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Static cleaning tests as the first step to optimize RO membranes cleaning procedure

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ABSTRACT

Membrane fouling is an intrinsic problem of membrane technology which affects process performance and causes a substantial rise of the operating costs. This becomes central in seawater desalination processes. This study is focused on applying a membrane chemical cleaning protocol to obtain the most adequate cleaning conditions to recover the permselective properties of an irreversibly fouled reverse osmosis membrane from a seawater desalination plant. The research was carried out in three steps: fouling characterization by scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDX), atomic force microscopy, attenuated total reflectance Fourier transform infrared spectroscopy and elemental analysis; static cleaning tests; and characterization of the membrane surface after the cleaning step. The cleaning process was performed in a static way as a preliminary step. Five of the most widely used cleaning agents were tested (NaOH, citric acid, HCl, sodium dodecyl sulfate [SDS], and ethylenediaminetetraacetic acid disodium salt) at a broad range of concentrations and two different temperatures (25 and 40˚C). SEM-EDX analysis showed that foulant compounds were mainly silica, iron silicate, and aluminum silicate, which indicated that fouling was mainly colloidal and inorganic. The best cleaning results were achieved by SDS 0.5% w/w at 40˚C, as the greatest recovery of the membrane permselective properties (permeability and salt rejection index) was obtained.

Keywords: Reverse osmosis; Desalination; Chemical cleaning; Fouling; Static tests

1. Introduction

Membrane fouling is the main problem present in the operation of reverse osmosis (RO) desalination plants, causing a decrease in process performance. The most important consequences of membrane fouling are: permeate flux drop, salt rejection reduction, power costs increase due to the need to raise the driving force to keep the operating conditions, more cleaning frequency, and reduction of the membrane life, what entails a considerable increase of the operation costs. In fact, the costs related to the RO membrane cleaning can represent up to 50% of the total operation costs in seawater desalination plants [1].

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Therefore, investigating the operating conditions of the installation is of great interest, in order to minimize the membrane fouling consequences.

Currently, a lot of studies focused on the analysis of the fouling phenomenon have been performed. Most of them investigate substance deposition mechanisms [2–5], the type of compounds that show greater contribution to this phenomenon, as well as their interaction with the membrane active layer [6].

Membrane autopsy is the most efficient method to characterize membrane fouling and optimize membrane cleaning processes. This is a destructive characterization method, offline, and quite expensive. Due to those disadvantages, this method is only recommended when complex fouling is present; when the cleaning protocols do not achieve an acceptable permeate flux recovery [7,8]; or when the cleaning process becomes harmful to the membrane active layer.

Membrane autopsy consists of several tests and characterization methods able to identify the fouling nature. The most widely used techniques are: scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDX), atomic force microscopy (AFM), attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR), and target energy-dispersive X-ray analysis. Several authors have employed these techniques to identify the chemical nature of the fouling layer and its formation mechanisms [9–14], as well as to optimize the cleaning process.

Although a large number of membrane cleaning methods are available [15], the chemical method is the most widely used [16,17]. The parameters that have greater influence on the chemical cleaning efficiency are: temperature [18], cleaning agent concentration, operating pressure, cross-flow velocity during cleaning step, and cleaning step duration [19,20].

The chemicals that are most often used for the chemical cleaning of RO membranes are acids, alkalis, surfactants, and chelating agents. Inorganic acids easily dissolve inorganic fouling deposits, whereas organic acids, such as citric or oxalic acid, dissolve organometallic foulants [21]. On the other hand, alkaline solutions are more efficient removing organic matter, since they are able to dissolve weakly acid organic matter (which mainly contains phenolic or carboxylic functional groups) and fragment polysaccharides and proteins into smaller sugars and amides [8–22]. Chelating agents are used for complexing metallic ions and are commonly applied in combination with other compounds, such as NaOH or sodium dodecyl sulfate (SDS) [23]. Anionic surfactants, such as SDS, are very efficient in removing organic foulants, since they eliminate or significantly reduce the adhesion forces between foulants and membrane surface [24]. These surfactants are very suitable to clean RO membranes used for wastewater treatment. Their efficiency to remove colloidal fouling has also been probed [25] and has been attributed to their emulsifier power, since they modify the surface tension of water and facilitate the detachment of fouling layers [26].

Due to the large variety of cleaning protocols and operating conditions available, analyzing each particular case (fouling type and membrane nature) becomes critical. The most suitable cleaning protocol can be obtained by testing and optimizing the cleaning solution and the operating conditions during the cleaning step (temperature, pressure, cross-flow velocity, and duration).

The objective of this work is to determine the most suitable protocol to clean RO membranes that show irreversible fouling after a long time of operation in a seawater desalination plant. For this purpose, static cleaning tests have been performed as a preliminary step.

2. Materials and methods

2.1. Membranes

In this work, a spiral-wound RO membrane module with a diameter of 8 inches, Hydranautics SWC3 (USA) was used. It had been retired from a desalination plant that treated seawater with the physicochemical composition shown in Table 1.

The samples to perform the characterization of the fouling layer and the cleaning tests were pieces cut from the commercial RO membrane.

Table 1
Chemical composition of the feed stream (seawater) treated in the desalination plant where the membrane module operated

Parameter	Value	Units
pH	7.51	pH unit
Conductivity 20°C	50,700	μS/cm
Chlorides	21,610	mg/L
Sodium	12,210	mg/L
Calcium	440	mg/L
Carbonates	0	mg/L
Bicarbonates	240	mg/L
Sulfates	3,200	mg/L
Boron	5.1	mg/L
Iron	14	μg/L
Barium	97.7	μg/L
Manganese	9.6	μg/L

2.2. Membrane surface characterization

Firstly, the characterization of membrane fouling was carried out by SEM-EDX (Jeol JSM-6300, Japan) and AFM (Veeco Multimode, USA). For this purpose, 24 membrane samples (12 samples from a new membrane and 12 samples from the fouled membrane) were analyzed. Eight of these samples were gold-coated to perform the SEM characterization, eight of them were carbon-coated for the SEM-EDX analysis, and the remaining eight samples were analyzed by AFM. A quantitative chemical analysis of the fouling layer deposited on the membrane surface was performed by the SEM-EDX characterization. AFM images were processed by Nanoscope software (NanoScope Services Ltd, UK) to determine different roughness parameters: R_a (average roughness), R_q (mean square roughness), and R_{max} (maximum ridge and valley height).

In addition, the fouling layer was analyzed by means of ATR-FTIR spectroscopy (Nicolet 710, USA). For this purpose, 600 mg of the fouling layer was collected from 0.75 m^2 of membrane surface.

Organic fouling was also analyzed by using an EuroEA C,H,N,S Elemental Analyzer (Germany). To perform the elemental analysis, approximately 1 mg of the collected fouling layer was used.

SEM-EDX and AFM were also used to analyze the membrane surface after the cleaning tests in order to check the efficiency of the cleaning protocol to remove the fouling layer from the membrane surface.

2.3. Cleaning tests and characterization of the permselective properties

A protocol to clean and characterize the membrane properties was defined. Several parameters that affect the cleaning efficiency were considered, such as the cleaning agent, its concentration, and temperature. This protocol was composed of four steps, shown in Fig. 1: cleaning solution preparation, static cleaning test, rinsing with water, and permselective properties characterization test.

Five cleaning agents, eight concentrations and two temperatures (25 and 40°C), were tested, as it is shown in Table 2. The selection of the cleaning agents was carried out from a revision of the cleaning processes in the literature. Every cleaning protocol was tested on eight-membrane samples and the results displayed are the average values (the mean relative error was 2.88%).

Additionally, distilled water at the two temperatures tested was included as cleaning agent in order to determine the effect of the operating temperature on the membrane cleaning without the addition of any chemical agent.

Static cleaning tests were carried out according to the methodology defined by Arnal et al. [27,28]. The cleaning consisted of soaking the samples into the cleaning solution for 1 h at constant temperature. After that, the samples were rinsed with distilled water for 1 h, renewing the water every 20 min.

Once the cleaning step was performed, the characterization of the membrane permselective properties (permeate flux and salt rejection index [SRI]) was carried out in order to determine the process efficiency. The characterization test was performed according to the manufacturer test conditions (55 bar, 32,000 mg/L of NaCl and 25°C). The test lasted 1 h. During the test, permeate flux (J_P) and SRI were measured every 15 min. A diagram of the pilot plant used for membrane characterization is shown in Fig. 2.

The pilot plant employed to characterize the permselective properties of the membrane was composed of the following elements: 100 L feed tank, 25 μm microfilter (Cintropur NW32, Airwatec SA, Belgium), high pressure pump (CAT 3CP1241, USA), and a plate-and-frame membrane module with capacity for eight-membrane samples. This pilot plant includes manometers before and after the membrane module, a thermometer, a flowmeter, and pressure regulating valves to control the process.

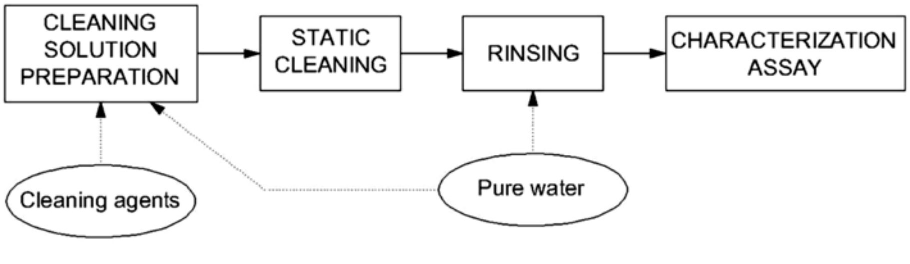


Fig. 1. Block diagram of the experimental methodology followed to carry out the cleaning tests.

Table 2
Cleaning solutions tested at 25 and 40˚C

Cleaning agent*	Concentracions tested (% w/v)							
Citric acid	0.01	0.05	0.1	0.2	0.5	1	2	4
Disodium salt of ethylenediaminetetraacetic acid (Na_2-EDTA)	0.01	0.05	0.1	0.2	0.5	1	2	4
Sodium dodecyl sulfate (SDS)	0.01	0.05	0.1	0.2	0.5	1	2	4
NaOH	0.01	0.2	0.4	0.8	2	4	8	16
HCl	0.01	0.02	0.04	0.08	0.1	0.2	0.3	0.6

*All chemicals were supplied by PANREAC (Spain).

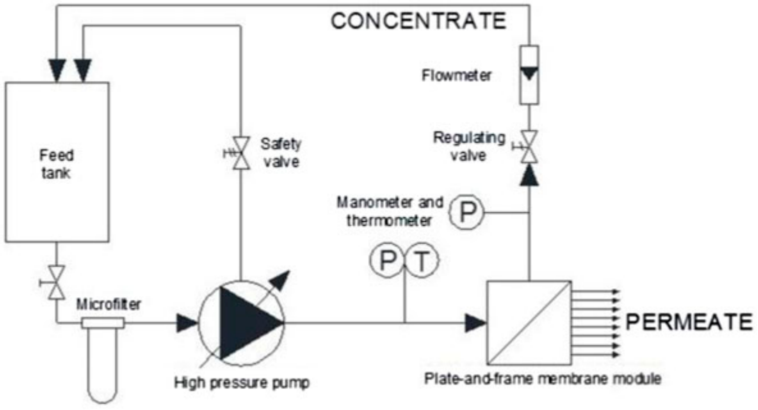


Fig. 2. Flow chart of the pilot plant used for membrane samples characterization.

The cleaning efficiency was calculated by means of the percent of recovery of permeate flux and SRI with respect to the blank (fouled membrane), for every temperature tested, according to these equations:

$$J_{\mathrm{p}}\,\text{recovery}\,(\%) = \frac{J_{\mathrm{p}} - J_{\mathrm{p0}}}{J_{\mathrm{p0}}} \times 100 \tag{1}$$

$$\text{SRI recovery}\,(\%) = \text{SRI} - \text{SRI}_0 \tag{2}$$

where J_{P} represents the membrane permeate flux after static cleaning, J_{P0} represents the permeate flux of the fouled membrane (considered as blank) for every temperature tested (25 and 40˚C), SRI represents the SRI of the cleaned membrane, and SRI_0 represents the rejection index of the fouled membrane for each temperature tested (25 and 40˚C).

Table 3
Results from elemental analysis of the fouling layer

N (%)	C (%)	H (%)	S (%)	Total weight (mg)
3.331	25.706	4.069	1.524	1.302

The results were compared with the properties of the virgin membrane, provided by the membrane manufacturer (shown in Table 3).

3. Results

3.1. Membrane fouling characterization

Membrane fouling was first characterized by SEM. In Fig. 3(a) (corresponding to the virgin membrane), the membrane active layer with no fouling deposits can be observed, while in Fig. 3(b) (corresponding to the fouled membrane), fouling deposits on the membrane surface can be noticed.

The composition of the fouling deposits was determined by SEM-EDX, as Fig. 4 shows. Silica, aluminum and iron silicates, and aluminum and iron oxides and hydroxides were detected. Therefore, inorganic and colloidal fouling was present on the membrane surface.

AFM analysis performed on virgin and fouled membranes is shown in Fig. 5. In Fig. 5(a) (corresponding to the virgin membrane), the surface roughness of the membrane active layer can be clearly distinguished. The deposition of foulants on the membrane surface (as it is shown in Fig. 5(b)) provided more heterogeneity to the membrane surface. An

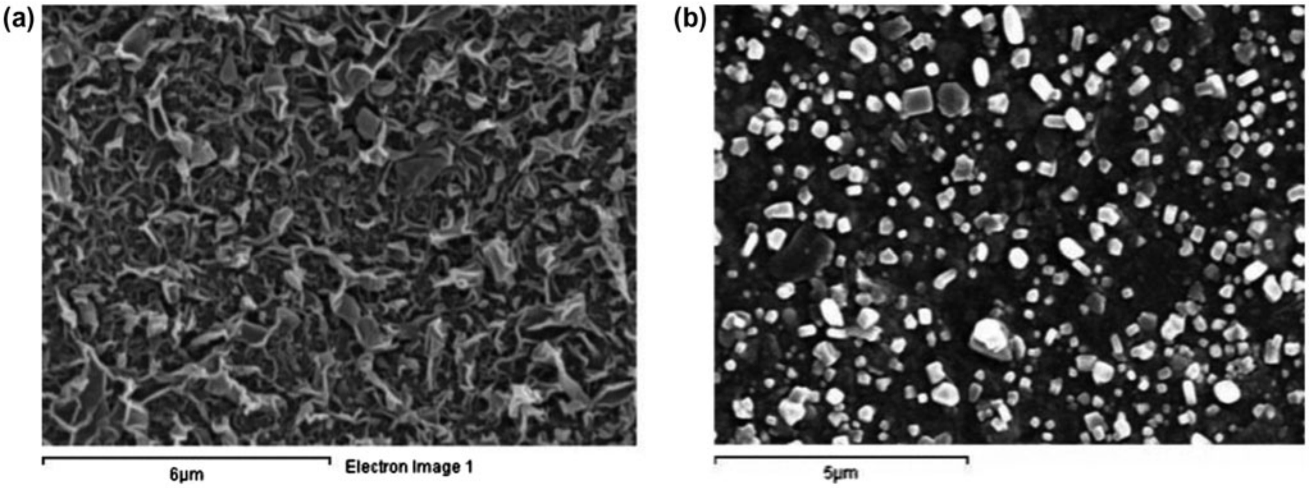
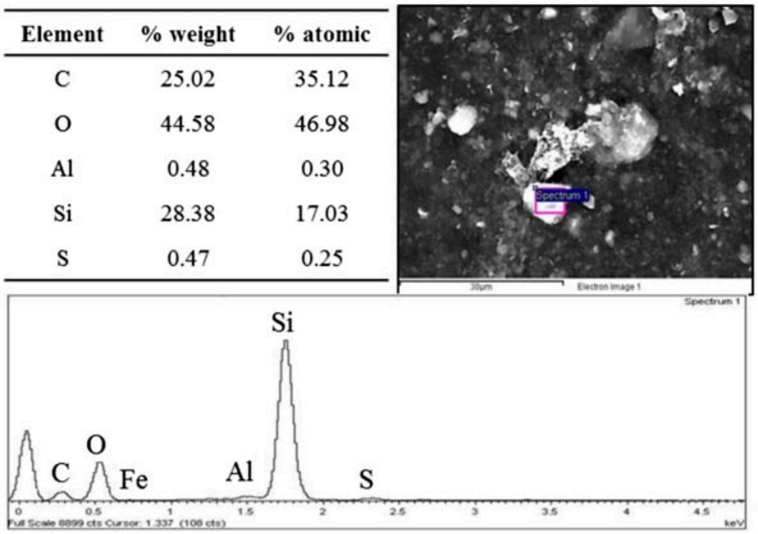


Fig. 3. SEM micrographs ×10,000 magnification. (a) Virgin membrane. (b) Fouled membrane.



Element	% weight	% atomic
C	25.02	35.12
O	44.58	46.98
Al	0.48	0.30
Si	28.38	17.03
S	0.47	0.25

Fig. 4. SEM-EDX microanalysis spectrum of a silica deposit found on the surface of a membrane sample.

increase in surface roughness due to membrane fouling was observed in the AFM analysis. The value of R_a increased from 127 to 133 nm, R_q increased from 160 to 174, and R_{max} from 1,253 to 1,775 nm.

The analysis of the fouling layer by ATR-FTIR is shown in Fig. 6. The following peaks can be emphasized:

(1) One peak of adsorption at 3,436 cm^{-1}, which is attributed to stretching of H_2O or more specifically to O–H bond in hydroxyl functional groups.
(2) Smaller peaks at 2,954 and 2,920 cm^{-1}, which are due to symmetric and asymmetric C–H stretching vibrations (non-aromatic, linear CH–structure, as $–CH_3$ and $–CH_2$ groups).
(3) Peak at 1,652 cm^{-1} that can be assigned to H_2O+NH or H_2O+C=O stretching vibrations.
(4) Peak at 1,550 cm^{-1} attributed to NH or COO– functional groups.
(5) Peaks between 1,460 and 1,380 cm^{-1} attributed to CO_3^{2-}, $CH_2–CH_3$, PO_4^{3-}, NH_4^+, COO^-
(6) A sharp peak at 1,037 cm^{-1} most likely originated by Si–O bonds of silicate materials.

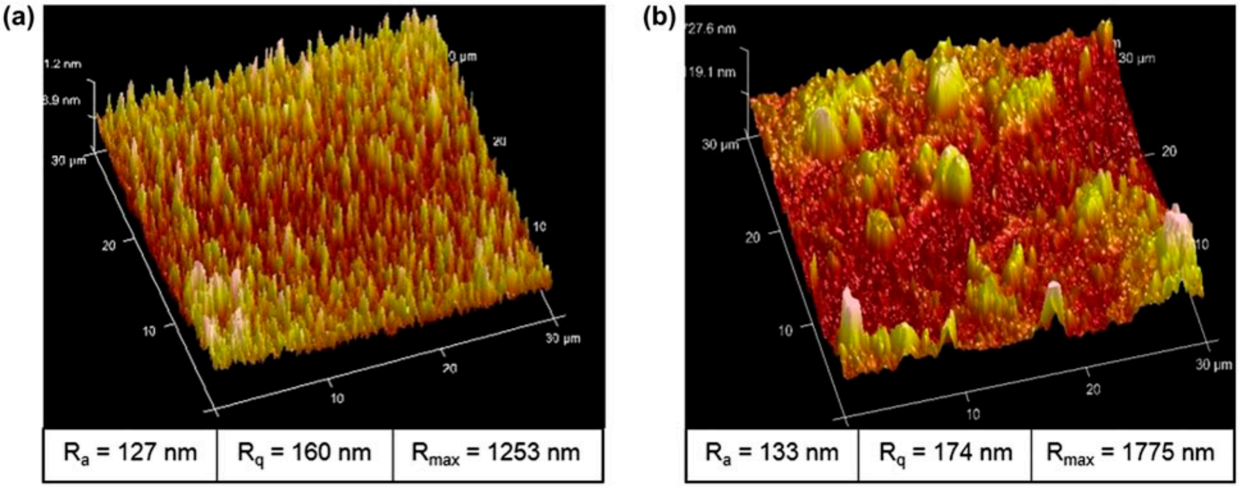


Fig. 5. Membrane surface roughness characterization by AFM. (a) Virgin membrane. (b) Fouled membrane.

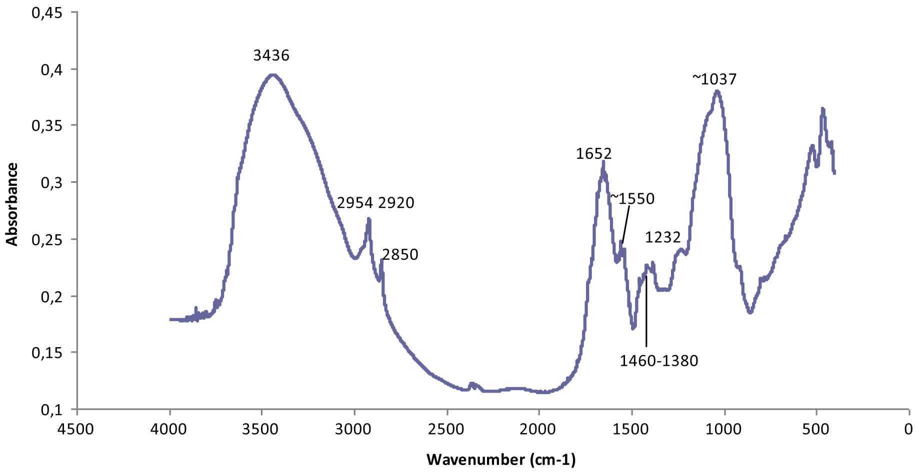


Fig. 6. ATR-FTIR spectrum of the fouling layer.

From the ATR-FTIR spectrum, it can be seen that membrane organic foulants such as silicate materials and natural organic matter (NOM) compounds are very commonly present when treating seawater. Analogous peaks on the ATR-FTIR spectrum were obtained by other authors when silicates, proteins, and polysaccharide materials were part of the fouling layer [29–31].

The results of the elemental analysis of the fouling layer are shown in Table 3.

From Table 3, it can be observed that around 30% of the fouling deposits were organic, composed by carbon, followed by hydrogen, nitrogen, and sulfur. The rest of the fouling materials deposited on the membrane surface (70%) were inorganic compounds specially silicates as it was confirmed from ATR-FTIR and SEM-EDX analysis.

3.2. Cleaning tests and characterization of permselective properties

3.2.1. Membrane initial state

The permselective properties of virgin and fouled membranes are shown in Table 4. Comparing the values obtained for the fouled membrane (blank) at 25˚C with the manufacturer specifications for the virgin membrane at the same temperature, membrane permeability (J_P) was observed to decrease by 49% and membrane selectivity (SRI) by about 8% due to fouling. Similar results (J_P reduction of 36% and SRI reduction of 10%) were observed at 40˚C.

Due to the severe fouling of the blank, cleaning tests are needed in order to determine the most suitable cleaning protocol.

Table 4
Permselective properties of the virgin and fouled membranes

	J_P (L/h m^2 bar)	SRI (%)
Virgin membrane*	0.4915	99.6
Blank at 25℃	0.2503	92.18
Blank at 40℃	0.3151	89.17

*According to the technical specifications bulletin of the membrane manufacturer.

3.2.2. Cleaning tests

The best results in terms of permeate flux and salt rejection recovery at 25 and 40℃ are shown in Figs. 7 and 8, respectively. The dashed line in the graphics represents the values for the virgin membrane obtained from the membrane manufacturer.

As it is shown in Fig. 7(a), the solution that reached the highest permeate flux recovery at 25℃ was a 2% (w/w) NaOH solution. The permeate flux values obtained when the membrane was cleaned with this solution are similar to those indicated for the virgin membrane by the manufacturer. Other solutions that showed high J_P recoveries were 0.4% NaOH, 1% SDS, 4% SDS, and 2% SDS for which J_P recoveries varied between 53 and 34%.

Fig. 7(b) shows the results on SRI recovery at 25℃. The largest SRI recovery at 25℃ was 2.54% and corresponded to the cleaning with a 4% SDS solution. The solution that achieved the greatest J_P recovery (2% NaOH) only reached a 0.84% recovery of the SRI. Citric acid, EDTA, and hydrochloric acid were not efficient to clean the membrane at 25℃ independently on the concentrations tested.

The cleaning efficiency improved considerably when the cleaning step was carried out at 40℃, as it is shown in Fig. 8. For the five cleaning agents tested at 40℃, the agents that showed no significant improvements when the cleaning was performed at 40℃ were NaOH and HCl.

As Fig. 8(a) shows, the best values of permeate flux recovery at 40℃ were achieved by means of cleaning with solutions containing 4% SDS, 2% SDS, 0.5% SDS, and 1% SDS (from 111 to 86% recovery), even exceeding the permeate flux value of the virgin membrane. This result is consistent with previous literature, and in the case of SDS and EDTA it can be explained by the enlargement of the membrane pores and/or an increase in the porosity of the membrane

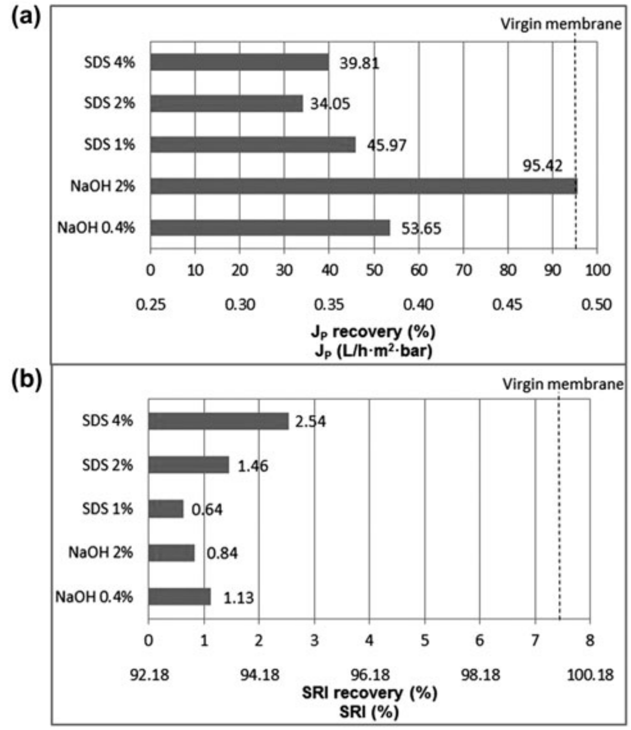


Fig. 7. The most efficient cleaning solutions at 25℃. (a) J_P recovery. (b) SRI recovery.

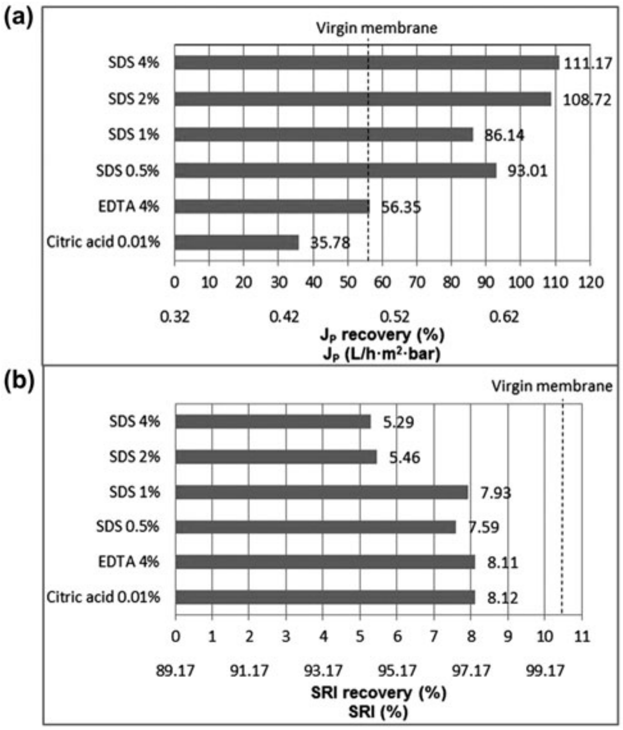


Fig. 8. The most efficient cleaning solutions at 40℃. (a) J_P recovery. (b) SRI recovery.

skin layer due to the adsorption of surfactants and chelating reagents to the membrane active layer [32]. However, the values of SRI were lower than that of the virgin membrane.

The greatest recoveries of SRI were achieved by cleaning with 0.01% citric acid, 4% EDTA, and 1% and 0.5% SDS. The recovery of SRI was larger than 7.59%, as it can be observed in Fig. 8(b). The recovery of SRI at 40℃ was much higher than the recovery obtained at 25℃.

If the values of J_P and SRI recovery are considered as a whole, it can be concluded that the best cleaning protocol corresponded to the cleaning with a 0.5% SDS solution at 40℃, obtaining a permeate flux of 0.6083 L/h m^2 bar (J_P recovery of 93%) and a SRI of 96.76% (SRI recovery of 7.59%).

Finally, Table 5 compares the results obtained by the different cleaning agents tested.

Negative values of J_P recovery or SRI recovery are considered "Detrimental," since it indicates that the values of J_P or SRI after cleaning are lower than those of the blank; "Irrelevant" means that the values of J_P or SRI recovery are about zero, since it indicates that the values of J_P or SRI after cleaning are equal to those of the blank; "Light recovery at 25℃" indicates J_P recovery between 10 and 50% or a SRI recovery between 1.5, and 4%; "Light recovery at 40℃" indicates J_P recovery between 10 and 40% or SRI recovery between 2 and 6%; "Good recovery at 25℃" indicates J_P recovery between 50 and 100% or SRI recovery between 4 and 6%; "Good recovery at 40℃" indicates J_P recovery between 40 and 70% or SRI recovery between 6 and 9%; "Excellent recovery at 25℃" indicates J_P recovery higher than 100% or SRI recovery higher than 6%; and "Excellent recovery at 40℃" indicates J_P recovery higher than 70% or SRI recovery higher than 9%.

Table 5
Recovery of the permselective properties of the membrane by the cleaning agents tested at two different temperatures

	25℃		40℃	
	Rec (J_P)	Rec (SRI)	Rec (J_P)	Rec (SRI)
Citric acid	0	0	+	++
NaOH	++	0	0	−
EDTA	0	0	++	++
SDS	+	+	+++	++
HCl	0	−	0	−

Notes: (−) detrimental; (0) irrelevant; (+) light recovery; (++) good recovery; (+++) excellent recovery.

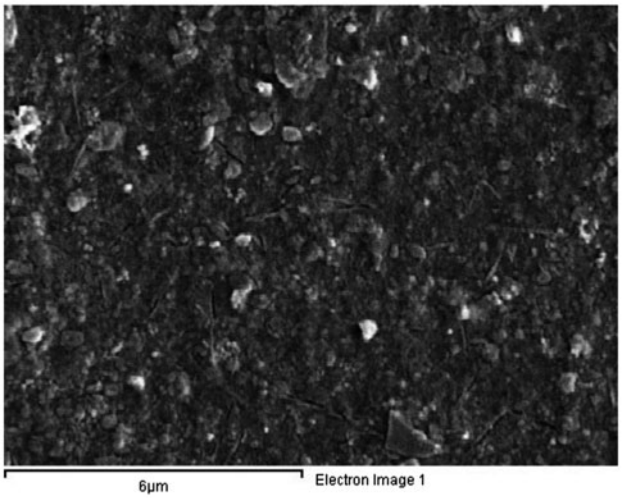


Fig. 9. SEM micrograph (×10,000 magnification) of a membrane cleaned with 0.5% SDS at 40℃.

It can be noticed that the best solution at 25℃ was NaOH while the best solution at 40℃ was SDS, followed by EDTA. This can be explained because most of the fouling was colloidal and probably organic fouling was present as well. Thus, the highest recovery was reached by means of the alkaline, surfactant, and chelating agents. As it was reported by other authors, NaOH is effective in the removal of organic foulants because it causes their hydrolysis and solubilization [17] and it enhances the charge repulsion of negatively charged foulants from the deprotonated membrane surface.

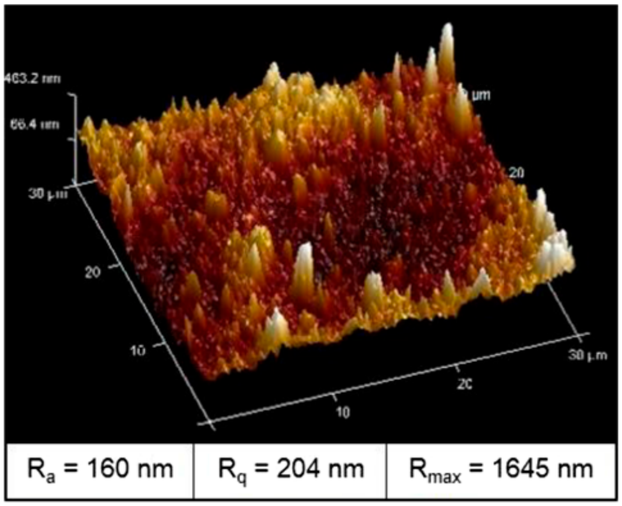


Fig. 10. AFM analysis of the membrane that was cleaned with 0.5% SDS at 50℃.

Table 6
Percentage distribution and variation of the elemental composition of membrane surface when it was cleaned with 0.5% SDS at 40˚C, measured by SEM-EDX

Element	Atomic % fouled membrane	Atomic % cleaned membrane	Variation (%)
O	76.30	82.32	6.03
S	7.17	5.81	−1.36
Si	7.01	2.52	−4.49
Fe	3.29	4.83	1.54
Al	2.33	1.26	−1.07
Na	0.94	1.33	0.39
Mg	0.88	0.32	−0.57
P	0.67	0.42	−0.25
Cl	0.70	0.74	0.04
K	0.29	0.18	−0.12
Ca	0.32	0.28	−0.04
Cr	0.11	0.00	−0.11

In addition, surfactants as SDS can solubilize macromolecules by means of the formation of micelles and they are able to reduce the adhesion forces between the foulants and the membrane surface [24]. Alkaline metal-chelating reagents, such as EDTA, are able to complex metal ions, removing them from the fouling layer, and they disrupt intermolecular foulant–cation bridges and support the opening of the organic foulant layer [32].

Regarding the effect of the temperature, an increase in the temperature of the cleaning solution improves J_P and SRI recovery values, due to a better solubility and transport of the foulants to the bulk solution [17]. The rate of the chemical reaction between the cleaning agent and the foulants increases with temperature as well. Moreover, as temperature raises, the cleaning solution penetrates more easily into the fouling layer due to the reduction of the viscosity. This effect was observed for all the solutions tested except for HCl and NaOH solutions.

3.3. Characterization of the membrane surface after the chemical cleaning

Figs. 9 and 10 show the results of the SEM and AFM characterization of the membrane that was cleaned with 0.5% SDS at 40˚C (the cleaning solution that showed the best results in terms of the recovery of the permselective properties of the membrane).

In Fig. 9, the surface of the membrane that had been treated with 0.5% SDS at 40˚C is shown. By comparing this image with Fig. 3(b) (fouled membrane), it can be observed that membrane cleaning was able to remove a considerably amount of the fouling deposits, although rests of the deposits can still be noticed on the membrane surface. No damage of the membrane surface was observed after cleaning with this solution.

The average elemental composition of the membrane surface after the cleaning with 0.5% SDS at 40˚C was obtained by SEM-EDX analysis. In Table 6, it is compared with that of the fouled membrane.

In general terms, partial remotion of those elements being part of the membrane fouling layer was observed. Silicon was the element that showed the largest removal from the membrane surface. Other elements related to fouling that have considerably reduced their presence on the membrane surface were sulfur, aluminum, and magnesium. This variation indicated a notable removal of sulfates and silicates of aluminum and magnesium. It is noticeable that the percentage of oxygen and iron on the membrane surface increased. This may be due to the reduction of the percentage of other elements and not to an increase of their presence as a consequence of cleaning. From the rest of the elements no significant conclusions can be obtained.

Finally, the AFM analysis of the membrane that was cleaned with 0.5% SDS at 40˚C showed a greater surface homogeneity compared with the fouled membrane, as can be observed in Fig. 10. The maximum ridge and valley height (R_{max}) was reduced from 1,775 to 1,645 nm by cleaning, although no significant changes in R_a and R_q parameters were observed.

4. Conclusions

The RO membrane coming from a desalination plant showed mainly inorganic fouling, but also showed organic fouling in lower proportion. Inorganic fouling deposits were fundamentally composed of silica, aluminum, and iron silicates and, to a lesser extent, of aluminum and iron oxides and hydroxides. Organic fouling was fundamentally composed of NOM compounds.

By means of the static cleaning tests it could be concluded that the most efficient cleaning agent was SDS, and the best cleaning conditions were a concentration of 0.5% and a temperature of 40˚C for 1 h of static cleaning. After the cleaning the permeate flux was increased up to 0.60 L/h m^2 bar and the SRI up to 96.76%. Considering the power costs required to raise the temperature of the cleaning solution to 40˚C during the cleaning step, the cleaning with 2% NaOH at 25˚C can be an alternative cleaning protocol, which allows to increase the membrane permeate flux up to 0.48 L/h m^2 bar and the rejection index up to 93.02%. The cleaning protocol developed in this research, employing the selected cleaning solutions and operation conditions, becomes efficient to recover the permselective properties of the membrane and does not damage the membrane active layer, as the SEM, SEM-EDX, and AFM analysis have demonstrated.

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