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Performance of hydrophobic ultrafiltration membranes in the treatment and protein recovery from palm oil mill effluent (POME)

Abdul Wahab Mohammad[a]*, Peng Tong Yap[a], Ta Yeong Wu[b]

[a]*Scale-Up and Downstream Processing Research Group, Department of Chemical and Process Engineering, Faculty of Engineering and Built Environment, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor Darul Ehsan, Malaysia*
Tel. +60 (3) 89214529; Fax +60 (3) 89214660; email: wahabm@eng.ukm.my, wahabm@yahoo.com
[b]*Chemical and Sustainable Process Engineering Research Group, School of Engineering, Monash University, Jalan Lagoon Selatan, Bandar Sunway, 46150, Selangor Darul Ehsan, Malaysia*
email: wu.ta.yeong@eng.monash.edu.my, tayeong@hotmail.com

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ABSTRACT

An attempt was made to investigate the effect of molecular weight cut-off (MWCO) and applied pressure on the treatment and protein recovery from palm oil mill effluent (POME). Hydrophobic ultrafiltration (UF) membranes, namely polyethersulphone and polysulphone membranes were used in this study although hydrophilic membranes are generally used in the wastewater treatment and protein recovery. In order to reduce significantly the total suspended solids from POME before proceeding with the dead-end UF process, the raw effluent was first subjected to physical pretreatment processes (depth and surface filtrations) and microfiltration process. Then, a polysulphone UF membrane (20,000 MWCO) as well as polyethersulphone UF membranes (10,000 MWCO and 2,000 MWCO) were used in the study at different applied pressures, ranging from 1 to 10 bar. This study indicated that MWCO and applied pressure imposed a direct effect on permeate flux, POME treatment and protein recovery. In general, the hydrophobic UF membrane with the highest MWCO (20,000 MWCO) and operated at the highest applied pressure (10 bar) gave the best performance of POME treatment and protein recovery, in which case the highest reduction of total suspended solids, turbidity, chemical oxygen demand, total dissolved solids and protein recovery could be obtained up to 98.3%, 96.2%, 82.0%, 41.2% and 78.0%, respectively.

Keywords: Hydrophobic membrane; Palm oil mill effluent (POME); Protein recovery; Ultrafiltration; Wastewater treatment

1. Introduction

Palm oil production is a basic source of income for many of the world's developing countries in South East Asia, Central and West Africa as well as Central America. As such, the Malaysian palm oil industry represents a pillar of Malaysian economies and a catalyst for rural development where the country today is one of the world's leading producers and exporters of palm oil. However, the extraction of palm oil involves a number of processing procedures and a large amount of process water. It is estimated that for 1 tonne of crude palm oil produced, 5–7.5 tonnes of water are required, and more than 50% of the water will end up as palm oil mill effluent (POME) [1].

Raw POME is a colloidal suspension containing 95–96% water, 0.6–0.7% oil and 4–5% total solids including 2–4% suspended solids that are mainly consisted of

* Corresponding author.

debris from palm fruit mesocarp generated from three main sources, namely sterilizer condensate, separator sludge and hydrocyclone wastewater [2]. If the effluent is discharged untreated, it can certainly cause considerable environmental problems [3] due to its high biochemical oxygen demand (25,000 mg/L), chemical oxygen demand (53,630 mg/L), oil and grease (8,370 mg/L), total solids (43,635 mg/L) as well as suspended solids (19,020 mg/L) [4]. On the other hand, high compositions of bioresources (such as protein) found in the raw POME render it possible to be reused and transformed into different added-value products through cleaner production and environmentally sound biotechnologies [5].

Therefore, there is an urgent need to find a compromising way that will enable the balance between the environmental protection and recovery of bioresources found in the raw POME. In relation to the above matter, ultrafiltration (UF) together with the suitable pretreatment may offer a sustainable approach for managing POME. This is because UF not only plays an important role in wastewater treatment but also in recovery of macromolecules from the respective wastewater [6,7]. Recently, UF is being applied to an increasing degree for treating various types of municipal [8] and industrial [9] wastewaters.

The effect of hydrophobicity of the membrane material on UF of protein has also been the subject of many studies and hydrophilic membranes are preferentially used because of their low-binding properties. Nevertheless, Hanemaaijer et al. [10] reported a higher increase in retention values for polysulphone membranes (hydrophobic membranes) after contact with protein compared to the case with regenerated cellulose membranes (hydrophilic membranes). Lo et al. [6] and Wu et al. [7] also found that polysulphone UF membranes were able to treat and reclaim the protein from poultry processing wastewater and POME, respectively with significant efficiency.

The main aim of the present study was to investigate the potential use of hydrophobic UF membranes with 3 different types of molecular weight cut-off (MWCO), namely polyethersulphone (10,000 and 2,000 MWCO) and polysulphone (20,000 MWCO) membranes in the treatment and recovery of protein in the POME, in which case the recovered protein has been proven to be a usable fermentation substrate in protease production [11,12]. In addition, the effect of applied pressure (1, 5 and 10 bar) on membrane performance would also be investigated in this study.

2. Experimental

2.1. Pretreatment of POME

Raw POME was obtained from a local palm oil mill factory (Seri Ulu Langat Palm Oil Mill Dengkil, Kajang, Selangor) and the typical characteristics of raw POME are shown in Table 1. The effluent was first pre-filtered by means of depth filtration to remove the coarse solids found in the suspension. The raw POME was initially passed through a filter bed, which consisted of minor stones with the average size of 0.7 cm. Then, the collected filtrate was passed through another filter bed that consisted of mixture of minor stones and sand (average diameter size of 300600 μm) in the ratio of 1:2. Later, the filtrate from the second filter bed was subjected to surface filtration, under vacuum through a Whatman No. 41 filter paper (20–25 μm) and finally a Whatman No. 40 filter paper (8 μm) before proceeding to the microfiltration (0.45 μm) process at 1–1.5 bar. The filtrate after undergoing the microfiltration process was named as pretreated POME.

Table 1
Characteristics of raw POME obtained from a local palm oil mill factory

Parameter	Mean
TSS, mg/L	21,200 ± 1,320
Turbidity, NTU	17,500 ± 1,024
TDS, mg/L	36,800 ± 504
COD, mg/L	110,100 ± 6,647
Total protein, g/L	91.4 ± 18.9

Note: Values represent means of triplicate determination ± standard deviations

Pretreated POME was analyzed for total suspended solids (TSS), turbidity, total dissolved solids (TDS), chemical oxygen demand (COD) and total concentration of protein. Three repeatable experiments were conducted and the average values were recorded.

2.2. UF of pretreated POME

The flat sheet UF membranes used in the study were sourced from DSS Alfa Laval. These membranes are categorized as hydrophobic membranes by Alfa Laval and the characteristics of these UF membranes are summarized in Table 2. The experiment was carried out using a dead-end membrane filtration unit, namely a SEPA stirred membrane cell (maximum volume = 300 mL) from Osmonics Inc. (USA). The effective membrane area was 15.2 cm^2.

Before starting the experiment, the membranes were soaked overnight in distilled water to remove impurities left over from the manufacturing process or additives used for stabilization. The next day, the membranes were wetted out again by circulating distilled water at 10 bar for 30–60 min. The procedure prevents membrane compaction during permeation or separation experiments.

After this conditioning step, 300 mL of pretreated POME was subjected to UF at the pressure of 1, 5 and

Table 2
The characteristics of the UF hydrophobic membranes

Designation	Membrane material	MWCO	Operation limits		
			pH range	Pressure (bar)	Temperature (°C)
GR70PP	Polysulphone	20,000	1–13	1–10	0–75
GR81PP	Polyethersulphone	10,000	1–13	1–10	0–75
GR95PP	Polyethersulphone	2,000	1–13	1–10	0–75

10 bar (±0.5 bar). The duration of each experiment was 6 h. In all trials, the stirring speed was fixed at 6.67 Hz (or about 400 rpm). The system was pressurized with nitrogen and the temperature was maintained constant at room temperature of 25°C (±2°C).

The total permeate collected was analyzed accordingly for TSS, turbidity, TDS, COD and total concentration of protein. Three repeatable experiments were conducted and the average values were recorded.

2.3. Analytical methods

The total filtrate and permeate were collected and analyzed. Hach DR/2010, Portable Datalogging Spectrophotometer, USA was used for measuring TSS and COD of the samples. The TSS and COD were analyzed using photometric method (Hach Method 8006) and reactor digestion method (Hach Method 8000), respectively. Hach DR/2000 direct reading spectrophotometer, USA was used for measuring turbidity of the samples by applying absorptometric method. The TDS of the samples were determined by using electronic probe (inoLab, WTW GmbH).

The total concentration of the protein was estimated by Modified Biuret method using bovine serum albumin (BSA) as the reference standard [13]. Biuret assay was slightly modified by adding 100 μL of a 5% (w/v) deoxycholic acid ($C_{24}H_{40}O_4$) solution made up in 0.1 M KOH into 1.0 mL of a diluted sample. Next, 4.0 mL of original Biuret reagent, consisting of potassium hydroxide (KOH) and copper (II) sulfate ($CuSO_4$) as well as potassium sodium tartrate ($KNaC_4H_4O_6{\cdot}4H_2O$), was mixed with the 1.0 mL of the above diluted sample. Then, the mixture was incubated at room temperature for 30–45 min and the absorbance of the sample was determined at 540 nm. The concentration of the protein was determined from the BSA standard curve.

The reduction for TSS, turbidity, TDS, COD and protein in pretreated POME was calculated by using the following equation:

$$R_{\text{pretreatment}}(\%) = \left(1 - \frac{C_{\text{pretreated POME}}}{C_{\text{raw POME}}}\right) \times 100 \quad (1)$$

where $C_{\text{pretreated POME}}$ is the concentration of the pretreated POME and $C_{\text{raw POME}}$ is the initial concentration of the raw POME.

To justify the efficiency of UF process alone in reducing TSS, turbidity, TDS, COD as well as recovering the protein from pretreated POME, Eq. (1) with similar expression would be used:

$$R_{\text{UF}}(\%) = \left(1 - \frac{C_{\text{permeate}}}{C_{\text{pretreated POME}}}\right) \times 100 \quad (2)$$

where C_{permeate} is the concentration of the permeate and $C_{\text{pretreated POME}}$ is the concentration of the pretreated POME.

3. Results and discussion

3.1. Quality of pretreated POME

The total reductions for TSS, turbidity, TDS, COD and protein in pretreated POME are illustrated in Fig. 1. The results obtained are average percentage reduction from reproducibility data of three tests.

Fig. 1 shows that the pretreatment processes, consisting of depth and surface filtrations as well as microfiltra-

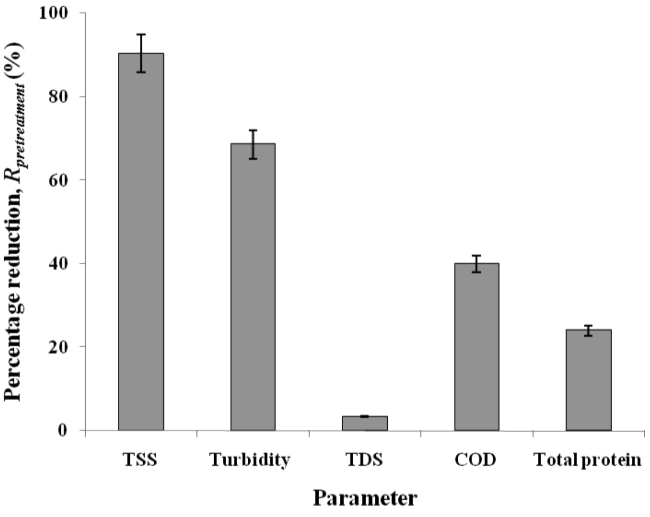

Fig. 1. Percentage reduction of TSS, turbidity, TDS, COD and protein after pretreatment processes. The percentage reductions are average values of triplicate tests.

tion, played a significant role in reducing almost 90.3% of TSS in POME. It is also shown in Fig. 1 that pretreatment processes were capable of reducing turbidity up to 68.6%. The great reduction of TSS in raw POME was necessary for reducing the possibility of fouling in UF membranes.

Only 3.40% of TDS could be reduced insignificantly via physical treatments and microfiltration (Fig. 1). The presence of significant amount of dissolved organic matter gave the overall low reduction of COD up to 40.0% (Fig. 1). The organic reduction efficiencies observed in this study are well comparable with those reported for depth and surface filtrations with average COD reduction efficiencies varied from 35.5 to 46.9% [7].

In this study, some of the protein (24.0%) in the raw POME was retained after the pretreatment processes. The availability of protein as nitrogen source in the suspended solids might be the reason why the solids could be reused as animal feed and fertilizer [5].

3.2. UF of pretreated POME

3.2.1. Effect of MWCO and applied pressure on permeate flux of pretreated POME

Fig. 2 shows the effect of MWCO and applied pressure on permeate flux of pretreated POME after 6 h of the UF process. In general, it is observed that the permeate flux increased with increasing applied pressure but the permeate flux started to level off at a higher applied pressure (>5 bar). This could be attributed to the gradual build-up of a cake layer on the membrane surface, especially at a higher applied pressure, in which case the cake layer formed at a higher pressure was denser and stable to disruption [7,14,15]. This would eventually accelerate membrane fouling and nullify the effect of higher applied pressure on permeate flux [9,16]. When the membrane was fouled, the permeate flux was usually difficult to stabilize [16]. As a result, the permeate flux could not form a linear relationship with the applied pressure.

The influence of different MWCO of the membranes on permeate flux could be deduced from Fig. 2. As expected, the polysulphone membrane with 20,000 MWCO showed the highest permeate flux (for all applied pressures involved) in comparison with the other hydrophobic membranes with lower MWCO. As a consequence, the permeate stream had the lowest resistance to its flow across the 20,000 MWCO membrane. However, it is envisaged that at higher applied pressure, the MWCO of the hydrophobic membranes would be greatly reduced thus giving higher resistance to their flows due to the occurrence of membrane fouling. According to Wu et al. [7,17], the rapid flux decline would take place about a few seconds or minutes once certain (hydrophobic) membrane was fouled by protein. This meant that once fouling occurred, the 100,000 MWCO membrane would immediately behave like a 10,000 MWCO membrane.

3.2.2. Effect of MWCO and applied pressure on the quality of permeate

In general, Fig. 3 indicates that more than 96% of TSS could be reduced by UF regardless of applied pressure and MWCO, though the highest reduction of TSS (up to 99.0%) happened at the highest MWCO and applied pressure of 20,000 and 10 bar, respectively. A similar result was also observed in the study carried out by Wu et al. [7] and Ahmad et al. [18], who claimed that a UF membrane was able to reject TSS exceeding 96% in the pretreated POME.

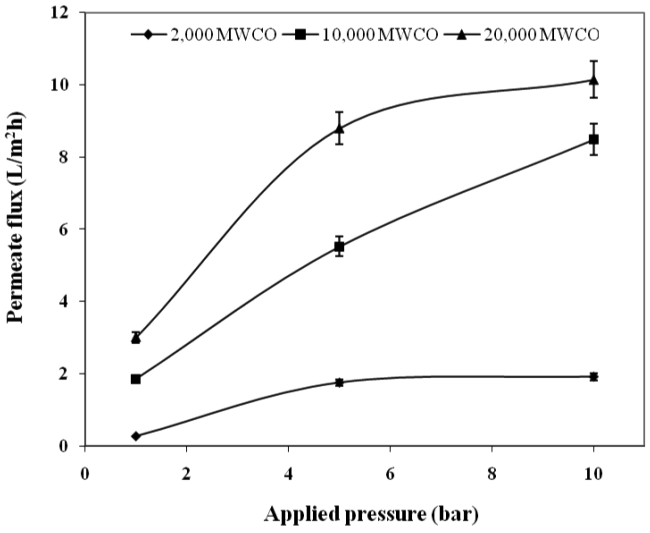

Fig. 2. Effect of applied pressure and MWCO of hydrophobic membranes on permeate flux. The permeate flux are average values of triplicate tests.

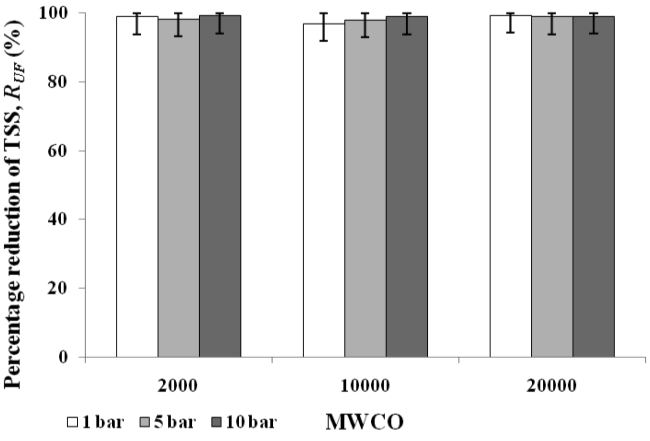

Fig. 3. Percentage reduction of TSS after UF process at 1, 5 and 10 bar for polysulphone membrane of 20,000 MWCO as well as polyethersulphone membranes of 10,000 MWCO and 2,000 MWCO. The percentage reductions are average values of triplicate tests.

The turbidity of pretreated POME after undergoing UF (Fig. 4) was further reduced, with up to 98.0% turbidity reduction could be observed at the highest MWCO and applied pressure of 20,000 and 10 bar, respectively. The high reduction of turbidity in this study was better than the result obtained by Ghosh and Balakrishnan, who used a polyethersulphone membrane (20,000 MWCO) in UF of sugarcane juice [19], in which only 31% reduction of turbidity could be achieved in the permeate of sugarcane juice.

The color of the pretreated POME (dark brown) after UF was significantly different before being treated at different MWCO and applied pressures. At the highest MWCO of 20,000 and pressure of 10 bar, the color of the permeate was light yellow, which corresponded well to the values obtained from the turbidity analysis (Fig. 4). Cassano et al. who used a polysulphone membrane (100,000 MWCO) in UF of 'grape must', also found that a decrease of the color intensity was associated with an increase of the clarity when the pressure was raised [20].

The percentage reduction of TDS (Fig. 5) was the lowest, comparing to the other parameters. This is because the UF membrane was suitable for extensive removal of TSS content, but it had difficulty in removing dissolved organics [18]. This is also the reason why the percentage reduction of COD (Fig. 6) was only within the range of 78.7–84.4%. In general, the reduction of COD increased with the increase in the applied pressure. This phenom-

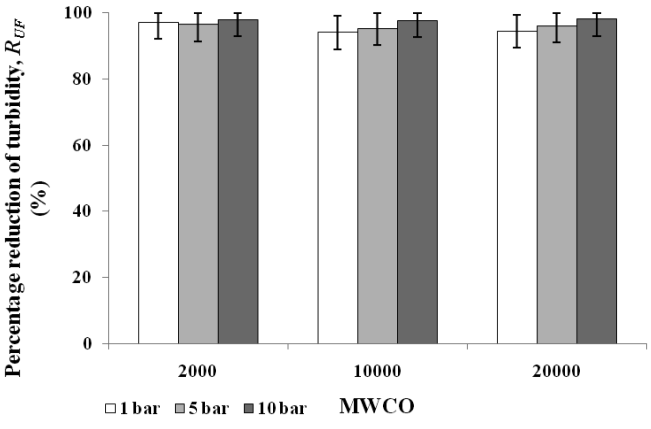

Fig. 4. Percentage reduction of turbidity after UF process at 1, 5 and 10 bar for polysulphone membrane of 20,000 MWCO as well as polyethersulphone membranes of 10,000 MWCO and 2,000 MWCO. The percentage reductions are average values of triplicate tests.

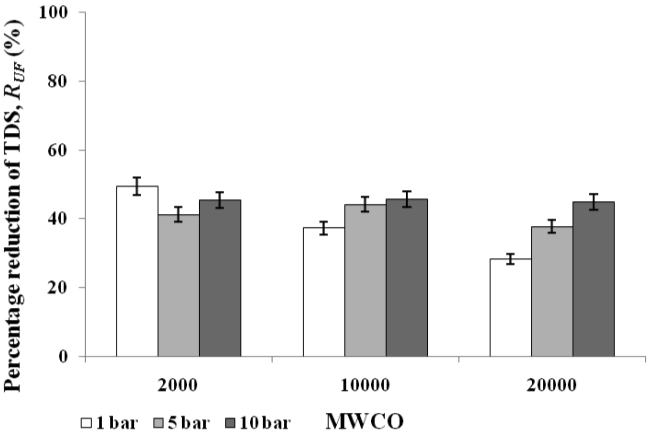

Fig. 5. Percentage reduction of TDS after UF process at 1, 5 and 10 bar for polysulphone membrane of 20,000 MWCO as well as polyethersulphone membranes of 10,000 MWCO and 2,000 MWCO. The percentage reductions are average values of triplicate tests.

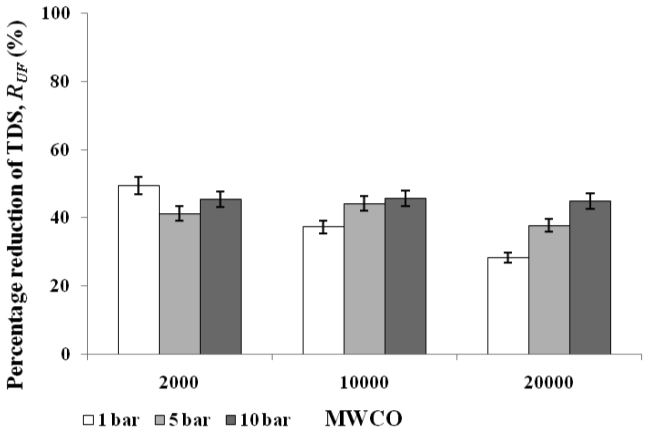

Fig. 6. Percentage reduction of COD after UF process at 1, 5 and 10 bar for polysulphone membrane of 20,000 MWCO as well as polyethersulphone membranes of 10,000 MWCO and 2,000 MWCO. The percentage reductions are average values of triplicate tests.

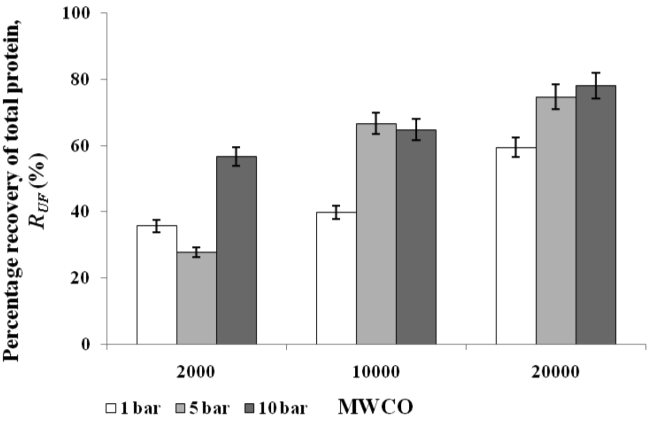

Fig. 7. Percentage recovery of total protein after UF process at 1, 5 and 10 bar for polysulphone membrane of 20,000 MWCO as well as polyethersulphone membranes of 10,000 MWCO and 2,000 MWCO. The percentage reductions are average values of triplicate tests.

enon might be influenced by the existence of a fouling layer and pore plugging, which could be exaggerated at a higher pressure [14]. However, Ahmad et al. [18,21] showed that transmembrane pressure did not influence the reduction of COD in the pretreated POME by using crossflow filtration.

Fig. 7 shows the effect of MWCO and applied pressure on protein recovery in the pretreated POME. In general, the protein recovery increased in line with increasing the applied pressure and MWCO, with the highest recovery of protein (78.0%) occurring at 10 bar by using the membrane of 20,000 MWCO in the UF process. These results indicated that solute rejection was greater at the higher filtration pressure, in qualitative agreement with the classical (Spiegler–Kedem) convection/diffusion model for solute transport, which predicts that solute rejection by a partially retentive membrane should increase with transmembrane solvent flux [7,14].

This study directly shows that MWCO and applied pressure would influence the percentage reduction of pollutants in pretreated POME and recovery of protein. From the results obtained (Figs. 3–7), the highest applied pressure of 10 bar would usually give the highest reduction of TSS, turbidity, TDS and COD as well as recovery of protein in the pretreated POME. This phenomenon might be influenced by the existence of fouling layer and pore plugging, which could be exaggerated at a higher operating pressure [7,14]. In general, the smallest MWCO of the membrane at the lowest applied pressure would have better reduction of pollutants and recovery of protein, compared to the larger MWCO of the membrane at the lowest pressure. However, further investigation implies that the membrane with the highest MWCO of 20,000 gave the best performance in pollutant reduction at the highest applied pressure of 10 bar. According to Harrison et al., the use of the membrane with higher MWCO would encourage not only the deposition of solutes on the surface of the membrane, but also the adsorption and blockage of solutes within the pores of the membrane [22]. In addition, Mousa and Al-Hitmi highlighted that fouling caused by the particles adsorbed within the membrane pores was more important than fouling caused by the cake layer formed on the membrane surface [23]. Therefore, fouling occurring at the highest MWCO and applied pressure might eventually lead to the best rejection of pollutants and recovery of protein in the pretreated POME.

4. Conclusions

Pretreatment processes consisting of depth and surface filtrations as well as microfiltration showed promising results in reducing TSS and turbidity up to 90.3% and 68.6%, respectively. Pretreatment processes could not reduce TDS (3.40% reduction) and COD (40.0% reduction) efficiently as the processes were designed mainly to retain the high concentration of suspended solids available in the POME. About 24.0% protein was retained together with the suspended solids as insoluble matters, which could be reused as fertilizer and animal feeds.

This study also indicated that the MWCO and applied pressure of the membranes imposed direct and significant effects on protein recovery, wastewater treatment as well as permeate flux of pretreated POME. In general, the highest reduction of TSS, turbidity, TDS and COD as well as recovery of protein occurred at the highest applied pressure and MWCO of 10 bar and 20,000 MWCO, respectively. Also, the permeate flux started to level off at higher applied pressure (> 5 bar). All these phenomena might be due to the higher accumulation of the retained solutes on hydrophobic membrane (that acts as a fouling layer) [24,25] and pore plugging, which could be exaggerated at higher pressure and MWCO, respectively [7,14]. However, if it is the objective to retain the lower molecular solutes in the retentate and for better wastewater treatment, higher applied pressure and MWCO can be used in a positive sense to increase the recovery of the solutes, thereby reducing the environmental risks from the effluent.

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