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Additional Information

Desalination of brackish water and reverse osmotic retentate by the use of nanofiltration membranes. Investigation on the effects of TMP and salts concentration on the performance of the treatment process

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ABSTRACT

Nanofiltration membrane with low-pressure operating conditions and high flux permeability seems to be an attractive alternative for water softening and desalination. In this work, two commercial flat sheet nanofiltration membranes (NF90 and NP030) were evaluated. Real brackish waters and reverse osmosis retentate were analysed and used in this purpose. The effect of transmembrane pressure (TMP) and concentration on the permeate flux and salts rejection were investigated. The effect of TMP and ions concentration was studied for NF90 because this membrane has higher rejection and permeability compared to NP030. The results obtained demonstrated that the permeate flux and the rejection increased with increasing in TMP, while the permeability decreased with increasing in water ions concentration without significant effect on the membrane rejection. Concerning the salts rejection, NF90 results showed that the sulfates had the highest rejection (99%) compared to the other ions. This was due to separation mechanism that occurs during the treatment to keep the electro-neutrality. For studied brackish waters, the rejection of all ions was more than 80%. The scaling problem was studied by checking the permeability after each cleaning step and by observing the surface of the employed membrane by FE-SEM/EDS. The images showed salt precipitation on the membrane surface after filtration experiments. When cleaning the NF90 membrane by the use of distilled water, the membrane permeability was slightly lower than the initial one.

Keywords: Nanofiltration; Desalination; 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 hold many advantages such as operating at low pressures in comparison with the reverse osmosis, having high permeate flux and high rejection of bivalent 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 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. A recent paper co-authored by Nicolini et al. [6] 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. These authors evaluated rejection of salts typically present in seawater (NaCl, Na₂SO₄, MgSO₄, K₂SO₄, and CaSO₄) at various transmembrane pressures. They found that sieve mechanisms, ionic

electrical exclusion, dielectric effects and permeate flux coupling of ions to keep electroneutrality contribute simultaneously to ions selective rejection. Additionally, the results revealed that permeation with mixture of mono and divalent ions through membranes with higher pore size, NP010 and NP030, showed that high sulfate exclusion leads to increase in the rejection of all cations present in the solution. Conversely, for NF90 membrane, steric effects are predominant and high sulfate rejection leads to reduction of monovalent ions rejection. On the other hand, Hilal et al. [7] 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). Highly concentrated (NaCl) salt solutions up to 25,000 ppm, a salinity level similar to that of seawater, were used. The main parameters studied were feed pressure and salt concentration. According to their results, for a salinity of 5000 ppm and a pressure of 9 bars, the experimental results showed that NF90 could achieve a salt rejection up to 95%, whereas its rejection dropped to 41% at a salinity of 25,000 ppm and the same pressure. Rejection levels achieved by NF270 have been in range of 11–29%, while N30F gave the lowest rejection in the range of 3–6%. Galanakis et al. [8] studied the nanofiltration of brackish groundwater by using a polypiperazine membrane. They collected samples of different hardness and salinity values (up to 762 mg CaCO₃ and 1803 mg NaCl/L, respectively) and treated them under low transmembrane pressures (6–10 bar), in a cross-flow nanofiltration module. According to their results, the membrane was able to provide high hardness retention coefficients (70–76%), satisfactory permeate fluxes (15–47 L/m²·h) and high mineral fouling resistance. A disadvantage of the process was the relatively low removal of salinity (44–66%) for brackish groundwaters that restricts the application in samples possessing salinity not much higher than ~1100 mg NaCl/L. Schaep et al. [9] 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%. Moreover, a rejection of 94% was found for calcium. Concerning the effect of the temperature and the recovery on the permeate flux, they supposed that it could be described by one equation, relating the flux to water viscosity and net pressure difference. Additionally, it was found that a small concentration of organic compounds can cause a substantial flux decrease. Recently, Song et al. [10] evaluated the nanofiltration softening performance of two NF membranes denoted as DK (termed as NF1) and DL (termed as NF2). The influences of transmembrane pressure (0.6–2.2 MPa), inlet tangential flow velocity (0.087–0.384 m/s) and feed water temperature (7–35°C) on the softening efficiencies were investigated. Their experimental results revealed that the calculated total hardness and the bivalent ion (SO_4^{2-} , CO_3^{2-} , Mg^{2+} and Ca^{2+}) concentrations in NF2 product water on the same operation were all slightly lower than those in NF1 permeate in two schemes. Additionally they found that pH value of NF permeate decreased prominently than those in feed water with increasing of transmembrane pressure, inlet tangential flow velocity or decreasing of feed water temperature within the testing scope. Pérez-González et al. [11] examined experimentally and theoretically, the effectiveness of NF for sulfate/chloride separation in highly concentrated saline solutions. The study was carried out with the NF270 (Dow Filmtec) membrane. The latter proved to be very effective providing sulfate rejection in the range 75–96% and chloride rejections between 2% and 11%. Moreover, they found that the increasing in the salt concentration (NaCl) reduced the retention ability of the membrane due to the decrease in Donnan effect.

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. This leads to filtration optimization in order to obtain high permeate flux with high rejection of different ions present in the brine water.

2. Materials and methods

2.1. Feed samples

Well water and reverse osmotic (RO) retentate (Brine 1) supplied by a tanning industry in Valencia (Spain), and RO retentate concentrated by evaporation (Brine 2) were treated in this work by nanofiltration. 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 set-up

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 and pump for feed circulation in a horizontal membrane module. Two manometers are put in the inlet and outlet of the membrane to have the transmembrane pressure (TMP) which is controlled by a valve. 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. The membrane material of NP030 is polyethersulfone (PES) with operating pH range between 0–14 and 95°C as maximal operating temperature. Concerning the membrane material of NF90 it's polyamide with operating pH range between 4–11 and 35°C as maximal operating temperature.

Before starting the desalination experiments, the NF membranes were immersed in osmotic water for 24 hours. Then, the NF system was equipped with the membrane and pretreated with osmotic water as feed solution. NF90 was compacted at 12 bars for 4 hours 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 osmotic water at different TMP range from 4 to 12 bars 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 equation (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).

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 osmotic 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 2kV. In addition, several little areas in the used membrane for desalination were analyzed with the EDX at 20kV. 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 21p and EC- Meter GLP 31p (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 bars. Each pressure takes one hour with 0.07 m³/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 (Fig. 2). 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 [13]. In the other hand, different samples with different concentrations were used (Brines 1 and 2).

3. Results and discussion

3.1. Membranes permeability

It was found that deionised water permeate flux increased linearly with the operating pressure as expected and that the permeability coefficients are about 2.62 and 4.99 L/h.m².bar for NP030 and NF90 membranes, respectively (Fig. 3). 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 explain by its high hydrophilicity, compared with NP030, which play an important role in the water transport through the membrane [6]. 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 which in absence of solute, the osmotic pressure effect becomes zero and pure water flux becomes proportional to operating pressure difference across the membrane [14,15].

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 $7.43 \times 10^{13} \text{ m}^{-1}$ and $3.56 \times 10^{14} \text{ m}^{-1}$ for NF90 and NP030 respectively. These results are in agreement with those

obtained by Nicolini et al. [6]. According to the contact angle and energy free surface of the membranes, they expected a lower water transport resistance for NF90 compared to NP030.

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. 4 for NP030 and NF90 membranes. The effect of TMP on salts retention is presented in Fig. 5. 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. 5).

The saline rejection reached with NF90 membrane is higher than that obtained by the use of NP030 membrane (Fig. 5). 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 bars 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 bars while this value was only 2.27% when NP030 was used.

NP030 and NF90 are negatively charged membranes [6]. Hence, chlorate, sulfate and nitrate, which are co-ions, are repulsed from the membrane while the counter-ions are attracted. 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. Regarding the divalent cations (calcium and magnesium), the results show that they have also high rejection. This is due in one hand to small pores of the membranes that lead to size exclusion, because the hydrated diameters

for calcium and magnesium are 0.824 nm and 0.856 nm, respectively [3]. On the other hand, the divalent ions are not driven through the membrane in high proportion to keep the electro-neutrality.

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. In addition, after the experiments, the membrane permeability was checked again just after rinsing the membrane with deionised water. After each cleaning step, the permeability slightly decreased from the initial membrane permeability (Fig. 6).

Fig. 7 shows that the permeate flux decreased with increasing ions concentration in the well water (Table 4). 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 some cases such as for magnesium, chloride, sodium and nitrate there is a slight increase in the rejection percentage. This was due to the diminution of the water flow through the membrane.

3.3.2. Study with different brines concentration

Brine 1 and brine 2 were chosen to obtain more significant results. The brine 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. 8). In this case, higher transmembrane pressure is needed in order to reach higher permeate flux. This is due to the concentration polarization phenomenon. The latter affects membrane permeation by the change in composition at the membrane interface. In the presence of solutes in the feed, the TMP is reduced by the osmotic pressure difference between the feed at the membrane interface and the permeate (Eq. 4).

$$J_{\text{solvent}} = \frac{TMP - \Delta\Pi_m}{R_m} \quad (4)$$

where J_{solvent} is the solvent flux through the membrane (L/h.m^2) and $\Delta\Pi_m$ is the osmotic pressure (bar).

This phenomenon lead to decrease in the permeate flux. That is the reason why, when the feed concentration increases the permeate flux decreases.

To know whether flux decrease is due to salts precipitation, the membrane was taken from the plant 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. 9), it seems clear that there is salt precipitation that had led to the decreasing of the membrane permeability after each experiment. Thus, the membrane should be cleaned after desalination experiments to keep permeate fluxes similar to the original ones (before brines 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.

Fig. 10 shows a small difference between the water permeation lines related to the new membrane and that cleaned after the desalination tests. This obviously reflects the effectiveness of the cleaning method applied. The results from EDX (Fig. 11), of different areas of the surface of the membrane, showed that carbon and oxygen were the majority elements on the membrane surface. These are the main components of the filter material. 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 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 bars with three brine waters having different ions concentrations. The results obtained show that NF90 had the highest permeability and salt rejection for both mono and divalent ions. The results also showed that the permeate flux and rejection increased linearly with increasing in TMP. On the contrary, the permeate flux decreased with increasing salts concentrations. However, the salts rejection remained high for all the studied concentrations. A disadvantage of this filtration process was the scaling problem that decreased slightly the membrane permeability after filtration experiments. The membrane NF90 was characterized by a high permeability and allows reaching high rejection rates for waters from 1293 $\mu\text{S}/\text{cm}$ up to 3.69 mS/cm . For concentrated water up to 10 mS/cm , higher pressures were required in order to obtain higher permeate flux.

In general terms, it can be concluded that NF of well water could be a competitive alternative to the more used RO processes. With NF 90 both flux and ions rejection were high and lower transmembrane pressures than in RO processes are required.

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Table 1. Physicochemical characteristics of feed waters.

Parameters	Well water	Brine 1	Brine 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

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 [12] ; ^c From [6]

Table 3. Final brine characterization (VCF= 2.58)

Parameters	Feed before NF	NF Permeate
Conductivity ($\mu\text{S}/\text{cm}$)	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

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 percentage of the ions in the brine waters after treatment with NF90 membrane.

Parameters	Brine 1		Brine 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

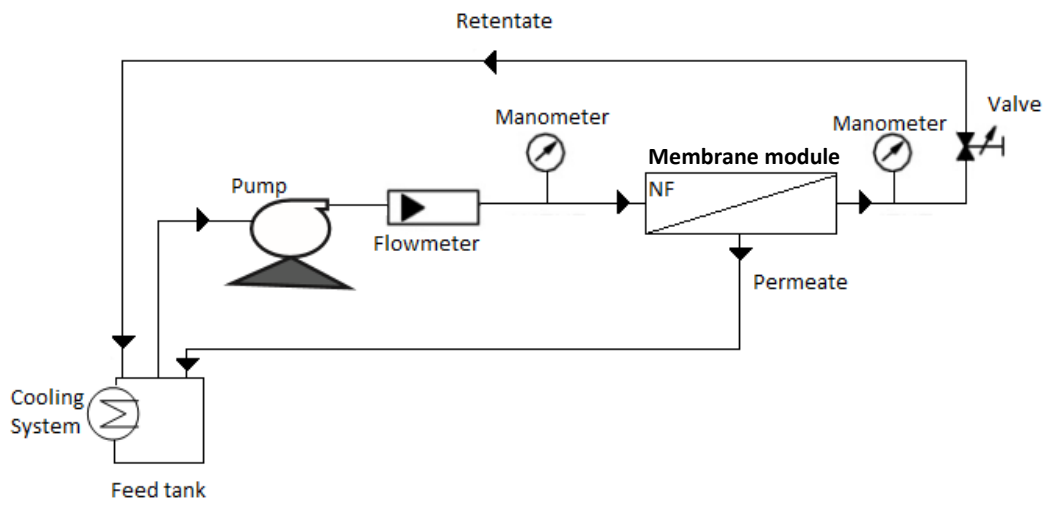


Fig. 1. Schematic diagram of Nanofiltration pilot plant.

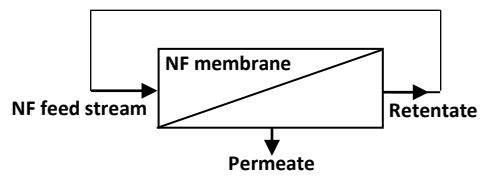


Fig. 2. Feed concentration without recirculation.

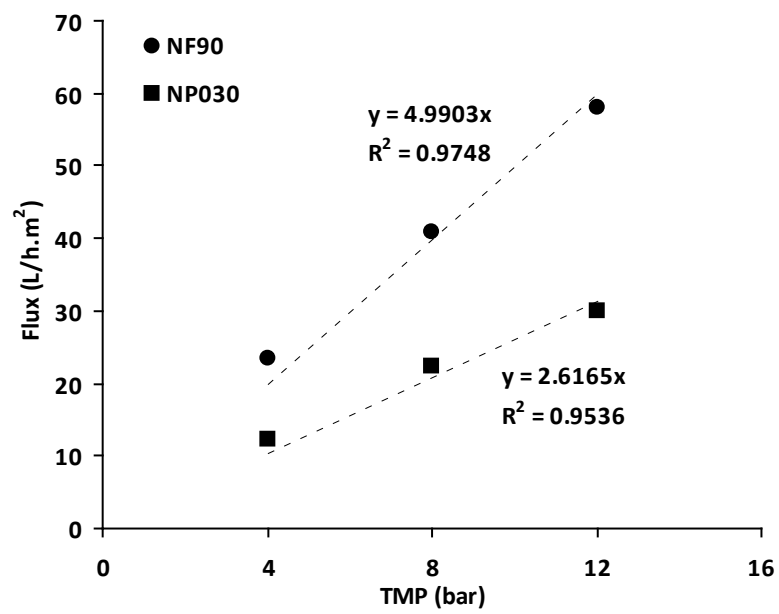


Fig. 3. Deionised water permeate fluxes as a function of TMP for NF90 and NP030 membranes.

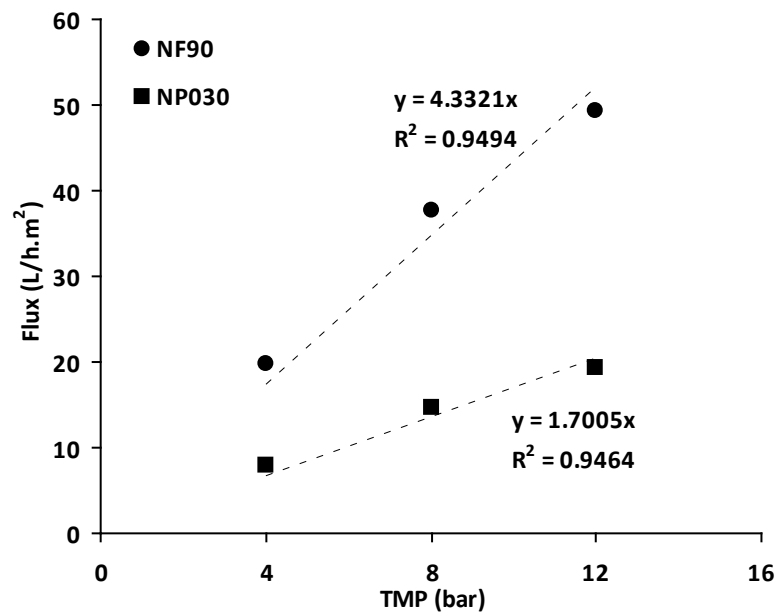


Fig. 4. Permeate fluxes as a function of TMP during desalination of well water by NP030 and NF90 membranes.

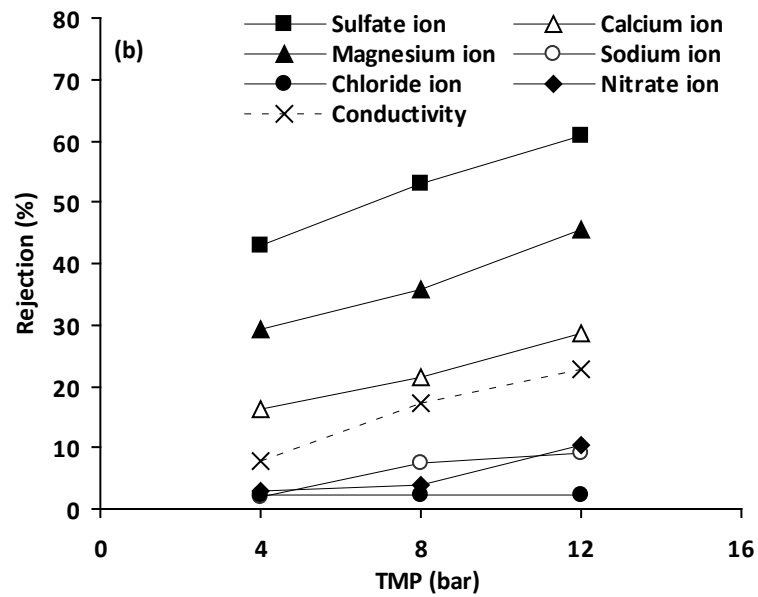
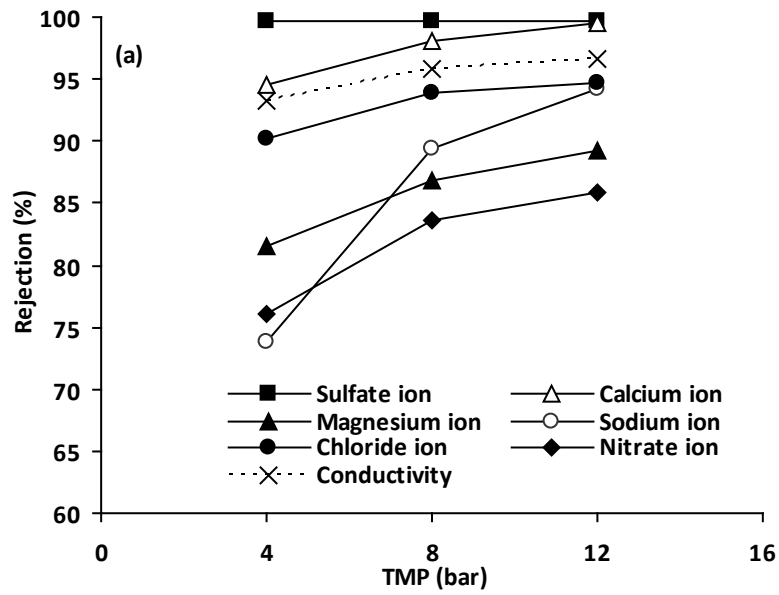


Fig. 5. Effect of TMP on salts retention for (a) NF90 and (b) NP030 membranes.

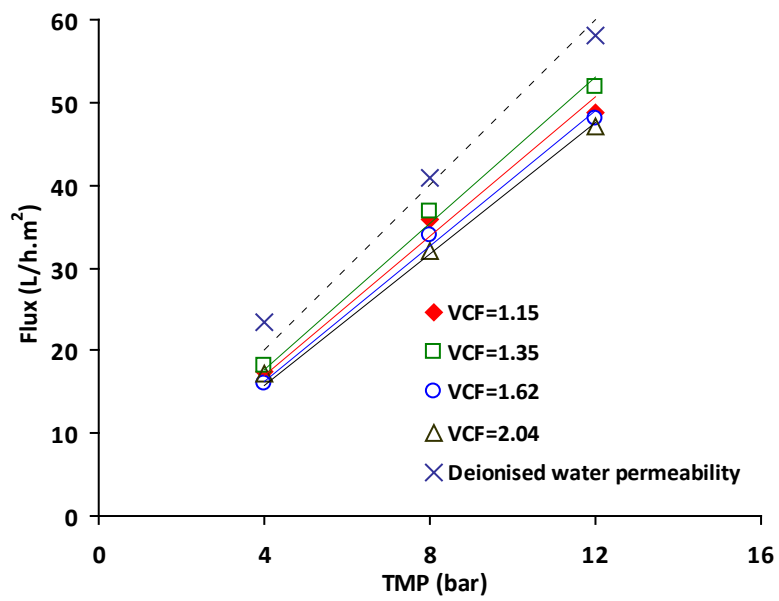


Fig. 6. Evolution of the permeate flux through NF90 membrane after cleaning step vs. TMP for each VCF.

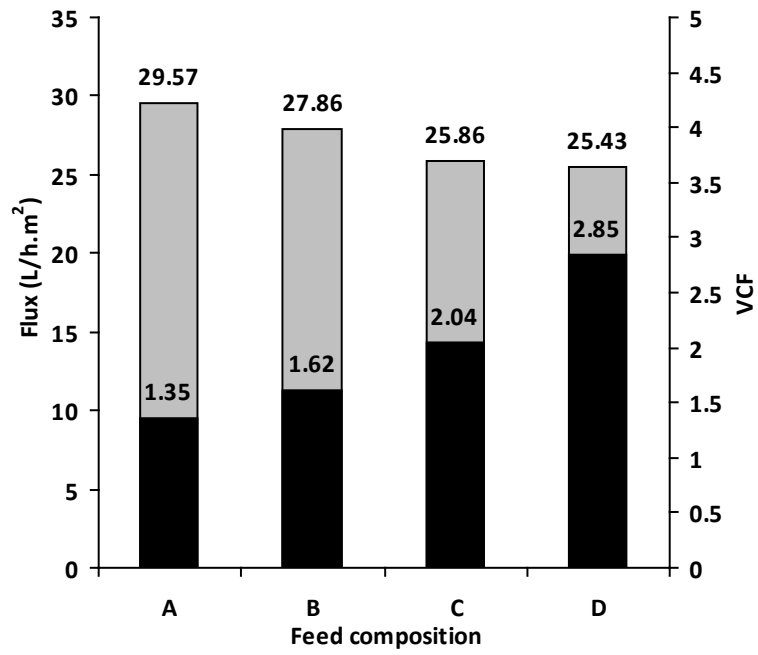


Fig. 7. Evolution of permeate flux of well water, through NF90 membrane, and VCF vs. ions feed concentration.

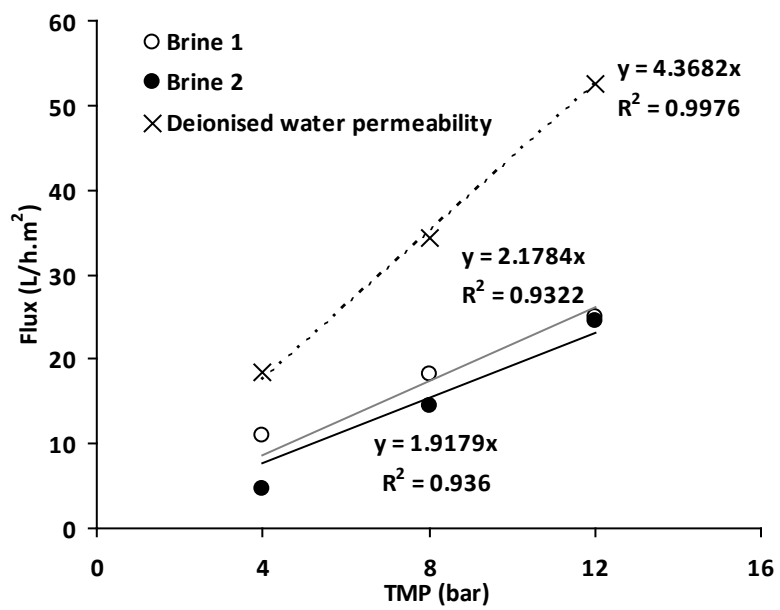


Fig. 8. Evolution of the permeate flux vs. TMP when treating brine waters.

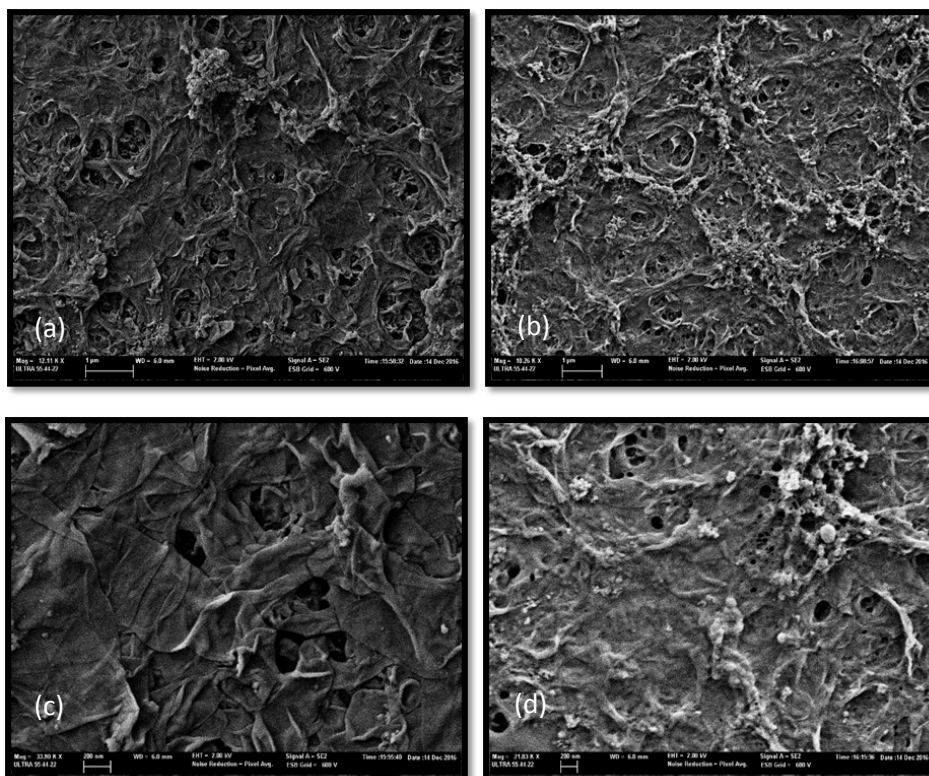


Fig. 9. FE-SEM images of the membrane NF90: (a) and (c) New membrane before using it; (b) and (c) Membrane after desalination.

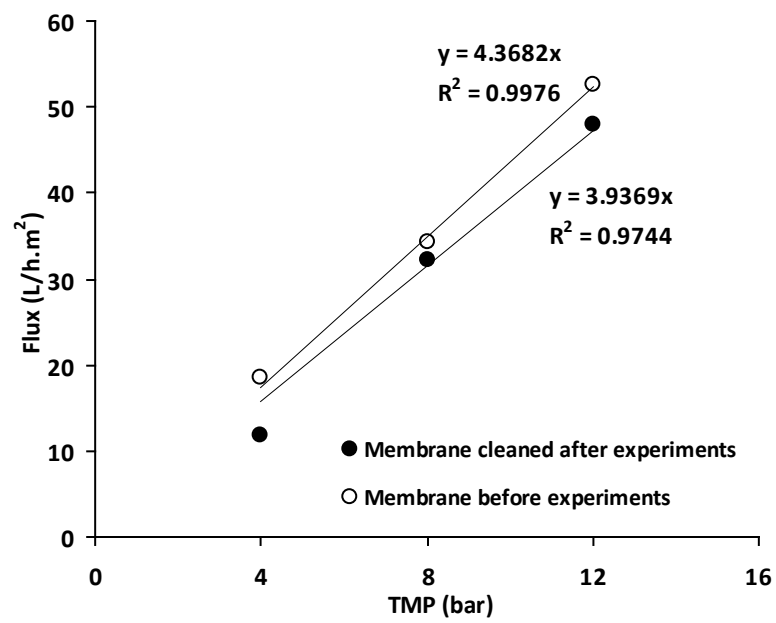


Fig. 10. Effect of cleaning process on the deionised water permeability of NF90 membrane.

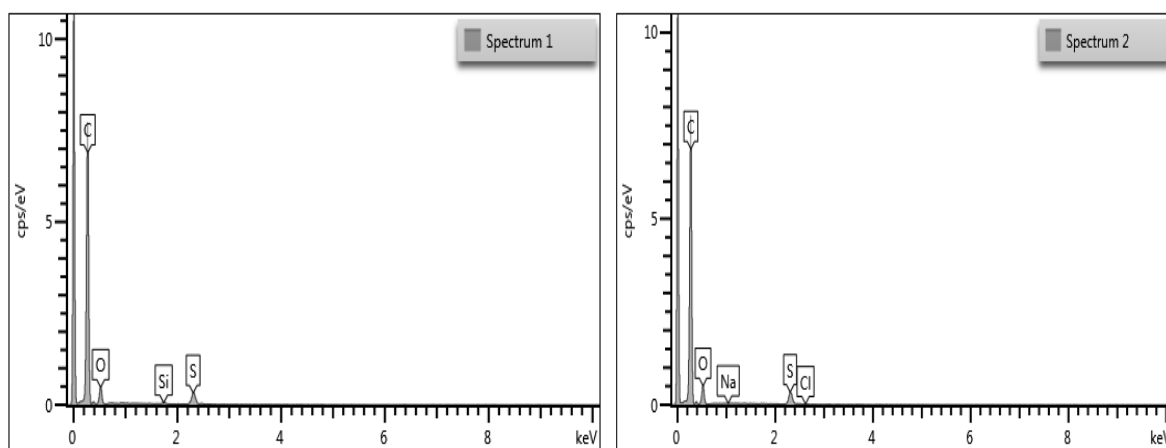


Fig. 11. EDS spectra of the NF90 membrane used for desalination after cleaning process.