultaneously. The

pond treatment usually used by the mill normally has a long hydraulic retention time (HRT) of 40 to 50 days, and to treat POME this way requires a large land area and low treatment efficiency in comparison to what is required by the membrane technology (Chin *et al.* 1996; Rupani *et al.* 2010).

The RO permeate obtained met both the discharge limit and the water reuse standard. Although the treated POME complied with the standard discharge limit, it would be more beneficial and valuable to reuse the water than to discharge it into the river. Moreover, all the parameters required were below the maximum for standard water reuse. The reclaimed water can be used for many purposes in the mill. Moreover, the high cost of membrane unit setup and maintenance can be overcome by the reduction in the cost for raw water supply and its further treatment. In addition, the pollutant from POME can be reduced effectively.

CONCLUSIONS

- 1) The flux value and pressure obtained for the best quality UF membrane permeate were 33.38 L/m²h and 5 bar, respectively, with the pH, turbidity, suspended solid, and BOD of 7.12, 1.08 NTU, 505.74, and 390 mg/L, respectively. The best quality RO permeate was found when the pressure was at 30 bar and the flux value 49.08 L/m²h. The results of the parameters analysis for pH, turbidity, and BOD were 6.67, 0.05 NTU, and 30 mg/L, respectively.
- 2) The present studies significantly show that UF and RO membrane technologies coupled with coagulation and flocculation as pre-treatment successfully reclaimed water from POME and allowed for its reuse. The water can be reused for many purposes in the mill.
- 3) It was found that the turbidity and BOD₅ of water reclaimed using the suggested method was significantly lower than those of the final discharged POME.
- 4) The crystal clear water that is obtained can be reclaimed, as it complies with the WHO water reuse standard.

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