

Performance study of ceramic microfiltration membrane for oily wastewater treatment

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Received 14 July 2006; received in revised form 15 September 2006; accepted 7 October 2006

Abstract

Cross-flow microfiltration (MF) processes were studied with oily wastewater using a ceramic ($\alpha\text{-Al}_2\text{O}_3$) membrane with 50 nm pore size. The influence of parameters such as transmembrane pressure (TMP), cross-flow velocity (CFV), oil concentration in feed, pH and salt concentration on the separation behaviors were investigated by the measurements of permeate flux, total organic carbon (TOC) removal efficiency, and size and zeta potential of the emulsion droplets. The results showed that there were different degrees of effect on the permeate flux by these parameters. The TOC removal efficiencies higher than 92.4% were achieved under all experimental conditions. A non-steady model of the accumulation volume of permeation was developed. It was found that the predicted values from the model were in good agreement with the experimental results. A sensitivity analysis (SA) of the model was also conducted to identify the degree of influence of the parameters on the accumulation volume of permeation. The results showed that the accumulation volume of permeation was significantly affected by the transmembrane pressure, indicating the model was reliable.

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Keywords: Ceramic membrane; Oily wastewater; Separation

1. Introduction

There has been increasing attention to the application of vegetable oils in industries since last decade because vegetable oils are renewable, non-toxic and environmentally friendly resources [1–3]. Vegetable oils are considered to be potential candidates to substitute conventional mineral oil-based lubricating oils and synthetic esters as a result of the stringent requirement of resource conservation and environmental protection [4–7].

Vegetable oils are low-cost candidates for the biodegradable replacement of mineral oils [8,9]. However, the limited applications of vegetable oils in the natural form are due to poor thermal/oxidation stability [10] and low temperature behavior [11]. Modifications of vegetable oils have been developed for industrial applications. These modifications include the use of substances such as acids, alkali, salt and organic additives. The composition of oily wastewaters becomes more complex due to

various industrial applications of vegetable oils, so oily wastewaters are difficult to be treated by conventional methods.

Several common techniques are used in oil–water separation and treatment. The gravity settling separation and mechanical coalescence methods are the well-known traditional treatment processes, the efficiency of which depends on the size of the oil droplets in wastewater. Chemical emulsion breaking is an effective way under proper application [12,13]. The coagulation and air flotation [14,15], electrostatic and electrocoagulation separation methods [16–19] were also applied in the oily wastewater separation processes. However, these methods would lead to a huge production of sludge and complicated operation problems. Other methods such as microwave treatment [20,21], ultrasonic wave treatment [22,23], thaw and heat treatment [24,25] have been occasionally applied in special oily water (sludge) treatment in recent years.

Membrane separation has enjoyed great popularity over the last 30 years and is becoming a promising technology. This technology has several advantages including stable effluent quality and small area requirement. Moreover, no chemicals addition is required. Many studies of membrane separation for oily wastewater treatment have been reported, particularly in ultrafiltration (UF) and reverse osmosis (RO) with organic membranes

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[26–29]. However, few studies are related to the application of inorganic ceramic membranes on oily wastewater treatment.

In this paper, the separation of oily wastewater was carried out using ceramic membrane. The effects of transmembrane pressure (TMP), cross-flow velocity (CFV), oil concentration in feed, pH and salt concentration on the permeate flux and TOC removal efficiency were studied. A non-steady model of the accumulation volume of permeation with TMP, CFV, oil concentration, and separation time was developed. A sensitivity analysis (SA) was also conducted to identify the degree of influence of the first three parameters on the accumulation volume of permeation.

2. Experimental

2.1. Materials

The tubular ceramic (α -alumina) membrane and module used in this study were produced by the Nanjing University of Technology, China. The ceramic membrane was 425 mm in height and 30 mm in diameter with 19 lumens. Each lumen was 4 mm in diameter. The nominal pore size was 50 nm and layer thickness ranged from 20 to 50 nm, which provided 0.1 m² effective areas for filtration.

The oily wastewater (feed solution) was prepared by mixing edible oil with distilled water and surfactant for 15 min at a speed of 5000 rpm using a homogenizer. The temperature of the feed solution was controlled between 20 ± 1 °C by means of a small heat-exchanger immersed into the feed tank.

2.2. Experimental set-up and operation

The schematic diagram of the experimental set-up is shown in Fig. 1. The experiments of oily wastewater separation were operated in the total recycle mode. The permeate sample collected in the sample vessel was measured and then returned to the feed tank for recycling. A total of 14 samples were collected

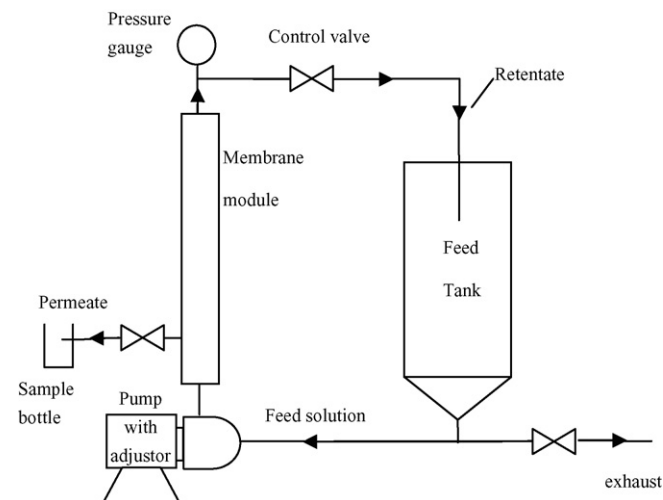


Fig. 1. Schematic diagram of the ceramic membrane separation unit.

at 5 min interval before the permeate flux reached a steady-state condition. The volume of the feed solution was 16 L. The applied TMP was in the range of 0.05–0.30 MPa, and the CFV was in the range of 0.21–1.68 m s⁻¹. The effect of ionic strength on the process performance was investigated by the change of NaCl concentration from 0.001 to 0.05 mol L⁻¹. The influence of pH on the performance of the ceramic membrane was determined by four various pH values of the solution adjusted with 2 mol L⁻¹ NaOH and 2 mol L⁻¹ HCl solutions.

After each MF run, the membrane was regenerated by the following procedure adopted by [30]. The oily wastewater was drained off from the system, and the membrane was rinsed with hot tap water at 40 °C for 30 min. Then the membrane was washed with 3% NaOH solution for 2 h in order to restore the membrane permeability. Finally, the washing process was completed by rinsing with deionized water until the permeate reached neutral pH. The initial water flux was measured after each regeneration, ensuring the results with high reproducibility.

2.3. Analytic methods

2.3.1. Permeate flux (J)

The permeate volume was measured during the separation process. The permeate flux was calculated by dividing the permeate volume by the product of effective membrane area and the sampling time:

$$\text{permeate flux } (J) = \frac{\text{permeate volume collected}}{\text{membrane area} \times \text{time}} \quad (1)$$

2.3.2. TOC removal efficiency (R_{TOC})

The TOC concentration was determined by a total carbon analyzer (TOC-5000, Shimadzu, Japan). The oil removal efficiency was evaluated according to the value of TOC removal efficiency (R_{TOC}), which is defined as

$$R_{\text{TOC}} = \frac{\text{TOC}_{\text{Feed}} - \text{TOC}_{\text{Ave}}}{\text{TOC}_{\text{Feed}}} \times 100 \quad (2)$$

where TOC_{Feed} is the TOC concentration in the feed solution, TOC_{Ave} is the average TOC concentration of the permeate (mg l⁻¹) and is defined as follows:

$$\text{TOC}_{\text{Ave}} = \frac{1}{\sum_t V_t} \int_0^t V_t C_t dt \quad (3)$$

where V_t is the instantaneous permeate volume (l), C_t the instantaneous TOC concentration of the permeate (mg l⁻¹) and t is the filtration time (min).

2.3.3. Sensitivity analysis (SA)

The relative SA ($S_X; p$) of an output variable X with respect to a parameter p is the change ΔX in X produced by a change Δp in p relative to the original values of X and p [31], i.e.

$$SA(X, p) = \frac{\Delta X / X}{\Delta p / p} \quad (4)$$

The higher SA value of a certain parameter indicates the model is more sensitive to that parameter [32].

2.3.4. Statistical analyses

The one-way ANOVA was used to test any significant difference in TOC removal efficiencies in the effluent among various operating conditions, including TMP, CFV, oil concentration, pH and salt concentration at the 95% confidence level. All statistical analyses were performed using SPSS^R for Window Release 10.1.

3. Results and discussion

3.1. Effect of TMP on permeate flux

Fig. 2 shows the variations of permeate flux at TMP from 0.05 to 0.3 MPa. The steady permeate flux was highly dependent on TMP. It was also found that the increase of permeate flux under lower TMP was greater than that under higher TMP. When the TMP was greater than 0.2 MPa, the rate of increase of permeate flux was lessened.

This is because the increase of TMP had both positive and negative effects on the permeate flux. Higher TMP allowed droplets (both solvent and solute) to pass rapidly through the membrane pores. However, more oil droplets accumulated both on the membrane surface and in the membrane pores, leading to membrane fouling. According to Fig. 2, the positive effect on the permeate flux was predominant at low TMP levels. For most suspensions, the flux increases with increasing pressure and then approaches to the limiting flux [33–35]. Beyond the threshold pressure, the flux increases nonlinearly with TMP due to concentration polarization [36,37]. The flux was independent of TMP under higher pressure condition due to gel polarization which could cause a flux decline under excessive pressure drop [38]. Meanwhile, membranes that had previously been used under high pressure were subjected to increase the fouling probability [39]. Therefore, it is suggested that the TMP at 0.2 MPa is suitable for this type of MF.

Fig. 2 also presents the average TOC removal efficiencies under various TMP values. The TOC removal efficiencies were

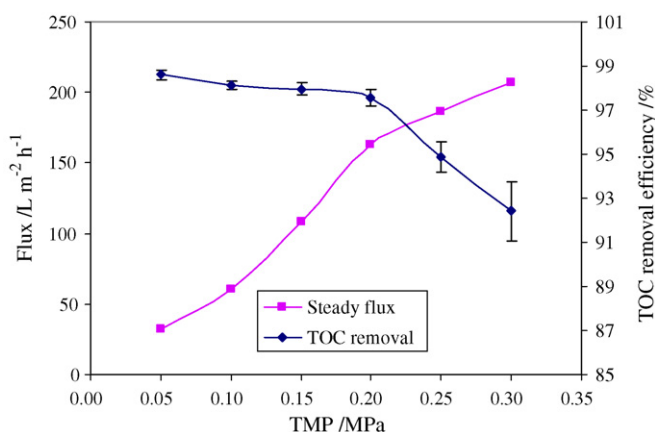


Fig. 2. Effect of TMP on permeate flux. The error bars were the standard deviation (S.D.) of the mean (CFV: 1.68 m s⁻¹, oil conc.: 500 mg L⁻¹) (n = 14).

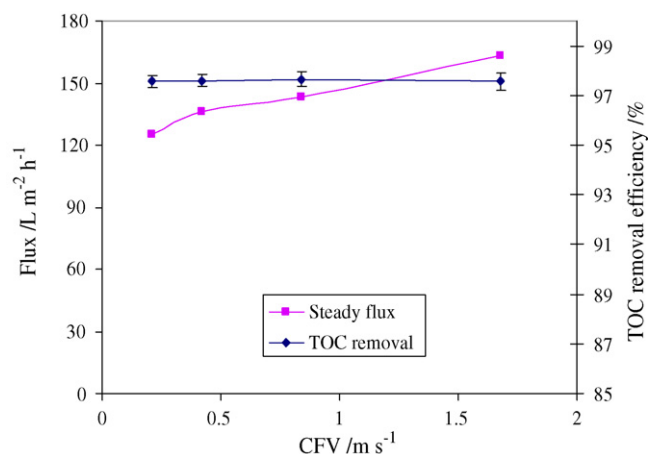


Fig. 3. Effect of CFV on permeate flux. The error bars were the standard deviation (S.D.) of the mean (TMP: 0.2 MPa, oil conc.: 500 mg L⁻¹) (n = 14).

between 92.4 and 98.6%. There was no significant difference in the TOC removal efficiency among the TMP of 0.05, 0.1, 0.15 and 0.2 MPa ($p > 0.05$). However, the TOC removal efficiencies decreased rapidly from 98 to 92% when the TMP increased from 0.2 to 0.3 MPa. This also revealed that the TMP above 0.2 MPa was inappropriate for the sake of good effluent quality.

3.2. Effect of CFV on permeate flux

The effect of CFV on the permeation flux with the CFV ranged from 0.21 to 1.68 m s⁻¹ is shown in Fig. 3. The results indicated that the higher CFV led to a higher steady permeate flux. This could be explained by the change of Reynolds number (Re). Re is defined as follows:

$$Re = \frac{\rho v d}{\mu} \quad (5)$$

where ρ is the density of the liquid, v the velocity of the liquid, d the diameter of the pipe, and μ is the viscosity of the liquid.

Therefore, the greater CFV resulted in a larger Re . Theoretically, turbulent flow is defined as if the Re exceeds 4000. When the CFV value increased from 0.21 to 1.68 m s⁻¹, the Re changed from 836 to 6680, correspondingly. The turbulent flow, which weakened the effect of concentration polarization [40], occurred as the CFV increased to 1.68 m s⁻¹, resulting in the increase of permeate flux.

Although high CFV can enhance the permeated flux, forceful turbulent is not recommended in the microfiltration membrane process. The main reason is that turbulent flow may consume TMP of the system, causing the decline of permeate flux. Hence, it is suggested that the CFV should be maintained at 1.68 m s⁻¹ for the operation of this kind of MF.

The average TOC removal efficiencies under various CFV values are shown in Fig. 3. The TOC removal efficiencies were maintained around 97% under different CFV conditions. The results of one-way ANOVA showed that the average TOC removal efficiencies had no significant difference among various CFV values ($p > 0.05$).

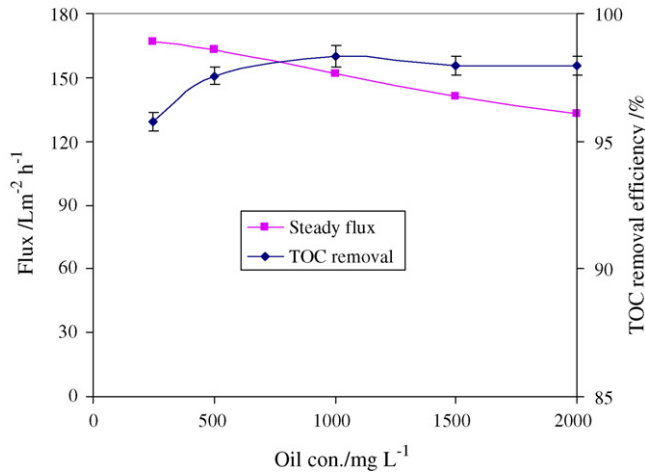


Fig. 4. Effect of oil concentration on permeate flux. The error bars were the standard deviation (S.D.) of the mean (TMP: 0.2 MPa, CFV: 1.68 m s⁻¹) ($n = 14$).

3.3. Effect of oil concentration on permeate flux

The variations of permeate flux with different oil concentrations in feed solution are illustrated in Fig. 4. The permeate flux showed a little dependence on oil concentration. Lower steady permeate flux was obtained under the higher oil concentration in the feed solution. It was because serious membrane fouling such as membrane adsorption and pore plugging occurred under high oil concentration. The concentration polarization on the membrane surface was also one of the factors.

Fig. 4 shows the average TOC removal efficiencies at various oil concentrations. The TOC removal efficiencies increased from 95.8 to 98% with the oil concentration increasing steadily from 250 to 1000 mg L⁻¹ before levelling off at about 98% to the concentration of 2000 mg L⁻¹.

3.4. Effect of pH on permeate flux

The influence of pH on the membrane filtration is shown in Fig. 5. The steady permeate flux was highly dependent on the pH of the feed solution. The steady flux increased sharply with

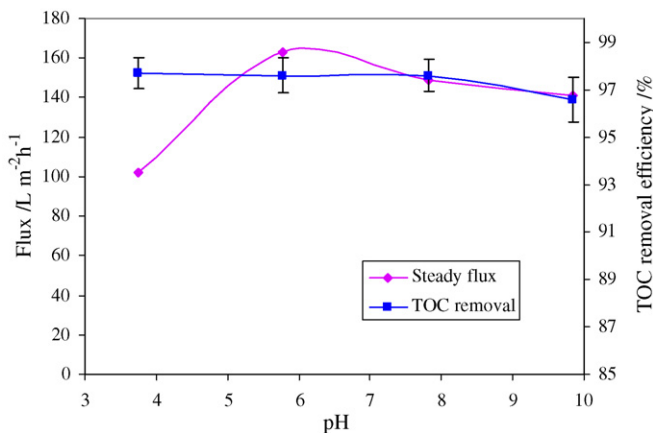


Fig. 5. Effect of pH on permeate flux. The error bars were the standard deviation (S.D.) of the mean (TMP: 0.2 MPa, CFV: 1.68 m s⁻¹, oil conc., 500 mg L⁻¹) ($n = 14$).

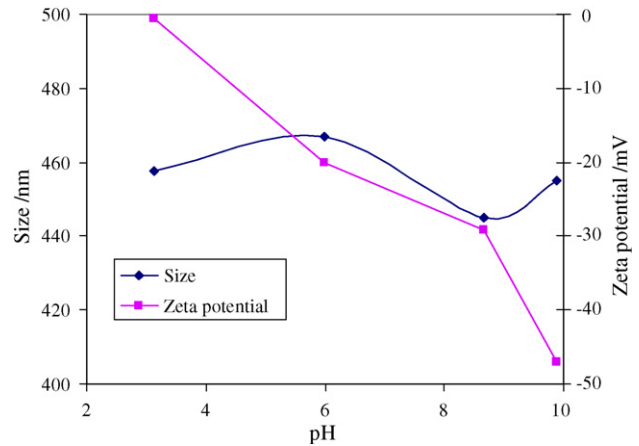


Fig. 6. Effect of pH on size and zeta potential of emulsion droplets.

the increase of pH from 3.8 to 5.8, and then reduced slightly from 163 to 141 L m⁻² h⁻¹ with the increase of pH from 5.8 to 9.9. The steady flux at pH of 5.8 reached a peak and was 45% greater than that at pH of 3.8. However, there are some arguments in the previous studies on the pH effect. Zhao et al. [41] found the steady flux decreased as the pH increased in a range of 2–10. However, Moossemiller et al. [42] reported that the maximum permeability of alumina membrane was observed at pH around 8–10. The experimental results in this study were somewhat consistent with the Moossemiller's conclusion.

The permeate flux under various pH values was affected not only by the characteristics of membrane but also by the properties of the solute (droplet). The size and zeta potential of emulsion droplets are shown in Fig. 6. The results of size determination of oil-in-water emulsion droplets indicated that no obvious variation existed in the average size of droplets under various pH values (Fig. 6). However, the stability of the oil-in-water emulsion was reported more stable at pH of 4–6 than that at pH of 6–10 [43]. The coagulation of emulsion did not happen under stable condition (i.e. pH 3.8) as the zeta potential of emulsion droplet was low in absolute value. Therefore, the lower level of steady flux was observed at low pH. While the emulsion droplets had the higher negative charge at higher pH (Fig. 6). The cake layer became more "open" at high pH due to the inter-droplet repulsion, and this increased the permeability, resulting in higher permeate flux. Meanwhile, the inter-droplet repulsion prevented the particle from depositing, and led to the reduction of the thickness of cake layer [44]. These factors reflected that the higher steady flux could be obtained at higher pH value.

The average TOC removal efficiencies under various pH values are shown in Fig. 5. The TOC removal efficiencies remained at the range of 96.6–97.7%. The results of one-way ANOVA indicated that the average TOC removal efficiencies had no significant difference under different pH conditions ($p > 0.05$).

3.5. Effect of salt concentration on permeate flux

Fig. 7 shows the variations of the permeate flux at salt concentrations from 0.001 to 0.05 mol L⁻¹. As shown in Fig. 7, the higher salt concentration gave a lower steady flux. The steady

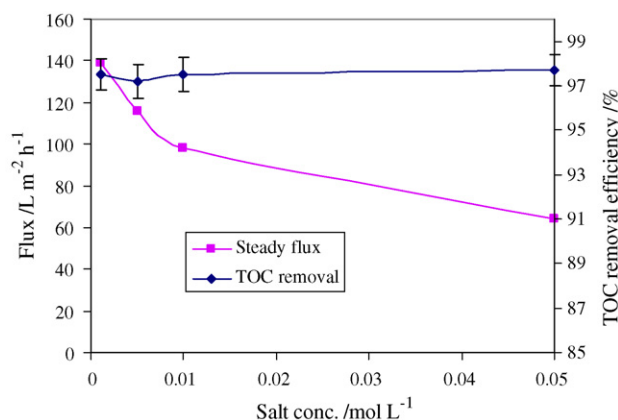


Fig. 7. Effect of salt concentration on permeate flux. The error bars were the standard deviation (S.D.) of the mean (TMP: 0.2 MPa, CFV: 1.68 m s⁻¹, oil conc., 500 mg L⁻¹) ($n=14$).

flux at higher salt concentration (i.e. 0.05 mol L⁻¹) was only 45% of that at lower salt concentration (i.e. 0.001 mol L⁻¹).

There is still much debate on the effect of salt concentration on permeate flux among previous researchers. Zhao et al. [41] found that the increase of ionic strength resulted in a higher steady flux. Tambe and Sharma [43] observed high ionic concentration tended to diminish the thickness of the double layer around the emulsion droplets, thereby reducing the electrostatic barrier to coalescence, causing high permeate flux. However, Elzo et al. [44] reported the opposite results, high permeate fluxes were observed at low salt concentration.

The size of the emulsion droplet was not uniform and the micelles carried charges due to the reaction of surfactants. Some small emulsion droplets could become smaller as the reduction of the double layer thickness under high ionic strength. Therefore, the smaller droplets entered the membrane pores or vacant spaces easily, resulting in membrane fouling and decrease of permeate flux.

The effect of salt concentration on the TOC removal efficiency is shown in Fig. 7. The average TOC removal efficiencies were fluctuated between 97.2 and 97.7% when the salt concentration changed from 0.001 to 0.05 mg L⁻¹. The results of one-way ANOVA indicated that the average TOC removal efficiencies had no significant difference among various salt concentrations ($p>0.05$).

4. Modeling development and sensitivity analysis

4.1. Modeling development

Numerous models are developed to characterize the membrane filtration processes [45,46]. The empirical model of permeate flux in a form of exponential decay function is utilized in the modeling of membrane fouling [47,48]. Pastagia et al. [49] developed a non-steady mass transfer model to predict the permeate flux for two-component dye mixture nanofiltration (NF).

Under normal condition, the oily wastewater is often in neutral pH and very low salinity. Therefore, the effects of pH and

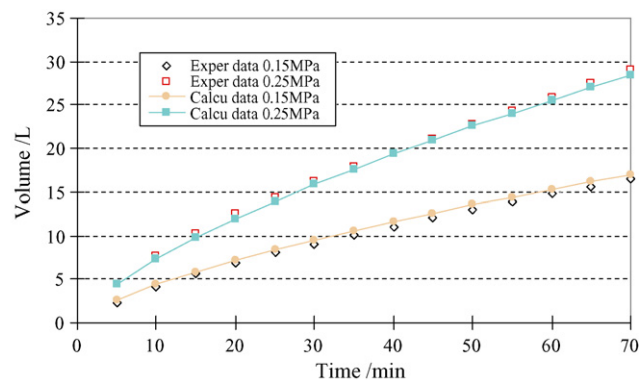


Fig. 8. The accumulation volume of permeation under various TMP.

salt concentration on the accumulation volume of permeation were not considered in the model proposed in this research. A non-steady model of the accumulation volume of permeation was developed by the regression method according to the experimental results under various CFV, TMP and oil concentrations. The procedure was as follows: (1) analyzing the time-dependence permeate flux curve and finding out nonlinear regression between the permeate flux and time, $J=f(t)$; (2) adding the effect of TMP on the permeate flux into the model, $J=f(t, \text{TMP})$; (3) considering the effect of CFV and oil concentration on the permeate flux, $J=f(t, \text{TMP}, \text{CFV}, \text{oil concentration})$; (4) optimizing the regression equation (largest value of R^2). The model was expressed as follows:

$$V = 6.35 \times \text{TMP} \times \left[0.85 + 1.32 \frac{\text{CFV}^{0.6}}{\text{Conc}^{0.4}} \right] (t - 0.5)^{0.68},$$

$$R^2 = 0.9919 \quad (7)$$

where V is accumulation volume of permeate (L), TMP the pressure (MPa), CFV the velocity of feed (m s⁻¹), Conc. the oil concentration (mg L⁻¹) and t is the working time (min).

Validity of the equation: TMP was at a range of 0.05–0.3 MPa, CFV was less than 1.68 m/s, and oil concentration was less than 2000 mg/L.

Figs. 8–10 show the experimental data and predicted accumulation volume of permeation under typical values of TMP, CFV and oil concentrations. It was found that the predicted values were in good agreement with the experimental results. The

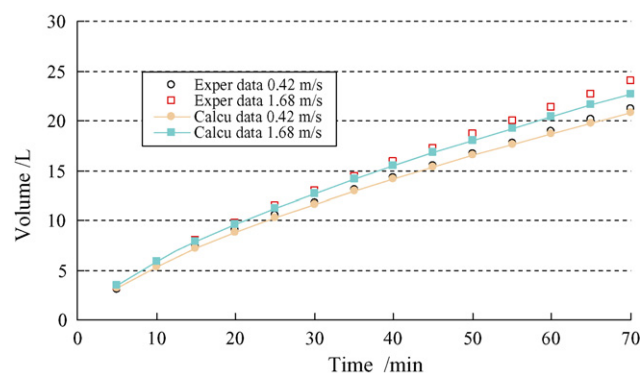


Fig. 9. The accumulation volume of permeation under various CFV.

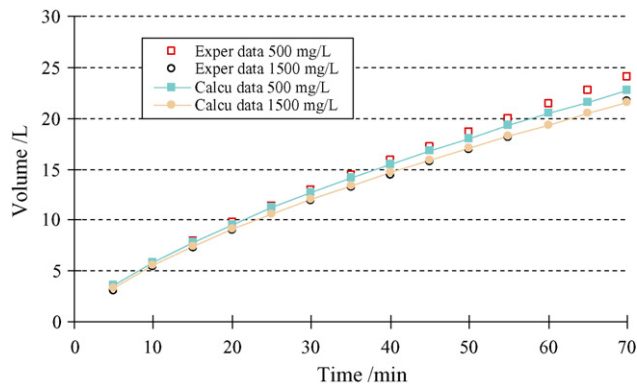


Fig. 10. The accumulation volume of permeation under various oil concentrations.

model is helpful for users to understand the change of the accumulation volume of permeation during the filtration process; to determine the time for the membrane cleaning; to understand the effect of TMP, CFV and oil concentration on the permeate flux; and to optimize the operating conditions of the filtration process.

4.2. Sensitivity analysis (SA)

Three parameters (TMP, CFV, and oil concentration) in the model were selected for sensitivity analysis. Table 1 shows the definition and standard operating values of parameters of the membrane separation processes in this study. Because the accuracies of the SA method depend on the parameter change which are made of +1% and –1% of the assigned values [50]. Table 2 presents the results of SA from the parameter changes. Among

Table 1
Parameters used in SA

Parameter	Definition	Assigned range	Assigned value
TMP (MPa)	Transmembrane pressure	0.05–0.3	0.2
CFV (m s^{-1})	Cross-flow velocity	0.21–1.68	0.95
Conc. (mg L^{-1})	Oil concentration in the feed stream	250–2000	1125
t (min)	Membrane separation time	0–70	35

Table 2
Results of computation of SA

Parameters	Variation	SA value
TMP	1%	1.00 (+)
	–1%	1.00 (+)
	Average	1.00 (+)
CFV	1%	0.05 (+)
	–1%	0.04 (+)
	Average	0.045 (+)
Conc.	1%	0.03 (–)
	–1%	0.03 (–)
	Average	0.03 (–)

(+/-) indicates a positive/inverse relation between the change in the parameter and the change in the flux or volume.

the three parameters, SA value from TMP was the highest, while SA value from oil concentration was the lowest. The SA value from TMP was 22 times higher than that from CFV and 33 times higher than that from oil concentration. The results of SA showed that the model was the most sensitive to the change of TMP and relatively insensitive to the changes of CFV and oil concentration. This indicated that the effect of TMP on the accumulation volume of permeation is the most significant among these three parameters.

5. Conclusions

The microfiltration process with ceramic membrane was successfully applied for the oily wastewater treatment in this study. The high permeate flux was achieved under high TMP, high CFV and low oil concentration. The results also indicated that the permeate flux decreased either under high salt concentration or under low pH value in the feed solution. The largest steady flux at pH of 5.8 was 45% greater than that at pH of 3.8. The steady flux at high salt concentration (0.05 mol L^{-1}) was only 45% of that at low salt concentration (0.001 mol L^{-1}). The TOC removal efficiencies were higher than 92.4% for all experimental conditions.

A non-steady model of the accumulation volume of permeation with four parameters (TMP, CFV, oil concentration and time) was developed. It was found that the predicted values from the model were in good agreement with the experimental results. The results of SA indicated that the model was the most sensitive to the change of TMP and relatively insensitive to changes of CFV and oil concentration. The results showed that the accumulation volume of permeation was significantly affected by the TMP. Therefore, the selection of an optimal value of TMP during the operation is very important for the membrane filtration process. This result indirectly supported that the prediction model was reasonable.

Acknowledgements

The authors gratefully thank the Research Grants Council and the Hong Kong Polytechnic University Central Research Grant for the financial support.

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