

Application of membrane distillation for the treatment of oil field produced water

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Abstract

Direct contact membrane distillation (DCMD) was investigated for the treatment of oil-field produced water using a hydrophobic polypropylene (PP) membrane with 0.2 μm pore size. The DCMD performance was studied under different feed temperatures ranged from 40 $^{\circ}\text{C}$ to 80 $^{\circ}\text{C}$ while the cooling temperature was maintained at 23 $^{\circ}\text{C}$. Increasing the feed water temperature resulted in a higher permeate water flux. Stable and reliable DCMD membrane performance was observed for all used membranes. The obtained results indicated the great potential of DCMD to treat hypersaline oil-field produced water with an overall rejection of salts higher than 99.9% and that of total organic carbon (TOC) greater than 93.3%. This was due to the presence of volatile organic compounds in oil-field water. Pre-treatment of produced water using 0.45 μm filter did not show much effect on the DCMD performance. A slight gradual reduction of the permeate flux was observed due to fouling phenomenon. A simple washing the membrane with de-ionized water was found to be an effective method for cleaning the membrane and restoring the permeate flux indicating the absence of irreversible fouling.

Key words

Oil-field produced water; Membrane distillation; Polypropylene membrane; Water treatment; Fouling

1. Introduction

Oil-field produced water is the largest by product that is inevitably generated during oil and gas extraction operations [1, 2]. In general, around 250 million barrels of this produced water is extracted each day around the world (40% of it is disposed to the environment [3]) while the corresponding produced oil is 80 million barrels [1, 2]. The produced water contains different organic and inorganic materials including salts, dissolved oil, heavy metals, dissolved gases, dispersed oil, treating chemicals and production solids [3-8]. The geological field layers and the type of produced hydrocarbons affect the concentration of these compounds in the produced water as well as its physical and chemical properties [3-5]. Discharging this water to the environment lead to many serious environmental impacts. Therefore, stringent regulatory standards have been adopted for the discharge of this produced water to the environment resulting in a major challenge to oil and gas industry worldwide [6]. Furthermore, the required huge quantities of water for oil production aggravates the water scarcity in various countries. Therefore, the high required standards for the oil-field produced water before its disposal together with the need for clean water in various areas with limited fresh water resources led to the application of necessary extensive water treatments [1, 3-5].

Various separate and combined methods have been used for produced water treatment including chemical coagulation, biological treatment, thermal processes and membrane separation processes [1, 3, 4, 9]. The application of these technologies is constrained by their high cost, use of toxic chemicals, large installation space and secondary pollution [1, 4, 10]. It is worth noting that although the pressure-driven membrane based processes have been reported as the 21st century convenient technologies for the treatment of produced water [1], membrane fouling and the associated high energy

costs limited the performance of these processes [9, 11]. For instance reverse osmosis (RO) is limited by the high salinity while the other processes nanofiltration (NF) and microfiltration (MF) are unable to remove all dissolved components including hydrocarbons [1, 11].

Membrane distillation (MD) is a thermally-driven membrane based method exhibiting various recognized advantages such as its high salt(s) rejection factor (i.e. 99-100%) and its capability to treat high saline aqueous solutions up to super-saturation [12, 13]. Moreover, MD operates at temperatures below the boiling point of the processing aqueous solutions, permitting the use of available energy sources from other processes such as waste heat and solar energy [12, 14-16], and at hydrostatic pressures lower than that applied in pressure-driven membrane technologies (MF, NF, RO) enabling it to treat highly concentrated solutions [11, 14, 17-19]. As it is well known MD requires the use of hydrophobic porous membranes and only water in vapor phase can pass through [13, 14, 20]. Due to the surface tension forces of the hydrophobic membrane, liquids should not enter its pores and liquid-vapor interfaces are formed at their entrances [13, 14, 16, 20, 21]. This process is driven by the vapor pressure difference and different configurations are considered to apply this driving force (e.g. direct contact membrane distillation, DCMD; air gap membrane distillation, AGMD; sweeping gas membrane distillation, SGMD; vacuum membrane distillation, VMD) [13, 14, 22-24]. Among them, DCMD is the most simple to operate [11, 13, 14].

In general, MD has been mostly considered for desalination and only few researches are available on its application to treat produced water. Gryta and Karakulski [25] studied the impact of oil concentration (0-2000 ppm) on the MD performance at different operating conditions and found that the permeate flux was affected adversely by

increasing the oil concentration. It was claimed that this reduction could be limited in some degree by increasing the feed temperature. Han et al. [9] investigated the influence of the main produced water components (oil, surfactants and salts) on the MD performance in terms of process stability, production rate and effluent quality. They concluded that the main responsible for the MD performance reduction is the combined presence of salts and surfactants and suggested to pre-treat produced waters by either removing salts or surfactants before applying MD. Macedonio [1] carried out DCMD tests using different membrane materials with different pore sizes under different operating conditions for the treatment of hyper saline oily water with 247900 mg/L salts concentration. They observed a stable and reliable DCMD performance in all conducted tests insuring the lack of membrane wetting. In terms of permeate quality, a high rejection factor was obtained for both total dissolved solids (> 99% rejection) and total organic carbon (> 90% rejection). AlKhudhiri et al. [26] used AGMD to treat hyper saline produced water with 187,440 ppm salts content and obtained a maximum salt rejection of 99.99% and 98.6% for organic carbon rejection. They finally stated that AGMD was an effective process to treat produced water. However, still there is a much concern about the MD application of real produced water processing as most studies were conducted using synthetic waters. The present study is aimed to investigate the efficiency of MD process to treat real produced water from an oil-field located north of Oman. Different water qualities have been investigated to build up a clear comparison study. The effects of various parameters affecting the permeate flux including the feed temperature and feed salinity have been studied. The stability of polypropylene (PP) membranes has been investigated carrying out a long-term study. Furthermore, the fouling tendency of these membranes was also studied.

2. Experimental

2.1. Produced water

The produced water from an oil-field in the north of Oman was supplied by Petroleum Development Oman (PDO). It was collected from the effluent of skim tanks whose characteristics are summarized in Table 1.

Table 1. Basic characteristics of the used produced water.

Component	Concentration
Total dissolved solids (TDS)	135,000 - 140000 ppm
Total hardness	47,000 - 49,000 ppm as CaCO ₃
Temperature	23 – 25 °C
TOC	39 ppm
BOD	20-30
COD	250-500
Oil in water (max)	76
Oil in water (average)	< 25 ppm
Sulphate (SO₄)	350 - 400 ppm
Calcium (Ca)	15,000 - 19,000 ppm
Magnesium (Mg)	260 – 280 ppm
Boron as B	20-35 ppm

2.2. Polypropylene Membrane

The used membrane was a flat-sheet polypropylene (PP) microporous hydrophobic membrane supplied by Sterlitech Corporation (PP022005). Its characteristics

are provided in Table 2. The surface morphology of the membrane was examined before and after the DCMD experiments by Scanning Electron Microscopy (FESEM, JSM-7600F, Tokyo, Japan) coupled with Energy Dispersive X-ray spectroscopy analysis (EDS) in order to determine its surface elemental composition. Attenuated total reflection Fourier transform infrared spectroscope (ATR-FTIR, PerkinElmer, FT-IR spectrometer Frontier, USA) was employed to study possible interactions between the feed components and the membrane material. The hydrophobicity of the membrane was studied by water contact angle measurements before and after DCMD experiments using a ThetaLite tension tensiometer (Biolin Scientific, Sweden).

Table 2. Basic characteristics of the used PP membrane.

Material	Polypropylene (PP)
Pore size (μm)	0.2
Porosity (%)	70
Thickness (μm)	110
Contact angle ($^{\circ}$)	114.3 ± 3.9

2.3. DCMD experiments

DCMD tests were performed using the experimental set-up schematized in Fig. 1 with different feed solutions: de-ionized water, simulated saline water and produced water (with and without pre-treatment). The pre-treatment experiment consists on filtration through a $0.45 \mu\text{m}$ filter (Whatman®, nitrocellulose membrane). The feed temperature was varied from $40 \text{ }^{\circ}\text{C}$ to $80 \text{ }^{\circ}\text{C}$ with an interval of $20 \text{ }^{\circ}\text{C}$. The stirring rate and permeate temperature were maintained at 1500 rpm and $23 \text{ }^{\circ}\text{C}$, respectively. Each experiment was

run for 5 h, and the data were recorded after 45 minutes to allow the stability of the permeate flux, which was measured every 30 minutes to check both the operational stability and performance of the used membrane. The total dissolved solids (TDS) and electrical conductivity were measured by means of MYRON L COMPANY conductivity/TDS Ultrameter II. The TOC content was measured by TOC-VCPN analyzer (Shimadzu, Japan).

The stainless-steel membrane cell is composed of two double walled compartments (i.e. feed and permeate). The membrane supported by a stainless-steel grid was placed between the two compartments. The permeate water was collected in a graduated cylinder and the mass of the collected permeate was measured using a digital balance. The temperatures of both the feed and permeate were maintained constant by their continuous circulation using a magnetic stirrer inside each compartment connected to the corresponding recirculation bath. These temperatures were monitored by Temperature sensors and the stirring rates were maintained the same in both compartments.

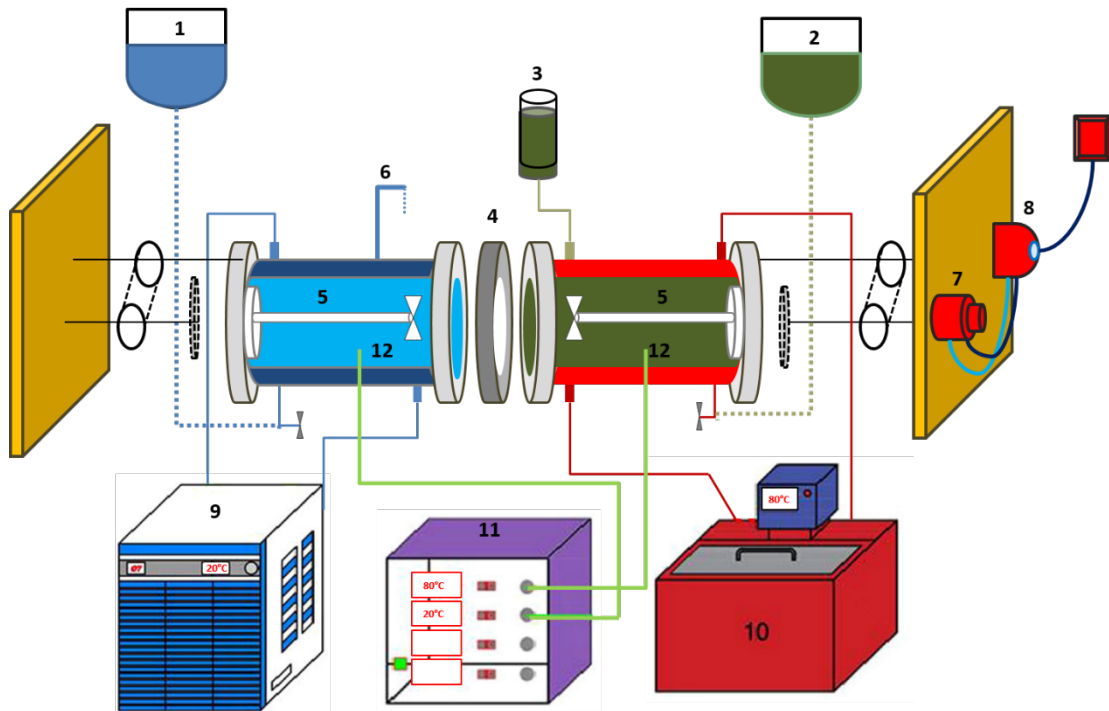


Figure 1: Schematic of DCMD Set-up: (1) permeate container, (2) feed container, (3) feed supplier during DCMD test, (4) membrane Holder, (5) stirrers and magnets, (6) permeate product, (7) stirring rate regulator, (8) motor for stirring feed and permeate, (9) chiller (cryostat), (10) heater thermostat, (11) digital multimeter, (12) temperature sensors.

3. Results and discussions

3.1. Synthetic salt solution

Before carrying out DCMD tests using real produced water as feed, a model NaCl aqueous solution with a concentration of 135,000 ppm similar to that of produced water was used. Figure 2 shows the effect of NaCl concentration on the permeate flux and salt rejection at different operating feed temperatures. For comparison, the permeate flux of de-ionized water used as feed was also presented for the same feed temperatures. Results show that the permeate flux corresponding to the NaCl aqueous solution was lower than that of deionized water due to the reduction of the water chemical potential (reduction of

the water vapor pressure) as shown in Table 3, Equations S.4 - S.6). Permeate flux was approximately constant for the full time of operation (120 min deionized water and 300 min for 135,000 ppm NaCl synthetic water). Average permeate flux was 1.5, 4.8 and 9.3 kg/m².h at 40, 60 and 80 °C respectively. Figure 2 shows that the average NaCl concentration in the product was 7.1, 6.2 and 13.2 ppm at feed temperatures of 40, 60 and 80 °C, respectively. These results correspond to NaCl rejection of >99.99 wt% for the studied operating temperature range. Permeate flux and rejection results demonstrate the stability of DCMD performance of the tested polypropylene membrane at the tested operating conditions. In addition, they demonstrate the absence of membrane pore wetting and leakage.

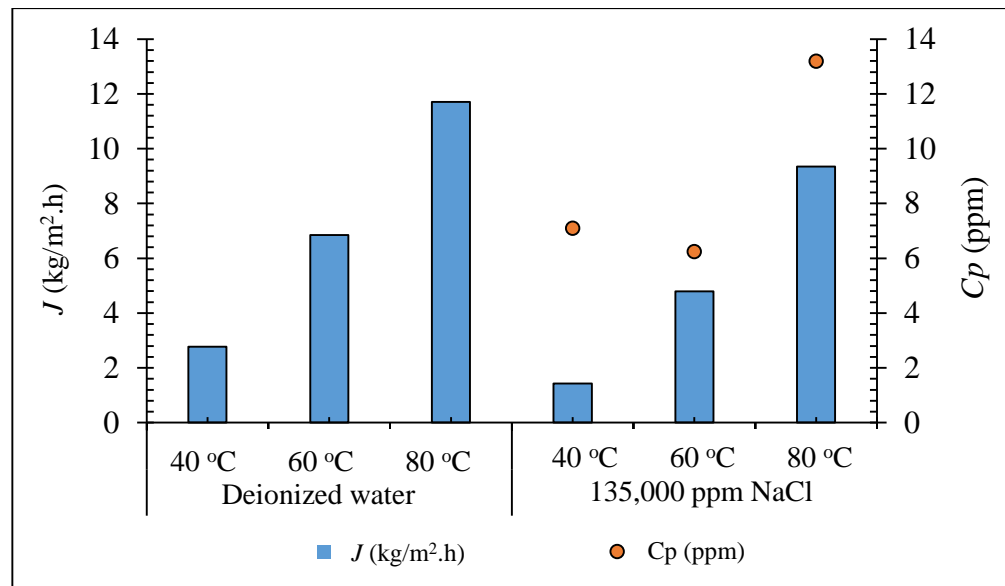


Figure 2. Effect of salt (NaCl) on DCMD permeate flux (J) and product concentration (C_p) at different feed temperatures after 120 min filtration time.

Figure 2 shows a reduction in water flux for synthetic NaCl solution compared to deionized water for the tested operating temperature range. The average reduction is approx. 49.3%, 28.3% and 23.5% at 40 °C, 60 °C and 80 °C, respectively showing that

80°C operating temperature has the highest flux efficiency. At 80 °C feed temperature, DCMD thermal efficiency is higher than at lower temperature. The increase in DCMD thermal efficiency at higher temperature lead to a reduction in NaCl concentration effect on permeate flux [16].

Table 3. Vapor pressure difference of DI water and saline water at different temperatures.

Temperature (°C)	Vapor pressure difference (kPa)	
	DI water	NaCl solution (135,000 ppm)
40	1.90	1.75
60	5.78	5.33
80	14.9	13.8

3.2. DCMD treatment of produced water

The produced water with all its components, namely, oil, salts and suspended particles was tested in DCMD. Figure 3 shows samples of the DCMD feed and permeate as well as the membrane appearance before and after the DCMD desalination tests.

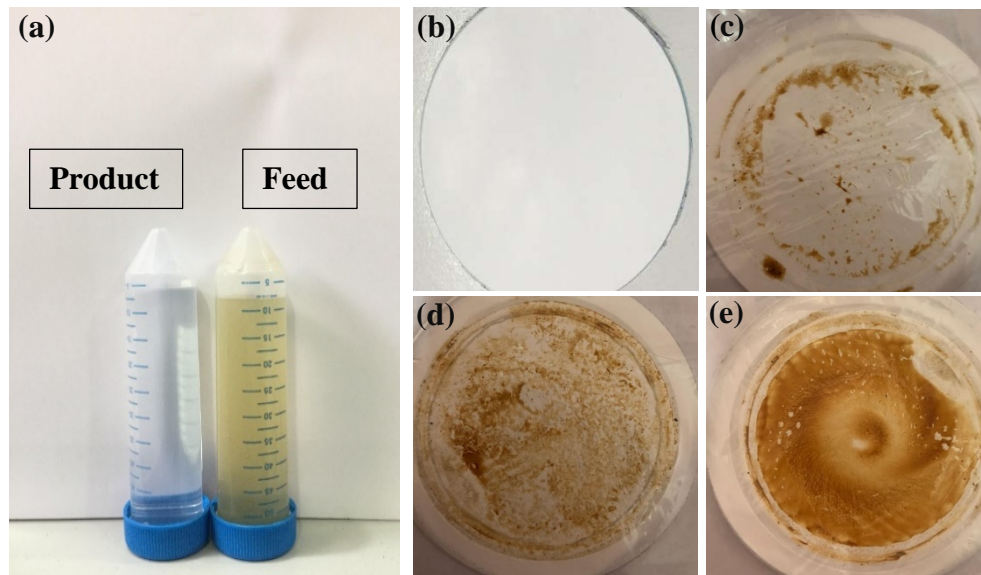


Figure 3. Pictures of feed and permeate samples of the DCMD (a), membrane appearance before (b) and after DCMD experiments at different feed temperatures 40 °C (c) 60 °C (d) and 80 °C (e).

Figure 4 shows the change of the DCMD permeate flux depending on the feed solution at different temperatures. Using oil-field produced water as feed resulted in a permeate flux reduction from that of deionized water of 53.5%, 34.9% and 28.6% at 40, 60 and 80 °C, respectively. This reduction is greater than that obtained when using NaCl aqueous solution as feed (i.e. 49.3%, 28.3% and 23.5% at 40, 60 and 80 °C, respectively) indicating that the presence of other contaminants (i.e. oil and suspended particles) induced a greater decline in the permeate flux. The reduction in flux is partly due to the deposited fouling layer on the membrane surface, as confirmed by the SEM images presented in Figure 5. The deposited fouling layer subsequently reduced the available membrane area for evaporation. To support the previous observation, EDS analysis associated to SEM was carried out to detect the elements present in the deposited layer at the membrane surface. The results are shown in Fig. 6. It was observed that the membrane surfaces are mainly covered by salts (Na^+ , Mg^{2+} , Ca^{2+} , Cl^-) deposition indicating that scaling was the main cause of membrane fouling which conforms with a previous study by Gunko *et. al.* [12]. The permeate fluxes are approx. 1.36, 4.15 and 7.74 $\text{kg/m}^2\cdot\text{h}$ at 40, 60 and 80 °C, respectively and the corresponding salt rejection was > 99.9% for the tested feed temperature range. It must be pointed out the stable permeate flux and high salt rejection demonstrate the absence of membrane pore wetting and leakage.

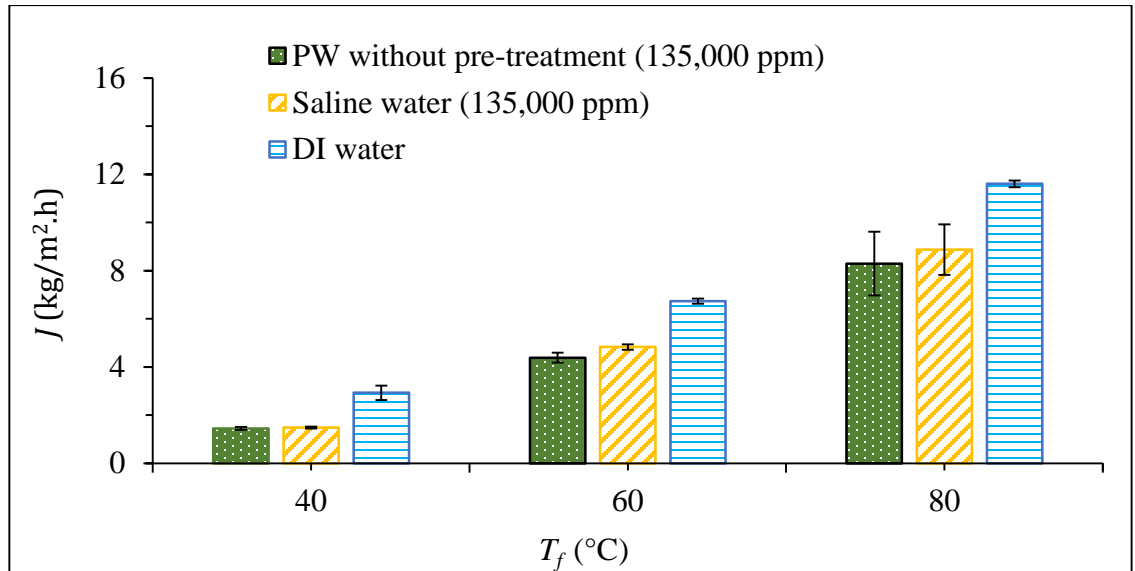


Figure 4. Permeate flux (J) after 300 min DCMD operation of different feed solutions: de-ionized water (DW); Saline water (135,000 ppm NaCl solution) and 135,000 TDS of Oil-field produced water (PW) without pre-treatment at different feed temperatures (T_f) (40, 60 and 80 °C).

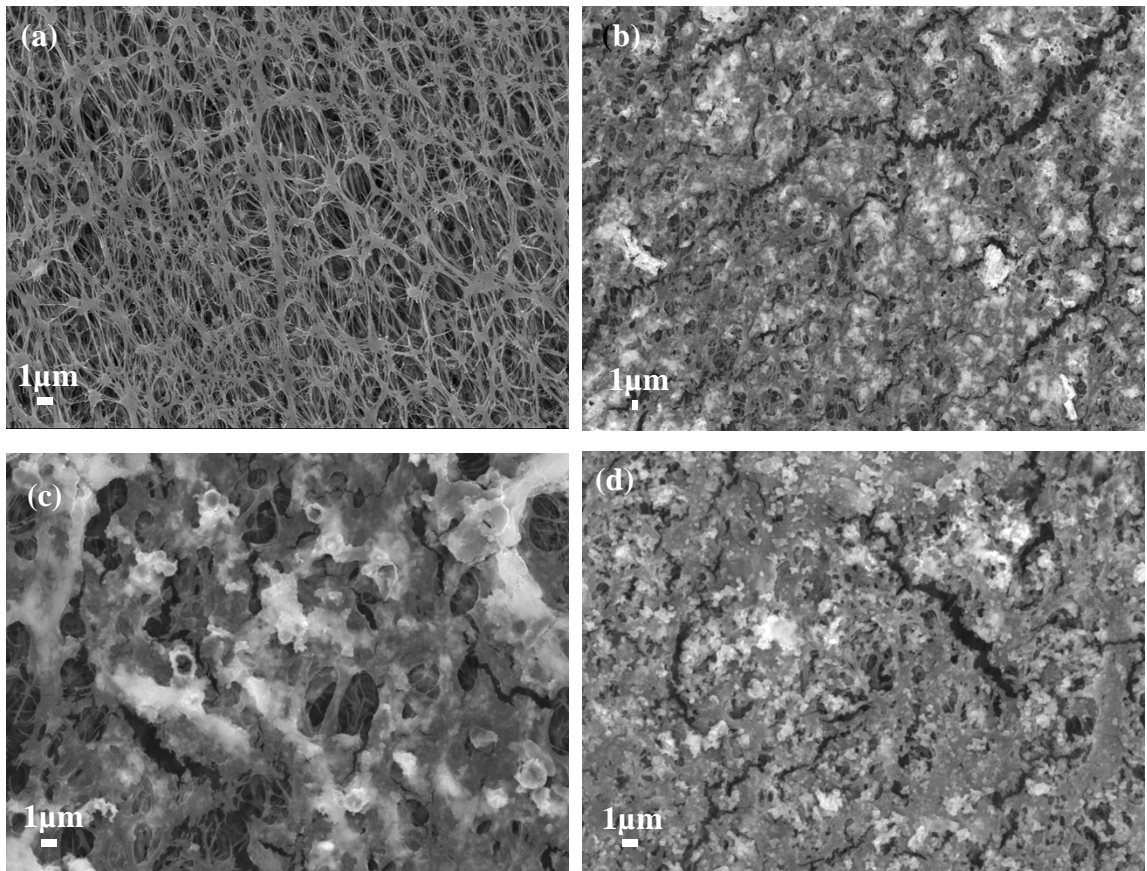
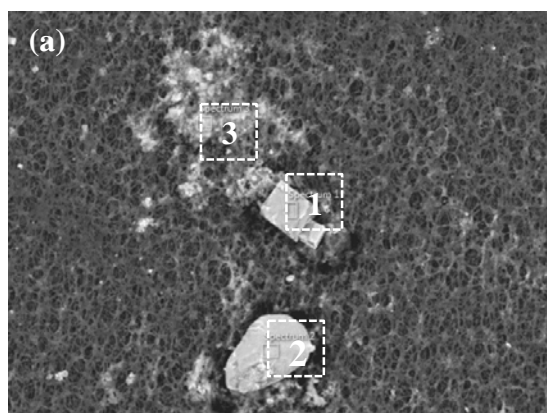
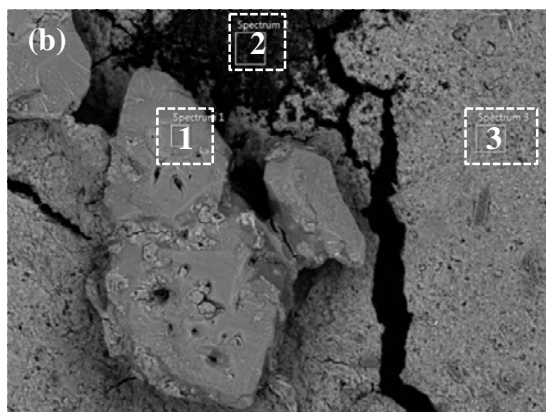


Figure 5. SEM images of the membrane surface in contact with the feed solution. (a) virgin PP membrane and used PP membrane after DCMD experiment of produced water at different feed temperatures, (b) 40°C, (c) 60°C and (d) 80°C.

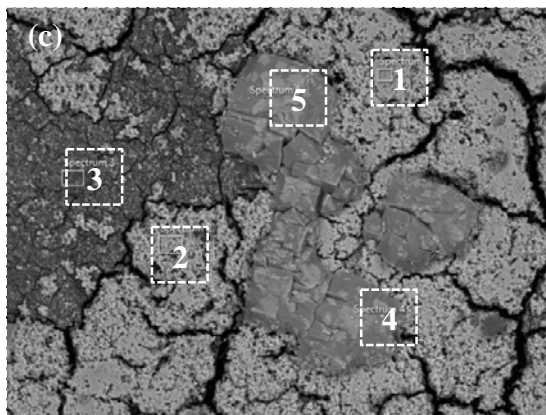
The amount of organic carbon in the permeate (1.73, 2.31, 2.58 ppm at 40, 60 and 80 °C, respectively) and salt rejection (95.53%, 94.04% and 93.35 at 40, 60 and 80 °C, respectively) indicate that the DCMD process is quite efficient in rejecting organic compounds present in oil-field produced water. The transport of these compounds through the membrane may occur by one of the three following ways: i) transport through wetted hydrophilic pores, ii) passage of oil droplets through the membrane pores from the area covered by oil droplets and/or iii) evaporation of volatile compounds and transport through the membrane pores in vapor phase. The first possibility can be proven by measuring the permeate electrical conductivity as salt(s) will also be transported simultaneously through the membrane pores with organic matter. This was not the case as the permeate flux was almost stable and the salt(s) rejection was always >99.9%. The affinity between the hydrophobic membrane surface and the hydrophobic oil nature might cause the second possibility. The build-up of the oil droplets on the membrane surface can cause a gradual permeate flux decline with time. Again, this wasn't the case. Therefore, the third possibility was the most probable cause due to the presence of volatile organic carbons (VOCs) in oil-field produced water. This is supported by the increase in VOCs concentration in the permeate at higher feed temperature. At higher feed temperature, a higher driving force is achieved due to higher vapor pressure which allows more VOCs transfer to the permeate side.



Element	Wt.%		
	1	2	3
Cl	60.7	58.8	62.4
Na	39.4	41.2	4.6
C	-	-	-
Ca	-	-	28.3
Mg	-	-	4.7



Element	1	2	3
	Cl	44.2	1.9
Na	31.7	-	6.7
C	23.3	97.1	-
Ca	0.81	0.72	25.2
Mg	-	0.31	4.7



Element	1	2	3	4	5
	Cl	44.9	45.7	11.7	43.6
Na	1.9	1.9	1.1	26.9	41.8
C	31.5	31.4	81.2	27.0	-
Ca	19.1	18.1	31.4	2.6	-
Mg	2.6	2.9	18.1	-	-

Figure 6. SEM images together with the corresponding EDS analysis of the used PP membrane after DCMD of produced water at different feed temperature, (a) 40°C, (b) 60°C and (c) 80°C

Figure 7 shows the FTIR-ATR spectra of the virgin PP membrane and the used ones after DCMD operation of produced water at different temperatures. The spectra were found to be similar indicating no clear interaction between the feed components and the membrane surface during DCMD treatment of oil-field produced water. In addition, the hydrophobicity of the membrane was preserved as all the used membranes exhibited water contact angles higher than 90° and almost close to the virgin PP membrane (Figure 8).

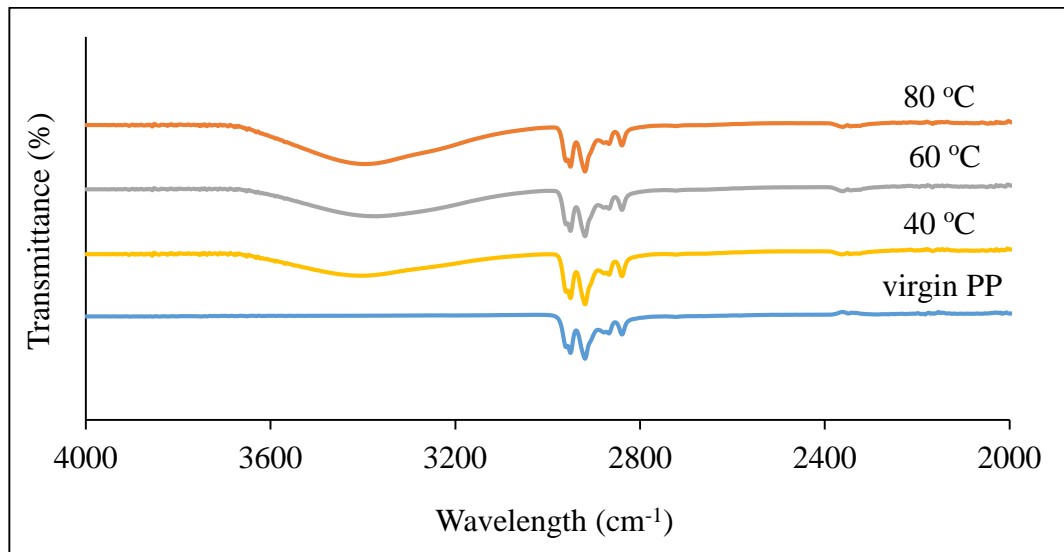


Figure 7. ATR-FTIR spectra of virgin and used membranes after DCMD desalination of produced water at different feed temperatures.

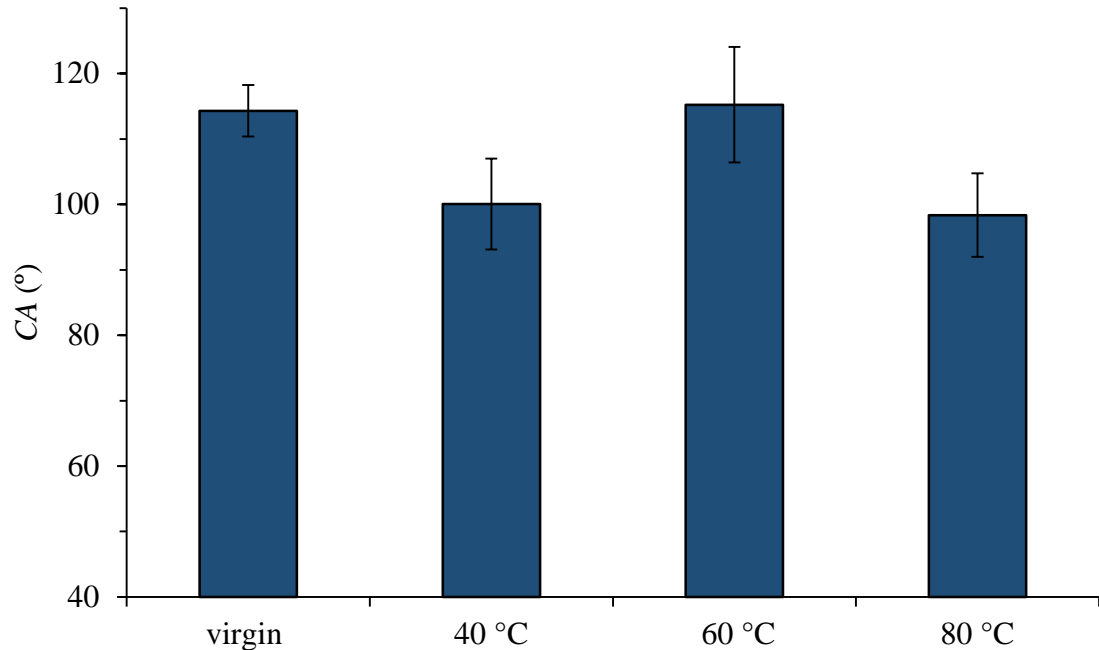


Figure 8. Water contact angle (CA) of the virgin and used membranes after DCMD experiments of produced water at different feed temperatures.

3.3. DCMD of pre-treated oil-field produced water

Although the results of the previous section indicated the small effect of the suspended particles and oil on the DCMD performance of PP membrane, the reduction of the permeate flux might be significant for long-term treatment of produced water. Therefore, the effect of the pre-treatment of produced water on the DCMD performance was investigated. As mentioned earlier, the pre-treatment consisted on filtration through 0.45 μm filter. Figure 9 shows the obtained permeate flux of the pre-treated feed produced water compared to that without pre-treatment and NaCl aqueous solution (135,000 ppm). The difference between the permeate flux of produced water with and without pre-treatment at 40 and 60 °C was nearly negligible (i.e. 2.6 % and 2.9 % for 40°C and 60°C, respectively). However, it reached 4.4 % at 80 °C. This greater reduction of the permeate

flux at higher temperature might be attributed to the fact that the membrane was more likely to be fouled for higher permeate fluxes associated to higher feed temperatures. This was observed clearly from the photos of the membrane surfaces showing the denser deposited layer at 80°C compared to those at lower temperatures when treating produced water without pre-treatment (Figure 3). In other words, it may be stated that the application of the pre-treatment step before DCMD treatment of produced water was significant at a higher feed temperature resulting in a lower membrane fouling and a greater DCMD performance.

It was found that the salt rejection factor was almost the same when using as feed the oil-field produced water with or without pre-treatment. Minimal effects of oil and suspended particles were detected. The obtained TOC concentration in the permeate were 1.07, 1.44 and 1.46 ppm at 40, 60 and 80 °C with the corresponding rejection of 89.71, 86.23 and 86%, respectively. Results proves the effectiveness of DCMD for the treatment of produced water even without pre-treatment.

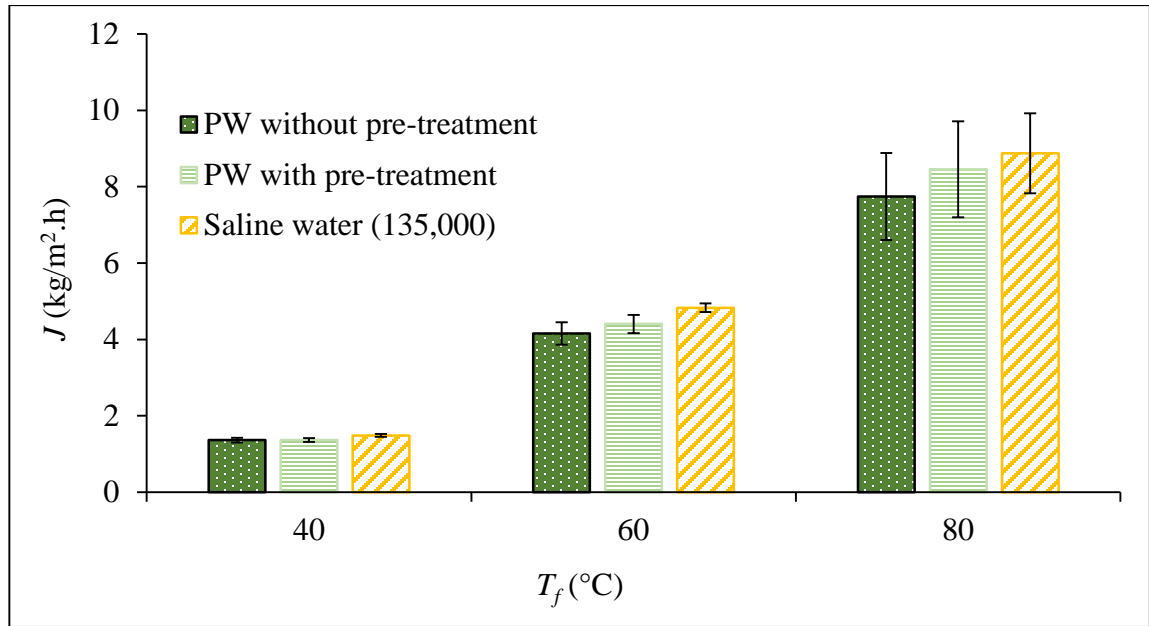


Figure 9. Permeate flux (J) of NaCl aqueous solution (135,000 ppm) and oil-field produced water (PW) with and without pre-treatment obtained after 300 min DCMD operation at different feed temperatures (T_f).

3.4. Longer time experiment, fouling and membrane cleaning

The stability of the polypropylene membrane during the treatment of oil-field produced water without pre-treatment was tested for a continuous 8 h time period maintaining the feed and permeate temperatures at 60 and 23 °C, respectively. As shown in figure 10, the permeate flux and salt rejection were maintained stable during this DCMD operation period. The permeate had a TDS value of 14.6 ppm, which corresponds to a salt rejection of approx. 100%. Moreover, the distillate contained around 5.31 ppm of TOC with a rejection factor of 86.39%.

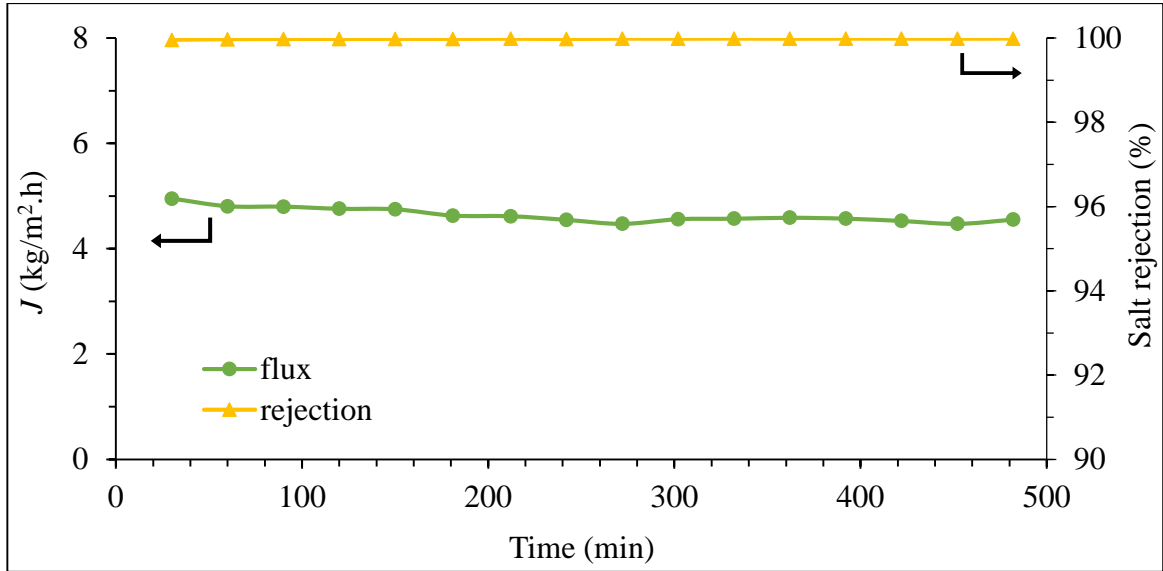


Figure 10. Variation of permeate flux (J) of PP membrane in a continuous 8 hours of DCMD experiments using oil-field produced water as a feed solution, the stirring rate of the feed and permeate aqueous solutions is 500 rpm, the permeate temperature (T_p) is 23 °C and the feed temperature (T_f) is 60 °C.

Irreversible fouling was studied using first deionized water as feed during about 200 minutes DCMD operation (period A). Then, the deionized water was replaced by oil-field produced water and change in permeate flux was measured during 330 minutes (period B). Subsequently, the membrane surface was cleaned using deionized water and the permeate flux was measured again using deionized water as feed (period C). Depending on the obtained results in this last step the type of fouling can be elucidated and decide therefore whether any chemical cleaning would be required or not. Figure 11 shows a slight gradual decrease of the permeate flux in period B occurred when the oil-field produced water was used as a feed partly due to fouling caused by salts scaling shown previously in Fig. 9 and discussed in Section 3.2. In this DCMD period, salt rejection was

higher than 99.9% and permeate had only 22 ppm TDS, which proved the absence of membrane wetting. The permeation of the fouled membrane could be recovered in period C by simple rinsing with deionized water. This indicates the absence of irreversible fouling. Therefore, it can be claimed that DCMD is a promising process for the treatment of produced water with a good performance that can be recovered by a simple physical washing with deionized water.

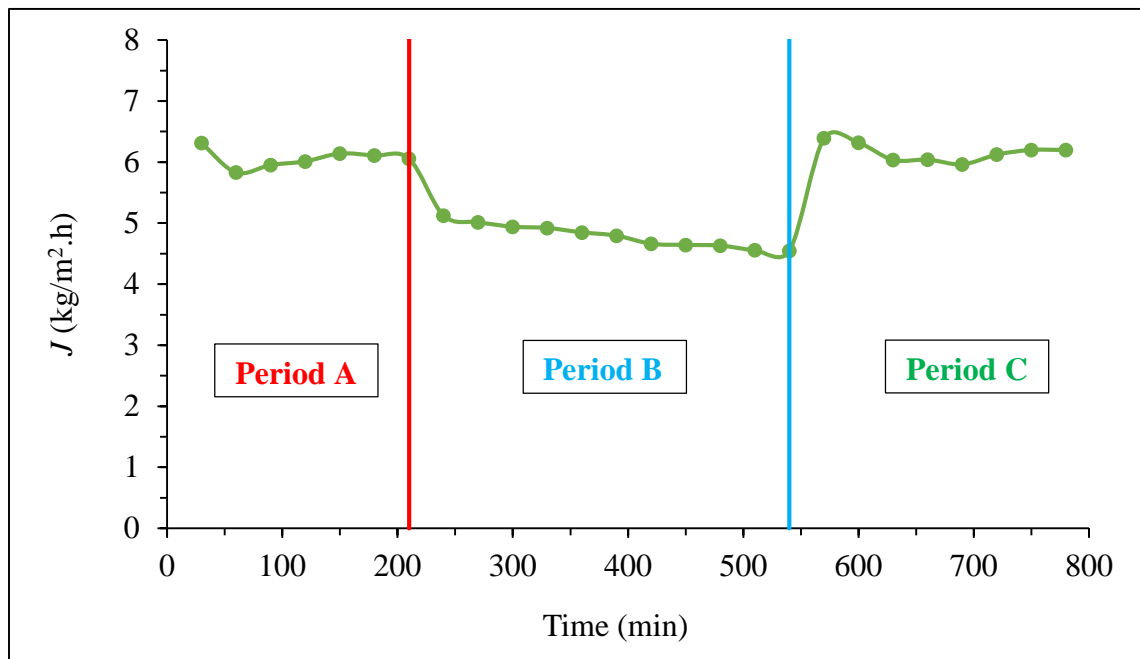


Figure 11. Efficiency of cleaning the fouled membrane with de-ionized water on the variation of the DCMD permeate flux (J)

4. Conclusions

Membrane distillation (MD) technology has been considered in a wide range of applications. However, it has been applied for the treatment of oil-field produced water in only very few studies and in most of them simulated oil-field produced waters have been

used. This study investigated the potential of DCMD in treating real produced water from an Omani oil-field.

Results show that DCMD has a great potential in treating oil-field produced water. Greater than 99.9% and 93.3% rejection was achieved for NaCl and TOC, respectively. The possible reason behind the higher passage of TOC in comparison to NaCl is the evaporation and the subsequent condensation of volatile crude oil derivatives (VOCs). In addition, no membrane pore wetting was detected.

Longer-term test showed DCMD performance stability and minimal reversible fouling cause by salts scaling of the salts present in oil-field produced water. A simple washing by deionized water can recover the initial permeate flux of the membrane. These indicated the high potential of MD for oil-field produced water treatment.

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