Filtration analysis and fouling mechanisms of PVDF membrane for POME treatment

Mohd Azwan Ahmad, Bidattul Syirat Zainal, Nashrah Hani Jamadon, Thomas Choong Shean Yaw and Luqman Chuah Abdullah

ABSTRACT

Palm oil mill effluent (POME) is a hazardous wastewater which contains high organic constituents and salt concentrations. The ultrafiltration (UF) process is a promising treatment design used for secondary treatment such as POME. However, membrane fouling is the major problem which limits the performance of the UF. This paper describes a detailed investigation of polyvinylidiene fluoride (PVDF) membrane for the treatment of POME. The fouling behavior was analyzed by water flux, fouling mechanism, scanning electron microscopy (SEM), particle size distribution (PSD) and Energy Dispersive X-ray (EDX). It was found that a significant reduction in the permeate flux was caused by the build up of a fouling layer. Study on the fouling mechanism shows that cake filtration dominated the fouling activities on the membrane surface, compared to standard blocking, intermediate blocking, and complete blocking. This result is supported by membrane autopsy through SEM, PSD and EDX.

Key words | blocking mechanism, palm oil mill effluent, polyvinylidiene fluoride, ultrafiltration

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HIGHLIGHTS

- The treatment of palm oil mill effluent via PVDF membrane was discussed.
- Membrane fouling was investigated by the fouling model, SEM, PSD and EDX.
- Cake filtration was the main fouling model compared with standard, intermediate and complete blocking.

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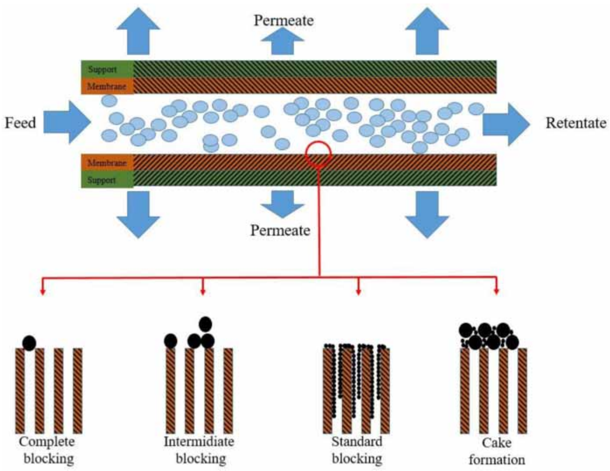
GRAPHICAL ABSTRACT



INTRODUCTION

Malaysia is known as one of the largest exporters of palm oil with an average production of crude palm oil of more than 13 million tonnes per year (Subramaniam *et al.* 2018). However, the production of wastewater from the palm oil mills, known as palm oil mill effluent (POME), contributes to the highest pollution load that is discharged into rivers all over the country (Taha & Ibrahim 2014; Ghani *et al.* 2017). There are three major processes contributing to the production of POME which is hydrocyclone, sterilizer condensate, and oil clarification in a ratio of 1:9:15, respectively (Wu *et al.* 2010). POME is a thick brownish colloidal with a mixture of oil, suspended solids and water, that is discharged at a temperature of 80–90 °C, and considered as a non-toxic wastewater because no chemicals were used during the extraction processes (Alrawi *et al.* 2013). The properties of POME are 4–5% of total solids, 0.6–0.7% of oil and grease and 95–96% water. Even though POME is a non-toxic wastewater, it contains soluble elements with different types of liquids, waste, residual oil and suspended solids that are particularly unsafe to the environment, either in the form of soluble gases (such as ammonia (NH_3), sulphur dioxide (SO_2) and methane (CH_4)), soluble solids or liquids, with concentrations exceeding the threshold limit values (Mohammed & Chong 2014). When the water quality declines due to untreated wastewater such as POME, it often leads to outbreaks or waterborne infectious diseases (Pons *et al.* 2015). It is noted that 85% of the palm oil mill in Malaysia uses a ponding system as a treatment method for POME, but it requires a high retention time and large area of land (Abdurahman & Azhari 2018).

The application of membrane separation processes have increased significantly in recent years and it has been adopted in many industries because of its ability to treat water and wastewater efficiently, it is economical and easy to operate (Shad *et al.* 2019). Generally, a membrane can be identified as a thin layer of semi-permeable material which separates substances and allows one part of a mixture to permeate the membrane, while inhibiting the other when a driving force is employed (Hai *et al.* 2014). Up to now, there are four different pressure-driven membrane types used in water and wastewater treatment, namely microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO). UF is a cross-flow membrane process which is able to eliminate all microbiological species, viruses and humic materials (Daisuke *et al.* 2017). Previous studies show that UF membranes are able to treat wastewater and maintain the turbidity value of the effluent below 0.1 NTU, and also eliminate particulate metals such as iron and manganese effectively (Ioannis *et al.* 2017). In the treatment of bilge wastewater, Grypta *et al.* (2001) found that UF membranes are able to reduce the oil content below 5 ppm and completely remove the oil pollutants when the second stage of filtration is applied. In the treatment of oil-water/wastewater, the UF membrane has been validated as a promising filtration technique on account of the appropriate pore sizes (usually in the range of 2–50 nm) and the ability of eliminating emulsified oil beads with no de-emulsification forms (Racar *et al.* 2017).

In the treatment of wastewater using UF as a filtration system, polyvinylidiene fluoride (PVDF) has been chosen to fabricate UF membranes compared to other polymers because of strong chemical resistance and mechanical properties (Kim *et al.* 2015). Buscio *et al.* (2015) applied the PVDF for the treatment of textile wastewater and their result showed that chemical oxygen demand (COD) and color removal was 60 and 30%, respectively. Additionally, no fouling was detected during their experiments (Buscio *et al.* 2015). In another PVDF membrane treatment on water contaminated by lead, Zhao *et al.* (2016) discovered that PVDF with a surface area of 12.56 cm^2 and an influent concentration of 224.5 mg/L was able to treat 13.9 L of lead contaminated water (equivalent to 73,000 of bed volumes) that meet the maximum contaminant level of 15 mg/L. It shows that the PVDF membrane was able to remove lead with significant results and had a better reusability in its applications (Zhao *et al.* 2016). The use of UF membrane is very effective in water treatments, however the major drawback of the system performance is the fouling behavior during the treatment processes (Kasi *et al.* 2017; Lee *et al.* 2018). Membrane fouling can be defined as the accumulation of foulant on the surface or pore of the UF membrane. In terms of fouling mechanism, basically there are four types of fouling model which have been used to analyze the fouling of membranes by different types of water reclamation with complex compositions (Huang *et al.* 2008). Those four types of fouling models are cake filtration, standard blocking, intermediate blocking and complete blocking. If the particle size of the foulant is larger than the size of the membrane pore, the deposition of this particle on the membrane surface will contribute to the growth of cake layer. On the other hand, if the particle size of foulant is smaller than the size of membrane pore, particles can enter the membrane pore and block the pores. The fitting equation of a fouling mechanism are shown in Table 1 (Iritani 2013). The main objective of this study was to investigate the PVDF performance in the treatment of POME and fouling behavior during the treatment processes. The equation in Table 1 was used to determine the type of fouling mechanism. A 0.0125 μm pore size PVDF membrane with a molecular weight cut-off (MWCO) of 200 kDa was chosen for the treatment process. The sample used was from the final discharged pond effluent of palm oil mill.

MATERIAL AND METHODS

Feed solution

POME was obtained from the Seri Ulu Langat Palm Oil Mill, Dengkil, Malaysia. The effluent sample was collected at the point of discharge to the anaerobic ponding system. Initially, the visual inspections were carried out at multiple points of the sampling site in order to evaluate the presence of a constitution, sample color, odors, etc. After collection, the sample was then tested for COD, total suspended solids, pH, color and turbidity according to the standard

Table 1 | Summary of the fitting equation of fouling mechanism

Fouling model	Model equation[a]	Model description
Cake filtration	$\frac{1}{Q}=\frac{1}{Q_i}+K_cV$	Particles deposit on each other and settle down on the surface of membrane, blocking the membrane pore and form a cake layer
Standard blocking	$\sqrt{Q}=\sqrt{Q_i}-\left(K_c\sqrt{Q_i}V/2\right)$	Small particles attach on the interval walls of the membrane pores
Intermediate blocking	$\frac{1}{Q}=K_ct+\frac{1}{Q_i}$	Approaching particles contact with the existing particles on the surface of membrane and block the membrane pores
Complete blocking	$Q=Q_i-K_cV$	Particles deposit on the membrane surface that is larger than the membrane pores

[a]K_c is blocking filtration constant, V is permeate volume, Q is the volumetric permeate flow rate, and Q_i is the initial volumetric permeate flow rate.

methods. The sample was then restored at 4 °C for further use. The percentage reductions of the sample parameters were calculated by the following equation:

$$C(\%) = \left(1 - \frac{C_b}{C_a}\right) \times 100$$

where C_b is the concentration of the permeate solution and C_a is the concentration of the feed sample.

UF materials and procedures

The filtration system was connected to two tubular crossflow UF membrane modules (PCI Systems, UK). Before the experiment began, the UF membrane was washed with clean water to eliminate any unwanted materials left after the manufacturing procedures. Later, the UF membranes were washed out again by circulating clean water for 60 min at 100 kPa. This procedure can avoid membrane compaction during the permeation or separation process. Experiments were run according to the total experimental configuration where retentate and permeate were both recycled back into the feed tank to assure a steady state in the composition and volume of the feed solution.

The experiments were performed based on two main parameters, namely applied pressure and feed flow rate. For each experimental run, the cross flow velocity and applied pressure were controlled by adjusting the pressure and flow regulator valve. The applied pressure beyond the membrane was monitored by the manometer at the inlet and outlet of the UF system, while the cross flow velocity was monitored by a flow rate indicator located before the membrane module. The output of each experiment was measured over 2 h (time is based on pre-experimental runs which shows static value after 2 h) wastewater filtration experiments, through analytical analysis with concentrate and permeate samples. The membrane concentrates stream was returned back to the feed tank to maintain constant oil concentration operation. Permeate flux was acquired by weighing permeate with an electronic balance. The experiments were repeated three times, and the average values were recorded. At the end of each experiment, chemical cleaning of the UF membrane was executed for further use.

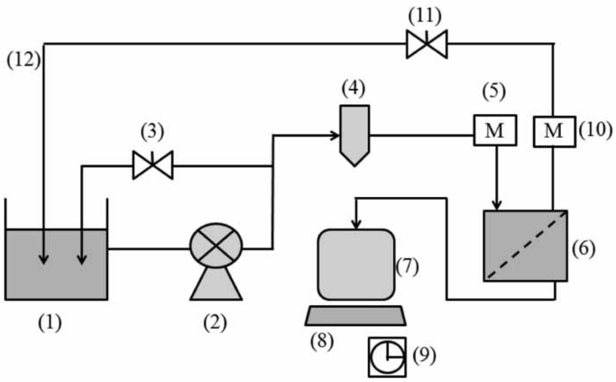


Figure 1 | Schematic diagram of experimental setup. (1) Feed tank, (2) gear pump, (3) valve, (4) flow rate indicator, (5) manometer, (6) UF membranes module, (7) permeate tank, (8) electronic balance, (9) stop watch, (10) manometer, (11) valve, and (12) return of concentrate stream.

Investigation on membrane fouling

The fouling experiments were carried out using a laboratory scale cross-flow membrane filtration unit as shown in Figure 1. A new UF membrane module was used for every fouling experiment. Based on the fouling model as stated in Table 1, cake filtration, standard blocking, intermediate, and complete blocking model at each applied pressure of 50, 100, 150 and 200 kPa with the model equation from Table 1 were performed in describing and quantifying the fouling mechanism controlling the membrane processes. At the end of each experiment, each of the fouled membranes were taken out for further studies.

Fouled UF membrane analysis

After fouling, the UF membrane was taken out from the membrane module. The membrane surface was wiped with a plastic sheet to physically get rid of the cake from the membrane surface, and later this fouled membrane was soaked in an alkaline solution (1M NaOH) for 24 h in order to complete the desorption of the remaining foulants. The 'foulant solution' was processed accordingly for the particle size distribution (PSD) analysis. PSD measurement of foulant solution and sludge were evaluated using a laser light scattering analyzer, Malvern Mastersizer 2000 (Hydro 2000 MU, Malvern Instruments Limited, UK), which measures materials from 0.01 to 1,000 μm.

Scanning electron microscopy (SEM) analysis

After the experimental works, a small part of the membrane was cut from the middle for SEM analysis. Before the viewing procedures, the samples were coated with gold using a sputter coater (BAL-TEC SCD005; Bal-Tec Co., Balzers; Vaduz, Liechtenstein). Later, the coated membranes were analyzed using a scanning electron microscope (Hitachi S-3400N, Tokyo, Japan).

Energy dispersive X-ray (EDX) analysis

The chemical compositions of the fouling layer were analyzed using an EDX spectrometer (Thermo Electron Corporation Instrument, USA) attached to an SEM (Hitachi S-3400N, Tokyo, Japan).

Membrane cleaning

The cleaning procedure was executed at the end of each experiment for the prevention of membrane drawback such as fouling and compaction. The membrane module was circulated with distilled water in order to flush out the remaining POME in the membranes. Later, the membrane was circulated with a chemical solution mixed with 1% w/w NaOH and 0.6% w/w NaClO for 30 min. The procedure was performed at room temperature with a constant pressure of 100 kPa and crossflow velocity at 1 m/s. Afterwards, the membrane was soaked in an acidic solution (HCl 0.5%) for 24 h, and later washed again with distilled water for 30 min. Finally, the water filtration of the cleaned membrane was calculated before and after cleaning for the verification of the cleaning effectiveness.

RESULTS AND DISCUSSION

Characteristic of POME

As shown below, Table 2 presents the POME samples which were collected from Seri Ulu Langat Palm Oil Mill, Dengkil, Malaysia. It is noted that the POME samples collected from this mill contain high COD, turbidity, TSS, and the color was also unsatisfactory.

Table 2 | Characteristics of POME

Parameter	POME
COD (mg/L)	17,500
TSS (mg/L)	5,000
Turbidity (NTU)	200
pH	4
Color (PtCo)	5,380
Temperature (40 °C)	45
Oil and grease (mg/L)	1,020

Effect of applied pressure on permeate flux

The investigations on the relationship of flux-pressure are presented in this section. The effect of applied pressure on the filtration performance with respect to the POME treatment is given in Figure 2.

As observed in Figure 2, the flux reductions in the first hour of filtration were 19.45% (58.50–47.12 L/m^2·h), 17.30% (68.12–56.33 L/m^2·h), 16.03% (75.41–63.32 L/m^2·h), and 15.08% (81.88–69.53 L/m^2·h), at applied pressures of 50, 100, 150 and 200 kPa. It was noticed that the increase in applied pressure may result in the development of foulant layers which can consolidate over time, and lead to the permanent fouling of the UF membranes (Norhan *et al.* 2011). The main reason for this phenomenon is because as the feed solution filtered through the UF membranes, the solutes from the feed accumulated on the membrane surface and led to the build up of a gel layer. The further increase of applied pressure may result in a greater concentration of gel layer and cause irreversible fouling as the gel layer becomes thicker or more compact. Due to that, the increase in applied pressure contributed to the higher resistance of gel layer and later, the permeation flux of the UF membrane became constant. Similarly, Vladisavljević & Rajkovic (1999) found that an increase in applied pressure drastically resulted in an increase of the solute concentration at the surface of the membrane, which consequently increased the fouling effects by the absorption of protein or gelation.

As shown in Figure 3, the COD removal decreased from 75.23 to 63.65%, turbidity removal decreased from 87.80 to 86.20%, and color removal decreased from 58.78 to 55.58%. This finding was potentially a result of the formation of a

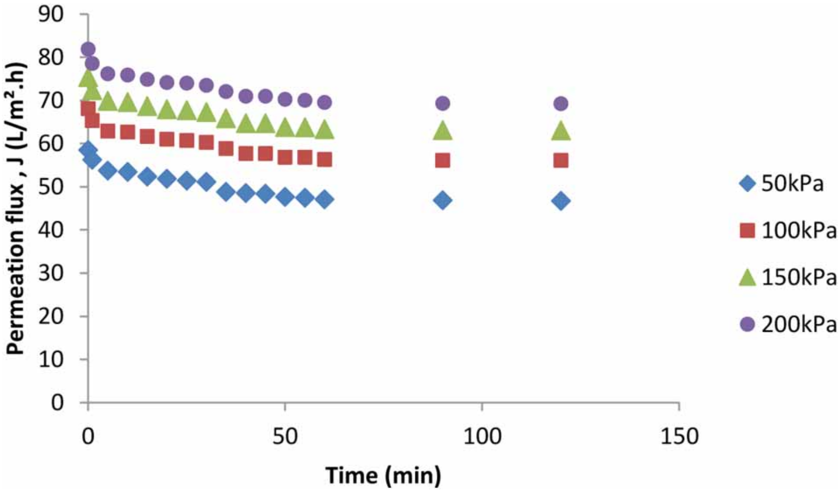


Figure 2 | Effect of applied pressures on permeate flux for PVDF membrane.

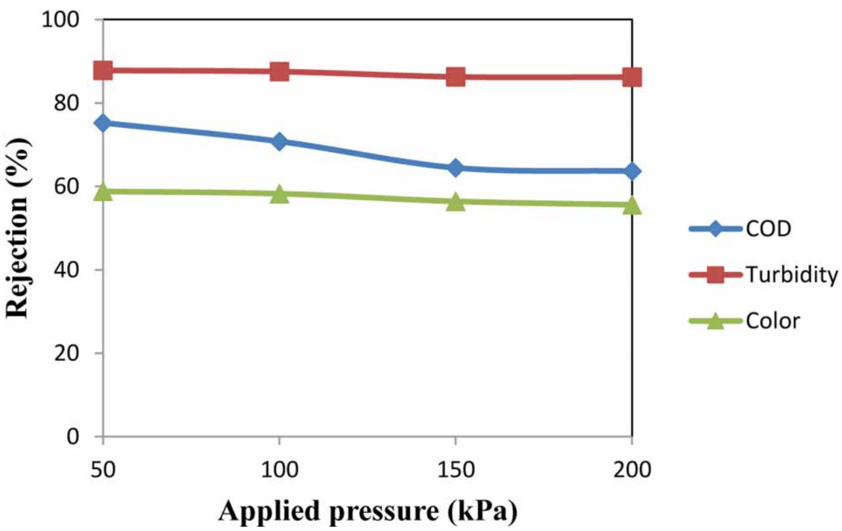


Figure 3 | Effect of applied pressure on percentage rejection of pollutants of PVDF membrane.

fouling layer and pore plugging on the membrane surface which blocked the organic matter from passing through the filtration membrane. A similar result was also discovered by Ratanatamskul & Kaweenantawong (2001), as they study the effect of UF membrane in textile wastewater treatment, where the highest rejection of COD and TOC occurred at the lowest applied pressure of 200 kPa. Also, in another research by Srisuwan & Thongchai (2002), as they studied the removal of heavy metal using the UF membrane treatment, they found that the highest removal of metal occurred at the lowest applied pressure of 50 kPa. This indicates that an increase in applied pressure may cause the enhancement of a gel layer, where this layer acts as a membrane protection and blocks incoming organic matter, and leads to the decrease of COD, turbidity and color removal.

Table 3 | Fitting of fouling model of PVDF membrane

Applied pressure (kPa)	R^2 of fouling model			
	Cake filtration	Standard blocking	Intermediate blocking	Complete blocking
50	0.9610	0.9547	0.9597	0.9520
100	0.9700	0.9652	0.9688	0.9631
150	0.9697	0.9650	0.9682	0.9630
200	0.9707	0.9659	0.9688	0.9640

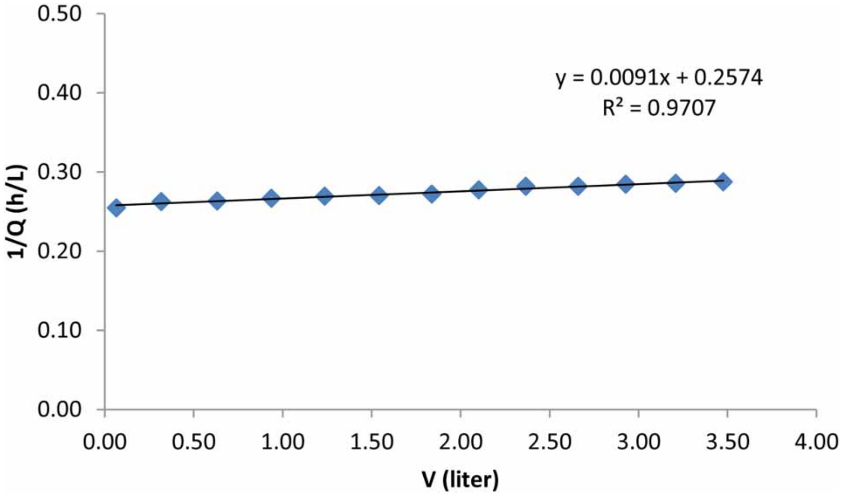


Figure 4 | Cake filtration model of PVDF membrane of applied pressure of 200 kPa.

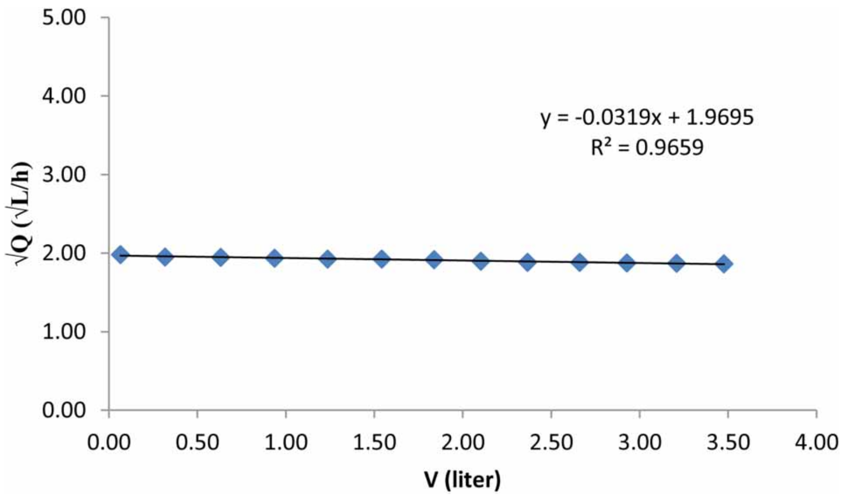


Figure 5 | Standard blocking model of PVDF membrane of applied pressure of 200 kPa.

Mechanism of membrane fouling

The ability to predict the fouling phenomenon on the UF membrane could be a key factor which can reduce or control fouling behavior during a plant operation or running at a design stage. This is because many factors may affect the existence of membrane fouling such as retention of smaller matters, formation of cake layer, plugging on the membrane pore, and concentration polarization (Maria *et al.* 2014). The standard blocking, intermediate blocking, complete blocking and cake filtration was used to evaluate the fouling mechanism for his study. Model equations in Table 1 were applied to study the plots of the fouling mechanism. The UF membranes used were fitted to the filtration experiments varying in applied pressures of 50, 100, 150 and 200 kPa. By evaluating all the experiments, the highest fitting situations at these applied pressures were chosen based on the highest determination value of R^2 (close to 1.0) as the most suitable model. Table 3 shows the fouling mechanism of PVDF membrane at various applied pressures assessed by the determination of the regression of the fouling models.

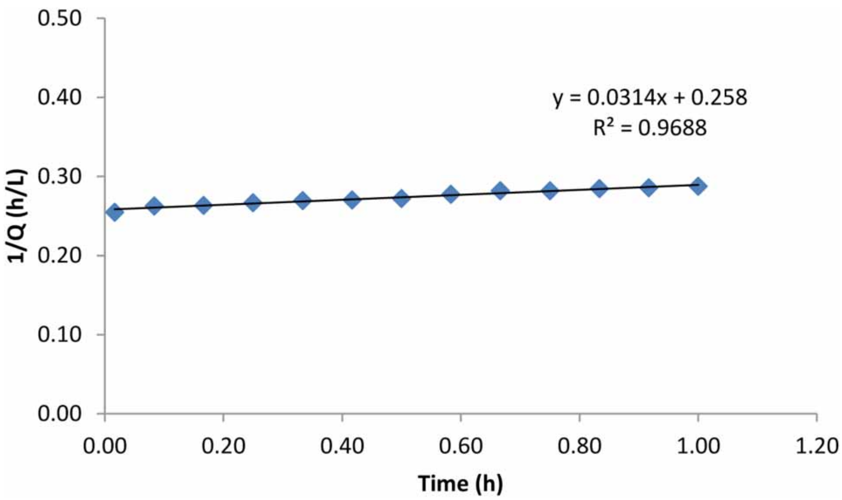


Figure 6 | Intermediate blocking model of PVDF membrane of applied pressure of 200 kPa.

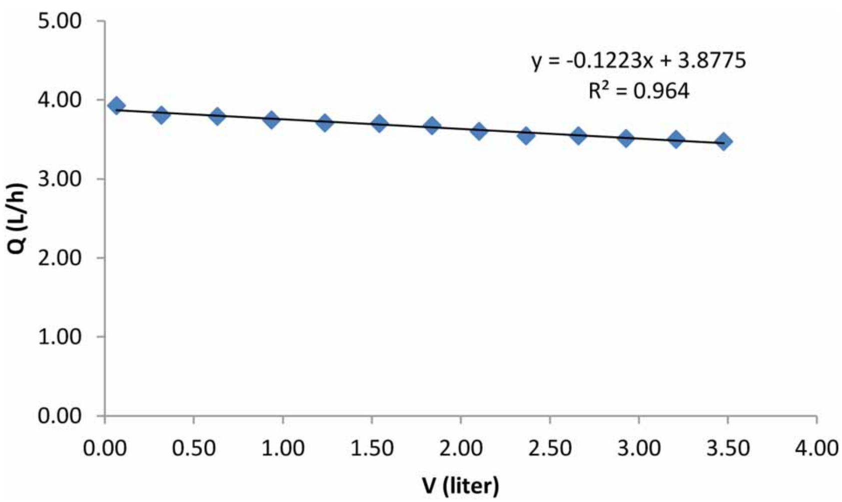


Figure 7 | Complete blocking model of PVDF membrane of applied pressure of 200 kPa.

Based on Table 3, it was acknowledged that the highest stability was noticed at an applied pressure of 200 kPa with a cake filtration mechanism having the best R^2 value of 0.9707. The model plots that describe the fouling mechanism of PVDF membrane at an applied pressure of 200 kPa are presented in Figures 4–7.

As shown from the plotted graphs above, the cake filtration was governed by the fouling mechanism of PVDF membrane with the greatest value of R^2 coefficient. The data from Table 3 also shows that the intermediate blocking model was the nearest model to the cake filtration, which agreed with the model from the principle and served as a confirmation of the formation of the cake layer on the membrane surface. The nearest R^2 value after the cake filtration was 0.9688, which is from the intermediate blocking mechanism at the same applied pressure. This means that the intermediate blocking had contributed to the cake layer formation at the surface of the membrane, and because of the principle, the intermediate blocking will participate in the affected area between the transition phase of the complete blocking and standard blocking, including the cake filtration mechanism. Similar results were also reported by

Boerlage *et al.* (2002) as they studied the effect of 11 UF membranes on the fouling mechanism and found that the cake filtration was proven to be the dominant mechanism. The studies carried out by Said *et al.* (2015) found that the optimum conditions of the UF membrane to treat POME were at applied pressure of 5 bars, 40 °C and pH of 9.05. At this stage, they also noticed that the cake layer was the best fit for the fouling mechanism with a R^2 value of 0.974, followed by intermediate blocking with a R^2 value of 0.951, standard blocking with a R^2 value of 0.914 and complete blocking with a R^2 value of 0.857. This also suggests that the PVDF membrane could be recommended as a reference membrane in describing the fouling mechanism of the POME treatment. However, the end decision can only be made after those membranes were subjected to a comprehensive experimental test. Furthermore, the evaluation of the cake filtrations model as the most universal model in explaining the fouling mechanism of the wastewater was verified by previous research (Vela *et al.* 2009; Rosas *et al.* 2014; Jin *et al.* 2015).

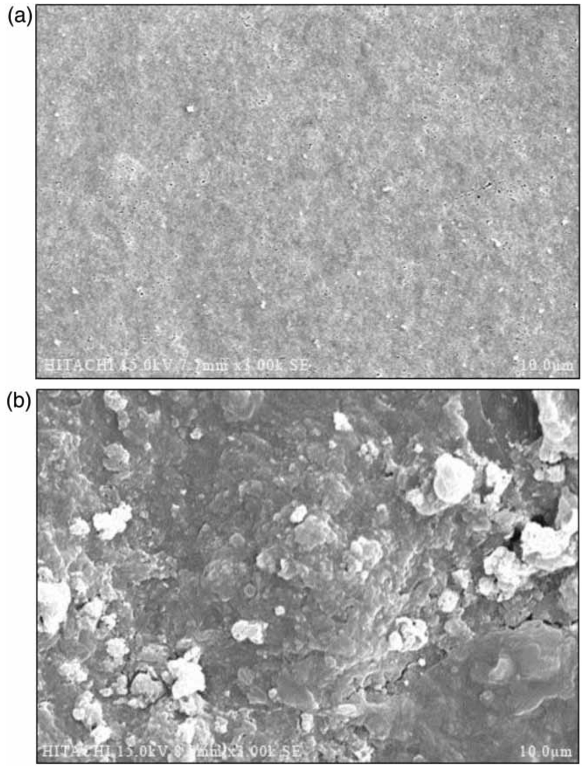


Figure 8 | SEM image of PVDF membrane: (a) new membrane; (b) fouled membrane.

SEM analysis

The SEM images of the surface of the PVDF membrane specimens were taken in order to describe the morphology on the membrane surfaces, which are given in Figure 8. The comparison of the new membrane and fouled membrane can be seen through the SEM images which explain the fouling phenomenon that occurred on the surface of the membranes.

In the SEM images of Figure 8(a), it can be observed that the surface of the PVDF membrane is clean and free of any particles. The characteristic of the membrane surface could be seen like a wave and valleys which could block microbial flocs, macromolecules and inorganic colloids. From Figure 8(b), the image of the fouled membranes showed the membrane surface had been covered with the cake layer which was caused by the occurrence of pore plugging if compared to the image of the clean membrane. In some parts of the membrane there was complete blockage where no pore appearance of the micrograph image was observed. Similar results were reported by Wu *et al.* (2007) as they studied the effect of the UF membrane with 20 kDa of MWCO to treat POME, where pore plugging was the main fouling that occurred after the treatment process. In other research, through SEM observation, Li & Chen (2010) discovered that the foulant from the municipal wastewater was deposited onto the UF membrane surface and into the membrane pore. With the help of SEM, they also found that biofilm fouling led to the pore blocking and later caused the build up of cake layer which dominated the fouling aging.

PSD analysis

The PSD measurements of foulant liquid of the PVDF membrane are presented in Figure 9. Based on Figure 9, the particle size distribution analysis showed that the particles of the foulants were dominated by the particle size in the range of 10–50 μm with a PSD percentage of 47.39%. These data indicate that smaller particles tend to foul or deposit on the membrane surface compared to larger particles. Generally, one of the main factors that affected the fouling of the membranes depended on the particle size

(Bae & Tak 2005). This result was also in agreement with Hwang *et al.* (2008) as they monitored smaller particles which tend to accumulate on the membrane pore compared to larger particles. Similar results were also reported by Liu *et al.* (2014) as they had studied the effects of particle-size based foulants which caused the fouling phenomenon in the treatment of polluted raw water using UF membrane.

EDX analysis

The chemical compositions of the fouling layer were analyzed by the EDX analysis as shown in Figure 10. The data show that the initial value was C (14.90% w/w), O (36.69% w/w), and F (45.41). The fouled PVDF membrane after the POME treatment contained C (15.66% w/w), O (43.89% w/w), F (36.59% w/w), Al (0.55% w/w), Si (0.40% w/w), P (1.25% w/w) and Ca (1.09% w/w). These data indicated that after the treatment processes, the initial chemical composition was depleted and replaced with the composition of the feed solution and led to fouling of the membrane. Generally, inorganic fouling may occur in two different ways, such as chemical precipitation and biological precipitation (Meng *et al.* 2009). The relationship between various types of foulant such as organic and inorganic materials may increase the fouling rate and lead to the formation of cake layer on the surface of the membrane (Meng *et al.* 2007). It is noted that the fouling phenomenon may be caused by inorganic ion components such as Ca and Si in the form of silicate ($CaCO_3$) elements (Zuo *et al.* 2014).

Analysis on membrane cleaning

The application of chemical cleaning was able to bring back the quality of UF membrane used in this project to the initial flux with 95% restoration of distilled water flux. It was also noticed that the NaOH solution was able to control the fouling of the UF membrane for the next filtration processes into its initial active performance with flux recovery (FR) higher than 90%. The study by Madaeni *et al.* (2001) proved that the application of NaOH as a chemical cleaning agent for fouled membrane by inorganic matters had recovered the flux efficiently. Hence, the application of cleaning procedures on the UF membranes after the filtration process may lengthen the membrane lifetime, reduce maintenance operation and also repair cost.

CONCLUSIONS

The aim of this research was originally to study the effect of PVDF membrane in the treatment of POME. The laboratory work of filtration experiments provided excellent results in the permeation flux rate and removal of particulates which are present in POME. Study on membrane fouling found that an increase in applied pressure can seriously affect the membrane condition and continuous treatment may cause a reduction of the membrane performance. The rejection of pollutants from the membrane may also decrease as the fouling layer becomes the membrane

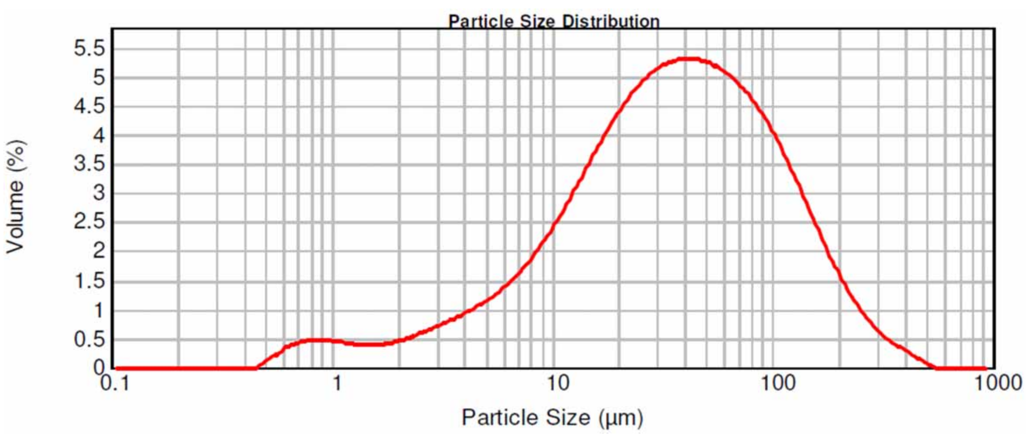


Figure 9 | Particle size distribution of foulant on PVDF membrane.

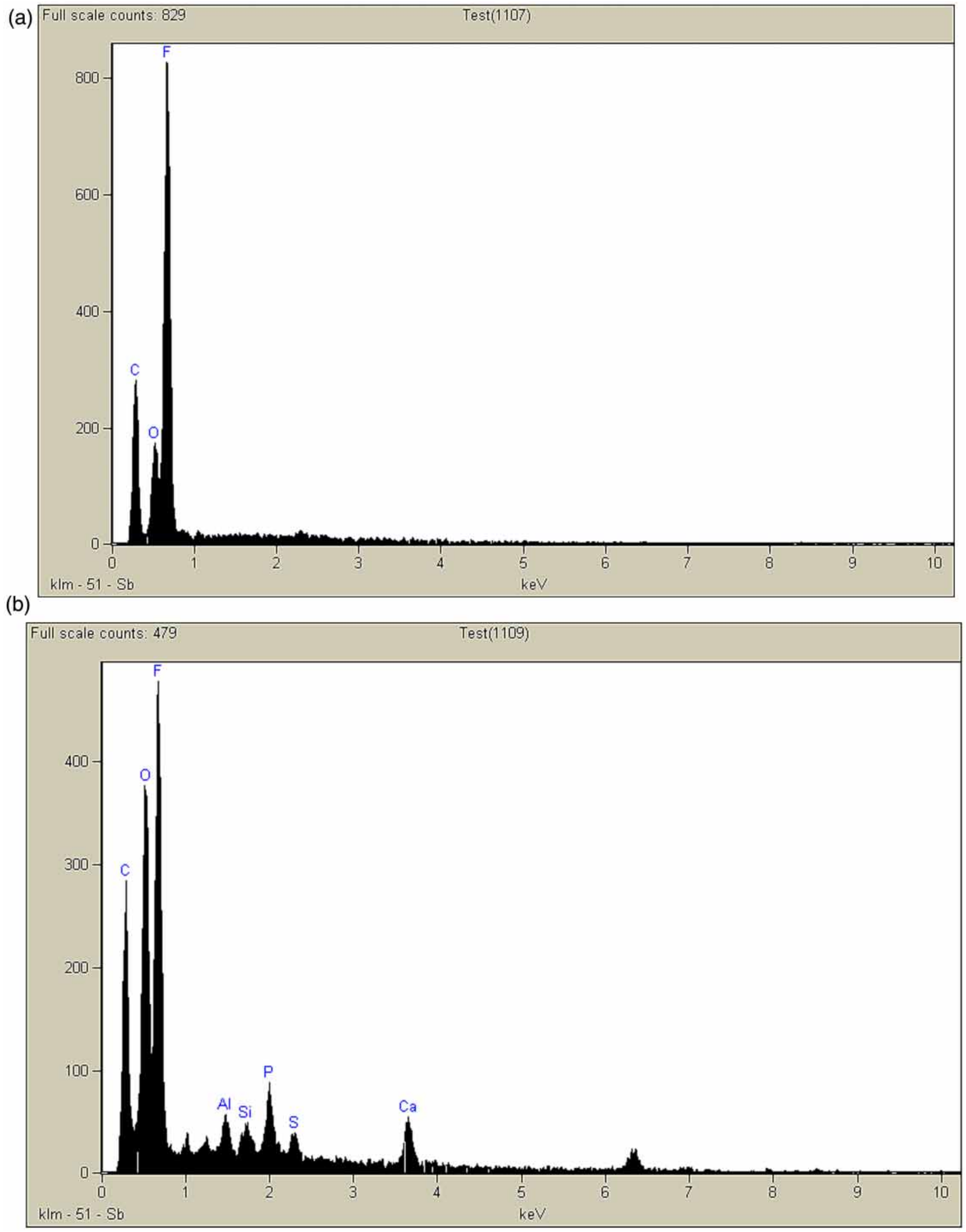


Figure 10 | EDX analysis of PVDF membrane: (a) new membrane; (b) fouled membrane.

protection and blocks other pollutants that come through. Based on the fouling model, SEM, PSD and EDX analysis, the fouled membranes has been dominated by the cake layer which was caused by small particles (ranges of 10–50 μm) from POME as they tend to accumulate on the membrane pores compared to larger particles. However, further treatment by membrane cleaning may return the membrane to the initial performance. Hence, further studies should get more attention to increase our knowledge on the relation between fouling behaviors and membrane performance, especially in the treatment of POME.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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