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A comparative study of ultrafiltration and physicochemical process as pretreatment of seawater reverse osmosis

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ABSTRACT

The effectiveness of two pretreatments for open intake seawater reverse osmosis (RO), aerated spiral wound ultrafiltration (ASWUF) and physicochemical (PC) pretreatment, were evaluated. Their efficiency in removing particulate materials was assessed by SDI, turbidity and Particle Size Distribution. Aerobic bacteria levels indicated the effectiveness of bacterial removal. Organic matter was characterized by TOC and UV_{254}. The apparent molecular weight (MW) distribution of organic matter was determined by centrifugal ultrafiltration fractionation and the effect of pretreated effluent on seawater RO flux decline was evaluated by a bench-scale test. Both pretreatments lowered feed water SDI to 2 and turbidity to 0.4 NTU and were highly efficient in removing particles larger than 1 µm. In general, ASWUF was more effective than PC, although PC removed TOC more efficiently. For both pretreatments, TOC fractions with a MW of under 3 kDa remained in treated water. Both pretreatments made effluents of excellent microbiological quality; however aerobic bacteria were isolated depending on the level of residual chlorine. Specific flux decline was slightly higher for seawater pretreated with PC and SEM revealed the presence of several foulants. Special care must be taken to prevent the contamination of the pretreated water zone (tanks, pipes…) in order to maintain the quality of influent to RO membranes.

Keywords: Ultrafiltration; Physicochemical pretreatment; Seawater reverse osmosis; Particle size distribution; Total organic carbon; Specific flux decline

1. Introduction

Reverse osmosis (RO) is the most widely used desalination process in the world today with an 80% share of the market [1]. Its main advantages are the simplicity of the process, normally composed of built-in modules and the costs involved compared to other desalination technologies. However, RO has certain membrane-related drawbacks, mainly, fouling.

Fouling is a crucial factor in the performance of RO plants, because it decreases the permeate flux and increases the concentration of solutes in the product. This causes operational costs to increase due to higher energy demands, and increases the membrane replacement rate. Considerable in-depth research has been done into fouling mechanisms, of which biofouling,

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organic fouling, particulate fouling and scaling are the most important [1–3].

Biofouling occurs when microbial cells accumulate and attach to the membrane by means of an extracellular polymeric substances matrix in which cells are embedded. Organic fouling is caused by natural organic matter, polysaccharides and aromatic compounds which may cause permeability decline by adsorption or irreversible fouling by complexation with calcium [3]. Particulate fouling is caused mainly by silica, iron oxide, aluminum silicate clays or colloids of iron [3], which may form compact cakes that create an additional barrier to filtration [4]. Scaling is caused by concentration polarization and scale layer formation when the product of the concentration of the soluble components exceeds their solubility level [2], although this problem is easily controlled due to the low recovery rates applied in seawater RO because of the limitations due to osmotic pressure [5].

The strategies aimed at improving the performance of RO plants therefore seek to prevent fouling. In order to achieve this, parameters such as SDI or turbidity have to be continuously monitored. The maximum allowable values in the feed water to RO are a turbidity of less than 0.2 NTU, SDI ≤ 3.0 or 4.0, absence of suspended solids and a sparingly soluble concentration that is lower than the saturation concentration [1]. In order to achieve this RO influent quality, it is essential to apply a pretreatment that is sufficient to ensure a complete removal of all the very finely dispersed organic and inorganic particulate matter and bacterial removal in combination with other antifouling strategies, such as chemical cleaning [6].

Usually open intake seawater RO units are preceded by a conventional pretreatment (CPT) procedure, which typically consists of acid addition, coagulant/flocculant addition, chlorination, media filtration, and cartridge filtration. In this type of pretreatment granular media filtration is considered as the heart of the treatment [6,7], and coagulation-flocculation are included to optimize the performance of the filtration process. Cartridge filtration is the last treatment before RO membranes, and it acts as a final polishing step to remove those particles that passed through media filtration. This combination of processes generates a suitable effluent and has been used traditionally [8–10]. However, variations in feed water can cause variations in effluent quality, which contribute to RO membrane fouling [1]. Often colloids and suspended particles pass through the CPT, and SDI values of between 4 and 5 are common [7]. When the quality of the influent is deficient this type of pretreatment is insufficient to produce an effluent with the required quality for the RO stage. [8,10]. Other pretreatment combinations include hydraulic flocculation and lamellar settlers combined with dual filtration (physicochemical (PC) pretreatment) for which SDI values of more than 3 have been reported [3].

Membrane pretreatment uses a different separation mechanism from CPT or PC. Ultrafiltration or Microfiltration form a barrier against suspended particles, colloidal materials and bacteria and provides an excellent treated water quality prior to RO [11]. Ultrafiltration membranes with a pore size of between 0.01 and 0.05 µm are a good bacterial retention barrier and guarantee a low, stable SDI value, even with significant fluctuations of raw water quality [8]. This type of membrane guarantee constant stability of microbiological quality better that microfiltration membranes [12]. A higher RO design flux, a reduced RO membrane replacement and reduced requirement for RO cleaning are advantages of membranes as pretreatment [3].

PC process and ultrafiltration membrane separation are both technologies that produce better results in terms of RO feed water quality than CPT [1]. So, the aim of this paper is therefore to compare the effectiveness of these two processes as pretreatment for open intake seawater RO working in real operational conditions.

2. Materials and methods

2.1. Description of the pilot-scale installations

The comparative study was carried out at pilot scale (Fig. 1) using raw seawater from the Mediterranean sea (Melilla, Spain). Water was obtained by open intake from an underwater intake pipe located at a depth of 15 m.

Ultrafiltration pretreatment consisted of an aerated spiral wound membrane (ASWUF) of polysulphone (20 kDa of MWCO, flux 45 l/m^2 h) with the capacity to treat 16 m^3/h, and with a macro-filtration unit (90 µm) installed immediately before it on the line. The system consisted of 24 membranes with a unitary filtration surface of 16.2 m^2, working under vacuum conditions with a transmembrane pressure of between –0.1 and –0.27 bar. Working conditions consisted of production periods of 20 min (flux 41 l/m^2 h) with continuous aeration, followed by backwashing phases of 0.5 min using filtered water. Chemical cleaning was carried out every two days with chlorine (100 mg/l), and once a week using citric acid (pH = 4.5). Chlorine addition was considered to avoid contamination. PC treatment with a capacity to treat 8 m^3/h involved chlorination (1.5 mg Cl_2/l) $FeCl_3$ coagulation (4 mg/l), hydraulic flocculation, sedimentation in a lamellar settler (0.45 m^3/m^2 h of effective surface loading rate) and filtration with a dual media (anthracite and silica sand) pressure filter.

2.2. Experimental methodology

Samples of influent and effluent from each assessed pretreatment technology were collected daily during a

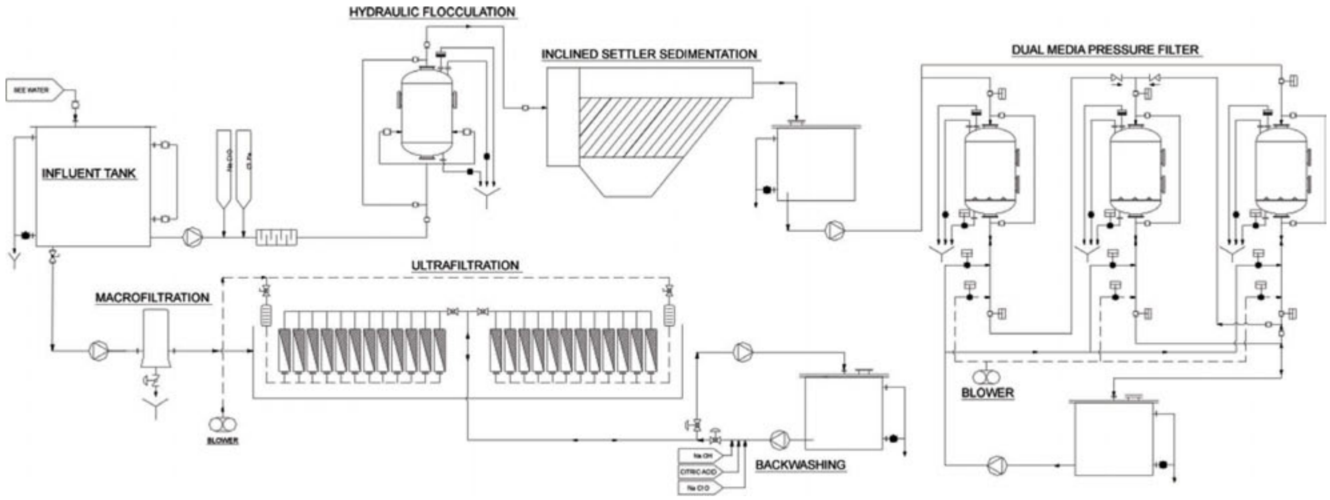

Fig. 1. Schematic diagram of an experimental pilot plant.

year. Turbidity, colour, suspended solids, UV_{254}, TOC were analysed as PC parameters. A quantitative diffuse radiation method described in Regulation UNE-EN ISO 7027: 2001 was used to determine turbidity, and a filtration method through 0.45 µm filters was used to determine the concentration of suspended solids. UV_{254} absorbance and true colour at $\lambda = 436$, 525 and 620 nm were measured over filtered samples (0.45 µm) and by means of a UV-visible spectrophotometer (ThermoSpectronic) with 1 cm quartz cell. TOC was measured using combustion TOC Analyser (SKALARTH). Residual free chlorine (RFC) was quantified by means of the volumetric evaluation method using *N*, *N*-diethyl-1, and 4-phenylenediamine, as described in Regulation UNE-EN ISO 7393-1. SDI was determined by the methods described by Fritzmann et al. [3].

The apparent molecular weight (MW) distribution of organic matter contained on samples of seawater was determined by fractioning with centrifugal ultrafiltration. Ultrafilters with 3 kDa, 10 kDa, 30 kDa, 50 kDa and 100 kDa of MW cut-offs were used (Amicon, Millipore Corp., Bedford MA). Water centrifugation was performed at 4000 r.p.m. for 10 min using a centrifuge (Eppendorf 5702) over a volume of 15 ml and filtrates of each ultrafilter were collected and analysed for TOC.

Particle Size Distribution (PSD) was conducted using a LiQuilaz-E20 particle counter (Particle Measuring Systems). The measuring principle is based on laser light extinction. A volume of 10 ml set at a fixed rate was analysed for each sample, which resulted in a minimum value for counted particles of 100 ml^{-1} and a maximum of 100,000 ml^{-1}. Particles ranged in size from 0.2 to 125 µm and the system was calibrated by inert latex particles of defined size.

For microbiological analyses, water samples were collected in sterile glass bottles (1 l) and analysed immediately after collection. Total aerobic bacteria count was carried out at 22°C by the membrane filtration procedure described in Regulation UNE-EN ISO 6222: 1999.

The effect of pretreated effluent on seawater RO flux decline was evaluated by a bench scale cross flow test using a RO membrane cell system with a test area of 0.01378 m^2 (9.5 × 14.5 cm). Microorganisms were reduced by chlorination and pretreated seawater was filtered by 0.5 microns prefilter. RO flux decline and pressure increases in an composite polyamide membrane (Hydranautics SWCS-2521) were measured for 24 h. Pretreated seawater was initially presurized at 52 bar with a flux of 22 l/m^2 h. A system recovery of 45% and a velocity cross flow of 0.3 m/s were considered for the experiment. Permeate and reject were reuse to maintain the experiment during 24 h and the seawater temperature was kept constant by a continuous recirculation of cold water into de main tank. Three replicates were achieved for each pretreated effluent.

After the bench test, 1 cm^2 fragments of tested evaluated RO membranes were analysed by scanning electron microscopy (SEM) using a Zeiss DSM 950 SEM operating at 5–30 kV, equipped with an Energy Dispersive Spectrometer (EDS Link Analytical Pentafet Si(Li)).

2.3. Statistical analysis

All data obtained in this study were analysed using the statistical program SPSS 15.0. Influent values were compared and correlated with those of the effluents. An analysis of variance (ANOVA) test was used to assess the homogeneity of variance with a significance level of 1% ($p < 0.01$). The least significant differences test (LSD-Test) was used to measure the differences between evaluated pretreatment technologies for the various parameters analysed.

3. Results and discussions

Our comparative study focused on the predictive parameters for three of the four main RO membrane fouling mechanisms, namely biofouling, colloidal and organic fouling. Scaling is usually controlled by the Stiff and Davis Saturation Index (S&DSI) [13], which for raw seawater ranged from 0.09 to 0.4 at 20°C. This means that no scaling problems should be expected at recovery of 40–45%. The ASWUF and the PC pretreatments were compared on the basis of effluent quality and potential RO membrane fouling problems. These parameters were analysed separately.

3.1. Particulate fouling

Table 1 shows a series of descriptive statistical analyses which provide evidence about the efficiency of both RO pretreatments. A high turbidity removal was observed for both pretreatments with performances of 82% and 79% for the ASWUF and the PC respectively. Similarly, SDI reduction was 63% by ASWUF and 51% by PC with average values of over two (Table 1), the optimum value for RO influent [7]. Analysis for the SS parameter illustrated a similar trend (Table 1).

Results from the ANOVA test showed no statistically significant differences between the effluents from the two pretreatments for turbidity, suspended solids and SDI (*p*-values 0.610, 0.126 and 0.566 respectively). However, there were differences between both effluents and the influent. Despite that, with ASWUF the effluent quality was slightly better and more consistent with standard deviation (S.D.) of all measured parameters lower than that of PC.

The quality of pretreated waters with ASWUF and PC was higher than that obtained by CPT. The same situation has been reported by Bonnelye et al. [7], Kim et al. [8] and Yang and Kim [10], which membrane pretreatment achieved effluent stability and SDI values lower than in CPT. With regard to the works of Kremen and Tanner [14] about the relationship between SDI and the flow resistance of foulants, both evaluated pretreatments provide water with a low propensity to particulate fouling.

Analysis of particle distribution may be considered a key element when comparing the quality of effluents from membrane technologies [15,16]. From the PSD analysis, four parameters have been underlined which are: the particle size where it was found the major count of particles (maximum count size or MCS); the maximum particle size (MPS) and the total count between 0.2–2 µm and 2–125 µm were considered for our study (Table 2).

Table 1
Physicochemical characterization of influent and effluents obtained from the experimental pretreatment

Parameters	Influent				ASWUF				PC			
	Max.	Min.	Average	S.D.	Max.	Min.	Average	S.D.	Max.	Min.	Average	S.D.
Turbidity, NTU	20.3	0.19	2.34^{a}	3.61	0.90	0.00	0.41^{b}	0.27	1.31	0.00	0.49^{b}	0.29
SS, mg/l	34.1	0.27	4.86^{a}	6.56	5.80	0.13	1.08^{b}	1.20	15.5	0.07	1.41^{b}	2.54
SDI	6.51	3.99	4.93^{a}	1.12	2.81	0.73	1.81^{b}	0.58	3.59	0.59	2.14^{b}	1.93
Colour$_{436}$, m^{-1}	7.70	1.00	5.53^{a}	1.37	7.00	0.00	5.02^{a}	1.58	7.10	0.00	5.05^{a}	1.69
Colour$_{525}$, m^{-1}	15.6	1.00	5.13^{a}	3.25	12.9	0.00	4.43^{a}	2.91	13.0	0.00	4.80^{a}	3.32
Colour$_{620}$, m^{-1}	6.40	0.00	3.77^{a}	1.24	5.80	0.00	3.29^{a}	1.33	5.70	0.00	3.41^{a}	1.41
TOC, mg/l	6.40	0.98	2.60^{a}	1.57	9.56	0.52	2.45^{a}	1.82	5.58	0.66	2.20^{a}	1.40
UV$_{254}$, m^{-1}	3.70	0.30	1.01^{a}	0.49	1.80	0.10	0.83^{a,b}	0.42	2.10	0.20	0.77^{b}	0.41

^{a,b} Groups with different letter show statistically significant differences between them (LSD-Test).

Table 2
Analysis of particle size distribution

	Influent			ASWUF				PC			
	Max.	Min.	Average	Max.	Min.	Average	R^{2}	Max.	Min.	Average	R^{2}
MCS, µm	1.00	0.20	0.39	0.50	0.20	0.36	0.12	0.70	0.20	0.38	0.18
MPS, µm	66.0	19.0	36.6	39.0	8.0	21.7	0.28	45.0	14.0	25.3	0.37
Particle size distribution, counts/ml × 10^{3}											
0.2–2 µm	37.1	10.5	19.4	24.7	5.10	16.9	0.23	25.8	8.48	17.6	0.21
2–125 µm	3.86	0.01	0.50	1.06	0.05	0.11	0.15	1.64	0.08	0.22	0.25

Distribution particles from the influent and effluents formed a logarithmic distribution with the majority of particles ranging in size from 0.2 to 2 µm. Particle removal was higher in the 2–125 µm range than in the 0.2–2 µm range; and the values for ASWUF were 77.9% and 13.0%, respectively, while those for PC were 55.8% and 9.0%. MCS remained almost constant after pretreatments, whereas MPS showed a slight difference with ASWUF accomplishing a size decrease from 66 µm to 39 µm, while PC reduced this value to 45 µm. These differences can be due to the different mechanism of action. ASWUF act by physical sieving, while PC works with addition of coagulant and dual media pressure filtration which can alter particles size distributions [17].

Schippers et al. [18] stated that particles of less than 0.05 µm are responsible for flux decline in RO membranes, which can be abundant in influent and effluent of pretreatment, in view of the 0.2–2 particles counts (Table 2). ASWUF acted as a screen preventing particles from getting through, and as established previously, the highest removal efficiency was for the highest particle size, as occurred with PC. However, it is important to point out that the effluent contained particles that were considerably larger than the membrane pore and of highly diverse origins.

Rojas et al. [19] observed that a large quantity of particles enter the effluent immediately after ultrafiltration membrane chemical cleaning or backwashing phases. This shows that a substantial proportion of the particles that are larger than the membrane pores come from the permeate zone after ultrafiltration. The origin of the particles swept into the effluent would appear to be incrustations in the permeate zone, the development of biofilms, the accumulation of organic matter, wear and tear of materials, etc. which means that the cleaning and maintenance of pipes and tanks, and the dosages in the pretreated water zone will affect final quality of the influent to RO and fouling development.

In the same way, effluent from PC increases particle counts, which together with a lower particle removal capacity gives rise to a poorer effluent quality. In view of this, PSD reveals that particulate fouling can occur on RO membranes despite ASWUF or PC pretreatment, although this is less probable after ASWUF due to better effluent quality.

3.2. Organic fouling

Organic matter removal by PC pretreatment was 15.4% for TOC and 23.8% for UV_{254} whereas with ASWUF it was 5.8% for TOC and 17.8% for UV_{254}. No significant colour removal was observed (Table 1). Coagulation by $FeCl_3$ helps organic colloids to join together which improves the organic matter removal by PC pretreatment [10,20].

In addition, a sieving mechanism allows the ASWUF membrane to remove organic matter. The limited effectiveness of ultrafiltration membranes in removing organic matter has been reported by Rojas et al. [19], who observed a strong correlation between the quality of the effluent and that of the influent. This high correlation was also observed in our experiment with seawater (Fig. 2).

Application of coagulation-flocculation as a pretreatment to ultrafiltration has proved to be highly efficient at increasing the natural organic matter removal capacity [21]. In this case, PC pretreatment showed a higher removal capacity than ASWUF. However, effluent quality also varied on the basis of influent quality (Fig. 2).

A 78% of the organic carbon concentration of raw seawater presented a MW of under 3 kDa (Fig. 3) and only 0.25% had weights of over 50 kDa, the cut-off for the ultrafiltration membranes we used. After pretreatments, the main concentration of remaining organic carbon was below 3 kDa, however, fractions with a

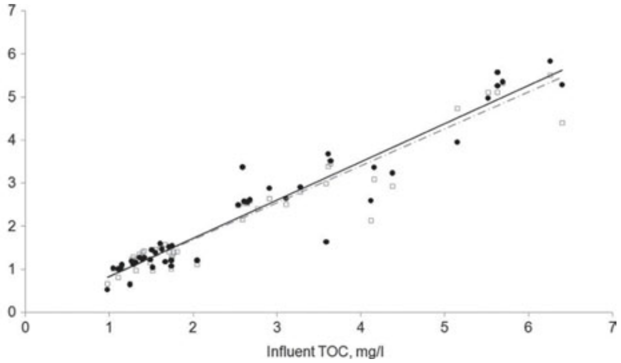

Fig. 2. Influent and Effluent TOC correlation for Physicochemical pretreatment (□, — —) and Aerated Spiral Wound Ultrafiltration pretreatment (●, ——).

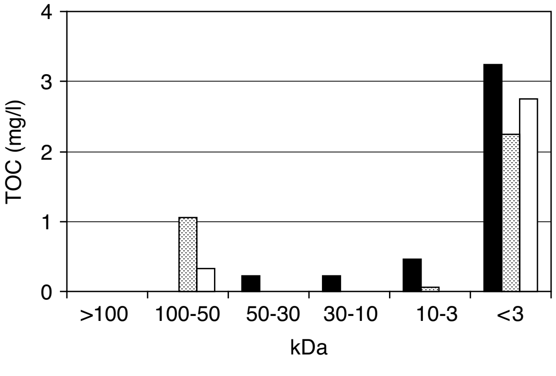

Fig. 3. MW distribution (kDa) of total organic carbon (TOC) in raw seawater ■, physicochemical effluent □ and ultrafiltration effluent ▨.

MW of between 50 and 100 kDa were observed after pretreatment, and represented 10 and 31% for PC and ASWUF effluent respectively. However, the origin of these larger particles is not from the influent because this fraction of MW was not observed in the raw seawater MW distribution.

As may be expected given the gradation of organic carbon in raw seawater, the ASWUF system would hardly be capable of eliminating organic matter. Cake formation over the membrane surface reduces the MW cut-off of the membrane, which is why the ASWUF membrane removes organic carbon fractions of over 3 kDa. The PC system removes organic matter by a different mechanism to ASWUF. Colloid fractions can be removed by coagulation/flocculation and the dissolved fraction can be partially adsorbed by anthracite. Organic matter, such as total aerobic bacteria or particles, ranging from 50–100 kDa appeared in the effluents, perhaps due to the contamination of the pretreated water zone (pipes or tanks), and this affects to the final organic matter concentration in the influent to RO. However, the high correlation of effluent TOC with respect to influent suggests that both pretreatments have low removal capacity for organic matter of low MW.

3.3. Biofouling

All influent samples analysed for total aerobic bacteria showed a positive count, ranging from 10 to 8000 cfu/ml whereas ASWUF and PC effluent samples were positive in 56.3% and 46.3%, respectively. Disinfection during PC pretreatment was achieved mainly by chlorination. This means that more positive counts were found when RFC concentration was lower, with 77.5% of positive samples located when RFC was zero. As soon as this concentration rises over 1 mg/l, positive samples fell to less than 5%.

A different situation was observed for ASWUF pretreatment: 29%, 38.7% and 32.3% of the positive counts analysed arose with RFC ranging 0 mg/l, 0–0.1 mg/l, 0.1–1 mg/l, respectively. Bacteria removal by ASWUF is based on a screening process which is highly effective at retaining bacteria [15]. However, aerobic bacteria are frequent after the membrane. This problem has been reported by authors such as Rojas et al. [19] and Gomez et al. [15] who found that the ultrafiltration membrane permeate side, the pipes and the tanks might be contaminated due to biofilm generation in the permeation zone. As the plant does not operate in sterile conditions, external conditions could lead to the development of biofilms, resulting in a loss of water quality. To prevent this situation a better cleaning procedure and maintenance are needed in that part of the plant.

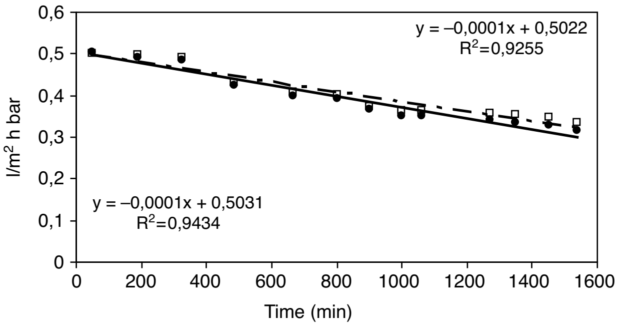

Fig. 4. Specific flux decline during bench experiment with seawater pretreated using the ASWUF (□, – · –) and the PC (●, ——) process.

3.4. Flux decline experiment

Fig. 4 shows the specific flux evolution during the 24 h bench experiment for both pretreated effluents. Permeability decline was slightly higher for seawater pretreated by PC, as shown by SDI analysis (Fig. 4), although no significant difference was observed in the rate of decline.

After the bench-scale experiment, the RO membrane surface was similar for both types of pretreated seawater. Fouling layers were not observed, however organic and inorganic deposits were occasionally detected, leading to the conclusion that foulant layers were not sufficient to cause flux decline. Ladner et al. [22] observed that during short bench-scale experiments, specific flux decline was mainly caused by changes in osmotic pressure and membrane compaction. The higher concentration of particles in the seawater pretreated using PC is the main difference between both evaluated pretreatments and maybe the cause of the slightly higher specific flux decline we observed.

4. Conclusions

Both ASWUF and PC open intake seawater pretreatments produce seawater with a quality that is higher than the seawater produced by CPT. Analysis of turbidity, suspended solid concentration and SDI revealed a similar effluent quality. However, PSD showed a better quality for effluent obtained by ASWUF, which suggests that particulate fouling is less probable in view of specific flux decline by bench scale tests. A low rate of organic matter removal was observed for both pretreatments due to the characteristics of influent organic compound (low MW).

Contamination of the permeate water zone is a source of organic matter, particles and bacteria. To counteract this effect, a treated water RFC concentration of over 1 mg Cl_2/l, and cleaning and maintenance of the pretreated water zone are required to preserve the quality of the influent to RO and avoid fouling.

Acknowledgements

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