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Application of Membrane Distillation for Secondary Effluent Treatment towards Water Recovery

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ABSTRACT

This paper presented the application of Direct Contact Membrane Distillation (DCMD) for the treatment of the secondary effluent of a municipal wastewater treatment plant (WWTP) to produce fresh water. The purification studies conducted at various feed temperatures demonstrated that the permeate water flux increased and that the water flux decreased quickly at the higher feed temperature. However, the electrical conductivity of permeate remained consistent at about 2.0 µS/cm. The majority of pollutants found in the secondary effluent, including SS, COD, nitrate, nitrite, phosphate, and total coliform, were entirely eliminated throughout the MD treatment process using a bi-composite membrane made of polytetrafluoroethylene and polypropylene at different feed stream temperatures. Ammonia had a limited rate of rejection, though. Protein and organic/inorganic aggregates made up the majority of the foulants were found on the membrane surface, not the inner pores. The long-term test, which involved an 18-day operation with a feed solution concentration of ten times, revealed that no wetness issue was seen despite a notable foulant deposit and reduction in water flux.

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1. Introduction

Fresh water is becoming more scare due to urbanization and population growth, which has increased the need to discover new, reliable freshwater sources. Currently, the majority of treated municipal wastewater amount is dumped straight into receiving aquatic bodies, wasting precious freshwater resources [1]. Recycling and reusing of municipal wastewater is one of the options for the water supply resource. This kind of water can be used for many non-potable applications, including surface water replenishment, industrial use, urban greening, agricultural irrigation, and even household use [2], [3].

As an emerging technique for desalination, membrane distillation (MD), a thermally driven membrane process, has drawn a lot of attention. It is especially well-suited for treating hypersaline solutions, such as brine from seawater reverse osmosis (SWRO) [4], [5]. The vapor pressure differential in the MD process propels the movement of volatile substances from the feed side to the permeate side while a hydrophobic porous membrane serves as a physical barrier between the hot feed and the permeate stream. Since MD does not require trans-membrane pressure, the process is not affected by feed concentration. Furthermore, MD can use low-grade or renewable thermal energy and operates at low temperatures below the boiling point of the feed water to generate the water vapor [6]-[8]. The fouling resistance and pollutant selectivity of the MD process make it potentially very beneficial for the treatment of wastewater. Some nations, including as Singapore, treated municipal wastewater as a valuable source of water to produce the high-grade reclaimed water, or NEWater, which is used for indirect portable uses and industrial applications [9]. Despite the enormous potential benefits for wastewater treatment, relatively few pilot plants were used for the MD process's practical use in the treatment of petrochemical, oil production, and gas refinery wastewater, as well as seawater desalination and fruit juice concentration [10]-[13]. Many earlier studies have noted that one of the main barriers to the practical application of MD systems is their lower energy efficiency compared to the current membrane process (i.e., RO). This is because MD systems based on reusable or waste heat, such as solar and geo-thermal energy, which can improve the capabilities of MD technology, have received the most attention [14]-[16].

There have been a lot of studies recently on the use of MD to treat the feed water samples containing different specific organic and inorganic chemicals that have been spiked, including humic acids, polysaccharides, proteins, NaCl, and $CaSO_4$. Srisurichanet et al. investigated humic acid, one of the main organic components in wastewater that caused fouling in MD [17]. M. Gryta et al. concentrated NaCl solution including natural organic matter using MD studies using polypropylene capillary membranes. It was stated that the fouling of MD membranes was induced by organic substances present in the feed [18]. Another research by M. Gryta indicated that water flux and feed solution temperature may have an impact on calcium sulfate scaling propensity in membrane distillation [19]. However no information is available in the literature on the specific municipal secondary wastewater treatment by MD using the superhydrophobic composite membranes. In addition, no studies explored the application of real wastewater as a feed for long-term MD operation.

This research investigated the effects of feed temperature and long-term operation on the water flux, water quality, and membrane fouling on the MD performances using the superhydrophobic-composite membrane made of polytetrafluoroethylene and polypropylene for the treatment of the municipal wastewater treatment plant (WWTP) secondary effluent. SEM-EDX (scanning electron microscopy-energy dispersive spectrometry), XRD (X-ray diffraction), and FTIR (fourier transform infrared) analyses were used for morphological and mineralogical characterization of the foulants on the membrane. This study would provide us meaningful information on the MD application for the reclamation of municipal WWTP secondary effluent.

2. Materials and Methods

2.1. Tested feed water

Municipal wastewater samples used throughout this study were collected from a secondary clarifier at a local municipal WWTP in Ho Chi Minh City. The obtained samples were immediately carried out with basic characterizations and stored at 4 °C for all MD experiments.

2.2. Membrane specification

Pall Corp. (USA) provided the composite membrane consisted of a polytetrafluoroethylene (PTFE) active layer and a polypropylene (PP) support layer for all experiments. Table 1 displays the membrane's primary characteristics.

Table 1. *Membrane specifications*

Characteristics	Values	
	Active layer	**Support layer**
Nominal pore size, μm	0.45	
Thickness, μm	36	53
Contact angle	140^o	118^o
Porosity	72.6 %	67.2 %

2.3. DCMD lab-scale setup and experimental methodology

The DCMD module was made from transparent acryl for visual observation. A flat sheet membrane coupon with dimensions of 0.029 × 0.079 m (length × depth) was placed in the middle of the cell. A schematic of the DCMD configuration is shown in Figure 1, which has the following components: a 6 L acrylic feed tank, a 6 L acrylic permeate tank, two gear pumps (75211-15, Cole-Parmer Instrument Company, USA), a heater and a chiller (Samheung Scientific, Korea). An analytical balance (CUX6200H, CAS corporation, Korea) was used to balance the permeate tank and quantify the continuously rising water weight. Water flux was calculated using permeate mass information. The produced water volume (L), operation time (h), and the membrane area (m^2) were used to compute water flux (J) in L m^{-2} h^{-1} (LMH).

The electrical conductivity of the feed and permeate streams were measured by an electronic conductivity meter (HQ40d, Hach, USA) connected with a probe (CDC40101, Hach, USA). The pressure and flow rate of the flow were continuously observed by using pressure gauges and flow meter. The flow rates were set at 1.6 and 1.2 L/min at the feed and permeate sides, respectively for all experiments.

For the experiments at various feed temperatures, 5 L of feed solution (real secondary effluents) and 1L of permeate solution (deionized water) were placed in 6 L tank, respectively (deionized water). Each batch underwent a 24 h test, after that water from the permeate tank was collected and examined the water's quality. In the long-term experiment, a 22 L tank holding 20 L of feed solution was used, and the permeate was extracted for testing the water's quality. The experiment was run continuously. Every time the MD system produced one liter of permeate, samples were taken. The feed was concentrated from 20 to 2 L and a total of 18 times of permeate were taken.

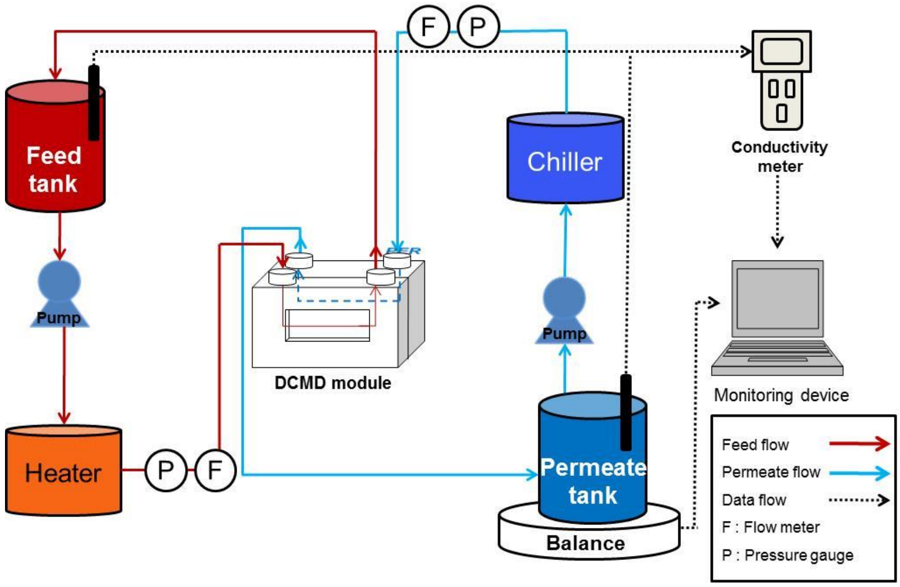

Figure 1. *The diagram of DCMD setup*

2.4. Analytical techniques for water quality analysis

Water samples underwent several analyses to determine physicochemical parameters such as pH, electrical conductivity, nitrate, nitrite, ammonia, phosphate, SS (suspended solid), COD, total coliform, and turbidity. A pH meter (AB15 Plus, Fisher Scientific) and a conductivity meter (HQ40d, Hach, USA) fitted with a probe (CDC40101, Hach, USA) were used to test the pH and conductivity, respectively. Nitrate and nitrite were analyzed by using IC (Ion chromatography). Hach Test Kits (Hach, USA) were used for ammonia, phosphate, and COD (21259-15) measurements. Turbidity was measured by a turbidity meter (Orion AQUAfast AQ45, Thermo Scientific Orion). Suspended solid (SS) and total coliform were measured following the Standard Methods for the Examination of Water and Wastewater [20].

2.5. Characterization of membranes

The fouled membranes, which were dried overnight at 50 ℃ in an oven, and the virgin membranes were investigated by various means of characterization. FE-SEM (field emission scanning electron microscope, S-4100, Hitachi, Japan) was used to characterize morphology of surface and cross section of membranes. Elemental analysis (EDX) was also conducted to investigate elements of membrane foulants. For the SEM-EDX analysis of the cross section of membrane, SEM image of cross section of fouled membrane were divided to 4 layers according to depth in which each layer has thickness of 8 µm and investigation of elements in each layer was conducted by EDX analysis. The analysis was operated at an acceleration voltage of 100 kV.

For XRD analysis, an X-ray diffractometer (Rigaku D/Max-IIA, Japan) equipped with Cu Kα radiation was utilized. At 50 kVp and 50 mV, continuous scans from 10° to 80° 2θ were gathered at a scan rate of 2° 2θ min^{-1}. The Joint Committee on Powder Diffraction Standards (JCPDS) cards were used to compare the obtained peaks and intensities of each mineral with the reference values.

For FTIR, an FTIR spectrometer (FTIR, Shimadzu IR Prestige-21) using the Attenuated Total Reflection (ATR) equipped with ZnSe crystal method was used to perform the scans at a resolution of 4 cm^{-1} at room temperature.

3. Results and Discussion

3.1. MD treatment of secondary MWWPT effluents at various feed temperatures

The secondary effluent was treated at feed temperatures of 40 ℃, 60 ℃ and 80 ℃ respectively, and at 20 ℃ of permeate side to find out the optimized feed temperature for MD operation in terms of water flux, quality, and membrane fouling.

Figure 2 shows water flux changes at various feed temperatures for 24 h MD operation of secondary effluent. Higher water fluxes decreased by 3.8 % (to 23.3 LMH), 14.4 % (to 46.3 LMH) and 70.6 % (to 29.5 LMH) respectively after 24 h of MD operation, whereas the permeate conductivity remains stable at 2.0 µS/cm for all runs. The high water flux at 60 ℃ and 80 ℃ could be a great advantage for the application of the MD process in treating WWTP effluents for water recovery purposes. However, the feed temperature at 80 ℃ led to the rapid decrease of water flux. This behavior was due to the thermodynamic effect of the temperature difference, vapor pressure difference and also the effect of feed concentration [4], [5]. The result suggested that the feed temperature should be controlled to less than 80 ℃ for suitable MD operation. Based on this investigation, the long-term experiment was performed at 60 ℃.

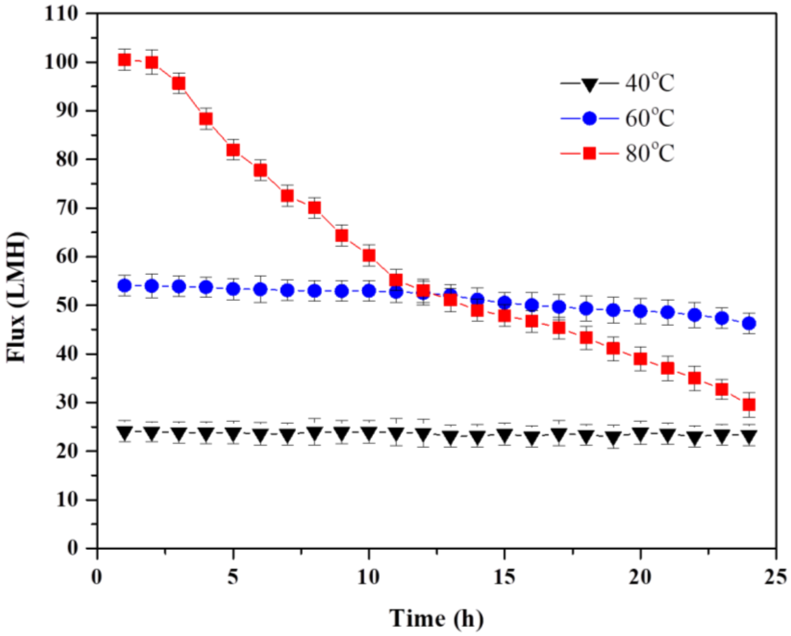

Figure 2. T*he permeate fluxes as a function of time at various feed temperatures*

In the results of the water quality, SS, nitrite, nitrate, and phosphate were detected below the detection limit in all permeates and generally lower rejection rates were observed at higher feed temperature for the ammonia and COD (Table 2). In the case of ammonia, rejection rates less than 40 % were observed at 60 ℃ and 80 ℃. The pH also increased at high feed temperatures due to the more penetration and dissolution of the ammonia in the permeate. As shown in Table 2, observed particulate and ionic contaminants in the feed stream such as SS (5.5 mg/L), nitrate (9.0 mg/L), nitrite (0.7 mg/L), phosphate

(2.4 mg/L) were completely removed after 24 h of MD treatment. The higher concentration of ammonia was detected in permeate with higher feed temperatures and pH also increased with feed temperature due to the more penetration and dissolution of the ammonia in permeate [21]. Concentrations of COD at 80 ℃ permeate were 0.5 mg/L (removal rate: 99.5 %), while no COD was detected in the permeate streams at the temperatures 60 ℃ and 40 ℃. It is believed to be due to the more permeation of volatile organic compounds at higher temperatures as presented in previous research [21]-[23]. No total coliforms were detected in all collected permeate streams at different operating temperatures.

Table 2. *Water quality of secondary MWWTP effluents treated by MD at various feed temperatures*

Parameters	**Feed**	**Permeate**					
		40 ºC		**60 ºC**		**80 ºC**	
		Value	Removal (%)	Value	Removal (%)	Value	Removal (%)
pH	7.24	7.26	-	7.6	-	8.13	-
COD (mg/L)	25.5	ND	100	ND	100	0.5	99.5
SS (mg/L)	5.5	ND	100	ND	100	ND	100
NO_3^- (mg/L)	9.0	ND	100	ND	100	ND	100
NO_2^- (mg/L)	0.7	ND	100	ND	100	ND	100
NH_4^+ (mg/L)	1.04	ND	100	0.63	39.4	0.66	36.5
T-N (mg/L)	11.14	ND	100	0.66	94.1	0.72	93.5
Phosphate (mg/L)	2.4	ND	100	ND	100	ND	100
Total Coliform (MPN/100ml)	1500	ND	100	ND	100	ND	100

Detection limits for COD, SS, nitrate, nitrite, and phosphate were 0.3, 0.1, 0.01, 0.001, and 0.05 mg/L, respectively.

3.2. Characterization of the fouled membranes

The transmembrane flux of the MD operation is one of the most important factors for the practical application of the MD process and is closely related to the fouling of the membrane over operating time. As shown in Figure. 3, more severe depositions of the brown-colored foulants were observed at higher feed temperatures. Regardless of the temperature, however, no crystalline material was detected from the SEM images and XRD results. According to element analysis using SEM-EDX, carbon and oxygen were the predominant elements in the foulants. The weight percent of the carbon increased from 26.0 to 48.9 % and that of the oxygen was sustained at around 38 % with increasing the feed temperature from 40 to 80 ℃. Generally, it was reported that microbial activity was inhibited at feed temperatures above 60 ℃ [24], [25], so more severe fouling at higher temperatures, in this study, could be due to organic substances such as humic acids, proteins, and polysaccharides as reported in previous research [17], [18], [26].

In the FTIR analysis, adsorption bands at 1020, 1540, 1650, and 3340 cm^{-1} were newly detected and increased with increasing feed temperature (Figure. 4). The peaks at 1540, 1650, and 3340 cm^{-1} indicate the deposition of proteins [18] and the peak at 1020 cm^{-1} means covalent bonds including oxygen, carbon and inorganic elements such as Ca, Si, and Mg. Furthermore, no detection of the peak of the carboxyl group which is one of the representative functional groups in humic acid, revealed that humic acid has a negligible effect on the fouling in this study.

For the inorganic materials, calcium and magnesium were major elements and the weight percentages of Ca and Mg decreased from 18.0 to 9.1 % and 11.5 to 3.2 %, respectively, with increasing feed temperatures from 40 to 80 ℃. Other minor elements such as P, Si, S, and Na were also detected with a weight percentage of less than 1 % indicating that the decrease in water flux with increasing feed temperature was due mainly to the proteins and organic/inorganic aggregates on the surface of the membrane. This result is similar to previous research in the literature [1], [4], [5], [27].

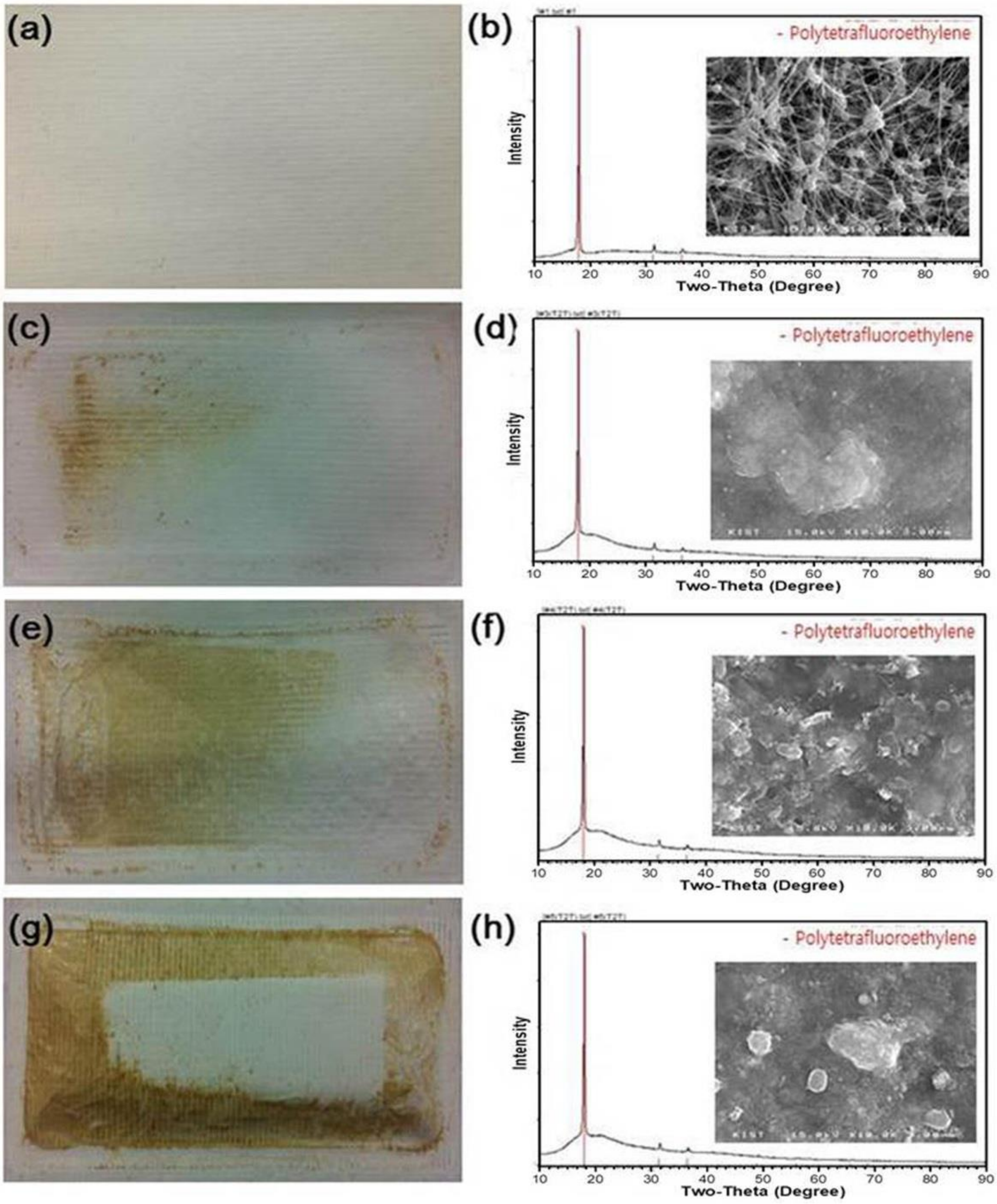

Figure 3. *Surface, XRD, and SEM results of membranes after MD treatment of secondary effluent at various feed temperatures ((a), (b): virgin membrane, (c), (d): 40 ℃, (e), (f): 60 ℃, (g), (h): 80 ℃).*

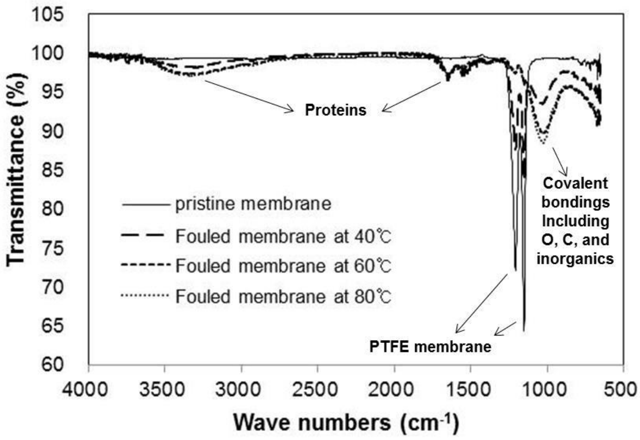

Figure 4. *FTIR results of fouled membrane operated at various feed temperatures*.

For further characterization of the membrane fouling, an investigation of the inner pores of the fouled membrane was performed by SEM-EDX analysis. SEM images and weight percentages of elements (EDX) according to the depth of the membrane cross section are indicated in Figure. 5. It showed SEM-EDX results of the virgin membrane (Figure. 5(a), (b)) and fouled membrane operated at 60 (Figure. 5(c), (d)) and 80 ℃ (Figure. 5(e), (f)). For the virgin membrane, only F and C which are original components of the PTFE membrane itself were detected with small amounts of oxygen (less than 2 %). In the case of the fouled membranes operated at 60 and 80 ℃, the inorganic elements such as Ca and Mg were detected only on the surface of the membrane cross section (top layer) and no inorganic elements were found in the inner layers of the membrane. Relatively high percentages (5~10 %) of oxygen were observed from the inner layers operated at 60 ℃, indicating that no penetration of inorganic components into the inner pores and the available contaminants in the permeate may be caused by the migration of organic substances during the MD operation at high temperature [5], [28].

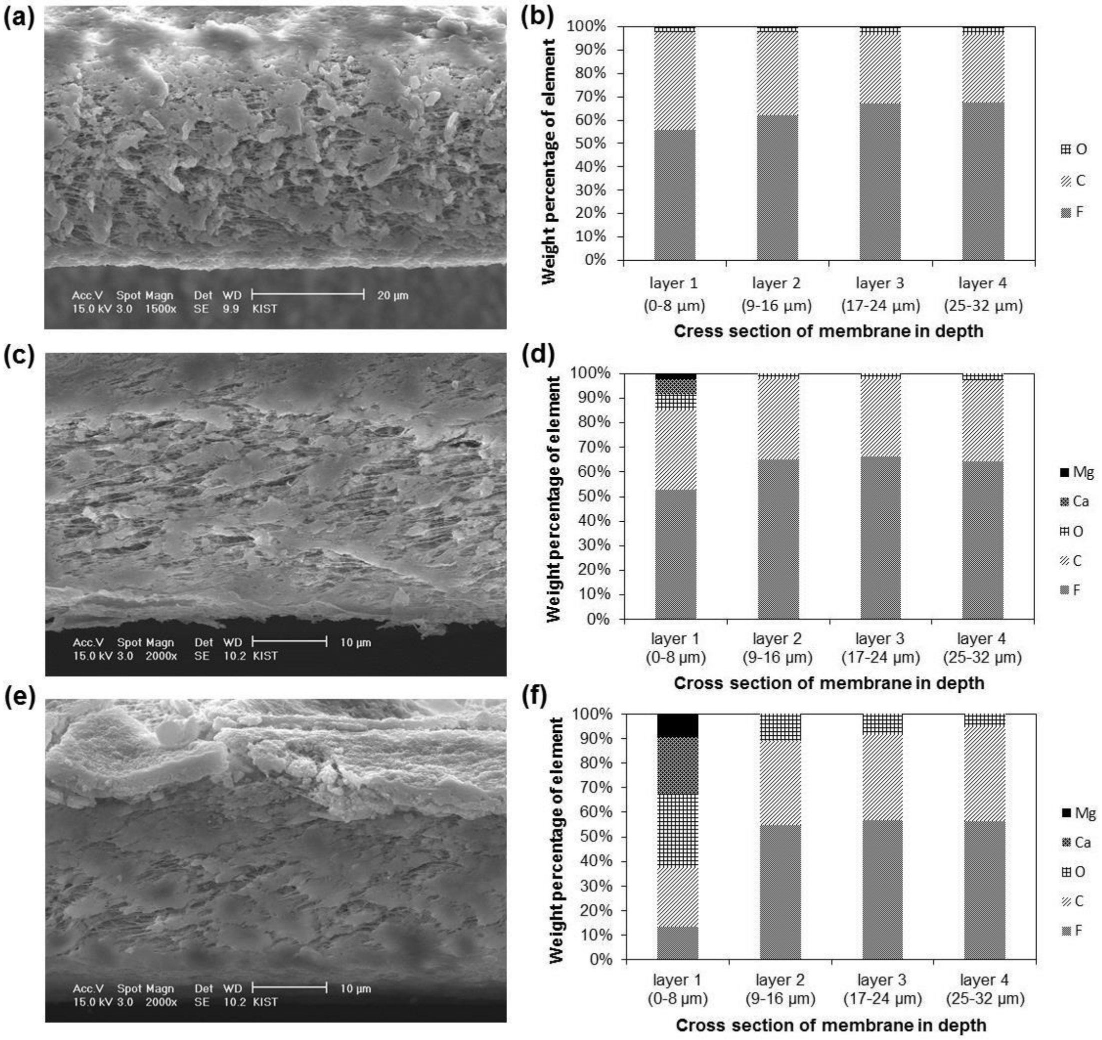

Figure 5. *Element distribution in cross section of membranes ((a), (b) virgin membrane, (c), (d) 2nd effluent treated by MD at 60 ℃, (e), (f) 2nd effluent treated by MD at 80 ℃)*

3.3. Long-term operation

A long-term MD operation with 20 L of secondary effluent without any pretreatment was conducted at the feed temperature of 60 ^{o}C to investigate the changes in water flux and quality in the permeate according to the fouling of the membranes. As shown in Figure. 6, the water flux decreased continuously from 52 to 10 LMH for 18 days of operation and 20 L of the initial feed solution was concentrated to 2 L. The electrical conductivity of permeate is stable at around 2.0 µS/cm indicating that no membrane wetting occurred during the long-term operation of MD.

For the water quality analyses, no COD, SS, nitrate, nitrite, and phosphate were detected from any permeate samples during the operation, and pH was maintained around 7.0 (Table 3). In the case of ammonia, concentrations in the first and second permeate samples were relatively high (0.72 and 0.7 mg/L) and ammonia concentrations in later samples were sustained at around 0.6 mg/L.

After at 10 times the concentration of feed solution, representative crystallized foulants (i.e. $CaCO_3$ and $CaSO_4$) that can cause scaling [29], [30] were not found. This is because the concentration of inorganic elements in the effluent was very low. During the long-term MD operation for 18 days, a sudden increase in the water flux and/or concentrations of contaminants in permeate which are the direct evidence for membrane wetting [27] were not found. The findings showed that when the foulants deposited on the membrane surface were appropriately regulated, long-term, stable operation free from wetting issues could be accomplished.

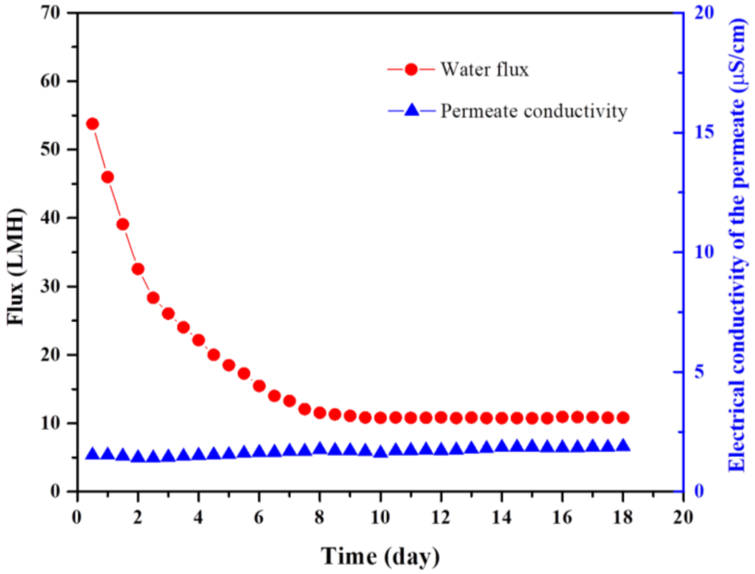

Figure 6. *Change in flux and electrical conductivity of the permeate in long-term MD experiment*

Table 3. *Water quality result of long-term MD operation in treating 2nd effluent*

Parameters	Feed	Permeate							
		1st	2nd	3rd	5th	7th	10th	14th	18th
pH	7.5	7.0	7.0	7.0	7.1	7.2	7.2	7.4	7.3
COD (mg/L)	23	ND	ND	ND	ND	ND	ND	ND	ND
SS (mg/L)	17	ND	ND	ND	ND	ND	ND	ND	ND
NO_3^- (mg/L)	10.2	ND	ND	ND	ND	ND	ND	ND	ND
NO_2^- (mg/L)	0.06	ND	ND	ND	ND	ND	ND	ND	ND
NH_4^+ (mg/L)	1.12	0.72 (35.7)	0.7 (37.5)	0.59 (47.3)	0.6 (46.4)	0.61 (45.5)	0.61 (45.5)	0.62 (44.6)	0.61 (45.5)
T-N (mg/L)	11.8	0.72 (93.9)	0.7 (94.1)	0.59 (95.2)	0.6 (94.9)	0.61 (94.8)	0.61 (94.8)	0.62 (94.7)	0.61 (94.8)
T-P (mg/L)	1.7	ND	ND	ND	ND	ND	ND	ND	ND

Detection limits for COD, SS, nitrate, nitrite, and phosphate were 0.3, 0.1, 0.01, 0.001, and 0.05 mg/L, respectively. Numbers in parenthesis indicate rejection rates (%).

4. Conclusions

This study suggested applying membrane distillation technology for further treatment of the secondary effluent to produce water with high quality for water reclamation purposes.

The permeate flux decreased more sharply at the higher feed temperature as the secondary effluent was purified by the DCMD process. The partial removal of certain pollutants at a feed temperature of 80 ℃ implied that the temperature should be controlled to a value lower than 80 ℃.

The organic and inorganic foulants attached on the membrane surface caused the reduction in flux permeate during the performance procedure, however, no evidences of membrane wetting was found.

Future research should examine the impact of membrane material on the performance of the DCMD for the treatment of secondary effluent at various concentration factors to determine the suitability of membrane material for such treatment settings. Significant amount of foulants accumulated on the membrane surface can lead to a barrier in particular applications of MD, therefore, additional pretreatments and strategies to mitigate the membrane fouling should be considered in future applications of MD for municipal wastewater secondary effluent treatment.

Conflict of Interest

The author declares no conflict of interest.

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