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Nanofiltration as pretreatment for scale prevention in seawater reverse osmosis desalination

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ABSTRACT

Seawater contains high concentrations of sparingly soluble salts which can cause scaling of the membrane surface, which in turn can limit the productivity and water recovery potential of seawater reverse osmosis (SWRO). Nanofiltration (NF) pre-treatment of seawater can prevent scaling via preferential removal of scale-forming ions. Several studies have shown that the rejection of scale-forming ions can vary considerably depending on the membrane. The main objective of this study was to test a number of commercially available NF membranes with synthetic seawater in order to compare their performance and identify optimum membranes and operational conditions for the removal of compounds that are known to cause scaling ($CaSO_{4\,(s)}$, $Mg(OH)_2$, $CaCO_{3\,(s)}$, etc.) in SWRO. Six membranes, supplied by several manufacturers, were tested in a laboratory-scale plant at various trans-membrane pressure differences. For the majority of membranes, the sulphate rejection was superior at 90%, which is very important for the prevention of scaling. Following on from the obtained results, a selection of the most suitable membranes for scaling prevention in seawater desalination can be done.

Keywords: Nanofiltration; Scale prevention; Desalination; Atomic force microscopy

1. Introduction

Seawater desalination is a separation process in which fresh water is extracted from saline water. Thus, the salts and hardness ions are retained in the concentrate stream, causing an increase in hardness and total dissolved solids (TDS). Due to the low solubility of hardness ions in seawater and depending on the conditions used, the hardness ions can precipitate on the desalination equipment resulting in scaling, which is a serious problem in desalination plants. For scale prevention in seawater reverse osmosis (SWRO) plants certain anti-scalants, such as polyphosphates or polycarboxylic acids, are added to the feed water, but even with this precaution, the product water recovery remains low and inorganic fouling is always present.

Nanofiltration (NF) is a pressure-driven membrane process which has a molecular weight cut-off between reverse osmosis and ultrafiltration. The rejection ability of NF membranes depends not only

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on the pore size but also on the charge of the membrane [1].

NF presents several advantages as compared to reverse osmosis, such as low operating pressures, higher fluxes, lower investment, operation and maintenance costs. Fairly high rejections of multivalent ions, especially anions such as sulphates, are also a characteristic of these membranes. Due to these properties, NF is considered a suitable pretreatment process for seawater desalination [2].

Hassan et al. [3] proposed the use of NF membranes as a pretreatment of desalination. They demonstrated that NF treatment of non-coagulated dual-media filtered seawater feed to desalination plants removed very fine turbidity, residual bacteria, scale forming hardness ions and lowered the TDS. These are the four factors that constitute the major problems in seawater desalination. Feeding the NF permeate using either thermal or membrane processes for desalination plants, could result in improved operation through less or no chemicals used and with a significant increase in their permeate and distillate recovery ratios. This improved operation could lead to lower energy consumption with the ultimate benefit of lowering potable water production costs.

The integration of NF with Multi Stage Flash (MSF) processes made it possible to operate MSF plants on NF product or SWRO reject from a NF-SWRO unit at a high distillation temperature of 120–160 °C with high distillate recovery, and again without chemical addition. Thus, MSF and NF SWRO-MSF could be operated at a top brine temperature of 120 °C without any scaling problems [4].

A demonstration plant was built in Umm Lujj, Saudi Arabia, consisting of six spiral wound NF modules (8 in. by 40 in.) followed by three SWRO elements. The results obtained from the demonstration unit confirmed the results previously obtained in the pilot plant study. Furthermore, the study allowed for the establishment of operating parameters for future NF-SWRO plants [5].

Macedonio et al. [6] analyzed seven different integrated membrane systems for seawater desalination namely: (1) Only the RO unit; (2) NF-RO; (3) MF-NF-RO; (4) MF-NF-RO and membrane crystallizer module on NF brine; (5) MF-NF-RO and membrane crystallizer module on RO brine; (6) MF-NF-RO and membrane crystallizer module on both, NF and RO brines; and (7) MF-NF-RO, membrane crystallizer module on NF brine and membrane distillation on RO brine. Through the introduction of NF as a pretreatment, the RO permeate increased due to the lower osmotic pressure of the water fed to the RO unit; more importantly, the increase of the water recovery was up to 52%.

However, there are various types of NF membranes, and it was necessary to study which ones are the most suitable for scale prevention. The aim of this study was to research, at laboratory scale, the productivity and the selectivity of various NF membranes in order to know which membranes are suitable for scale prevention in SWRO.

2. Materials and methods

2.1. Membrane characterization

Six commercially available NF membranes were used in this study. These membranes were kindly supplied as flat sheets by Dow Chemical, Alfa Laval Membranes, Koch Membrane Systems and Hydranautics. All membranes were stored in darkness at 4°C and each one was immersed in deionised water for at least 12 h before being used, except those that have specific cleaning procedures specified by the manufacturers.

2.1.1. Determination of membrane pore size

Pore size is one of the main parameters in membrane characterization. The experimental procedure described by Bowen et al. [7] was used in order to determine the pore dimensions of the studied NF membranes. Bowen et al. obtained membrane pore size from the limiting rejection of neutral solutes using the following equation

$$R_{\lim} = 1 - (1-\lambda_i)^2(2-(1-\lambda_i)^2)(1+0.054\lambda_i - 0.0998\lambda_i^2 + 0.441\lambda_i^3) \quad (1)$$

where $\lambda_i = r_s / r_p$; r_s is the solute radius and r_p is the pore size.

Three uncharged solutes, glucose, glycerine and 2-propanol, were used for characterising the six membranes used in terms of pore size. Their physical properties are given in Table 1.

Rejection experiments at different pressures were done in order to obtain the limiting rejection for each solute. The experimental system used was the same as the one described in Section 2.1, and the crossflow velocity was 0.5 m s^{-1}, which is sufficiently high for dismissing concentration polarisation.

Table 1
Solute radius and diffusion coefficient for uncharged solutes used [8]

Solute	r_s (nm)	D_s ($\times 10^{-9}$ m^2 s^{-1})
Glucose	0.358	0.67
Glycerine	0.260	0.95
2-Propanol	0.216	1.02

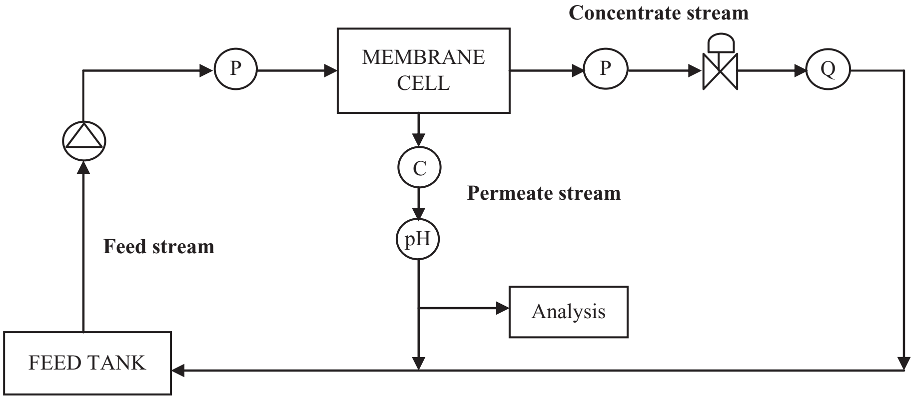


Fig. 1. Experimental system flow sheet.

2.1.2. *Roughness measurement with atomic force microscopy*

Atomic force microscopy (AFM) has been used for the determination of membrane roughness. The study of NF membranes using AFM has been reported in the literature [7,9–11].

AFM presents some advantages when compared to other techniques such as scanning electron microscopy (SEM) or transmission electron microscopy (TEM), AFM has a higher resolution, the sample preparation is minimal and the sample is not damaged by the electron beam [12].

With the AFM *VeecoNanoscope IV Multimode AFM*, using tapping mode, the surface roughness of each membrane was determined. Roughness is one of the most important surface properties as it has a strong influence on membrane fouling [9]. Vrijenhoek et al. [13] demonstrated through use of AFM analysis that the deposit of particles is higher for rough membranes than for smooth membranes when all test conditions are held constant.

2.2. *Ion rejection experiments*

All the experiments were carried out in a laboratory-scale test cell using a crossflow module (model SEPA-CF Osmonics). The flow sheet of the experimental set up is shown in Fig. 1. The crossflow velocity and the transmembrane pressure were measured by two pressure sensors and a flow meter, connected directly to a data acquisition card. Permeate and concentrate streams were all re-circulated into the feed tank in order to keep the feed concentration constant during the experiment. The transmembrane pressure was adjusted manually using a needle valve located in the concentrate line, and it was varied between 2 and 20 bars. The crossflow velocity was 0.15 m s^{-1} and was controlled by a PID using the Labview software. This value of crossflow velocity was chosen according to data proportioned by Dow Chemical, so this value is between the ones used normally in desalination plants. Conductivity and pH of the permeate stream were measured on-line, using a conductivity cell (Crison53 92) and a Ag/AgCl pH electrode (Crison 53 03). All the data was saved in a computer. At each operating pressure, a sample was collected and stored at 4°C until the corresponding analysis was completed.

Before starting the experiment, all membranes were pressurised at the maximum pressure for the experiment, 20 bar. The membranes were pressurised one hour with deionized water and then a further hour with the synthetic seawater.

2.3. *Analytical methods*

In order to determine the rejection of all the ions present in the feed water, several analytical methods have been used for the analysis of feed water and permeate samples.

Ion Chromatography, Dionex ICS-2000, was used to analyse anions (Cl^-, SO_4^{2-}, Br^-) and cations (Na^+, K^+, Ca^{2+}, Mg^{2+}), Total Carbon Analyzer, Shimadzu TOC-5050A, was used to analyse inorganic carbon and solute concentration in the pore size characterisation and Inductively Coupled Plasma Mass Spectrometry (ICP-MS), Agilent 9500cx, was used to analyze total boron and strontium.

Reference materials and spiked samples were analyzed together with samples in each analysis batch, and the recoveries were always between 90% and 110%.

Table 2
Composition of synthetic seawater

	mg kg^{-1}
Cl^-	19258 ± 1761
Na^+	10065 ± 1112
$SO_4{}^{2-}$	2497 ± 418
Mg^{2+}	1436 ± 130
Ca^{2+}	437 ± 19
K^+	401 ± 20
$HCO_3{}^-$	26 ± 1
Br^-	67 ± 4
Sr^{2+}	8.65 ± 0.52
H_3BO_3	23.37 ± 3.5
Conductivity (mS cm^{-1})	44 ± 2
pH	7.80 ± 0.01

2.4. Synthetic seawater

Synthetic seawater, containing only inorganic salts in order to avoid problems related to organic matter, was used as the feed solution in all the experiments. It was prepared following the procedure described by Kester et al. [14]. The prepared feed solution was analysed with the before mentioned analytical methods and its species distribution was calculated using the 2.15 version of PHREEQC program with Pitzer database [15]. The composition with the corresponding species of synthetic seawater prepared is shown in Table 2.The species distribution was also performed for the obtained permeates. This allowed all solutions to be checked to ensure that they maintained their electroneutrality, and that inorganic carbon and boron were present as hydrogen carbonate and boric acid, respectively.

3. Results and discussion

3.1. Membrane characterization

Table 3 shows the results obtained for membrane characterization in terms of pore size for the six membranes studied and the values found in the literature for doing a comparison with the experimental ones.

According to the data in Table 3, the values obtained in this study are very similar to the ones found in the literature. The only case that presents a higher difference between the experimental value and the one in the literature is for ESNA 1-LF2 membrane. For this membrane, a pore size of 0.49 nm has been obtained, while in the literature it was reported by Wang et al. [19] that this membrane has a pore size of 0.30 nm. This difference can be explained basically for two different reasons. On the one hand, the differences between two membrane pieces; and on the other hand, in Wang et al.'s study, different values of uncharged solute radius were used for pore radius calculation. Additionally, they used two different solutes than the ones used in this study.

Table 3
Obtained results for membrane pore size (r_p) and comparison with results in the literature

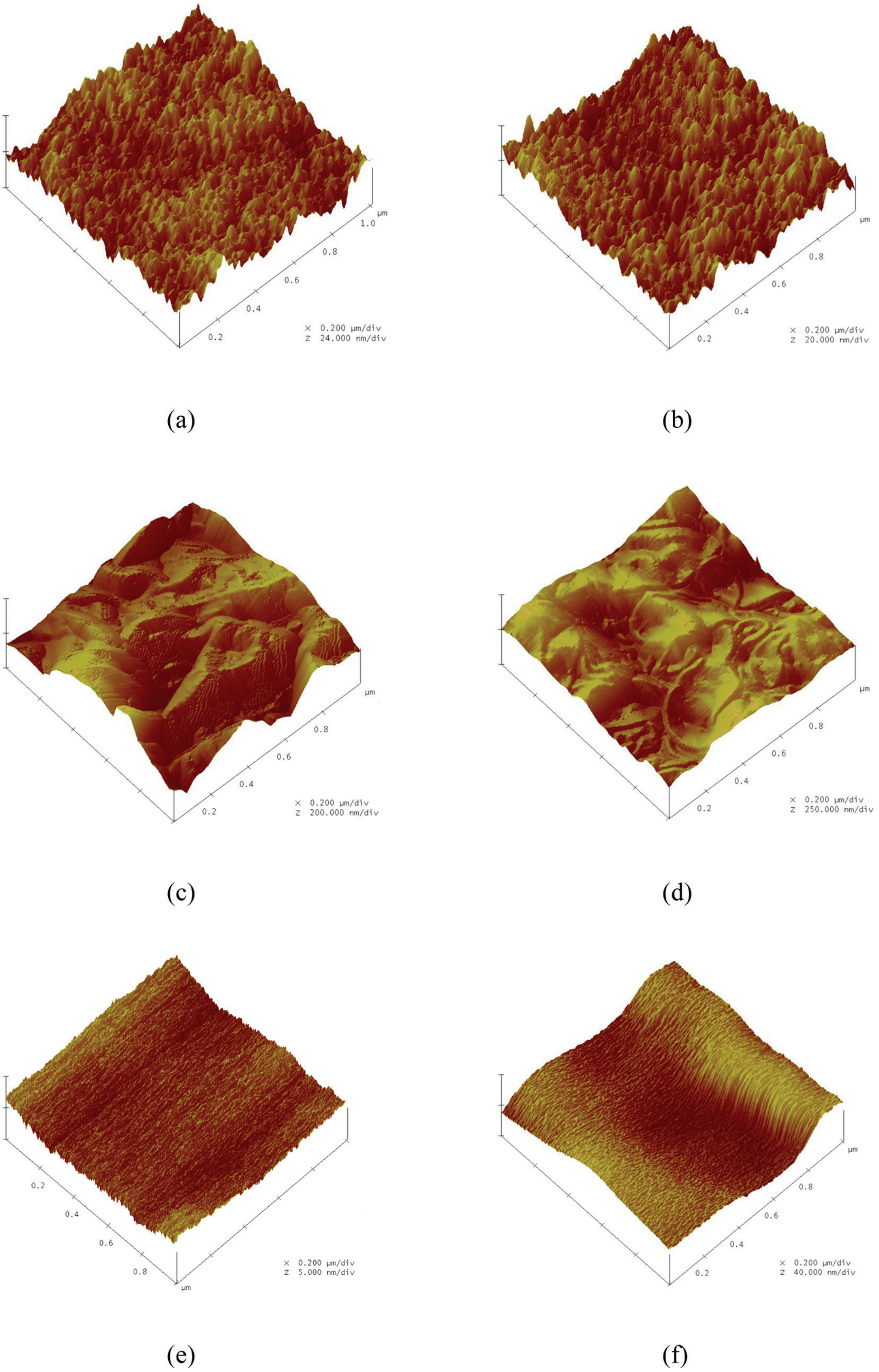
Membrane	Solute	Limiting rejection	r_p (nm)	r_p (ref) (nm)
NF270	Glucose	0.973	0.40	
	Glycerine	0.554	0.51	
	2-Propanol	0.332	0.60	
Average			0.50	0.48 [16]
NF200	Glucose	0.975	0.40	
	Glycerine	0.613	0.47	
	2-Propanol	0.440	0.50	
Average			0.46	0.50 [17]
NF90	Glucose	0.984	0.38	
	Glycerine	0.912	0.32	
	2-Propanol	0.800	0.31	
Average			0.34	0.38 [18]
K-SR2	Glucose	0.803	0.51	
	Glycerine	0.257	0.84	
	2-Propanol	0.237	0.74	
Average			0.69	0.64 [18]
ESNA 1-LF2	Glucose	0.740	0.55	
	Glycerine	0.567	0.49	
	2-Propanol	0.519	0.44	
Average			0.49	0.30 [19]
NF99HF	Glucose	0.945	0.42	
	Glycerine	0.631	0.46	
	2-Propanol	0.437	0.50	
Average			0.46	–

Table 4 and Figs. 2(a)–(f) show the results obtained for membranes roughness and the AFM images for each membrane.

As shown in Table 4, the values obtained were compared with other studies from the literature. It could be

Table 4
Obtained results for membrane roughness (Rms) and comparison with results in the literature

Membrane	Manufacturer	Rms (nm)	Rms (ref) (nm)
NF270	Dow Chemical	5.35	4.38 [11]
NF200	Dow Chemical	7.39	3.68 [11]
NF90	Dow Chemical	103.3	40.0 [18]
K-SR2	Koch Membrane Systems	0.76	0.45 [20]
ESNA 1-LF2	Hydranautics	49.07	55.0 [21]
NF99HF	Alfa Laval	12.29	–



(a) (b)

(c) (d)

(e) (f)

Fig. 2. AFM pictures of studied nanofiltration membranes: (a) NF270; (b) NF200; (c) NF90; (d) ESNA 1-LF2; (e) K-SR2; (f) NF99HF.

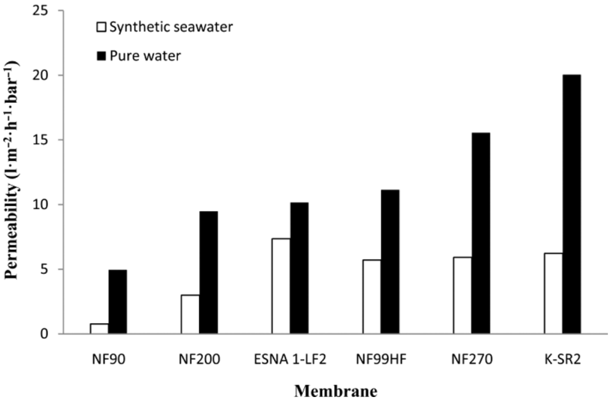


Fig. 3. Permeabilities of different NF membranes.

observed that the values obtained in this study have the same order of magnitude than the ones published in previous works. The value that shows the greatest difference from those in the literature is the roughness of NF90 membrane. The difference between the values obtained in this study and the ones found in the literature are attributed to the differences between the two membrane pieces.

There are noticeable differences between the roughnesses of the six membranes studied. The membrane with the highest roughness was found to be NF90, Dow Chemical, and the one with the lowest value for this parameter was K-SR2 from Koch Membrane Systems. Several studies [13,22–25] have demonstrated that colloidal fouling of RO and NF membranes are strongly correlated with the membrane surface roughness. Membranes with high surface roughness are more prone to fouling.

3.2. Membrane permeabilities

Fig. 3 shows the pure water membrane permeabilities as well as the membrane permeabilities with synthetic seawater, calculated with the slope of the linear part of dependence of transmembrane volume flow on the pressure difference of tested membranes. The membrane with the lowest permeability is NF90, whereas in the case of pure water membrane permeabilities KSR-2 demonstrates the highest value for this parameter. The other membranes have intermediate permeabilities between these two extreme cases.

3.3. Rejection results

Figs. 4(a)–(h) show the observed rejections for the seawater ions for the six membranes studied.

Scaling occurs when sparingly soluble salts become concentrated because of the retention and their concentrations that exceed their solubility limit [1]. Scales can be divided into two categories: an alkaline soft scale made of $CaCO_3$ and $Mg(OH)_2$ and non-alkaline hard scale consisting of $CaSO_4$, or $CaSO_4·½2H_2O$ or $CaSO_4·2H_2O$ [26], therefore in order to prevent scaling, it is important that membranes show high rejections of divalent ions, as well as bicarbonate.

As shown in Table 4, all membranes show a high rejection of divalent ions, especially sulphates, which are almost totally rejected (>95%) by the majority of membranes. This high sulphate rejection is important for the scaling prevention in SWRO plants.

The calcium rejection ranges from 10% to 95%, and with the majority of membranes this rejection is between 60% and 70%. The results observed in the case of magnesium rejection are similar to calcium, the lowest rejection of magnesium is about 40%, corresponding to ESNA 1-LF2 membrane, and the highest is 99% (NF90).

In addition to the rejection of multivalent ions, it is also important to know which percentage of monovalent ions present in seawater is rejected in NF stage because if seawater arrives less concentrated in the reverse osmosis membranes, the necessary pressure in this last stage will be lower and it will involve a reduction in energy consumption.

At first glance one can observe that the rejection of monovalent ions is clearly lower (20–30%) than the rejection of divalent ions, this therefore confirms that the rejection by NF membranes is dependent on the ion valence.

The rejection of bicarbonate is higher than the rejection of the other monovalent ions, for some membranes it reaches ca. 80%, and most membranes reject up to 60%. That is important for the scale prevention because $CaCO_3$ is one of the most important scalants in SWRO.

Hilal et al. [27] studied the rejection, at laboratory scale, of three different NF membranes with concentrated NaCl solutions. Two of these membranes were NF270 and NF90, which were also used in this study. Using a feed solution of 25,000 ppm of NaCl at 9 bar of pressure, Hilal et al. obtained the rejections of 41% for NF90 and 11% for NF270. These results are very similar to those obtained in this study with the NF of synthetic seawater at 10 bar, namely 32% for NF90 and 16% for NF270.

In a further study, Hilal et al. [28] investigated the performance of NF90 and NF270 in the treatment of synthetic and real seawater. The hydraulic permeabilities and rejections of monovalent ions from their investigations were very similar to this study, however, the rejections of divalent ions shown in their research were rather different from this study. This can

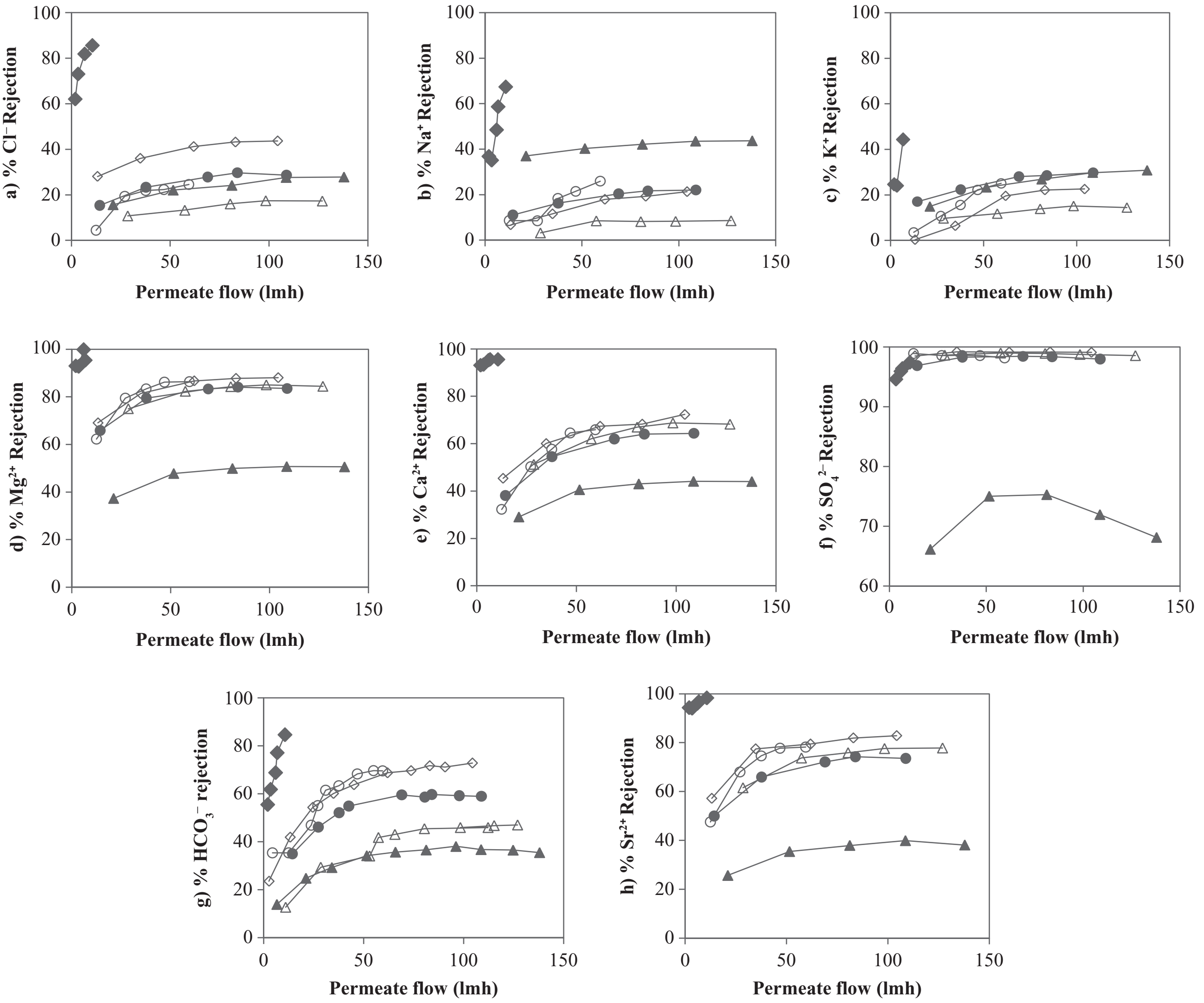


Fig. 4. Observed rejection for different ions and membranes (●: NF270, ○: NF200, ◆: NF90, ◇: NF99HF, ▲: ESNA 1-LF2, △: K-SR2).

be explained by the fact that Hilal et al. used a circular test cell with empty feed channel, whereas in this study we used a spacer-filled feed channel. This could give rise to quite different extents of concentration polarization, which is especially important for the strongly-rejected divalent ions.

3.4. Boron rejection

Another important solute to be considered is boron, which is present in seawater as boric acid, and under the experimental conditions, NF membranes had difficulty in rejecting it, as illustrated by Figs. 5(a)–(f).

The membrane with the highest boron rejection was NF270, which has a rejection of about 30%. The other membranes have rejections lower than 10% and even some of them present negative rejections. So, using NF270 as a pretreatment in SWRO could help reaching the maximum permissible concentration of 1 ppm in drinking water [29].

A parameter that plays an important role in boron rejection is pH. It is well known that the boron compounds in seawater, mainly boric acid, do not dissociate into ions at low to natural seawater pH. Consequently, the boron rejection is not sufficiently high [30]. However, at pH higher than 9.5, boron is present mostly as $H_2BO_3^-$, and it can be eliminated nearly 100% [31]. The problem at high pH is the high precipitation of calcium and magnesium salts [30], therefore the water has to be softened before raising the pH which could be another point in favour of using NF as a pretreatment in SWRO desalination plants.

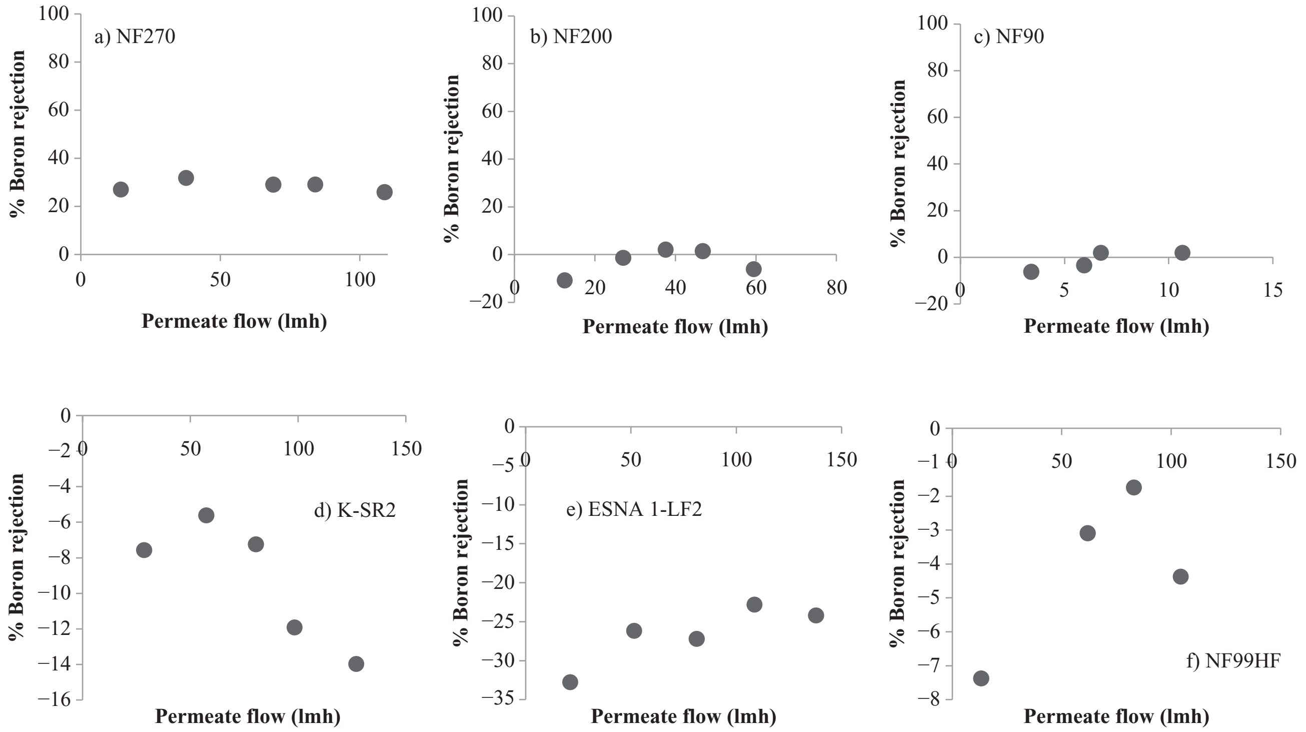


Fig. 5. Effect of permeate flux on boron rejection by various NF membranes.

5. Conclusions

Six different NF membranes have been studied in order to choose which ones can be suitable for the scaling prevention in SWRO. Synthetic seawater was filtered in a laboratory-scale plant and the rejection of different ions was analysed. The obtained results show that the rejection of divalent ions is high in all the membranes tested, which is highly important for the scaling prevention.

An overview of the results concludes that the most suitable NF membranes for anti-scaling pretreatment are: NF270 (Dow Chemical), K-SR2 (Koch Membrane Systems) and NF99HF (Alfa Laval). These three membranes have been selected due to their high rejections of scale forming ions as well as their highs permeate flux. However, more studies should be performed, especially at pilot scale, in order to define the optimal operating parameters of NF for pretreatment in SWRO desalination process.

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