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Evaluation of membrane-based desalting processes for RO brine treatment

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ABSTRACT

Membrane-based desalting processes including reverse osmosis (RO), forward osmosis (FO), and membrane distillation (MD) were systematically evaluated for concentrating RO brine. Basic characteristics of membrane processes were first examined. Commercial polyamide RO exhibited higher water and lower salt permeability coefficients than cellulose FO membrane. However, salt rejection by FO seemed to be higher than RO primarily due to the hindrance of reverse draw solute flux. The water flux of MD comparable to RO was obtained when temperature gradient was more than 20–30˚C. The applicability of RO, FO, and MD was further tested with real brine obtained from full-scale RO plant processing brackish water. Results demonstrated that water flux was not significantly reduced in MD, while severe flux decline was observed in both RO and FO at high recovery. To elucidate major causes of different flux behaviors, the fouled membrane surfaces were analyzed by scanning electron microscopy with energy dispersive X-ray spectroscopy and X-ray diffraction. Foulant analysis suggested that $CaCO_3$ scaling occurred particularly at high water recovery, which was in good agreement with water quality simulation. $CaCO_3$ scaling, however, had only small impact on flux behavior in MD. From these findings, MD could be suggested as the best option for concentrating industrial RO brine if low-grade heat (below 50–70˚C) is available.

Keywords: Reverse osmosis; RO brine; Forward osmosis; Membrane distillation; Membrane fouling

1. Introduction

Reverse osmosis (RO) membrane has been utilized in a wide range of applications including drinking and industrial water production as well as seawater and brackish water desalination [1–4]. The advantages of RO process are low energy requirement, easy operation, modular design, and small footprint demand [5,6]. However, RO process also suffers from some critical problems. RO brine is one of them since it could have serious detrimental impact on aquatic environments. Due to constituents of RO brine, eutrophication, pH variation, and accumulation of heavy metals can occur in receiving waters [7]. In order to decrease RO brine disposal and increase

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water recovery, 2nd stage RO process has been usually employed. However, since highly concentrated RO brine imposes high fouling potential and thus requires high energy for operation, 2nd stage RO process needs to be replaced by another desalting process for controlling fouling and reducing energy cost.

Recently, forward osmosis (FO) process is being considered as an alternative for a low energy, low fouling, and high recovery process to conventional RO process. In FO process, a highly concentrated draw solution is utilized to induce the driving force of separation and then is recovered at significantly reduced energy cost and fouling level [8–10]. However, FO process has many obstacles to be overcome. For instance, internal concentration polarization (ICP) should be reduced by developing more efficient FO membrane to improve water productivity [11–14]. For the reconstitution of draw solution, FO process is often integrated with a variety of separation processes such as membrane distillation (MD) [15–17]. Novel draw solutes have been proposed including magnetic nanoparticles for efficient recovery of draw solutes [18].

MD process is another candidate for RO brine treatment. This process is operated by temperature gradient between feed and permeate solutions which induces vapor pressure difference as a driving force across the hydrophobic membrane [19,20]. Since only water molecules in vapor phase can pass through the membrane pore, nonvolatile compounds (e.g., ionic compounds) can be completely removed [21]. MD process, however, has various drawbacks which include high energy consumption, low rejection of volatile compounds and wetting phenomenon [22,23]. Nevertheless, if low-grade heat from power-generating and industrial manufacturing plants is available, MD can be an excellent option for the treatment of industrial RO brine since MD process can be operated at relatively low temperature (below 50–70˚C).

Membrane-based desalting processes including FO and MD processes have been investigated for treating RO brine. It has been suggested that concentrating brine could be potentially achieved by FO process [24]. Fouling behavior under various operating conditions (i.e. temperature, pressure, and feed concentration) was studied for FO concentrating RO brine [25]. Martinetti et al. [26] also reported that fouling of MD and FO processes were significantly affected by RO brine properties. Besides membrane processes, advanced oxidation processes have been proposed for RO brine treatment, however, their applications were limited only to laboratory-scale [27]. Despite the recent efforts, systematic comparison of membrane-based desalting processes has not been performed, particularly for treating RO brine from real-scale industrial processes.

The main objective of this study was to evaluate membrane-based desalting processes for concentrating RO brine in an industrial RO plant. Basic characteristics of membrane processes were first examined, and then actual RO brine was treated by three different membrane processes (i.e. RO, FO, and MD) to select the optimum membrane process by evaluating maximum recovery rates. Lastly, major causes of severe flux decline observed at high recovery were elucidated by analyzing the fouled membrane surfaces using scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX) and X-ray diffraction (XRD).

2. Materials and methods

2.1. RO brine

Actual RO brine used for feed solution was obtained from an industrial RO plant in Korea which desalinates brackish water. The components of RO brine were measured by inductively coupled plasma ICP and titration methods according to American Society of Testing Materials (ASTM). The results of water analysis were summarized in Table 1.

2.2. RO experiments

The brackish RO membrane, SWC5 (Hydranautics, CA, USA), was used for treating RO brine. This membrane was thin film composite (TFC) polyamide membranes. For storage, membranes were immersed in deionized (DI) water at 4˚C and water was replaced regularly. Prior to each analysis, the membranes were put into 20–25˚C DI water for 2 h as all analyses in this study were carried out at room temperature. A

Table 1
Primary water quality of RO brine obtained from industrial brackish water treatment. Concentration of each component was determined by ICP and titration methods according to ASTM

Water quality parameter	Concentration
Turbidity (NTU)	5.05
TOC (mg/L)	14.68
Hardness (ppm $CaCO_3$)	434.5
Ca (ppm $CaCO_3$)	311.2
Mg (ppm $CaCO_3$)	123.3
Na (mg/L)	141.5
Cl (mg/L)	305
SO_4 (mg/L)	360
K (mg/L)	36.5
SiO_2 (mg/L)	28.8

laboratory-scale cross-flow RO membrane test unit, similar to that described in other publications [28,29], was employed for the fouling experiments. The rectangular plate-frame cell has dimensions of 14.6 cm length, 9.5 cm width, and 0.2 cm height. The system was operated in a closed-loop mode with only concentrate being recirculated into the feed water reservoir. Permeate flux was continuously monitored using a digital balance and recorded in real time with a laboratory computer. Operating conditions for fouling experiments were maximum recovery rate of 65%, initial flux of 20 LMH, operating pressure of 30 bar, and cross-flow velocity of 15.9 cm/s. The conductivity meter (HACH, Germany) and total organic carbon analyzer (TOC-V CPH, Shimadzu, Japan) were used to evaluate the rejection of ions and organics.

2.3. FO experiments

The FO membrane used in this study was provided by Hydration Technologies Innovations (Albany, OR, USA). This membrane is made of cellulose-based polymers with an embedded polyester mesh for mechanical strength. The total thickness of the membrane is approximately 50 μm. A detailed description of the structure and properties of the membrane can be found elsewhere [30]. The FO experiments were performed with the laboratory-scale FO system integrated with MD process for recovery of draw solution as depicted in Fig. 1. Detailed experimental descriptions of the cross-flow laboratory-scale FO systems used in the study are given elsewhere [8,17]. FO experiments were conducted in the FO mode in which the draw solution faced to the support layer, and the dilute feed solution faced to the active one. Since permeate flowed from the feed to the draw tank, feed solution was gradually concentrated while draw solution was progressively diluted. Therefore, MD process was employed after FO process for reconstituting draw solution. The FO cell had two symmetric channels on both sides of the membrane for co-current flows of feed and draw solutions. The cross-flow FO unit consisted of channels, 77 mm long, 26 mm wide, and 3 mm deep. Variable speed gear pumps (ISMATEC, Germany) were used to pump the liquids in a closed loop. Actual RO brine was used for feed solution and cross-flow velocity was set at 15.9 cm/s. Feed and permeate temperatures were set at 20 ± 0.5℃, and draw temperature was set at 50 ± 0.5℃. Three molar $MgCl_2$ was used as draw solution to produce the osmotic pressure driving force. The feed solution tank was placed on a digital scale, and the weight changes were measured by a computer in real time to determine permeate water flux.

2.4. MD experiments

MD membrane used in this study was Polytetrafluoroethylene (PTFE) membrane (FGLP, Millipore, Germany) with pore diameter of 0.22 μm. The MD

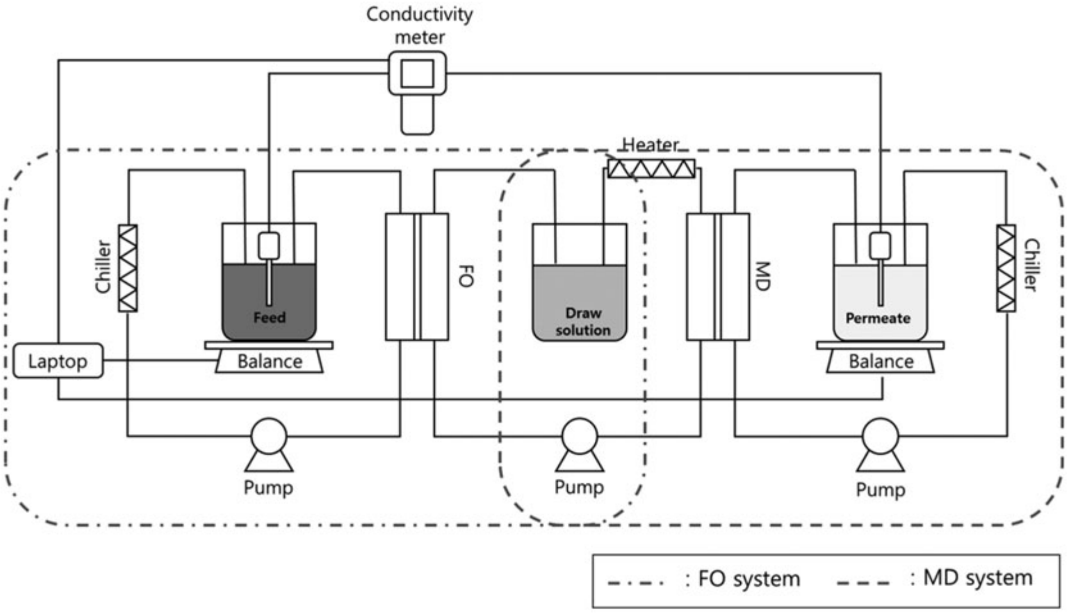

Fig. 1. Schematic description of FO system integrated with MD process for recovery of draw solution.

experiments were performed with the laboratory-scale direct contact membrane distillation (DCMD) system. Detailed experimental descriptions of the cross-flow laboratory-scale DCMD systems used in the study are given elsewhere [31]. The cross-flow MD unit consisted of channels, 77 mm long, 26 mm wide, and 3 mm deep. Variable speed gear pumps (ISMATEC, Germany) were used to pump the liquids in a closed loop. Actual RO brine was used for feed solution, and cross-flow velocity was set at 15.9 cm/s. Feed and permeate temperatures were set at 50 ± 0.5 and 20 ± 0.5˚C, respectively. The water productivity and selectivity were monitored as described in the RO and FO experiments.

2.5. *Surface analysis of fouled membrane by SEM-EDX and XRD*

For a surface analysis of fouled membranes, the membrane coupons were obtained after each experiment was finished, and dried in a desiccator. To investigate the surface image of fouled membranes, field emission scanning electron microscope (Hitachi S-4800, Japan) was utilized, and element analysis of foulants was carried out by EDX attached to SEM. For better understanding of fouling components, XRD analysis (Rigaku ATX-G, Japan) was also performed. Results of XRD analysis were compared with a library to find out which scaling appeared.

3. Results and discussion

3.1. *Basic performance evaluation of RO, FO, and MD membrane processes*

Prior to treating actual industrial RO brine, basic membrane properties were first evaluated in terms of water and salt permeability coefficients (i.e. A and B coefficients). The results are presented in Table 2 and showed that RO membrane had higher A coefficient than FO membrane, indicating that FO process presented lower water flux than RO process. Specifically, when water fluxes of RO and FO processes were measured with DI water as feed solution, water flux induced by NaCl 3 M (i.e. osmotic pressure 162 bar) in FO process was 19.01 ± 0.3 LMH, while RO process could exhibit the same flux at approximately 20 bar. This observation was expected since FO membrane used in this study was made of cellulose triacetate which normally displayed less productivity compared to TFC polyamide RO membrane.

Table 2
Comparison of FO and RO membrane properties

Membranes	Water permeability coefficient (A) (μm/s/bar)	Solute permeability coefficient (B) (μm/s)	Remarks
FO (HTI)	0.204	0.129	Cellulose triacetate
RO (SWC-5)	0.284	0.116	Thin film composite polyamide

In order to compare MD with other membrane processes, water flux was measured by increasing feed temperature from 30 to 80˚C with fixed permeate temperature of 20˚C. Results are presented in Fig. 2 and showed that water flux was exponentially increased with increasing temperature gradient, exhibiting similar tendency with partial vapor pressure difference [32]. It should be noted that water flux of 20–30 LMH could be obtained with 20–30˚C of temperature difference in MD process.

MD process also demonstrated great salt rejection close to 100% as shown in Fig. 3 because only vapor water molecules could pass through the membrane pores [19]. When comparing RO with FO, FO process exhibited higher salt rejection rate than RO process although RO membrane had lower B coefficient than FO membrane. This is probably due to the hindrance effect of reverse salt flux on forward salt flux. It has been reported that the transport of dissolved species from feed to draw is hindered by the reverse flow of draw solutes from draw to feed [33,34]. By inspecting basic membrane properties, it may be concluded that

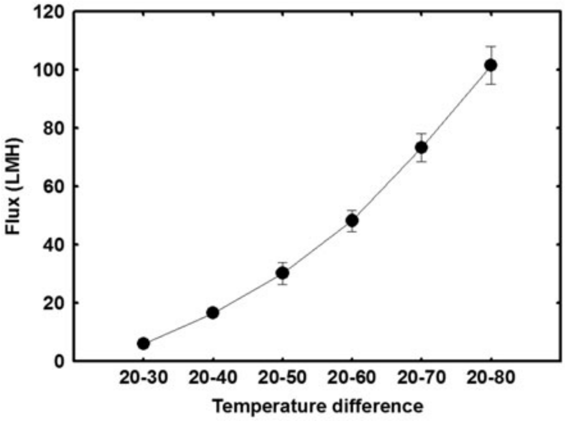

Fig. 2. Water flux in MD process with respect to temperature difference. Experimental conditions: feed and permeate solutions of DI water; cross-flow velocity of 15.9 cm/s; pH of 5.8; permeate temperature of 20˚C; and feed temperature ranging from 20 to 80˚C with an interval of 10˚C. Experiments were carried out 2 times, and the average value was reported.

MD process is the best option for treating RO brine in terms of water flux and rejection if low-grade heat is available to maintain temperature difference of 20–30°C.

3.2. Application of RO, FO, and MD processes for RO brine treatment

To evaluate the applicability of membrane-based desalting processes, RO brine was obtained from actual brackish RO plants producing industrial water and treated by RO, FO and MD membrane process under similar operating conditions, particularly identical initial flux (i.e. 20 LMH). Representative results were presented as a function of water recovery rate in Fig. 4. When treating RO brine by RO process, water flux slightly decreased at an initial stage; however, it started to decline significantly after reaching recovery rate of 40–45%. In case of FO process, water flux was rarely reduced at an early stage, however, it also started to decrease greatly at the recovery rate of 45–50%. The difference of fouling behaviors between RO and FO processes could be originated from the presence or absence of operating pressure, which means that pressurized conditions induced more severe fouling and lower fouling reversibility [35]. When treating RO brine by MD process, in contrast to RO and FO processes, severe flux decline was not observed even after 65% recovery. Therefore, it could be stated that MD process may be less affected by the formation of fouling layer, particularly scale formation at high recovery.

Water quality of RO brine (Table 1) was further analyzed to investigate major causes of severe flux decline at high recovery rate. The occurrence of scaling was expected due to high concentrations of divalent cations (i.e. Ca and Mg). To find out dominant scalants, saturation (%) with respect to water recovery rate was calculated using ROSA (Reverse Osmosis System Analysis) program which is a software to design and simulate RO process. Based on the ionic compositions of RO brine, SiO_2 and $CaSO_4$ were most likely scalants to be formed, however, the simulation results indicated that these scales would appear after the recovery of 75% as shown in Fig. 5. Langelier saturation index (LSI) with respect to recovery rate was also determined to evaluate the possibility of $CaCO_3$ scaling. As presented in Fig. 5, $CaCO_3$ scaling could possibly occur even at low recovery rate. However, severe flux decline in RO and FO processes was observed after 40% recovery. It was thought that the main reason of this disagreement was the inhibitory effect of magnesium ions on $CaCO_3$ scaling formation. Previous studies suggested that magnesium could be easily incorporated into $CaCO_3$ and thus disturb the growth of $CaCO_3$ crystals [36,37]. From these observations and speculations, it can be concluded that severe flux decline was greatly influenced by $CaCO_3$ scaling, however, it needs to be verified by analyzing foulants on the fouled membrane surfaces.

Lastly, to confirm whether the process can be used for RO brine treatment, rejection rates were evaluated in terms of inorganic ions and organics and are presented in Table 3. In RO process, ionic compounds and organic matters were rejected at 99.8 and 99.5%, respectively. Both ionic compounds and organic matters were perfectly removed when FO process was integrated with MD process for recovery of draw solution [38]. In MD process, ionic compounds and organic matters

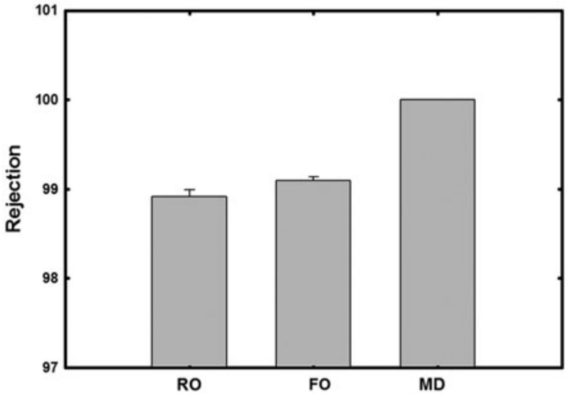

Fig. 3. Comparison of salt rejections in RO, FO, and MD processes. Experimental conditions: feed solution of NaCl 2,000 ppm; cross-flow velocity of 15.9 cm/s; pH of 5.8; and initial flux of approximately 9 LMH. Experiments were carried out 2 times and the average value was reported.

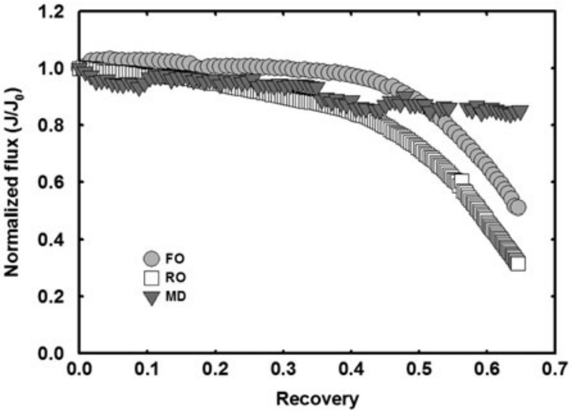

Fig. 4. Normalized water fluxes with respect to recovery rate in RO, FO, and MD processes. Experiments were conducted using actual RO brine (see Table 1). Initial fluxes of three processes were set at approximately 20 LMH for the same hydrodynamic condition.

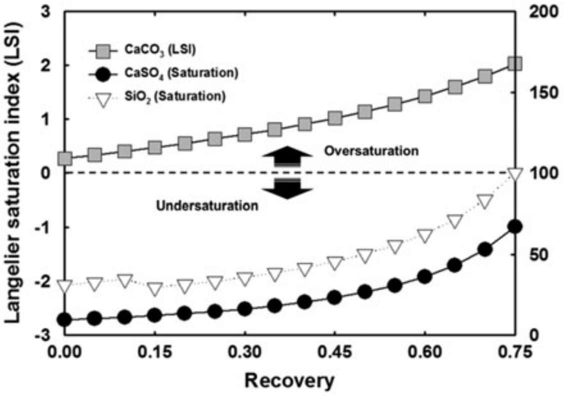

Fig. 5. Simulated LSI and saturation (%) of $CaSO_4$ and SiO_2 with respect to water recovery rate. LSI and saturation (%) were calculated by ROSA (Reverse Osmosis System Analysis) program.

were rejected 100 and 99.03%, respectively, implying that RO brine may contain some volatile organic compounds which were not entirely removed. Our experimental observation using actual RO brine demonstrated that MD process might be the best option for stable water productivity and good selectivity.

Table 3
Rejection rates in RO, FO, and MD processes when treating actual RO brine. FO process was integrated with MD process for recovery of draw solution

	RO (%)	FO with MD (%)	MD (%)
Conductivity	99.8	100	100
TOC	99.53	100	99.03

3.3. *Foulant analysis for the verification of scale formation*

In order to verify the proposed cause of severe flux decline (i.e. $CaCO_3$ scaling), the membrane surfaces of RO, FO, and MD processes were first analyzed by SEM-EDX. In RO process, fouled membrane coupons were obtained after recovery of 30 and 65% since fouling behaviors were differentiated with respect to recovery rate as shown in Fig. 4. At an initial period, a thin and irregular shaped organic fouling layer was observed on the membrane surface due to high level of TOC as shown in Table 1 (Fig. 6(a)). On the contrary, as shown in Fig. 6(b), a structured and crystal shaped fouling layer covered the membrane surface during high recovery operation. In order to ascertain components which covered the membrane surfaces, EDX analysis was conducted. Results indicated that carbon (C) and oxygen (O) were dominantly detected at the initial stage while calcium (Ca) was mainly observed at the latter stage. These results demonstrated that gradual flux decline at the initial stage was caused by deposited organic foulants and drastic flux decline after 45% recovery was originated from the scaling formation containing calcium. In case of FO process, fouled membrane coupons were collected after 65% recovery (Fig. 6(c)). Scales similar to crystals

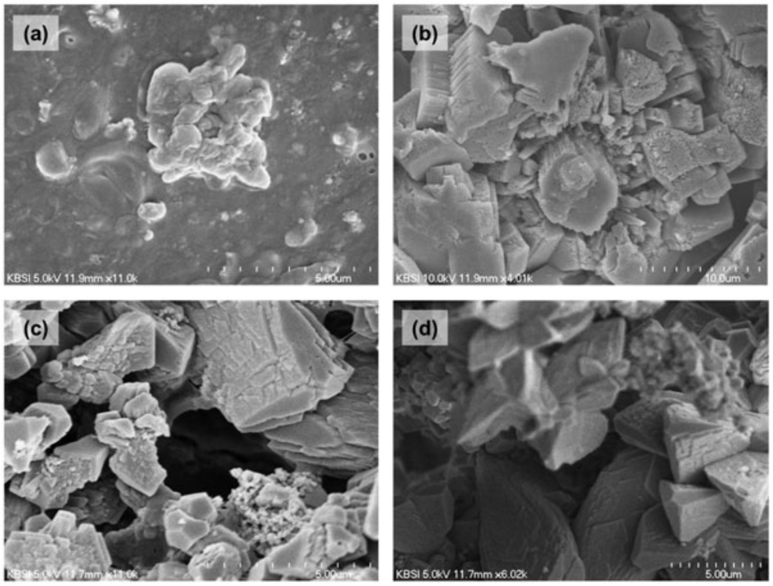

Fig. 6. SEM images of fouled membrane surfaces: (a) RO membrane at 30% recovery, (b) RO membrane at 65% recovery, (c) FO membrane at 65% recovery and (d) MD membrane at 65% recovery.

on RO membrane were observed through SEM images. From the literature, crystal structures on both RO and FO membrane surfaces seemed to be $CaCO_3$ scaling [39]. EDX results also suggested that these scales consisted of oxygen (O) and calcium (Ca) dominantly.

Similarly, significant fouling layer was also observed on the MD membrane surface despite less flux decline. Relatively large scales were found in the MD process compared with RO and FO (Fig. 6(d)). EDX results indicated that the fouling layer dominantly consisted of oxygen (O) and calcium (Ca) similar to those formed in other processes. The XRD analysis also confirmed the formation of $CaCO_3$ scaling on the MD membrane surface, as predicted by LSI calculation (Fig. 5). Interestingly, in only MD process, severe flux decline did not appear in spite of the fact that $CaCO_3$ scaling layer was formed. Similar observation was made in the recent study which reported that $CaCO_3$ scaling did not have a significant effect on permeate flux decline in MD process since wetting phenomenon was rarely found due to hydrophobic characteristic of $CaCO_3$ [40]. However, further research is needed to investigate the mechanisms of scaling formation with respect to various membrane processes.

4. Conclusions

In this study, various membrane-based desalting processes for the treatment of RO brine were evaluated by performing a series of basic performance and fouling experiments using actual RO brine. Major causes of flux decline were also investigated by foulant analysis using SEM-EDX and XRD. Primary findings drawn from this study are summarized briefly as follows:

RO membrane generally exhibited larger water transport coefficient and thus higher water flux than FO membrane. However, despite larger solute transport coefficient, FO membrane showed better salt rejection, probably resulting from hindering effect of reverse draw solute transport. MD membrane showed water fluxes comparable to RO with temperature gradients above 20–30˚C. When treating industrial brine from real brackish RO plant, water fluxes of both FO and RO processes were severely declined due to $CaCO_3$ scaling. In MD process, water flux declined much slower in spite of the occurrence of $CaCO_3$ scaling. Our experimental observations suggested that MD could be an excellent option for treating industrial RO brine if low-grade heat is accessible for driving MD process, although membrane wetting should be prevented in the long-term operation.

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