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Desalination of brackish water and reverse osmotic retentate using nanofiltration membranes: effects of TMP and feed concentration on the treatment

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ABSTRACT

Nanofiltration (NF) membrane with low-pressure operating conditions and high flux permeability seems to be an attractive alternative for water softening and desalination. In order to study the performance of NF membrane on treating real brackish water, with mixture of mono and divalent ions, two commercial flat sheet nanofiltration membranes (NF90 and NP030) were used. The experiments were carried out with transmembrane pressure from 4 to 12 bar with three brackish waters having different ions concentrations. The results obtained showed that the high hydrophilicity of NF90 and its small pore size were the main advantages that allow this membrane to have the highest permeability and salt rejection for both mono and divalent ions in comparison with NP030. The results also showed that the permeate flux and rejection increased linearly with increasing in TMP. NF90 membrane performance was assessed by studying the effect of feed ions concentration. It was observed that the permeate flux decreased with increasing in salts concentration due to concentration polarization. Additionally, the study of scaling problem showed that its contribution in permeate flux decreasing was not as much significant and that the membrane permeability, recovered after the cleaning step, was 90%. In the other hand, the salts rejection of NF90 remained high (more than 80%) for all the studied concentrations due to its separation mechanism.

Keywords: Desalination; Nanofiltration; Real brackish waters; Rejection of ions

1. Introduction

Membrane filtration is a pressure driven process in which membrane acts as selective barriers to restrict the passage of pollutants and allows relatively clear water to pass through [1]. Depending on their pore sizes, membranes processes have been classified into four categories: microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO) membranes. With properties in between ultrafiltration (UF) and reverse osmosis (RO), NF membranes possess pore size typically of 1 nm which corresponds to molecular weight cut-off (MWCO) of 300–500 Da [2]. This process holds many advantages such as operating at low pressures in comparison with the reverse osmosis, having high permeate flux and high rejection of divalent ions. These characteristics leads to low energy consumption and consequently to low operating cost. The main parameters that characterize the NF membrane are the permeate flux and the rejection. Depending on the surface membrane properties, many mechanisms can affect the separation performance such as size exclusion (steric effect), electrostatic effect (Donnan effect) and dielectric exclusion. The feed concentration, the nature

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of ions as well as the operating transmembrane pressure are also important parameters to take into account while studying the NF membranes.

NF process was applied in numerous fields from water production to various industrial processes and effluents [3–5]. It is mainly applied in drinking water purification for softening, decolouring and micro pollutant removal. Several studies had evaluated the efficiency of NF membrane in treating salt solutions. Walha et al. [6] had studied the possibility of producing drinking water using different processes: NF, reverse osmosis and electrodialysis. The results related to the NF tests on brackish water showed it's insufficient to obtain drinking water since the TDS of permeate was superior to the standard value authorized by the WHO. In this perspective, Mogheir et al. [7] had carried out tests that help to understand the behavior of two NF membranes using different samples (pure, real and synthetic solution) but focusing on real sample. The tested parameters were salt rejection (TDS), nitrate removal (NO_3^-), flux rate (L/m²·h), permeability (L/m²·h·bar) and flux recovery rate. In another work, Schaep et al. [8] investigate groundwater softening using commercial nanofiltration membrane for reaching drinking water quality. They found that UTC 20 membrane shows retentions higher than 90% for multivalent ions, whereas monovalent ions were retained for about 60–70%. Additionally, it was found that a small concentration of organic compounds can cause a substantial flux decrease.

A recent paper co-authored by Nicolini et al. [9] gives the characteristics and the performance of three negatively charged commercial NF membranes (NF90, NF030 and NP010) and explains the phenomena involved in ions permeation using synthetic aqueous solutions having single and mixed salts (NaCl, Na_2SO_4, $MgSO_4$, K_2SO_4, and $CaSO_4$). On the other hand, Hilal et al. [10] had also studied the treatment of highly concentrated (NaCl) salt solutions with salinity level similar to that of seawater by the use of three commercial NF membranes (NF90, NF270, N30F).

In the present work, natural brackish water, reverse osmosis (RO) retentate and RO retentate after its concentration by evaporation were chosen in order to study the parameters affecting the performance of NF90 and NP030 membranes since these membranes had shown their good efficiency in synthetic water treatment [9,10]. The efficiency of the chosen membranes in water desalination was evaluated by filtering real brackish waters. This leads to filtration optimization in order to obtain high permeate flux with high rejection of different ions present in the tested water samples.

2. Materials and methods

2.1. Feed samples

Well water and reverse osmotic (RO) retentate (Brackish 1) supplied by a tanning industry in Valencia (Spain), and RO retentate concentrated by evaporation (Brackish 2) were treated in this work by nanofiltration (NF). These water samples were chosen to more evaluate the concentration effect on the membranes performance. The physicochemical characteristics of the three feed samples are summarized in Table 1.

2.2. Experimental setup

The filtration experiments were done in bench scale cross-flow membrane filtration unit designed in the "Universidad Politécnica de Valencia". This system includes a feed tank (5 L) and pump for feed circulation in a horizontal membrane module. The automated NF laboratory plant was used to regulate transmembrane pressure (TMP), cross flow velocity and temperature. Two manometers, controlled by a valve, are put in the inlet and outlet of the membrane. The flow rate is measured within line flow meter. The feed temperature was monitored by sensor. In order to keep the feed at constant room temperature (25°C) a heat exchanger was used to counteract heat generated by the pump (Fig. 1). The permeate flux was determined gravimetrically as the change of permeate weight versus time by using a laboratory scale balance. The membrane specific area was 0.0072 m².

Two flat sheet nanofiltration membranes, NF90 from Dow-FILMTEC and NP030 from MICRODYN NADIR provider were used with the characteristics as shown in the Table 2 [9,11].

Before starting the desalination experiments, the NF membranes were immersed in osmotic water for 24 h. Then, the NF system was equipped with the membrane and pretreated with osmotic water as feed solution. NF90 was compacted at 12 bar for 4 h in order to avoid the membrane compaction during desalination experiments. The compaction is done when reaching a steady state.

2.3. Membrane characterization

2.3.1. Membrane permeability

In order to characterize the NF membranes, their hydraulic permeabilities were determined with deionised water at different TMP range from 4 to 12 bar before any experimental run. Each pressure takes one hour with 0.07 m³/h as flow rate. The permeate flux were calculated for each TMP using Eq. (1).

$$J = \frac{Q}{A} = M_P \times TMP \quad (1)$$

with J is the permeate flux (L/h·m²), Q is the flow rate (L/h), A the membrane surface (m²) and M_p the membrane permeability (L/h·m²·bar).

Table 1
Physicochemical characteristics of feed waters

Parameters	Well water	Brackish 1	Brackish 2
pH	7.61	7.92	7.86
Conductivity (mS/cm)	1.293	3.69	10.48
Na^+ (mg/L)	64.09	198.9	786
Mg^{2+} (mg/L)	45.3	170.7	760
Ca^{2+} (mg/L)	201.3	645	1250
Cl^- (mg/L)	132	327	3000
SO_4^{2-} (mg/L)	395	1110	3500
NO_3^- (mg/L)	31.8	85.2	140.5

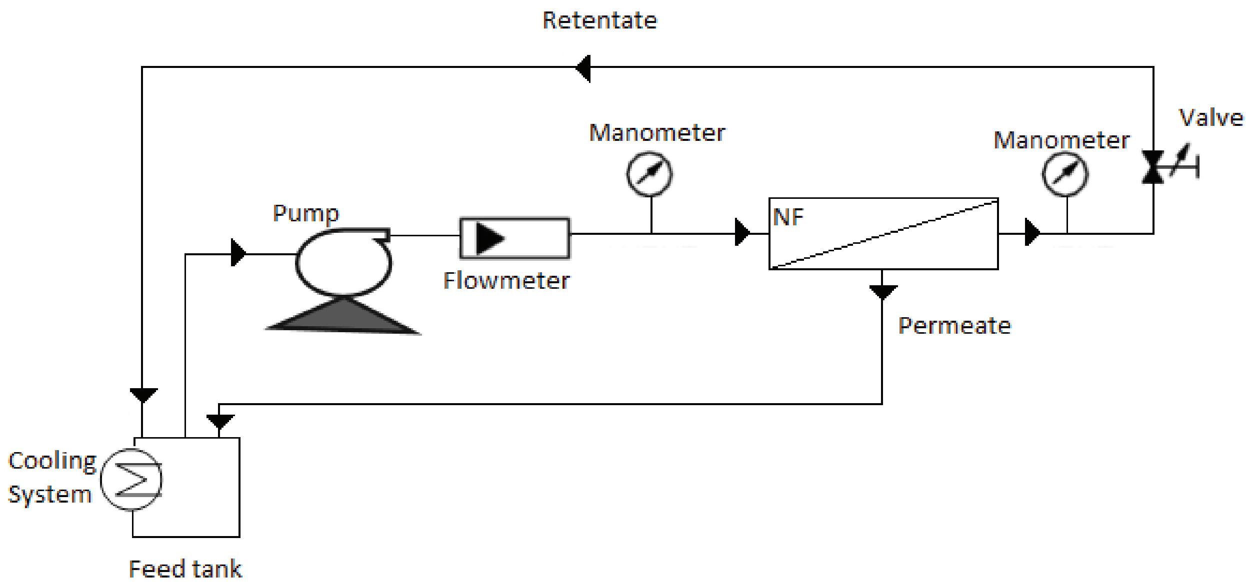

Fig. 1. Schematic diagram of Nanofiltration pilot plant.

Table 2
NF membranes specifications

Membrane	NF030[a]	NF90[b]
Manufactures	Microdyn Nadir	Dow/Filmtec
Material	PES	Polyamide
Maximum operating temperature (°C)	95	35
pH range	0–14	4–11
MWCO (Da)	400	200
Average pore radius (nm)	0.93[c]	0.68[c]

[a] From the provider; [b] From Ref. [11] ; [c] From Ref. [9].

Moreover, in order to study the possibility of membrane scaling, the membrane permeability was checked after each cleaning step.

The membrane resistance R_m was also determined by Darcy's law [Eq. (2)] which is a relation between deionised water permeates flux and TMP.

$$J = \frac{TMP}{\mu \times R_m} \quad (2)$$

with TMP is the transmembrane pressure (Pa), R_m the hydraulic resistance of membrane (m^{-1}) and μ the dynamic viscosity of pure water (Pa·s).

2.3.2. *Field Emission Scanning Electron Microscopy (FE-SEM) and EDX*

Membranes surfaces pictures of new NF90 and used NF90 for desalination experiments were done with several scale and magnitude using FE-SEM from Zeiss brand, Ultra 55 model with an extra high tension (EHT) of 2 kV. In addition, several little areas in the used membrane for desalination were analyzed with the EDX at 20 kV. The aim of this study is to confirm whether there is scaling problem in the membrane or not.

2.4. *Analytical methods*

The conductivity, pH and ions concentrations (calcium, magnesium, sodium, chloride, sulfate and nitrate) are the analyzed parameters in the feed and permeate samples. The pH and the conductivity were measured with a pH-Meter GLP 21þ and EC-Meter GLP 31þ (Crison Instruments, Spain). The ions (with the exception of sodium) concentration was measured using kit Merk, while the sodium was analyzed by flame atomic emission spectroscopy using S2 Series AA System Atomic Absorption Spectrometer (Thermo Electron Corporation, Cambridge, UK).

2.5. *Desalination experiments*

The desalination experiments were conducted with TMP range from 4 to 12 bar. Each pressure takes one hour with 0.07 m^3/h as constant flow rate. Samples from permeate and feed tank were analyzed in order to calculate the saline rejection percentage [Eq. (3)]:

$$R = \left(1 - \frac{C_p}{C_i}\right) \times 100 \quad (3)$$

with R is the rejection percentage (%), C_i is the feed concentration (mg/L) and C_p is the permeate concentration (mg/L).

Two parameters were taken into consideration in this work: the TMP effect and the concentration effect. The later was studied in two ways. In one hand, well sample was treated without recirculation in order to concentrate it. After each time, samples were taken from permeate and the feed tank in order to assess the evolution of the concentration and the rejection percentage until reaching a volume concentration factor (VCF) which is defined as the relation between the initial water volume and remaining water volume after the permeate extractions [12]. In the other hand, different samples with different concentrations were used (Brackish 1 and 2).

3. Results and discussion

3.1. Membranes permeability

It was found that the permeate flux of deionised water increased linearly with the operating pressure and that the permeability coefficients are about 2.62 and 4.99 L/h·m²·bar for NP030 and NF90 membranes, respectively (Fig. 2). Comparing these results with those found in the literature, they are very similar to Nicolini et al. [9] permeability values for both membranes.

These results show that even if NF90 have the lowest average pore size (0.68 nm), its permeability is higher than NP030 having the highest average pore size (0.93 nm). This could be explained by its high hydrophilicity, compared with NP030, which play an important role in the water transport through the membrane [9]. Previous researches had shown the influence of membrane surface characteristic on its performance. Ferjani et al. [13] have performed brackish water desalination using Cellulose Acetate (CA) membranes and compared it with the same materials modified by a thin layer of polymethyl hydrosiloxane (PMHS) onto the surface. The results showed the influence of the membrane surface in decreasing water flux and increasing the salt rejection due to PMHS hydrophobic surface layer and the blocking of small size pores by PMHS penetration.

Regarding the linear behavior between the transmembrane pressure and the pure water flux in both membranes, it could be explained by Spiegler-Kedem model. According to this model, the solvent transport is due to the TMP across the membrane and the solute transport is due to concentration gradient and/or convective coupling to the volume flow [14]. Consequently, in absence of solutes, the pure water flux becomes proportional to operating pressure difference across the membrane.

Moreover, the membrane hydraulic resistance R_m was calculated for NF90 and NP030. It was found that for 12 bars, the membrane hydraulic resistance was 7.43×10^{13} m^{-1} and 3.56×10^{14} m^{-1} for NF90 and NP030, respectively. These results are in agreement with the bibliography. According to Nicolini et al. [9], a lower water transport resistance for NF90 compared to NP030 was expected due to the contact angle and energy free surface of the membranes.

3.2. Effect of transmembrane pressure on salts rejection

The well water was used in this purpose. The permeate flux when treating the groundwater as function of transmembrane pressure TMP was plotted in Fig. 3 for NP030 and NF90 membranes. The effect of TMP on salts rejection is presented in Fig. 4. The results showed that the permeate flux of the saline water vary linearly with TMP and deviate slightly compared to the permeability of deionised water. Concerning the ions rejection, its value increases with increase of TMP for the both membranes. Moreover, the divalent ions have higher rejection percentage then monovalent ones (Fig. 4).

NP030 is negatively charged membrane for the whole pH range while NF90 for pH greater than 4 [9]. Moreover, the saline rejection reached with NF90 membrane is higher than that obtained by the use of NP030 membrane (Fig. 4). This is due to the smaller size pore of NF90 in comparison with NP030. For instance, the sulfate rejection by NF90 was 99.75% at TMP of 4 bar while in the case of NP030 this value was only 43.04%. Concerning the monovalent ions, the rejection in NF90 was obviously higher than in NP030. For example, the chloride rejection by NF90 was 90.15% at TMP of 4 bar while this value was only 2.27% when NP030 was used. This is in agreement with the bibliography. Nicolini et al. [9] has found, when studying the membrane salt rejection at the isoelectric point, that the electrical exclusion effect contributes strongly in the ionic permeation mechanism for

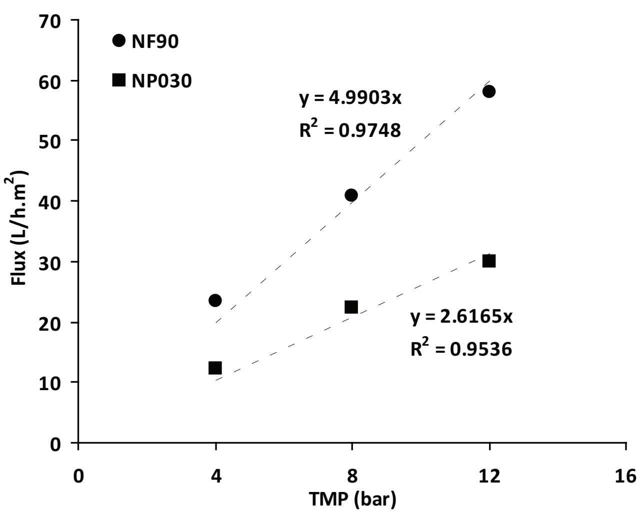

Fig. 2. Deionised water permeate fluxes as a function of TMP for NF90 and NP030 membranes.

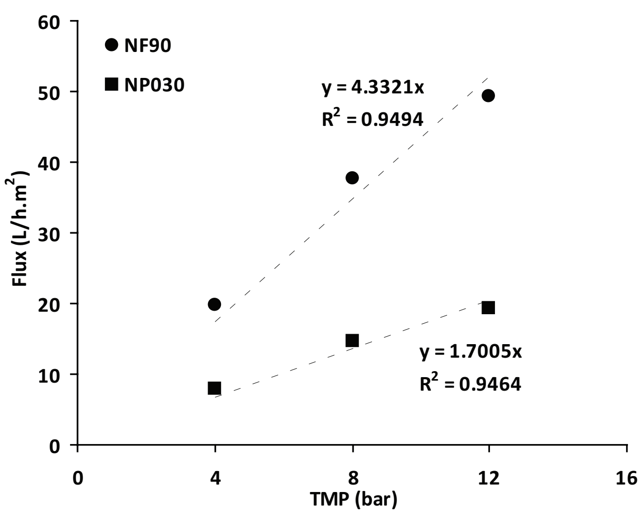

Fig. 3. Permeate fluxes as a function of TMP during desalination of well water by NP030 and NF90 membranes.

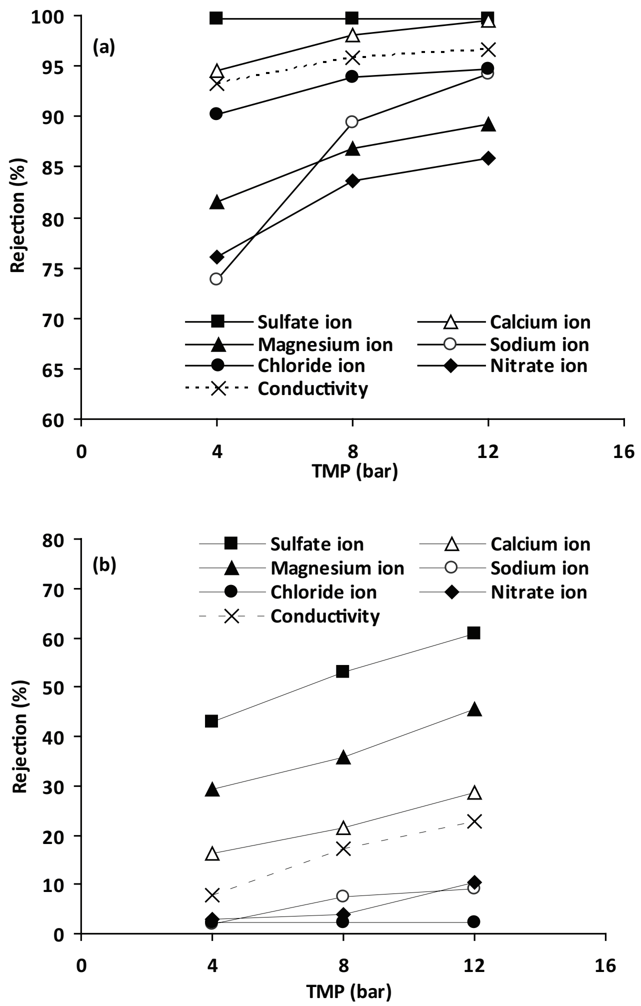

Fig. 4. Effect of TMP on salts retention for (a) NF90 and (b) NP030 membranes.

Table 3
Characteristics of well water before and after NF (VCF = 2.58)

Parameters	Feed before NF	NF Permeate
Conductivity (μS/cm)	1223	40.3
pH	7.73	7.29
Na^+ (mg/L)	64.09	7.00
Cl^- (mg/L)	106	12
SO_4^{2-} (mg/L)	350	2
NO_3^- (mg/L)	27.6	3.5
Ca^{2+} (mg/L)	132	4
Mg^{2+} (mg/L)	41.6	6.5

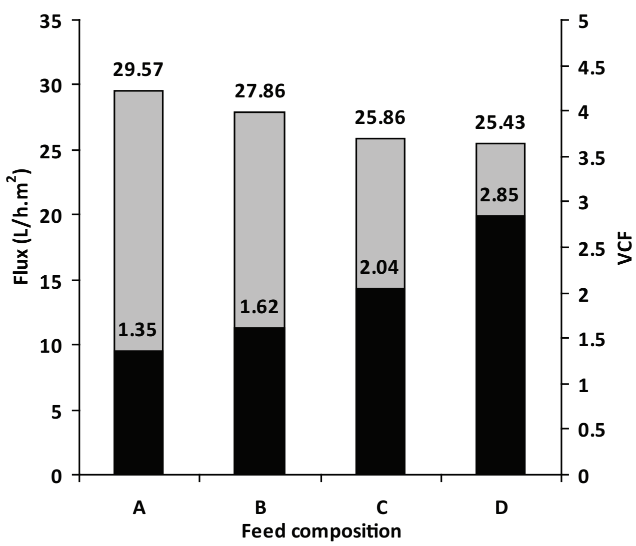

Fig. 5. Evolution of permeate flux of well water, *through* NF90 membrane, and VCF vs. ions feed concentration.

NP030 while the small pore size of NF90 is the main contributor in salt rejection. Consequently, the low MWCO of NF90 combined with the electrical exclusion leads to high salt rejection for this membrane in comparison with NP030.

Since both membranes are negatively charged in pH near to 7.5, co-ions (anions) are repulsed from the membrane while the counter-ions (cations) are attracted based on Donnan exclusion. However, because sulfate possesses higher ionic charge and higher ionic radius than chloride and nitrate, these latest have higher permeation and consequently they are transported through the membrane to maintain the electro-neutrality. That explains the high rejection of sulfates ions in comparison with chloride and nitrate in both membranes. Comparable results were found by other authors [9,15,16].

Regarding the divalent cations, the results show that they have higher rejection then monovalent cations for both membranes. The same behavior was concluded in several researches with synthetic mixture composition [6,15,17]. It was observed that the presence of Ca^{2+} and Mg^{2+} with Na^+ in the solution leads to decrease of sodium rejection and increase in divalent cations rejection in comparison with single salt solution. This is due in one hand to the steric effect which affects the cation permeation by resulting a diminution of Na^+ rejection. On the other hand, the small pores of NF90 lead to higher rejection in comparison with N030, because the hydrated diameters for calcium and magnesium are 0.824 nm and 0.856 nm, respectively [3].

3.3. Feed concentration effect

3.3.1. Volume concentration factor

The effect of the feed concentration on the NF membrane capacity is an important parameter to study. As NF90 membrane had the highest salts rejection, it was chosen for this study at a constant TMP of 8 bars. The well water was treated by NF90 membrane without permeate stream recirculation in order to concentrate it. During the filtration, samples were taken from permeate and the feed tank in order to assess the evolution of the concentration and the rejection percentage until reaching a volume concentration factor (VCF) of 2.58. The results are shown in Table 3.

Fig. 5 shows that the permeate flux decreased with increasing ions concentration in the well water (Table 4). A similar behavior was observed by Pérez-González et al. [16]. They have found that membrane permeability decrease exponentially with saline concentration. However, concerning the evolution of the rejection percentage, it seems clear from Table 5 that the membrane rejection was not affected by increasing the feed concentration. In order to obtain more significant results, more concentrated waters, brackish 1 and brackish 2, were studied.

3.3.2. Study with different brackish concentrations

The brackish waters were treated with the NF90 membrane at 8 bars. From Table 6, it seems clear that when the water was more concentrated the rejection was still high, however the permeate flux decreased significantly (Fig. 6). In this case, higher transmembrane pressure is needed in order to reach higher permeate flux. This is due to the concentration polarization phenomenon. The water flux changes due to the variation in osmotic pressure caused by the concentration of ions in samples. To maintain a constant volume flux, the TMP across the membrane has to be adjusted at every solute concentration [14]. Concerning the ionic rejection it was observed that there was no significant change. This result confirms that the small pore size of NF90 is the main factor that characterizes the salt rejection of this membrane.

In order to avoid that salts precipitation also participates in flux decrease, the membrane should be cleaned after desalination experiments to keep permeate fluxes similar to the original one (before brackish filtration).

The NF membrane was cleaned in two steps. Firstly, the impurities in the membrane surface were rinsed by passing deionised water through the membrane module without transmembrane pressure. The osmotic conductivity in the outlet of the pilot plant was checked each time until obtaining the initial conductivity of deionised water. Then, a transmembrane pressure of 1 bar was used in the second step in order to eliminate the salts that still left in the NF membrane. In the same way, the conductivity was checked until reaching the initial value.

Table 4
Water samples having different concentrations of ions

Ions (mg/L)	Feed composition			
	A	B	C	D
SO_4^{2-}	370	380	430	500
Cl^-	118	121	162	177
NO_3^-	29.4	30.4	31.5	33.9
Ca^{2+}	142	151	167	213
Na^+	65.51	67.01	74.15	80.98
Mg^{2+}	48.4	53.3	63.3	70.3

Note: A (VCF = 1.35); B (VCF = 1.62); C (VCF =2.04); D (VCF = 2.85).

Table 5
Rejection of ions vs. feed concentration

Anions	Feed concentration (mg/L)	Rejection (%)	Cations	Feed concentration (mg/L)	Rejection (%)
SO_4^{2-}	350	99.43	Ca^{2+}	132	96.97
	370	99.73		142	97.18
	380	99.74		151	97.35
	430	99.77		167	97.60
	500	99.80		213	98.12
Cl^-	106	89.62	Mg^{2+}	41.6	77.40
	118	89.83		48.4	80.79
	121	90.91		53.3	86.87
	162	92.59		63.3	88.94
	177	93.22		70.3	90.75
NO_3^-	27.6	84.78	Na^+	64.09	89.44
	29.4	87.76		65.51	89.52
	30.4	88.16		67.01	89.98
	31.5	89.52		74.15	91.28
	33.9	89.68		80.98	91.35

Table 6
Rejection rate of brackish waters ions using NF90 membrane

Parameters	Brackish 1		Brackish 2	
	Permeate	Rejection (%)	Permeate	Rejection (%)
Na^+ (mg/L)	17.42	91.24	108.9	86.15
Cl^- (mg/L)	10	96.94	135	95.50
SO_4^{2-} (mg/L)	1	99.90	8	99.77
NO_3^- (mg/L)	4.9	88.42	25.9	81.57
Ca^{2+} (mg/L)	4	99.35	12	99.04
Mg^{2+} (mg/L)	8.5	96.15	16.8	97.79
Conductivity (µS/cm)	105.6	97.14	638	93.91

To evaluate the effectiveness of the membrane cleaning, the NF90 membrane was taken from the plant after cleaning it and samples were analysed with FE-SEM/EDX to observe eventual scaling problems. As the pictures of the membrane before and after desalination experiments show (Fig. 7), it seems clear that there is salt precipitation. However, Fig. 8 shows a slight difference between the water permeation lines related to the new membrane

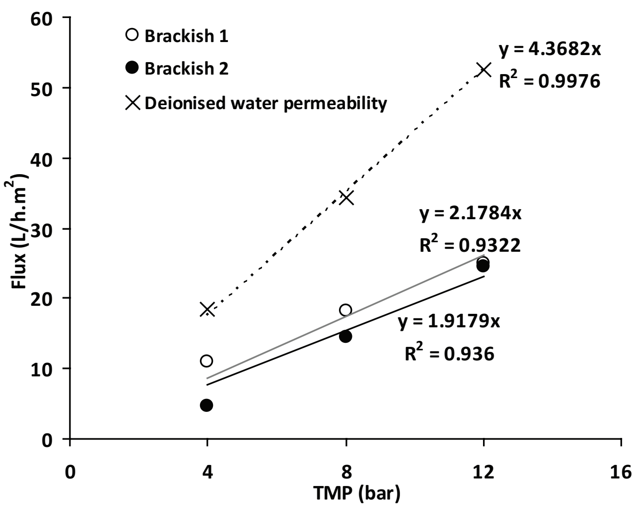

Fig. 6. Evolution of the permeate flux vs. TMP when treating brackish waters.

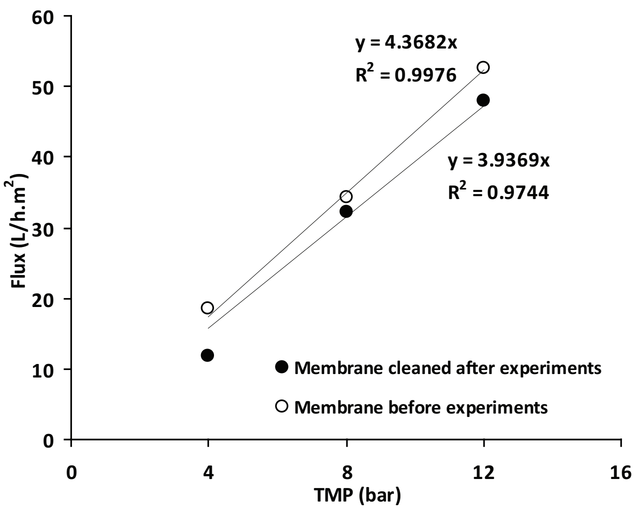

Fig. 8. Effect of cleaning process on the deionised water permeability of NF90 membrane.

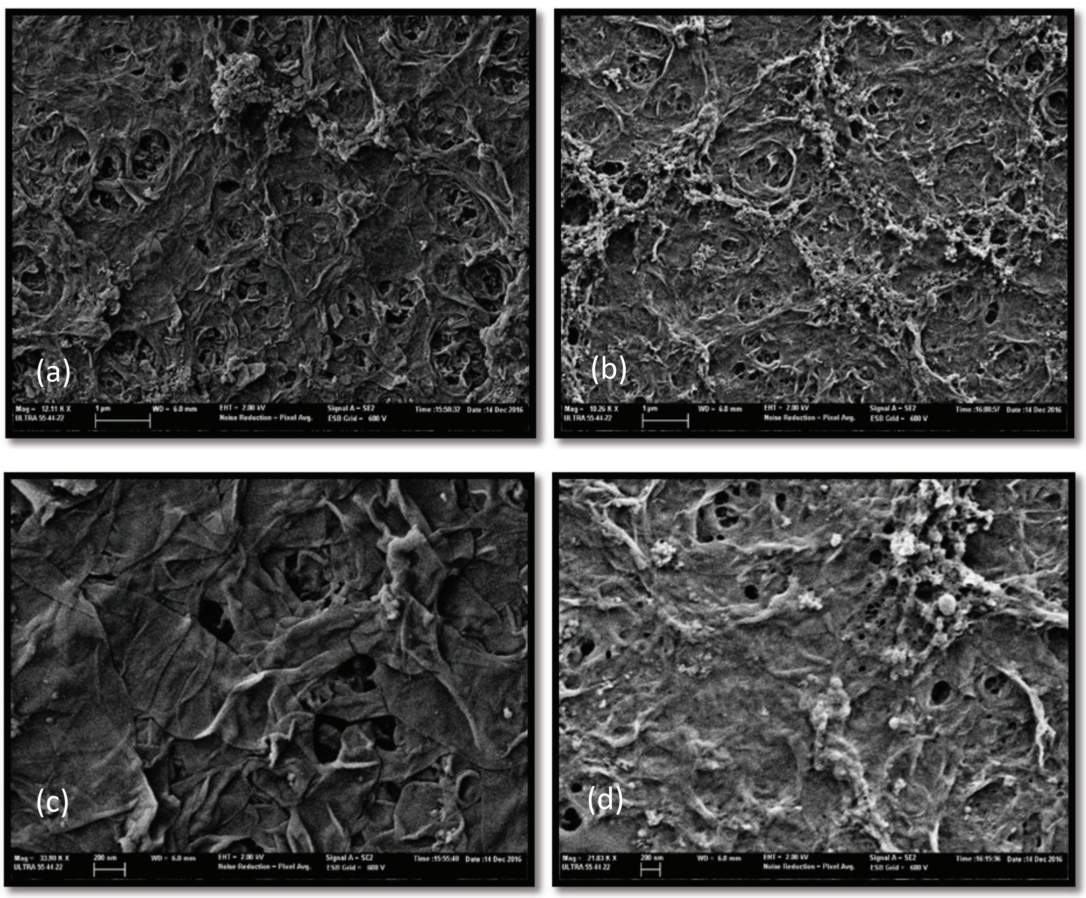

Fig. 7. FE-SEM images of the membrane NF90: (a) and (c) New membrane before using it; (b) and (d) Membrane after desalination.

and that cleaned after the desalination tests. Moreover, the calculation of membrane resistance showed that there is a minor increasing from 7.04×10^{13} to 8.92×10^{13} m^{-1} for 8 bar. This obviously reflects the effectiveness of the cleaning method applied. The results from EDX, of different areas of the surface of the membrane, showed that carbon and oxygen were the majority elements on the membrane surface (data not shown). These are the main components of the filter material (from provider). In some cases, sodium and chloride appears in very small weight percentage since these ions are present in small amounts, hardly appearing in the EDX spectrum. Results showed no presence of elements such as calcium and magnesium on the membrane surface. However, a small residual amount of sulfur was observed on some areas of the membranes but it remains without remarkable effect on the original water permeability of the membrane.

4. Conclusion

In this work, it was demonstrated that the membrane mechanism separation is the main factor that contribute in NF performance. It was found that the high permeability and salt rejection of NF90 in comparison with NP030 was mainly due to the small pore size and hydrophilicity of the membrane. Moreover, when assessing the efficiency of NF90 while increasing ions concentration, it was found that the permeability decreased without significant effect on the membrane rejection. This result confirms that the small pore size of NF90 membrane is the main factor that leads to high salt rejection. For all studied brackish waters, the rejection of ions was more than 80% for NF90. Concerning the permeate flux decreasing, this is due to the concentration polarization that changes the water flux due to the variation in osmotic pressure.

Finally, by observing the surface of the new and used membrane by FE-SEM/EDS, it was found that there is a salt precipitation on the membrane surface after filtration experiments. However, by checking the membrane permeability after the cleaning step, it was found that the permeability was slightly lower than the initial one (10%) which demonstrate the effectiveness of the cleaning process.

In general terms, it can be concluded that nanofiltration process of brackish water could be a competitive alternative to the more used RO processes. With NF90, both flux and ions rejection were high and lower transmembrane pressures than in RO processes were required.

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