

Rejection of salt mixtures from high saline by nanofiltration membranes

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Abstract—Nanofiltration (NF) membranes have recently been employed as pretreatment unit operations in seawater desalination processes and as partial demineralization to seawater. The present paper investigates the performance of selected commercial NF membranes to reject salts of high concentrations at salinity levels representative of brackish and sea water. Two commercial nanofiltration membranes (NF90 and NF270) have been investigated in detail to study their performance in filtering aqueous solutions containing different salt mixtures in a cross-flow NF membrane process within the pressure range from 4 to 9 bar. Spiegler-Kedem model (SKM) was used to fit the experimental data of rejection with the permeate flux. The results showed that NF90 membrane was shown to have a distinct ability to reject both monovalent and divalent ions of all investigated mixtures with very reasonable values but with relatively low flux. This will make NF90 more suitable for the application in the pretreatment of desalination processes. On the other hand, NF270 can reject monovalent ions at relatively low values and divalent ions at reasonable values, but at very high permeate flux. The SKM model only fitted well the experimental data of divalent ions in salt mixture. Based on the evaluation of the overall performance of NF90 and NF270 membranes, their distinct ability to reject salts at high salinity from seawater is considered an advantage in the field of pretreatment of seawater feed to desalination units.

Key words: Membrane, Nanofiltration, Salt Rejection, Pretreatment, Spiegler-Kedem Model

INTRODUCTION

A nanofiltration (NF) membrane is a type of pressure-driven membrane that has properties in between those of ultrafiltration (UF) and reverse osmosis (RO) membranes. NF membranes extend the applications of membrane processes and are considered promising for many future applications, including water treatment, wastewater reuse [1], and desalination applications [2]. Moreover, NF membrane processes have many desirable features such as low operation pressure, high flux, high retention of multivalent anion salts and dissolved organic molecular compounds [3] with relatively low molecular weight ($200\text{--}1,000\text{ g mol}^{-1}$), relatively low investment, operation, and low maintenance costs. A recent comprehensive review on the use of NF membranes in water treatment has been presented elsewhere [4].

NF has recently been used in pre-treatment units in both RO and thermal processes [2,5]. This will treat most of the desalination problems such as scaling, fouling, high-energy requirements and the requirement of high quality construction materials. NF has also been used to filter seawater directly for partial demineralization applications where the RO process cannot be used to carry out that [6,7]. Two different NF membranes (NF70 and NF200) were also employed [7] to partially demineralize seawater by measuring the permeate salinity and the rejection of three cations of Na^+ , Ca^{+2} , and Mg^{+2} . In comparison between NF70 and NF200, it was seen that the ability of the latter membrane to reject calcium ions is higher than that of magnesium ions.

In our previous work [8-10], the filtration of the single seawater salts at different concentration levels was investigated by using three NF membranes (NF90, NF270, and N30F). It was seen that the rejection of all investigated salts was increased with increasing pressure and decreasing concentration. The SKM model was used to fit the experimental results of rejection *versus* permeate flux and to find the fitting parameters of reflection coefficient (σ) and solute permeability (P_s) for each salt and the corresponding membrane. The results showed that there was a good agreement between the theoretical and the experimental data of all investigated salts for all membranes except one case. This case was for the membranes which have low rejection and low flux. In another study, Hagemeyer and Gimbel [11] investigated the rejection of two salt mixtures, $\text{NaCl}/\text{Na}_2\text{SO}_4$ and $\text{NaCl}/\text{CaCl}_2$, at relatively low concentration. For the former mixture, the results showed ion rejection of SO_4^{2-} is higher than Cl^- while the rejection of Na^+ depended on the mole fraction of NaCl in the mixture. With increasing mole fraction of Na_2SO_4 the rejection of Na^+ increased from the rejection of the pure NaCl to the rejection of Na_2SO_4 .

In this work, the filtration of two salt mixtures at high salinity using only NF90 and NF270 membranes was investigated. Two different mixtures were used to prepare ions at high salinity similar to the ions found in real seawater. Furthermore, the effect of pressure on permeate flux and rejection for each ion in the investigated salt mixtures was also addressed. The SKM model was applied to the experimental data of rejection *versus* permeate flux for each ion in all salt mixture in order to check its validity to the experimental data of each ion in the investigated mixtures and to find the fitting parameters of reflection coefficient (σ) and solute permeability (P_s) for each ion.

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THEORY

The transport of the solute through ultrafiltration [12], nanofiltration [8], and reverse osmosis membranes can be described by irreversible thermodynamics where the membrane is considered as a black box. Kedem and Katchalsky [13] introduced the relation of the volumetric flux J_v and the solute flux J_s through a membrane in the following equations:

$$J_v = L_p(\Delta P - \sigma \Delta \pi) \quad (1)$$

$$J_s = P_s \Delta C_s + (1 - \sigma) C_s J_v \quad (2)$$

where σ , P_s and L_p are the reflection coefficient, solute permeability and pure water permeability, respectively. Eq. (2) shows that the solute flux is the sum of diffusive and convective terms. Solute transport by convection takes place because of an applied pressure gradient across the membrane. A concentration difference on both sides of the membrane causes diffusive transport. When high concentration differences between the reject and the permeate exist, Speigler and Kedem [14] used the above equations and obtained the following expression of the rejection rate of the solute related to permeation flux:

$$R = \sigma \frac{(1-F)}{(1-\sigma F)} \quad (3)$$

Where

$$F = \exp\left(-\frac{(1-\sigma)J_v}{P_s}\right) \quad (4)$$

where R is the rejection. According to Eq. (3), the rejection increases with increasing the water flux.

The parameters σ and P_s can be determined from the experimental data of rejection (R) as a function of volume flux (J_v) using best-fit method. The reflection coefficient (σ) is a parameter that measures the degree of semipermeability of the membrane reflecting its ability to pass solvent in preference to solute. When an osmotic difference ($\Delta \pi$) across an imperfectly semipermeable membrane is compensated by an applied pressure (ΔP) so that the volumetric flow is zero (ΔP is smaller than $\Delta \pi$), the ratio between the two is defined as σ , as shown in Eq. (5).

$$\sigma = \left(\frac{\Delta P}{\Delta \pi}\right)_{J_v=0} \quad (5)$$

A value of $\sigma=1$ means that the convection solute transport does not take place at all. This is the case for ideal RO membranes where the membranes have no pores available for the convective transport. In an entirely unselective membrane in which a concentration gradient does not cause volumetric flow at all, $\sigma=0$. While for the UF and NF membranes which have pores, the reflection coefficient will be $\sigma < 1$, especially if the solutes are small enough to the entire membrane pores under the convective transport effect [15].

Since the polarization concentration was neglected according to the experimental conditions, the rejection, R , was calculated by using the following equation:

$$R = 1 - \left(\frac{C_p}{C_f}\right) \quad (6)$$

Where C_p and C_f are permeate and feed concentrations (ppm), respectively. The pure water permeability, L_p ($L \cdot h^{-1} \cdot m^{-2} \cdot bar^{-1}$), was calculated as:

$$L_p = \frac{V_p}{t \cdot A \cdot P} \quad (7)$$

where V_p is the Volume of permeate (L), t is time (h), A is effective membrane area (m^2) and P is the applied pressure (bar).

EXPERIMENTAL

1. Set-up and Chemicals

The filtration experiments were performed in a laboratory scale test cell using cross flow module. The same rig used in the filtration of single salts as presented in previous papers was used here [8-10]. A circular disc membrane with an effective membrane area of 12.6 cm^2 was employed. Two NF membranes (NF90 and NF270), which were supplied by DOW chemical company (USA), were used in this study. Both membranes are made from polyamide. Atomic force microscopy (AFM) was used to characterize the NF membranes. High resolution images and surface characteristic details of NF90 and NF270 membranes have been published by elsewhere [16]. Table 1 shows the pore size, pore size distribution, surface roughness (RMS), and the porosity obtained from AFM images.

The deionized water used for preparation of the salt solutions was obtained through demineralization using ion exchange followed by reverse osmosis. The conductivity of the water was lower than $1 \mu\text{S/cm}$. All salts which were used to prepare salt mixtures were obtained from Fisher Scientific-UK with purity higher than 99.5%. In this study, two different salt mixtures were prepared and filtered at concentration similar to seawater. Table 2 shows the concentration of anions and cations of the salt mixtures. Mixtures I and II contain the main anions and cations in seawater which have the highest concentration in the seawater. It was reported [13] that the most salinity (around 86%) of the seawater comes from two ions (Na^+ and Cl^-). Therefore, both investigated mixtures contain NaCl salt at relatively high concentration as a basic compound. Mixture I (NaCl and Na_2SO_4) contains three main ions (Na^+ , Cl^- , and SO_4^{2-}) found in the seawater at high concentrations nearly similar to their con-

Table 1. AFM Surface characteristics of the investigated membranes NF90 and NF270 [16]

Membrane	Pore size (nm)	RMS (nm)	%Porosity
NF90	0.55	27.75	17
NF270	0.71	3.68	16

Table 2. The ions concentration for the investigated salt mixture (Ion concentration in ppm)

Ion	Mixture I (NaCl+Na ₂ SO ₄) (ppm)	Mixture II (NaCl+MgCl ₂) (ppm)
Na ⁺	11460	11008
Cl ⁻	15194	18389
SO ₄ ²⁻	3379	-
Mg ⁺²	-	479
Total (ppm)	30033	29876

centrations in the seawater. Na_2SO_4 is replaced by MgCl_2 in mixture II which contains again three ions (Na^+ , Cl^- , and Mg^{+2}) at high concentration.

2. Filtration Procedure

In all filtration experiments, the membranes were immersed at least overnight in water before being used in any experimental work, and each membrane was pressurized to 9 bar for at least 2 hours by using pure water to avoid any compression effects and to establish leak tightness. The filtration experiment was carried out by circulating three liters of the feed solution using a stainless steel gear pump into the filtration cell. The trans-membrane pressure and volumetric flow rate were adjusted by using the concentrate (reject) outlet valve and variable speed key of the pump. The pressure was varied between high pressure at 9 bar down to low pressure at 4 bar at the specific pressure of (9, 8, 7, 6, 5, 4 bar). The experiments were done in total re-circulation mode, i.e., both the concentrate and the permeate streams were recirculated into the feed tank, so that the feed concentration was kept approximately constant.

3. Analytical Methods

Several analytical methods were used to determine the solute concentration of salt mixture and seawater samples in feed and permeate solutions in order to calculate the solute rejection. Coupled plasma-atomic emission spectrometry (ICP-AES) was used to measure the investigated ions (Na^+ , Mg^{+2} , and SO_4^{2-}) in salt mixtures. The chloride ion concentration was measured by chloride ion selective electrode [17]. A Russell chloride ion selective electrode Model 662-0632 with a double junction reference electrode Russell Model S8168 was used to measure the electrode potential of chloride ion in the feed solution and the permeate line. These electrodes were used specifically in the filtration experiments of mixtures to determine the concentration of chloride ion present in the solution. They allowed the measurement of electrode potential quickly, simply and accurately. A digital pH/mV meter Russell Model CD660 was used to measure the electrode potential given in mV up to one decimal place. An ionic strength adjustor (ISA) was used to provide a constant background ionic strength for both standard and sample solutions; the activity coefficient of the chloride ion in solutions will be similarly constant. It was made up from 5.0 M NaNO_3 solution. For the chloride ion solution, the 1,000 ppm chloride solution was used as a standard, as well as a 5 M ISA solution. A calibration curve

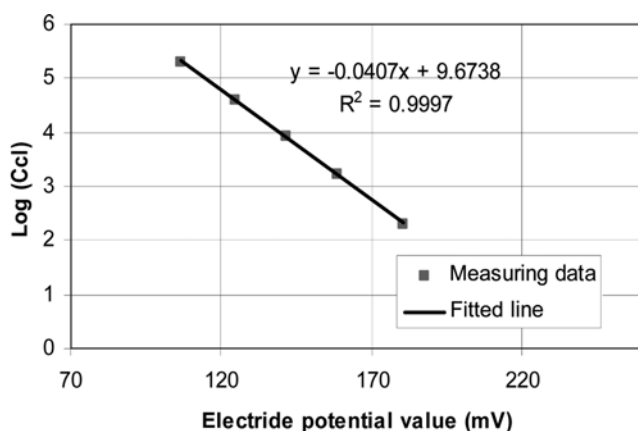


Fig. 1. The calibration curve for chloride concentration.

between the electrode potential reading and concentration of standard chloride solution on a log axis was prepared which will be linear over a range of analytical concentrations, as shown in Fig. 1. This curve was used to calculate the concentration of chloride ions in the investigated salt mixtures.

Once the chloride potential is taken, the concentration of chloride is calculated using the line or the linear regression equation shown in Fig. 1. More details about the analytical method are found elsewhere [18].

RESULTS AND DISCUSSION

This section will present the filtration results for the investigated salt mixtures shown in Table 1. In particular, the effect of pressure on permeate flux and observed ion rejection for NF90 and NF270 membranes will be discussed at pressure in the range of 4 to 9 bar. This will give an idea about the performance of both investigated membranes to be employed as pretreatment to desalination processes or as partial demineralization applications.

1. Mixture I (NaCl and Na_2SO_4)

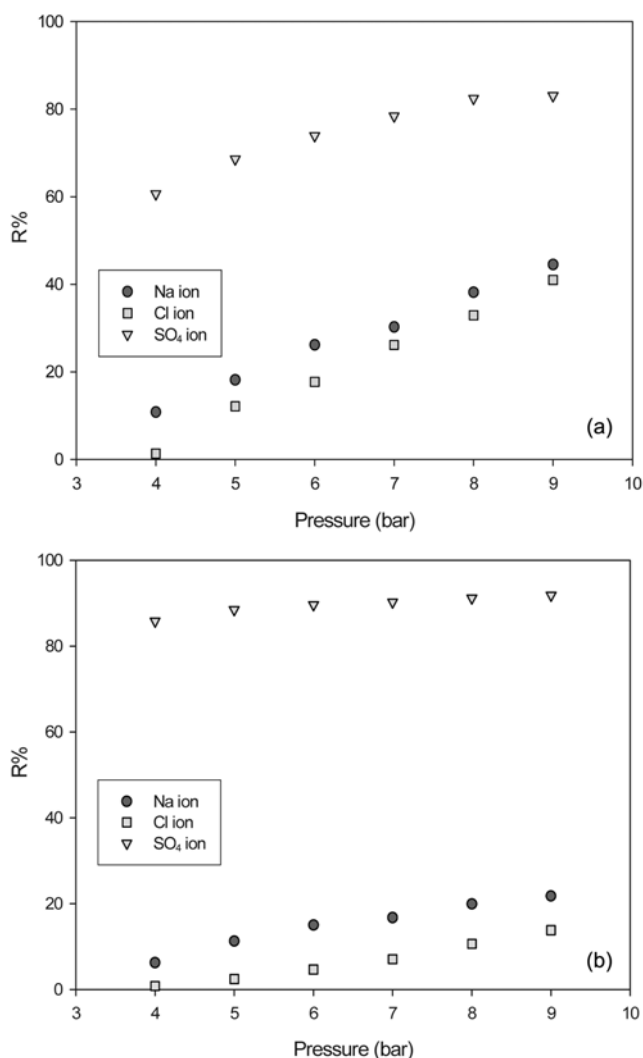


Fig. 2. Rejections of Na^+ , Cl^- , and SO_4^{2-} ions versus pressure for both membranes (a) NF90, and (b) NF270.

Fig. 2 shows the experimental data of rejection for each ion of mixture I versus pressure for both NF90 and NF270 membranes. The studied concentration of each ion is shown in Table 1. It is clear from this figure that the rejection increases with increasing pressure for both membranes NF90 and NF270. The increase was less in the case of NF270. This is similar to the findings in our previous work [19] for the rejection of single salts. In addition, both membranes are able to reject divalent anion (SO_4^{2-}) higher than that of the monovalent ions (Na^+ and Cl^-) due to the large-sized ions of sulfate ions (sieving effect) as shown in Fig. 2.

For the same reason, the rejection of Na^+ is higher than that of Cl^- for both membranes. This is consistent with findings derived from the AFM characterization; both membranes have different pore diameters, hence different rejections as shown in Table 1. In particular, for the NF90 membrane the rejection values for SO_4^{2-} anion were 83.1% at 9 bar and 60.6% at 2 bar, while they decreased for monovalent ions to be 44.5% and 41.0% at 9 bar for Na^+ and Cl^- ions, respectively (see Fig. 2(a)). On the other hand, the rejection values of SO_4^{2-} anion were 91.8% at 9 bar and 85.7% at 2 bar for NF270. For monovalent ions, the rejection values were 21.8% and 13.8% for Na^+ and Cl^- ions, respectively, for the latter membrane, as shown in Fig. 2(b).

It is worth mentioning that the rejection of monovalent ions using NF90 membranes is higher than that of NF270 membrane (sieving effect), while for divalent ion (SO_4^{2-}) the ion rejection for NF270 is slightly higher than the rejection for NF90 membranes. This means that both membranes are capable of preventing the scaling of sulfate compounds over the desalination equipment with the same efficiency. This supports the use of NF membranes as pretreatment for the desalination processes. The NF90 membrane is able to reject the monovalent ions more than NF270 membrane, which means that the NF90 membrane is preferred for partial desalination processes over the NF270 membrane. On the other hand, Fig. 3 shows the permeate flux of mixture I versus pressure for both investigated membranes.

It is clear from Fig. 3 that the permeate flux increases with pressure due to the increase of solvent flux. In addition, the permeate

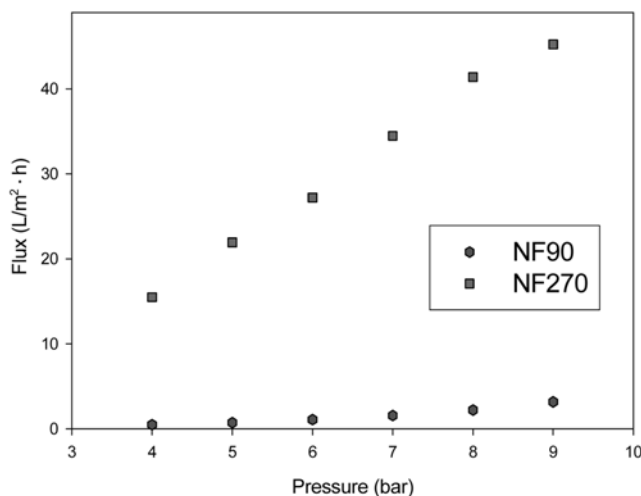


Fig. 3. The permeate flux of mixture I versus pressure for NF90 and NF270 membranes.

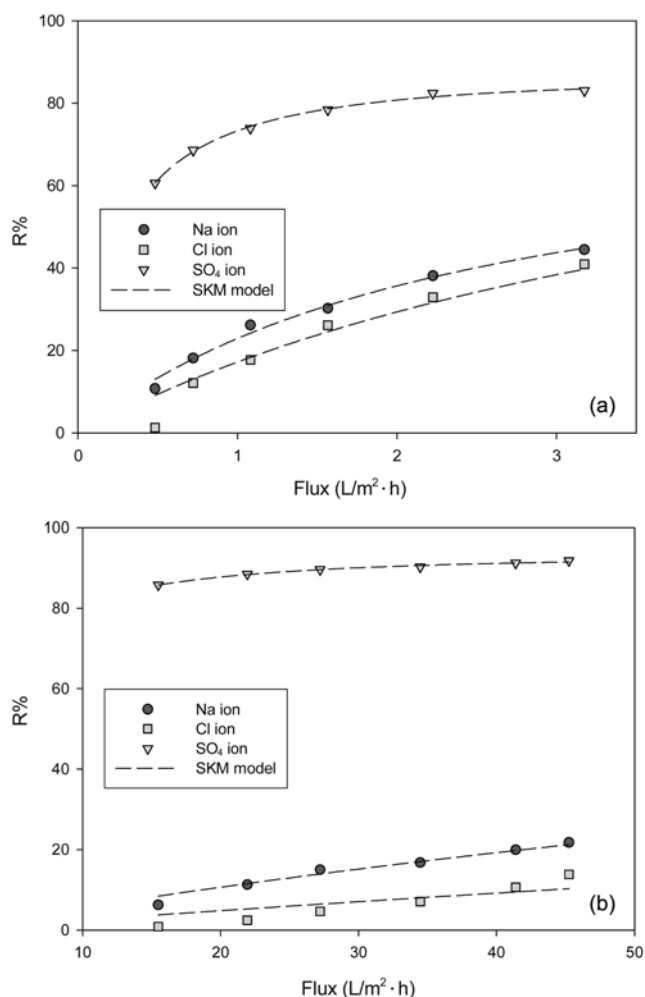


Fig. 4. Rejections of Na^+ , Cl^- , and SO_4^{2-} ions with the flux for both membranes (a) NF90, and (b) NF270.

flux for NF270 is extremely higher than that of NF90, as shown in Fig. 3. Over the pressure range of 2 to 9 bar, the flux was in the range of 15.5 to 45.2 ($\text{L}/\text{m}^2 \cdot \text{h}$) for NF270, while for NF90 the flux was in the range of 0.50 to 3.2 ($\text{L}/\text{m}^2 \cdot \text{h}$). As a result, the NF270 membrane will be better than NF90 membrane in preventing sulfate scaling. The rejection as a function of permeate flux for all anions and cations contained in mixture I by using two investigated membranes is shown in Fig. 4.

This relation is similar to the relation of the rejection with pressure for the same ions, as shown in Fig. 4, in which the rejection increases with increasing flux for NF90 and slightly increases with flux for NF270 membrane. The SKM model was used to fit the experimental data of rejection with flux for each investigated ions (Na^+ , Cl^- , and SO_4^{2-}) to determine the reflection coefficient (σ) and the solute permeability (P_s) parameters. The fitted line is shown as dashed lines in Fig. 4, which shows a good fitting for divalent ion of SO_4^{2-} for both membranes. However, for monovalent ions the fitting was not good for both membranes at low flux except one case. This case is the fitting of experimental data of rejection of Na^+ cation using NF90 as shown in Fig. 4(a). However, the reason for this poor fitting is due to their low rejection values as the SKM model is a valid correlative framework for membranes with only high rejections. The

Table 3. Reflection factor (σ) and solute permeability (P_s) for investigated ions of mixture I for NF90 and NF270 membranes

Membranes	NF90		NF270	
Ions with feed concentration (ppm)	σ	P_s (L/m ² ·h)	σ	P_s (L/m ² ·h)
Na ⁺ (11460)	0.689	2.16	*	168
Cl ⁻ (15194)	*	4.80	*	395
SO ₄ ²⁻ (3379)	0.854	0.23	0.927	1.760

* No obtained data

regression parameters σ and P_s for the two NF membranes under study for each investigated ion are presented in Table 3.

It is clear that values of P_s and σ are dependent on the type of ions and on the membrane used. Moreover, σ value is high for sulfate anion for both NF90 and NF270 membranes, which means that these membranes are very close to the ideal case in rejecting this anion. On the other hand, there are no obtained real values for σ and P_s for cases of monovalent ions for both membranes in which SKM model was not fitted well due to the invalidity of this model to these

cases.

2. Mixture II (NaCl and MgCl₂)

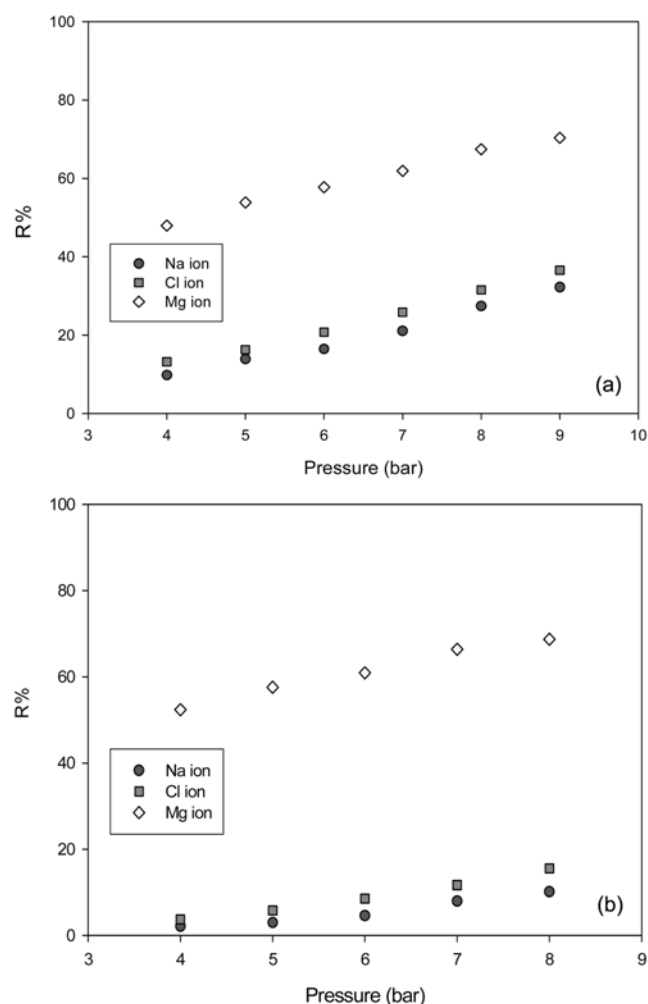
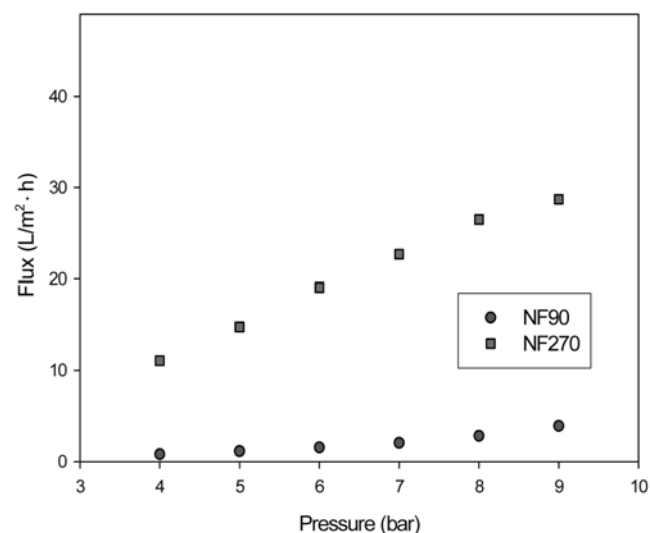
The ion rejection of mixture II (Na⁺, Cl⁻, Mg²⁺) with pressure for both investigated membranes is shown in Fig. 5. The studied concentration of each ion was previously shown in Table 1. It is clear that the rejection increases with increasing pressure for both membranes. Again, both membranes are able to reject divalent anion of Mg²⁺ higher than that of the monovalent ions due to the large size of magnesium cation (sieving effect) as shown in Fig. 5.

Furthermore, for both membranes the rejection of Cl⁻ ion is higher than the rejection of Na⁺—the opposite rejection to these ions in the filtration of mixture I. This is for the reason that Cl⁻ is the only anion in mixture II and for the neutrality which affects most of Cl⁻ anion rejected away with magnesium cation (charge effect). However, in mixture I, there is a competition between two anions (Cl⁻ and SO₄²⁻) in neutrality and the rejection. But due to the large size of SO₄²⁻ anion, its rejection is higher than that of Cl⁻ and most of latter anion passes with Na⁺ cation through the pores of the NF membrane due to the neutrality effect. The rejection of Mg²⁺ cation at pressure 9 bar was 70.3% and 74.5% for NF90 and NF270 respectively, which confirms the ability of these membranes to prevent the scaling through the desalination processes. However, it is recommended the experiment be performed at pressure higher than 9 bar in order to get rid off the divalent ions from the feed of the desalination processes completely. This is a recommendation for a future work.

The rejection of Na⁺ and Cl⁻ ions is (32.2% and 36.6%) and (12.4% and 19.0%) at 9 bar for NF90 and NF270 membranes, respectively. On the other hand, Fig. 6 shows the permeate flux of mixture II versus pressure for both investigated membranes.

Again, the permeate flux increases with pressure due to the increasing of solvent flux. As the filtration case of mixture I, the permeate flux for NF270 is much higher than that for NF90 as shown in Fig. 6.

The permeate flux values were 3.9 and 28.7 (L/m²·h) at 9 bar for NF90 and NF270 membranes, respectively; therefore, the use

**Fig. 5. Rejections of Na⁺, Cl⁻, and Mg²⁺ ions versus pressure for both membranes (a) NF90, and (b) NF270.****Fig. 6. The permeate flux of mixture II versus pressure for NF90 and NF270 membranes.**

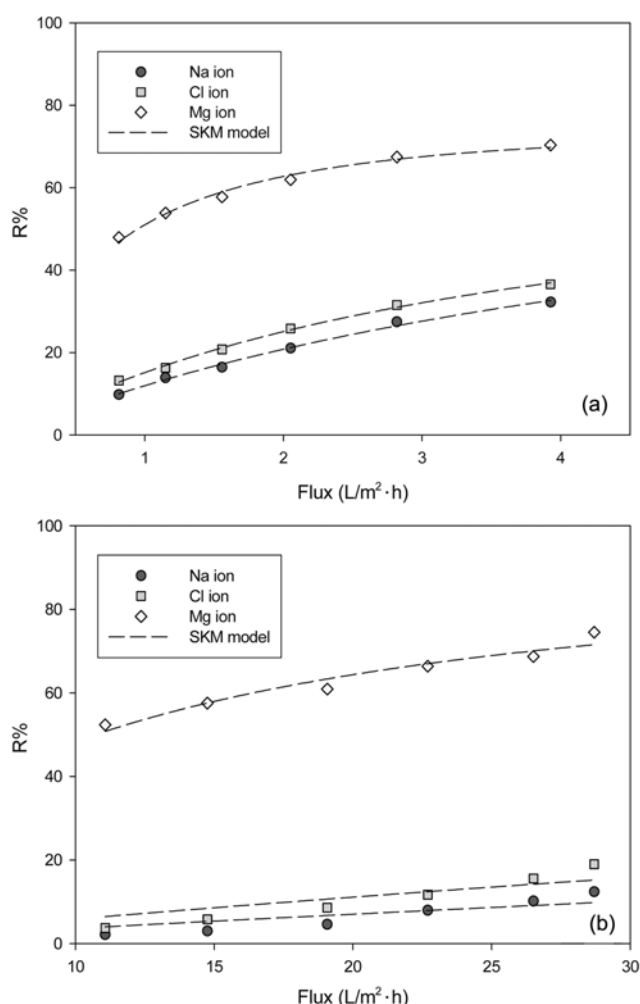


Fig. 7. Rejections of Na^+ , Cl^- , and Mg^{2+} ions with the flux for both membranes (a) NF90, and (b) NF270.

of NF270 is preferred to prevent magnesium scaling problems over NF90 in the desalination processes. Fig. 7 shows the rejection of ions of Na^+ , Cl^- , and Mg^{2+} versus permeate flux for the two investigated membranes. It is obvious that the rejection increases with increasing permeate flux for both membranes. The dashed line in the latter figure represents the fitted line of the SKM model for both membranes. A good agreement has been obtained between this model and the rejection of all ions for NF90, as shown in Fig. 7(a), while for NF270 the model only fits well the data of Mg^{2+} cation, as shown in Fig. 7(b). However, the model does not fit the monovalent ions (Na^+ , Cl^-) for NF270 for the reason that their rejection is not high. The model is only working for the membranes with high rejection values. Table 3 shows the parameter values of σ and P_s for each ion of mixture II for both investigated membranes. Real values of these parameters have only been obtained for the ions and membranes whose experimental data fit well the SKM model. According to Table 3, the parameters σ and P_s are dependent on the type of ions and on membranes. The σ value is high for magnesium cation for both NF90 and NF270 membranes due to their rejection values, while the σ value is medium for monovalent ions for NF90 membrane.

Table 4. Reflection factor (σ) and solute permeability (P_s) for investigated ions of mixture II for NF90 and NF270 membrane

Membranes	NF90		NF270	
Ions with feed concentration (ppm)	σ	P_s ($\text{L}/\text{m}^2\cdot\text{h}$)	*	P_s ($\text{L}/\text{m}^2\cdot\text{h}$)
Na^+ (11008)	0.677	4.82	*	160
Cl^- (18389)	0.594	3.125	0.931	264
Mg^{2+} (479)	0.731	0.557	*	9.6

* No obtained data

However, for NF270 membrane no logical values of σ have been obtained during the fitting of the latter ions. The values of P_s which are shown in Table 4 are also high but not logical and impossible for the reason that P_s , which represents the amount of permeable ion through the membrane, could not be higher than the total (solvent) flux, as shown in Fig. 6. Therefore, the SKM model is not valid for the experimental data of monovalent ions of mixture II for the NF270 membrane. As a result, it can be seen that the values of σ and P_s depend on the type of membrane and ions.

CONCLUSIONS AND FUTURE WORK

NF90 and NF270 membranes have been used to filter two different mixtures at salinity levels representative of seawater and pressure in the range of 4 to 9 bar. The results showed that the rejection increases with pressure for NF90 and slightly increases with pressure for NF270. The former membrane showed its ability to reject both monovalent and divalent of all investigated mixtures with very reasonable values but with relatively low flux. This makes NF90 more suitable for application in the pretreatment of desalination processes and in partial demineralization applications. On the other hand, NF270 can reject monovalent ions at relatively low values and divalent ions at reasonable values, but at very high permeate flux. So, it is recommended to use NF270 only as pretreatment for desalination processes. Finally, it is also recommended to carry out all filtration experiments at relatively higher pressure (>9 bar) in order to prevent all types of scaling in the desalination processes.

The SKM model was used to fit the experimental data of rejection of each ion in salt mixtures with the permeate flux in order to determine the fitting parameters of the reflection coefficient (σ) and the solute permeability (P_s). It is obvious that the model was not able to represent most experimental data of monovalent ions in salt mixtures for NF90 and NF270 due to their low rejection values, while the SKM model is a valid correlative framework for membranes with high rejections. However, for divalent ions the results showed that there was a good agreement between SKM fitting and the experimental data for both membranes. The values of the fitting parameter are high for both NF90 and NF270 membranes in the rejection of most divalent of single salt solutions, which means that these membranes are much closer to the ideal case that gives a complete rejection. For some filtration cases, there are no obtained real values for P_s and σ due to the invalidity of the SKM model in these cases. In a parallel work, both membranes (NF90 and NF270) were also investigated to study the rejection of both synthetic and real seawater in order to study their performance in treating seawater.

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NOMENCLATURE

A	: effective membrane area [m ²]
C _p	: permeate concentration [ppm]
C _f	: feed concentration [ppm]
ΔC _s	: solute concentration difference between the feed and the permeate [ppm]
C _s	: solute concentration [ppm]
J _v	: the volumetric flux [L·m ⁻² ·h ⁻¹]
J _s	: the solute flux [L·m ⁻² ·h ⁻¹]
L _p	: pure water permeability [L·h ⁻¹ ·m ⁻² ·bar ⁻¹]
P _s	: solute permeability [L·m ⁻² ·h ⁻¹]
P	: applied pressure [bar]
ΔP	: applied pressure on the membrane [bar]
R	: the rejection [%]
t	: time [h]
V _p	: volume of permeate [L]
σ	: reflection coefficient
Δπ	: osmotic pressure difference across the membrane [bar]

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