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Treatment of petroleum refinery wastewater using crossflow and immersed membrane processes

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ABSTRACT

Due to the dwindling water resources in the Kingdom of Saudi Arabia and increasing stringencies on the discharge of effluents, two pilot-scale membrane processes were installed in Riyadh petroleum refinery in order to treat wastewater discharged by the selected refinery after an API separator. One of the membrane processes was performed in a crossflow mode, while the other process was performed in an immersed mode. Results on permeate flux of both processes showed that the immersed membrane process was producing a stable flux value (more than $50\,\mathrm{l/m^2\,h}$) throughout the investigation period (more than 800 h). On the other hand, permeate flux of the crossflow membrane process was noticed to steadily decline after 600 h of running period. In terms of oil and grease contents, both membrane processes produced permeates that contained less than 1.4 mg/l. The immersed membrane process was found to be a potential process for treating oily wastewater, where the permeate flux was found to be almost stable at its initial value throughout the investigation. Moreover, the immersed membrane was effectively cleaned using a commercial detergent.

Keywords: Crossflow membrane; Immersed membrane; Oily wastewater; Membrane cleaning; Pilot-scale plant.

1. Introduction

Wastewaters produced from oil refineries include free and emulsified oil from leaks, spills, tank draw-off, and other sources. On the other hand, large volumes of water are used in petroleum refineries, especially for cooling systems. Furthermore, surface water run-off and sanitary wastewater are also generated. The quantity of wastewater generated and its characteristics depends on the process configuration. As a general guide, approximately 3.3–5 $\mathrm{m^3}$ of wastewater per ton of processed crude oil are generated when cooling water is recycled. Refineries generate polluted wastewater containing biochemical oxygen demand (BOD) and chemical oxygen demand (COD) levels of approximately 150–250 mg/l and 300–600 mg/l, respec tively; phenol levels of 20–200 mg/l; oil levels of 100–300 mg/l in desalter water and up to 5,000 mg/l in tank bottoms and other pollutants. From the above discussion, it is clear that large quantities of contaminated oily wastewater are produced from petroleum refineries. In order to protect the receiving water bodies and the environment in general, oily wastewater must be given a proper treatment.

Wastewater treatment facilities at refineries should be selected and installed so that pollutants in the wastewater can be effectively removed at points close to the sources of such pollutants. In recent years, new technique was implemented using different types of membranes to treat oil-containing wastewater. Abbasi et al. [1] investigated the performance of a MF ceramic membrane for treatment of oily wastewater, where the effects of different operating parameters were investigated. The results showed that rejection of total organic carbon (TOC) for the synthetic feeds was found to be more than 94 percent. The results also showed that by increasing temperature and pressure, the permeate flux increased. Hemmati et al. [2] used air sparging as a mean to solve the problem of fouling and decline in permeation flux in oily wastewater microfiltration. The results showed that an increase of up to 170 percent of permeation flux was achieved. Furthermore, increasing crossflow velocity (CFV), transmembrane pressure (TMP) and air sparging flow rate was reported to increase the permeation flux. Singh et al. [3] studied the use of MF to treat industrial oily wastewater. Effects of different operating parameters such as TMP on the steady-state permeate flux and oil rejection was investigated. The results showed that the steady state permeate flux increased with TMP. The oil concentration was found to be reduced from 192 to 4.5 mg/l after treatment. Nandi et al. [4] studied the performance of the separation of oil-in-water (o/w) emulsions using low-cost ceramic membrane. Synthetic oily emulsions constituting 125 and 250 mg/l oil concentrations were subjected to MF in batch mode of operation with varying TMP. The results showed that the membrane exhibited 98.8 percent oil rejection efficiency and 5.36×10^{-6} $m^3/m^2 s$ permeate flux after 60 min of experimental run at 68.95 kPa transmembrane pressure and 250 mg/l initial oil concentration. The use of crossflow membranes in treating oily wastewater was the subject of many published works [5–32]. With respect to the use of immersed membranes in the treatment of oily wastewater and up to the knowledge of the investigator, the literature lacks information in this area.

The progressive decline of flux with time cannot be avoided and eventually the flux becomes low and some type of cleaning or regeneration of the membrane must be carried out. The cleaning process and its frequency depend on the filtered product and on the chemical resistance of the membrane. Most cleaning procedures are a combination of hydraulic and chemical cleaning. Wang et al. [33] investigated the possibility of using polyvinylidene fluoride (PVDF) membrane to treat emulsified oily wastewater. They reported that the MF could effectively treat the laboratory prepared emulsified oily wastewater and the fouled membrane could be recovered by using conventional cleaning methods. Salahi et al. [34] proposed a cleaning procedure using a metal chelating agent (EDTA) and an anionic surfactant (SDS) which was able to regenerate the fouled UF membranes effectively. Yan et al. [35] reported that the addition of nano-sized alumina particles improved membrane antifouling performance, and the flux recovery ratio of modified membranes reached 100 percent washing with 1 weight percent of a surfactant solution (pH 10). More work on membrane cleaning can be cited in Al-Malack [36] and Lindau and Jonsson [37].

1.1. Fouling mechanisms

In crossflow MF processes, there occurs a formation of a secondary or dynamic membrane on top of the primary membrane. Fouling mechanisms, as a result of dynamic membrane formation, was divided into three categories by Tanny [38]. Class I dynamic membranes are formed when filtering suspensions where the particles have a particle size greater than the pore size of the membrane. This phenomenon is known as concentration polarization.

Class II dynamic membranes are created when filtering dilute suspensions of colloidal particles of particle size much smaller than the pore size of the membrane. In this case, the flux decline mechanism was found to behave according to an internal pore clogging phenomenon rather than cake build-up on the membrane surface. Visvanathan and Ben Aim [39] and other investigators [40–43] reported similar results. All investigators reported the following fouling model which represents the decrease in permeate volume:

$$\frac{t}{V} = \frac{1}{Q_0} + \frac{k_1 t}{2} \tag{1}$$

where V is the permeate volume, t is the filtration time, Q_0 is the initial flux rate and k_1 is the filtration constant. After some time, the colloidal particles will be brought-up to the membrane surface, and the flux behavior will proceed in accordance with the following classical cake filtration model [44]:

$$\frac{t}{V} = \frac{1}{K_1}(V - 2V_f) \tag{2}$$

where V_f is the volume of permeate which produces a hydraulic resistance equal to that of the membrane, and K_1 is the cake filtration constant. A different form of the cake filtration model was reported by Visvanathan and Ben Aim [39] and was as follows:

$$\frac{t}{V} = \frac{1}{Q_0} + \frac{K_1 V}{2} \tag{3}$$

Al-Malack and Anderson [45] investigated the formation of dynamic membranes with crossflow MF. They concluded that dynamic membrane formation obeys the standard law of filtration in the first few minutes of membrane formation (15 min). As time passes, the dynamic membrane formation was found to proceed according to the classical cake filtration model. Moreover, Akay et al. [46] investigated the removal of phosphate from water by red mud using crossflow MF. They evaluated the specific cake resistance in crossflow MF as a function of phosphate concentration by using the cake filtration model.

Class III dynamic membranes are formed when filtering polymers or polyelectrolyte molecules of equal size to the membrane size.

Based on the above literature review, it could be concluded that the immersed membrane filtration processes demands further attention for more rigorous investigation, particularly focusing on its feasibility in the treatment of oily wastewater.

2. Materials and methods

The membrane pilot plants were commissioned at Riyadh petroleum refinery. As per the instructions of the manual, the immersed membrane was cleaned with service water for one hour. At the end of the one-hour cleaning, the process tank was drained and again refilled with service water. An amount of sodium hypochlorite was added to maintain a concentration of 200 mg/l. The membranes were soaked in the tank for approximately 18 h at a pH range of 8–10.

The immersed membrane pilot plant comprised of peristaltic pump, process tank of 254 liter (67 US gal) capacity, control panel, immersed membrane module with an extended aeration tube, blower and back pulse tank of 27 liter (7 US gal). The general characteristics of the immersed membrane are given in Table 1. On the other hand, the crossflow pilot-scale crossflow membrane comprised of pumps, process tank (100 gal), permeate tank (50 gal), and crossflow membrane module. The general characteristics of the crossflow membrane are given in Table 2.

Table 1
General specifications of the immersed membrane module

Configuration	Outside/in hollow fiber
Membrane surface area	0.93 m^2
Weight of module (drained)	1.9 kg
Weight of module (wet)	2.1 kg
Permeate (Fiber side) hold up volume	0.13 liter
Nominal pore size	0.04 μm

Table 2
General specifications of the crossflow membrane module

Configuration	Eight 21 mm ID tubes in series
Membrane surface area 3 feet length	0.4 m^2
Membrane surface properties	Non-ionic and hydrophilic
Normal membrane pore diameter	0.1 micron
Maximum hold up volume	12 ft; 31.6 liter
Cleaning pH range	2–11

The two pilot–scale setups, namely immersed and crossflow membranes, were installed at the selected petroleum refinery to treat the effluent of the API separator. The study was conducted for six weeks. Samples from the API separator effluent and the membrane permeate were collected and analyzed for different water quality parameters at the laboratories of King Fahd University of Petroleum and Minerals. All analyses were conducted in accordance with the Standard Methods [47].

The cleaning procedure was followed once a week as per the instructions given in the pilot plant manual (with 200 ppm sodium hypochlorite). The cleaning procedure was not found to produce consistent results and, consequently, cleaning with sodium hypochlorite was discontinued. The two pilot-scale membrane processes were cleaned using commercially available detergents such as Tide, while maintaining the pH of the cleaning solution at a value around 8.5. The two membrane processes were cleaned once a week for 3 h. Back pulsing for 15 s was adopted during cleaning, every 15 min. The temperature of cleaning solution was maintained around 35°C.

3. Results and discussion

3.1. Permeate flux

With respect to the immersed membrane, Fig. 1 shows the permeate flux rate obtained over the investigation period (more than 800 h). The figure clearly shows that the flux values were stable throughout the investigation (>50 $l/m^2 h$). It is worth

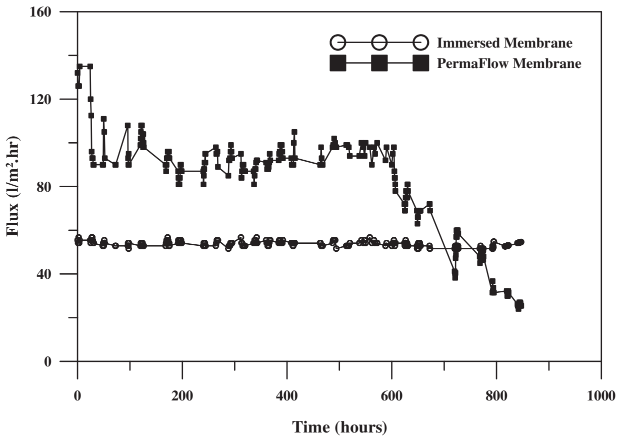

Fig. 1. Permeate flux with respect to time of both pilot-scale plants.

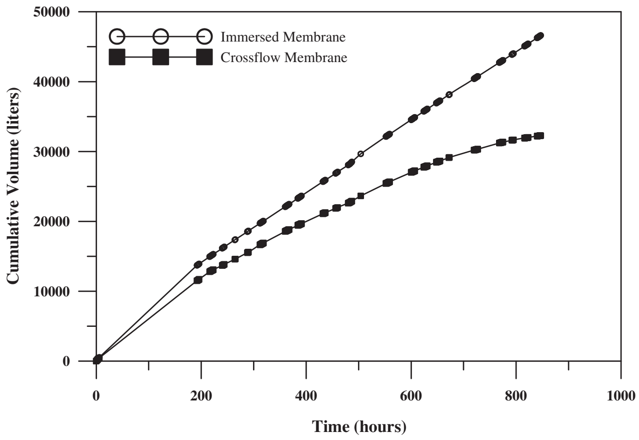

Fig. 2. Permeate cumulative volume produced by both membrane processes.

to mention that the investigation was carried out at the lowest possible pump flow rate. The flux stability could be attributed to presence of air diffusers at the bottom of the membrane module. The air diffusers were provided to prevent or even reduce fouling of the membrane and serve the purpose of mixing the reactor contents and maintain aerobic conditions in the reactor. The other reason could be the low TMP (less than 5 psi) that was maintained throughout the experimental period. Regarding the crossflow membrane, Fig. 1 also shows the permeate flux rate obtained over the investigation period (about 800 h). The figure shows that the flux values initially declined from 140 l/m^2 h to around 90 l/m^2 h within the first 100 h of operation. After about 600 h of operation, the permeate flux was found to decline steadily regardless of frequent backwashing and cleaning cycles. At the end of the investigation (more than 800 h), the flux was found to reach a value of about 20 l/m^2 h. The fast drop in the flux value could be attributed to either a decrease in the useful membrane area (due to clogging of membrane pores) or an increase in hydraulic resistance to filtration. This increase in hydraulic resistance can be caused either by narrowing of the pores (in depth clogging) or by cake formation on the primary membrane surface. The total hydraulic resistance comprises resistance caused by internal membrane fouling, and resistance caused by deposition of particles and or colloids on the primary membrane surface. Other reasons which could have caused membrane clogging include the size of the pores of the membrane, the surface charge of the membrane, the mechanism of adsorption of particles onto the membrane surface and the hydrophobic or hydrophilic nature of the membrane surface.

3.2. Cumulative volume

The collected permeate cumulative volume with respect to running time is shown in Fig. 2 for both membrane processes. The figure clearly demonstrates that, at the beginning of the experiment, the cumulative volume of the immersed membrane was marginally higher than that for the crossflow membrane process. After 400 h of the running period, the figure evidently shows that the differences in permeate volumes produced by both membrane process were becoming significant. By the end of the running time, 46.6 and 32.3 m^3 of permeate volume were collected from the immersed and crossflow membrane processes, respectively. This is mainly attributed to the fouling of the crossflow membrane process, which resulted in the steady decrease in the permeate flux.

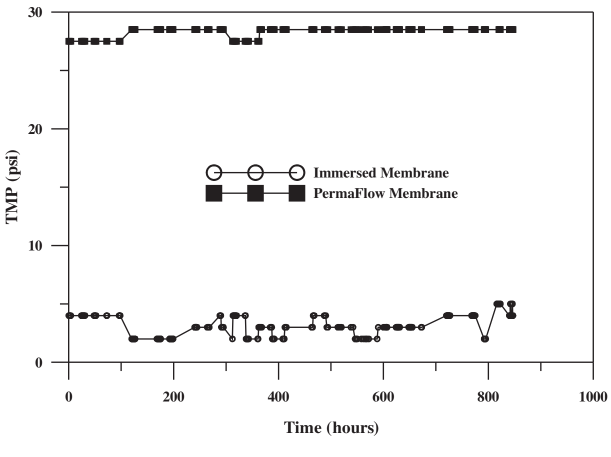

Fig. 3. Transmembrane pressure with respect to time of both pilot scale plants.

3.3. Transmembrane pressure

Fig. 3 shows the TMP throughout the investigation period. The figure shows that TMP, for the immersed membrane, was fluctuating around a value of less than 5 psi, which can be attributed to the automatic backwashing mode and the low adopted flow rate. On the other hand, Fig. 3 also shows the TMP, for the crossflow membrane, throughout the investigation period. The figure clearly shows that the TMP was fluctuating around a value of 27 psi, which can be attributed to the membrane fouling. It is worth mentioning that the crossflow membrane process was operated at an almost constant TMP, which is clearly reflected on the decreasing permeate flux values.

3.4. Hydraulic resistance

Fig. 4 shows the hydraulic resistance caused by deposition of oil and particles with time for both membrane processes. It is worth mentioning that the hydraulic resistance was calculated using the following equation:

$$J = \frac{\Delta P}{\mu \mathrm{R}} \tag{4}$$

where J is the permeate flux, ΔP is the TMP, μ is the liquid viscosity and R is the membrane resistance. The figure clearly shows that the hydraulic resistance of the immersed membrane was almost constant (0.272×10^{-10} per meter). On the other hand, the hydraulic resistance of the crossflow membrane processes was found to increase with time. At the end of the investigation period, the hydraulic resistance reached values of 0.272×10^{-10} and 2.77×10^{-10} per meter for the immersed and crossflow membrane processes, respectively. The sharp increase in hydraulic resistance of the crossflow membrane processes can be attributed to fouling of the membrane. It is worth to mention that membrane performance is dependent on more complex interactions between particle size, membrane surface, and performance history, as well as a number of other factors. There are several reasons which could have caused clogging of the crossflow membrane. Firstly, when comparatively small pore size membranes are used, the cake filtration mechanism will be dominant and clogging of the pores will be reduced, which will in turn lead to the alleviation of the flux decay and improvement in water quality. The hydraulic resistance results clearly indicate that cake filtration mechanism was not the predominant in this case. Secondly, it is suggested that membrane surface charge influences electrostatic interactions between particles in the feed and the primary membrane. Particles in water and wastewater possess electrostatic charges that are usually negative but, occasionally, may be positive. Thus, if the membrane has an opposite charge (positive), negatively charged particles will be attached to the membrane surface by electrostatic interactions. The third reason for the aforementioned clogging phenomenon of the crossflow membrane is particle adsorption onto the membrane surface. There are three mechanisms by which particles can be adsorbed onto membrane surfaces, namely electrostatic (ionic) attractions, hydrophobic interactions and hydrogen bonding, which is a much weaker mechanism.

3.5. Membrane backwash

The immersed membrane process was operated with automatic backwash cycles. Every 15 min, the

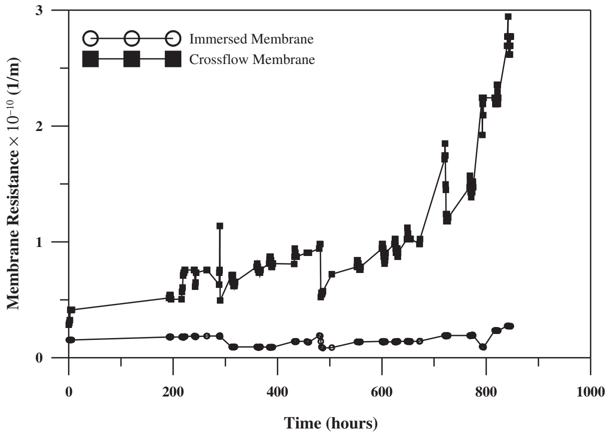

Fig. 4. Membrane resistance of crossflow and immersed membrane processes.

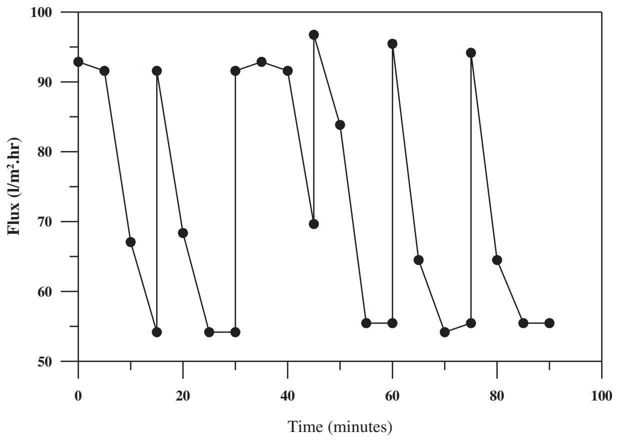

Fig. 5. Backwashing cycles of the immersed membrane.

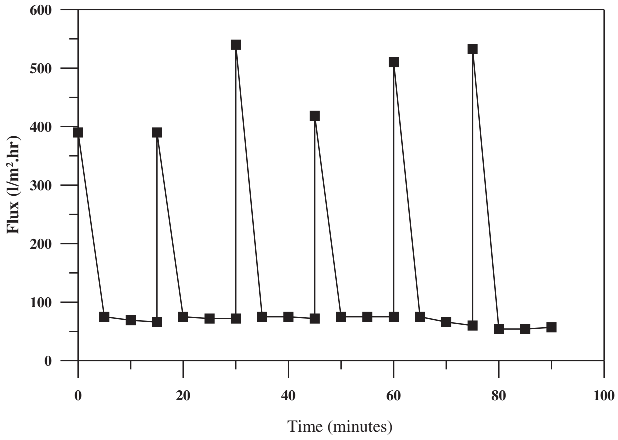

Fig. 6. Backwahsing cycles of the crossflow membrane.

process would backwash the membrane using the collected permeate for 15 s. It seems that the flux stability could be attributed to the automatic backwash (backpulse) cycles in addition to the presence of air diffusers at the bottom of the membrane module. Fig. 5 shows results on a typical backwash cycles collected over a period of 90 min for the immersed membrane process. The figure clearly shows the effect of backwashing on the flux values, where the flux was found to reach a value of more than 90 l/m^2 hr after backwashing, but eventually the flux was found to drop to its initial value within 10 min. Fig. 6 shows results on a typical backwash cycles collected over a period of 90 min for the crossflow membrane process. The figure clearly shows that backwashing was not found to have a significant effect on the permeate flux values. Immediately after cleaning, the permeate flux was noticed to reach values between 400 and 550 l/m^2 h, but it was also decreasing rapidly. As in the case of the immersed membrane process, the crossflow investigation was also carried out at the lowest possible pump flow rate. It is worth to mention that the crossflow process was operated with manual backwash cycles. The process was backwashed every 15 min for 15 s using the collected permeate. The flux decline can be attributed to the build-up of particles on the surface of the membrane. The cleaning results showed that the immersed membrane was cleaned effectively and the membrane could be brought to its initial conditions. In the case of the crossflow membrane processes, the cleaning procedure was not found to be effective at all, particularly after sometime of operation.

3.6. Permeate quality

Results on the quality of the permeate collected from the immersed and crossflow membrane processes are shown in Table 3. The investigation showed that both membranes used in the study had the ability to almost completely remove oil for the treated wastewater, regardless of the initial oil content in the

Table 3
Quality of permeate samples produced by both membrane processes

Parameters	API Effluent	Immersed membrane effluent			Crossflow membrane effluent		
		Minimum	Maximum	Average	Minimum	Maximum	Average
Conductivity	2,505	1997	2,578	2,341	1915	2,618	2,227
pH	8.925	8.29	10.09	8.79	8.22	12.06	8.57
TDS (mg/l)	1,471	1,228	1,707	1,505	1,225	1845	1,501
TSS (mg/l)	104.5	0	0	0	0	0	0
BOD (mg/l)	150.5	37.70	115	62.93	60	114	81
COD (mg/l)	228	44	278	113	76	295	153
NH_3-N (mg/l)	14.01	0.14	16.89	11.36	4.89	28.52	15.15
TKN (mg/l)	29.26	12.81	22.95	17.75	8.56	28.52	18.66
Cl (mg/l)	348.5	267	401	341	263	360	312
SO_4 (mg/l)	546.5	305	491	406	284	458	353
NO_3-N (mg/l)	15.685	24.38	20.06	10.06	1.65	10.05	4
Alkalinity (mg/l)	329.5	151	220	195	168	1,093	347
PO_4 (mg/l)	1.625	0.17	0.94	0.51	0.11	0.95	0.38
Si (mg/l)	16.45	2.36	7.30	5.05	1.95	6.65	4.58
NO_2-N (mg/l)	15.22	0	17.88	10.91	7.32	15.08	11.28
O & G (mg/l)	36	<1.4	2	<1.4	<1.4	2.4	>1.4
Phenol (mg/l)	0.92	<0.1	0.55	0.21	<0.1	0.71	0.44

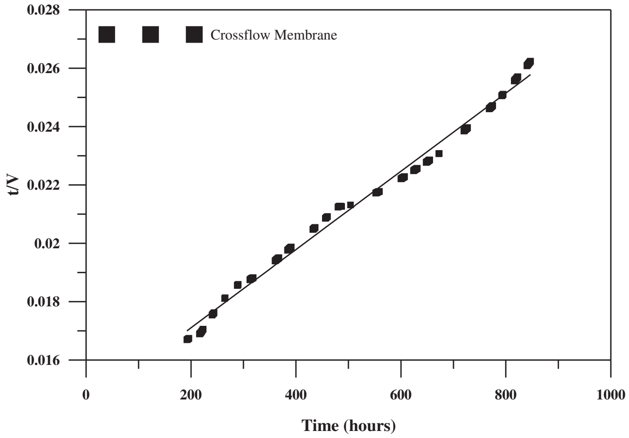

Fig. 7. t/V versus time for crossflow membrane process.

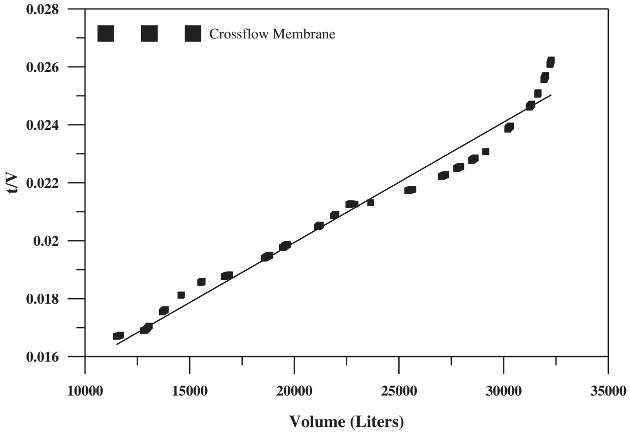

Fig. 8. t/V versus permeate cumulative volume for crossflow membrane process.

feed. The table clearly demonstrates that suspended solids were completely removed from the wastewater. The average concentrations of oil and grease were less than the detection limits of the method used throughout the investigation (1.4 mg/l). The total dissolved solids (TDS) in the permeate is expected to be the same as that of the feed due to the fact that those membranes were not made to remove dissolved solids. Permeate TDS values of 1,500 mg/l represent the TDS content in the effluent produced by the API separator at Riyadh refinery (1,470 mg/l). The BOD removal efficiencies were 58 and 46 percent for the immersed and crossflow membrane processes, respectively, while the COD removal efficiencies were 50 and 33 percent, respectively. Water quality results suggest that the permeate could be used as a feed to the reverse osmosis (RO) plant installed at the refinery.

4. Fouling mechanisms of crossflow membrane

The evaluation of the mechanism by which the primary membrane is being fouled when treating oily wastewater was carried out using the standard law of filtration and the classical cake filtration models. Figs. 7 and 8 show clearly indicated that obtained data can be represented using both models. The results indicate that there is no predominant model that can be used to describe the fouling mechanism of the crossflow membrane process. This could be attributed to the presence of particles of different sizes which will result in clogging the pore and, at the same time, forming a cake layer of dynamic membrane on the membrane surface.

Standard law of filtration

$$(\mathrm{t/V}) = 1.34 \times 10^{-5} \times t + 0.0144 \quad (R^2 = 0.99)$$

Initial flux rate, $Q_0 = 69.4$ liter/h
Filtration constant, $k_1 = 6.7 \times 10^{-6}$ (per liter)

Classical cake filtration

$$(\mathrm{t/V}) = 4.15 \times 10^{-5} \times V + 0.0116 \quad (R^2 = 0.96)$$

Initial flux rate, $Q_0 = 86.2$ (liter/h)
Cake filtration constant, $K_1 = 2.1 \times 10^{-5}$ (h/liter^2)

5. Conclusions

The performance of immersed and crossflow membranes in treating refinery wastewater was investigated. Two pilot-scale plants, namely crossflow and immersed membrane, were used in the current investigation at Riyadh petroleum refinery. The results showed that the immersed membrane process was found to perform well in treating oily wastewater, where the permeate flux was almost stable at its initial value throughout the investigation (more than 50 l/m^2 h). Moreover, the immersed membrane was effectively cleaned using a commercial detergent, while the sodium hypochlorite recommended by the manufacturer was not found effective in cleaning the membrane. On the other hand, the crossflow membrane process was not found to perform satisfactorily when treating oily wastewater produced from petroleum refineries. The permeate flux was found to decline rapidly from 140 to 90 l/m^2 h after 100 h of operation. After 600 h of operation, the permeate flux was found to steadily declining regardless of backwashing and cleaning cycles. Based on the obtained results of the investigation, the immersed membrane process is recommended as a potential process for treating oily wastewater produced from petroleum refineries. In addition, Pretreatment of the API separator effluent is expected to improve the performance of the immersed membrane and may prolong its service life. To further improve the quality of the permeate in terms of COD, it is highly recommended to investigate the use of the immersed membrane process in treating oily wastewater and other types of industrial wastewater as a membrane bioreactor (MBR). Furthermore, it is highly recommended to investigate the kinetics of activated sludge processes coupled with the immersed membrane. Fouling of the crossflow membrane process was found to proceed in accordance with the standard and cake filtration model simultaneously.

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