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## Spiral-wound Module Nanofiltration of Surface River Water

### ABSTRACT

The performance of the nanofiltration of surface water, using a spiral-wound module, was assessed for the particular case of the Tagus River surface water collected at ca. 40 km of Lisbon (Portugal). The nanofiltration experiments were performed in the nanofiltration laboratory unit with a spiral wound module with 2.6 m<sup>2</sup> of membrane area (Filmtec NF270-2540, DOW Chemical), for different transmembrane pressures, recirculation flow rates and water recovery rates. The permeate flux and the rejection coefficients of total organic carbon (TOC), adsorbable organic halogens (AOX) and inorganic ions were measured.

The rejection coefficient of bivalent ions was in the range of 80-98% and for monovalent ions was in the range of 20-70%. The rejection coefficient increases with the transmembrane pressure and is not dependent on the water recovery rate, for the range of operating conditions investigated. The recirculation flow rate had almost no effect on the rejection coefficient of the ions, indicating that the concentration polarization was not severe.

The NF process has a rejection of TOC higher than 80%, contributing to reduce strongly the formation of disinfection by-products precursors. The process is also adequate to remove partially the AOX from the surface water. Depending on the operating conditions, the rejection coefficient of AOX was in the range between 26% and 72% for transmembrane pressures higher than 400 kPa.

### KEYWORDS:

Surface water, Nanofiltration, Spiral-wound modules, AOX, TOC

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## RESUMO

Estudou-se o desempenho da nanofiltração de água de superfície usando um módulo enrolado em espiral (Filmtec NF270-2540, DOW Chemical), para o caso particular de água de superfície captada na região de Valadas do rio Tejo a cerca de 40 km de Lisboa. Os ensaios de nanofiltração foram efectuados numa unidade laboratorial de nanofiltração com um módulo enrolado em espiral com 2.6 m<sup>2</sup> de área de membrana (Filmtec NF270-2540, DOW Chemical), para diferentes valores de pressão transmembranar, caudais de recirculação e taxas de recuperação de água. Para cada condição operatória mediu-se o fluxo de permeado e os coeficientes de rejeição do carbono orgânico total (TOC), dos compostos orgânicos halogenados adsorvíveis (AOX) e dos iões inorgânicos.

O coeficiente de rejeição dos iões bivalentes estava na gama entre 80% a 98% e o dos os iões monovalentes estava na gama entre 20% a 70%. Para a gama de condições operatórias investigada o coeficiente de rejeição aumenta em geral com a pressão transmembranar e é independente da taxa de recuperação de água. O caudal de recirculação na gama investigada também não influencia significativamente o coeficiente de rejeição dos diferentes iões, o que mostra que a polarização de concentração não é significativa.

A nanofiltração apresenta um rejeição ao TOC superior a 80% contribuindo, por isso, para reduzir significativamente os precursores de sub-produtos de desinfecção presentes na água. Adicionalmente a nanofiltração também remove AOX, observado-se que o coeficiente de rejeição respectivo se encontrava entre 25% e 72%, para pressões transmembranares maiores do que 400 kPa.

## 1. INTRODUCTION

Over the last few years, nanofiltration (NF) has been introduced in the field of water treatment to improve the final quality of the drinking water [1-3]. Unlike conventional water treatment units, nanofiltration can remove micropollutants such as pesticides [4] and endocrine disruptors [5]. Furthermore, the nanofiltration removes also the total organic carbon (TOC) of the surface water, contributing to minimize the formation of disinfection by-products in a subsequent disinfection stage [6]. Spiral-wound modules are widely used in the nanofiltration of surface water for drinking water production due to its high packing density and relatively low price [7-9].

The effect of pH, transmembrane pressure on the performance of spiral-wound module nanofiltration of brackish water with has been studied by Koyuncu and Yazgan [10]. The study is, however, restricted to very low recovery rates and to a single value of the recirculation flowrate. Other studies have been focused on the membrane fouling [11] and scale formation [12,13]. Despite of the importance of spiral-wound modules for drinking water production, the detailed analysis of the influence of the feed pressure, recirculation flow rate and water recovery rate on the system performance are scarce in the literature.

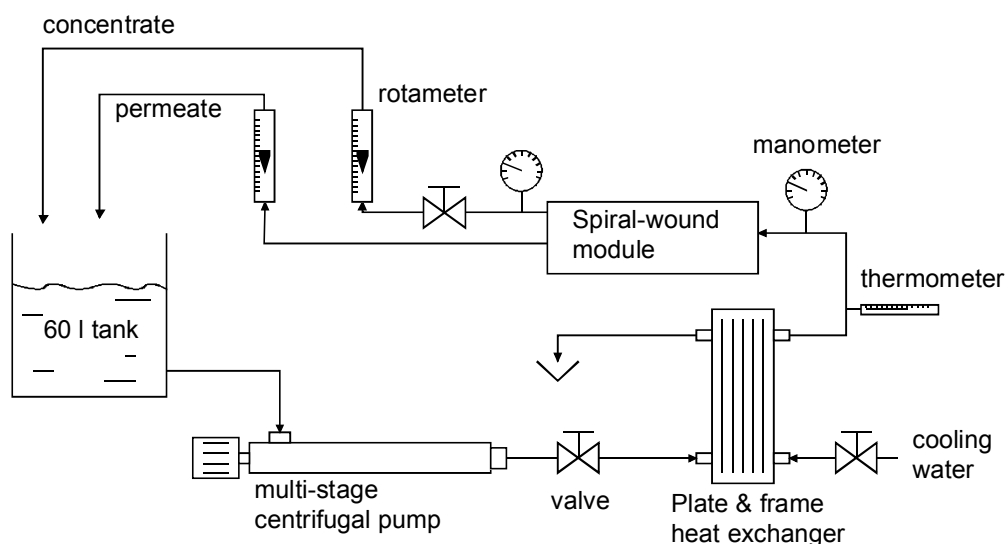
The objective of this work is to investigate the effect of operating conditions on the performance of a typical spiral-wound nanofiltration module (Filmtec NF270-2540 from

DOW Chemical), for the treatment of surface water collected from the Tagus River at ca. 40 km from Lisbon (Portugal). The experiments were performed in a laboratory nanofiltration unit using about 50 l of surface water. The permeate flux and observed rejection coefficients of TOC, adsorbable organic halogens (AOX) and ions were measured under different transmembrane pressures, recirculation flow rates and water recovery rates. The concentration polarization index and saturation indexes of precipitation reactions were also analysed, using a suitable mass transfer correlation and an advanced public domain water speciation software adequate for diluted waters.

## 2. EXPERIMENTAL

### 2.1 Apparatus and Materials

The nanofiltration experiments were performed in the nanofiltration set-up shown in Fig. 1. The feed solution was pumped by a multi-stage centrifugal pump (Tonkaflow, Model SS 1838G). The operating temperature was controlled by a plate-and-frame heat exchanger with a heat transfer area of 0.44 m<sup>2</sup>. The fluid pressure was measured with Bourdon Manometers with an accuracy of  $\pm 10$  kPa and the concentrate and permeate flow rates were measured with calibrated rotameters with an accuracy of  $\pm 20$  l/h and  $\pm 1.5$  l/h, respectively. The recirculation flow rate,  $Q_r$ , and the transmembrane pressure,  $\Delta P$ , were controlled by means of the valves placed after the pump and after the membrane module.



**Figure 1.** Nanofiltration set-up with spiral-wound module

The spiral-wound module Filmtec NF200-2540 from Dow Chemical Company has a membrane area of 2.6 m<sup>2</sup>, a diameter of 6.35 cm, a length of 1 m and feed channels with 0.71 mm height. The total cross-section open area of the feed channels is 9.2 cm<sup>2</sup>. The membrane has a nominal cutoff of 200 Dalton and a hydraulic permeability of  $2.3 \times 10^{-11} \text{ m s}^{-1} \text{ Pa}^{-1}$  at 25°C. This is an ultra low-pressure nanofiltration membrane that operates with typical transmembrane pressures lower than 1 MPa. The surface water was collected from the Tagus River, just before the river estuary, at ca. 40 km from Lisbon (Portugal) and was kept in full tanks of 20 l until being used in the nanofiltration experiments.

## 2.2 Procedure

The nanofiltration tests were performed at 25°C in total recirculation mode, with both the concentrate and the permeate streams recirculated to the feed tank, and in concentration mode, with continuous removal of the nanofiltration permeate. Approximately 50 l of river water were used in a nanofiltration test. Before the nanofiltration, the water was filtered through a 1-micron cartridge filter, to prevent the plugging of the membrane module with particles. The average transmembrane pressure,  $\Delta P$ , was varied between 200 kPa and 800 kPa. Samples of 1.5 l of feed water, concentrate and permeate were collected and preserved at 5°C until the chemical analysis. The water recovery rate,  $\eta$ , in the concentration mode is defined by the ratio between the total volume of permeate collected and the initial volume of surface water. As the retention volume of the unit was ca. 10 l, the water recovery rate could not increase above 80% in a nanofiltration experiment. After the nanofiltration of the surface water, the membrane was cleaned with tap water until recovering the initial hydraulic permeability. The tap water was previously filtered through an activated carbon filter, to remove the residual chlorine, and through a 1-micron cartridge filter.

## 2.3 Analytical methods

The water turbidity was determined by nephelometric method, using the turbidity meter TU 1100 (Standard Method 2130 B [14]) and the results are expressed in Nephelometric Turbidity Units (NTU). The pH was measured using a pH meter Russell, Model RL 150, the conductivity was measured at 20°C with the Metrohm 660 conductivity meter.

The adsorbable organic halogen (AOX) was determined in accordance with the specifications EN1485, using the IDC multi X 2000 AOX Analyzer, which allows the direct determination of organic halides adsorbable onto activated carbon. The total organic carbon (TOC) of the samples was measured using a Shimadzu TOC analyzer, model TOC-Vcsh. The AOX is expressed in mg Cl/l and the TOC is expressed in mg C/l.

The chloride ion was analyzed by the argentometric method (Standard Method 4500 B [14]), the sulphate ion was analyzed by the Turbidimetric Method (Standard Method 4500 E [14]), the nitrate was determined by Nitrate Electrode Method (Standard Method 4500 D [14]). The concentration of ions Na, K, Ca, Mg was determined by flame atomic absorption spectrometry with direct air-acetylene flame (Standard Method 3111 B [14]). The alkalinity, expressed in mg HCO<sub>3</sub><sup>-</sup>/l, was determined according to the Standard Method 2320 B [14].

### 3. SATURATION INDEX AND CONCENTRATION POLARIZATION

The analysis of the saturation indexes of the precipitation reactions in the water and nanofiltration concentrates was made with the public-domain computer program PHREEQC [15], which simulates chemical reactions and transport processes in natural or polluted water with low or moderate ionic strength. The saturation index (SI) is defined by  $\log(\text{IAP}) - \log K$ , where IAP is the ion activity product and K is the thermodynamic equilibrium constant of the reaction. The input data of the program is the detailed ionic composition of the water.

The rejection of the a solute by the membrane is quantified by the rejection coefficient given by  $R = (c_{Af,loc} - c_{Ap,loc}) / c_{Af,loc}$ , where  $c_{Af,loc}$  and  $c_{Ap,loc}$  are the local solute concentration in the feed channel of the module and in the permeate stream, respectively. Assuming that R is constant along the module feed channel, it is related with the water recovery rate in the module through [16]:

$$R = 1 - \ln(1 - \eta_m c_{Ap} / c_{A0}) / \ln(1 - \eta_m) \quad (1)$$

where  $c_{A0}$  and  $c_{Ap}$  are the experimental values of the solute concentration in the module inlet and in the permeate, respectively. The water recovery rate in the module is given by  $\eta_m = Q_p / Q_0$ , where  $Q_0$  and  $Q_p$  are the water flow rate in the module inlet and in the permeate stream, respectively.

The increase of solute concentration in the membrane surface due to the solute rejection by the membrane is quantified by concentration polarization index  $\Gamma = (c_{Am,loc} - c_{Af,loc}) / c_{Af,loc}$ , where  $c_{Am,loc}$  is the local concentration of solute in the membrane surface. According to the film theory [17] and assuming that  $\Gamma$  is constant in the module feed channel, the concentration polarization index is given by

$$\Gamma = R(e^{J_p / k_c} - 1) \quad (2)$$

where  $J_p$  is the permeate flux and  $k_c$  is the mass transfer coefficient. For the module used in this work, the following mass transfer correlation is adequate to compute the mass transfer coefficient [16]:

$$Sh = 0.33 Re^{0.5} Sc^{0.33} \quad (3)$$

where Sh is the Sherwood number, based on channel height, Re is the Reynolds number, based on the channel height and on the average superficial velocity in the module feed channel and Sc is the Schmidt number.

#### 4. RESULTS AND DISCUSSION

The analysis of the river water collected at three different months is shown in Table 1. As shown in this table, the turbidity, TOC and AOX assume rather high values. The salinity of the water increases strongly from January to July. This phenomenon is probably due to the reduction of the river flow rate and the subsequent contamination of the water with the downstream estuary saline water. The water is slightly hard, with the total concentration of  $Mg^{2+}$  and  $Ca^{2+}$  in the range from 40 to 70 ppm.

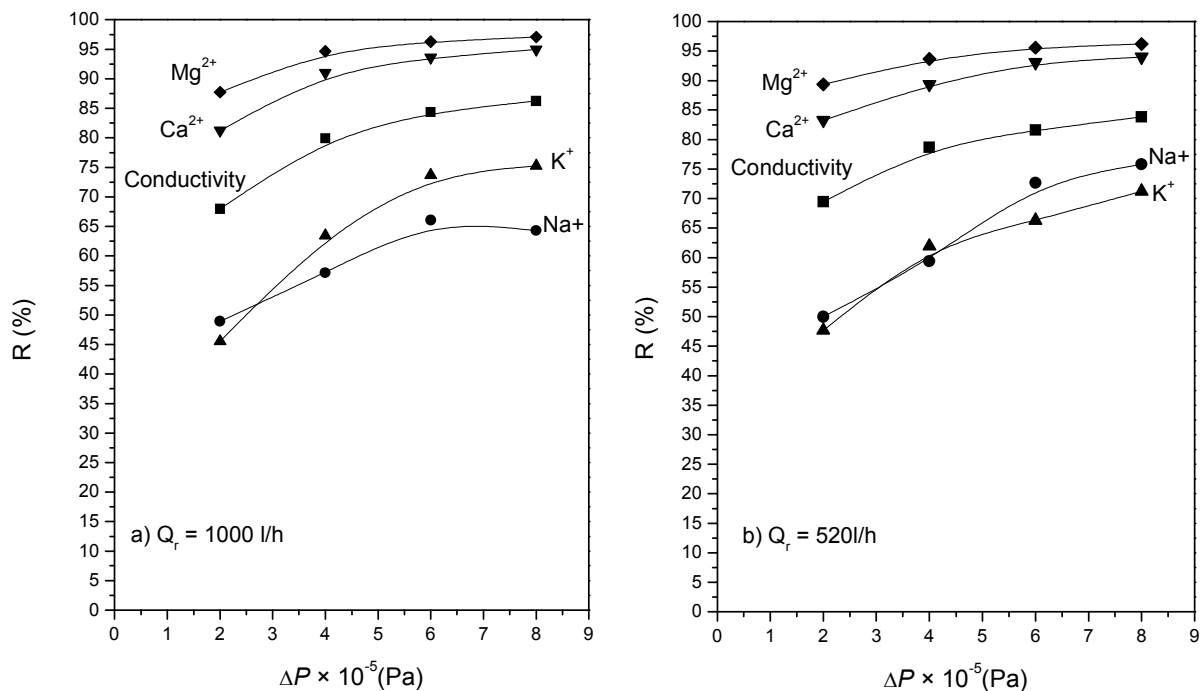
The effect of the feed pressure and recirculation flow rate on the surface water nanofiltration performance is displayed in Figures 2a, 2b and 3. As shown in Figure 2a and 2b, the observed rejection of the conductivity and of the ions increases with the transmembrane pressure. Furthermore, the rejection coefficients approach a plateau as the transmembrane increases.

**Table 1.** Analysis of three samples of surface water from Tagus river

	15 Jan-2005	7 May-2005	11 July-2005
pH	7.84	7.5	7.67
Conductivity ( $\mu S/cm$ )	271	558	647
Turbidity (NTU)	11.1	13.7	16.4
TOC (mg/l)		3.1	3.3
AOX ( $\mu g/l$ )		213	235
Ions (mg/l)			
$Cl^-$	28.4	53.3	110
$SO_4^{2-}$		90.8	143
$HCO_3^-$		137	135
$Na^+$	38	45	85
$K^+$	3.98	4.87	6
$Ca^{2+}$	32	52.3	55.5
$Mg^{2+}$	7.5	10.3	16
$Fe^{2+}$	0.05	0.21	0.05

This phenomena is typical of nanofiltration of multi-ionic solutions and has been explained based on the extended Nernst-Planck equations which incorporate the effects of diffusion, convection and migration inside the membrane, together with steric hindrance factors to convection and diffusion [e.g. 18,19]. The increase of the permeate flux through the increase of the transmembrane pressure has the effect of reducing the ions concentration of the treated water. As this effect is more important for the magnesium and calcium ions, the nanofiltration reduces the total hardness of the treated water.

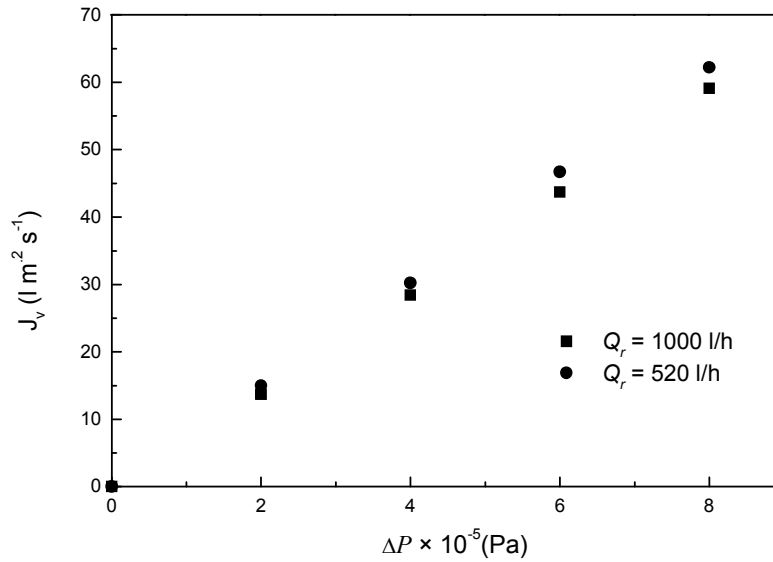
Comparing the results of Figure 2a with the ones of Figure 2b it is evident that the recirculation flow rate has no strong influence on the observed rejection coefficients of the ions. This means that at the feed flow rates tested, the concentration polarization of the ions is not severe. In fact, taking for example the case of a typical salt such as calcium carbonate, which has a diffusion coefficient of  $1.1 \times 10^{-9} \text{ m}^2/\text{s}$  [20], the concentration polarization at the highest transmembrane pressure (permeate flux of  $60 \text{ l m}^{-2} \text{ h}^{-1}$ ) assuming a rejection coefficient of 0.95, is only 0.24 at a recirculation flow rate of 1000 l/h ( $Re = 240$ ) and 0.38 at a recirculation flow rate of 550 l/h ( $Re = 130$ ). For lower transmembrane pressures, the concentration polarization is below these values and, therefore, has almost no influence on the observed rejection coefficients. For the spiral-wound module used, the recommended flux of to treat surface water is  $24 \text{ l m}^{-2} \text{ h}^{-1}$  and the minimum concentrate flow rate is 700 l/h ( $Re = 165$ ) [21]. With these conditions and for the same salt, the concentration polarization is only 0.11.



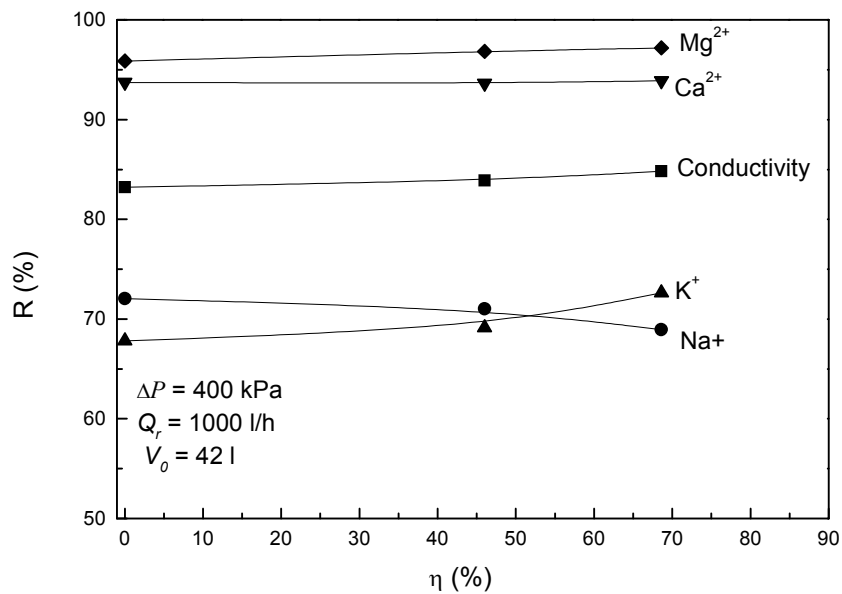
**Figure 2** Observed rejection coefficients vs. transmembrane pressure for two recirculation flow rates. The surface water was collected on 15 Jan 2005 and was filtered through 1-micron cartridge filter. The initial volume of water was 42 l.

The permeate flux versus the transmembrane pressure is displayed in Figure 3, for the two different values of recirculation flow rate. The variation with the permeate flux with the transmembrane pressure is linear and almost independent of the recirculation flow rate. No fouling effects were detected during the experiment as expected, because the time scale of the experiment (1 hour) is much lower than the typical time scale of the membrane fouling (about 1 month) observed for surface water treatment [22].

The influence of the water recovery rate on the NF performance is displayed in Figure 4. The results show that the observed rejection coefficients are not influenced by this parameter in the range from 0 to 68.6%. Therefore, for design purposes it is adequate to assume that the observed rejection coefficients are independent of the water recovery rate.



**Figure 3.** Permeation flux vs. transmembrane pressure. The surface water was collected on 15 Jan 2005 and was filtered through 1-micron cartridge filter.



**Figure 4.** Rejection coefficients vs. water recovery rate. The surface water was collected on 15 Jan 2005 and was filtered through 1-micron cartridge filter.



The nanofiltration performance of two water samples collected in May and July are displayed as a function of the transmembrane pressure and of the water recovery rate in Table 2 and 3, respectively. In general, the results show that the observed rejection coefficients of the ions follow the same behaviour already observed: they increase with the transmembrane pressure and are independent of the water recovery rate. The observed rejection of TOC is always higher than 80% and can increase up to 97% at high water recovery rates. This high rejection of TOC contributes strongly to reduce the formation potential of disinfection by-products in a final disinfection stage.

The AOX rejection coefficient increases with the transmembrane pressure. At 200 kPa the AOX rejection is very low, but for transmembrane pressures between 400 and 600 kPa, the rejection coefficient varies between 26% and 50%, at low water recovery rates. At high water recovery rates, the AOX rejection can increase up to 73%. These results indicate that the NF process removes partially the AOX at high transmembrane pressures and water recovery rates. Further removal of AOX requires a NF membrane with a lower molecular weight cut-off (MWC).

**Table 2.** Effect of transmembrane pressure and water recovery rate on the permeate flux and composition and rejection coefficients <sup>a</sup>

	Feed water	$\eta = 0\%$			$\eta = 64.5\%$	
		Permeate at $\Delta P$ (kPa) =			Conc. water	Permeate at $\Delta P = 400$ kPa
		200	400	600		
flux (l/m <sup>2</sup> h)		16.0	33.9	52.4		32.4
Cond. ( $\mu$ S/cm) <sup>b</sup>	558	226 (59.5%)	170 (70.9%)	145 (75.9%)	1000	281 (73.2%)
Turb. (NTU) <sup>b</sup>	13.7	0.3 (97.9%)	0.3 (97.9%)	0.3 (98.0%)	6.4	0.1 (98.5%)
TOC (ppm) <sup>b</sup>	3.10	0.13 (95.9%)	0.13 (96.1%)	0.22 (93.6%)	7.2	0.24 (96.9%)
AOX (ppm) <sup>b</sup>	0.213	0.21 (1.4%)	0.16 (26.2%)	0.11 (51.0%)	0.50	0.14 (72.8%)
Cl <sup>-</sup> (ppm) <sup>b</sup>	53.3	42.6 (20.6%)	32.0 (41.6%)	32.0 (42.5%)	67.5	49.7 (27.7%)
HCO <sub>3</sub> <sup>-</sup> (ppm) <sup>b</sup>	136	59.2 (57.5%)	39.7 (72.4%)	45.8 (68.8%)	267.2	97.0 (65.2%)
SO <sub>4</sub> <sup>2-</sup> (ppm) <sup>b</sup>	90.8	1.83 (98.1%)	1.73 (98.2%)	1.73 (98.2%)	228	2.63 (98.9%)
Na <sup>+</sup> (mg/L) <sup>b</sup>	45.0	31.5 (30.7%)	25.0 (46.1%)	21.5 (54.8%)	71	39.5 (46.0%)
K <sup>+</sup> (mg/L) <sup>b</sup>	4.87	2.6 (47.4%)	2.1 (58.5%)	1.8 (65.4%)	8	3.62 (56.4%)
Ca <sup>2+</sup> (mg/L) <sup>b</sup>	52.3	14.4 (73.1%)	9.35 (83.1%)	6.85 (88.0%)	101.8	16.4 (84.7%)
Mg <sup>2+</sup> (mg/L) <sup>b</sup>	10.3	2.10 (80.1%)	1.22 (88.8%)	0.92 (91.9%)	23.3	2.05 (91.7%)

<sup>a)</sup> The surface water was collected on 7 May 2005 and was filtered through 1-micron cartridge filter. The Initial volume of water was 48 l and the recirculation flow rate was 650 l/h.

<sup>b)</sup> The observed rejection coefficients are displayed in brackets.

**Table 3.** Effect of transmembrane pressure and water recovery rate on the permeate flux and composition and rejection coefficients <sup>a</sup>

	Feed water	$\eta = 0\%$			Conc. water	$\eta = 64.9\%$ Permeate at $\Delta P = 400$ kPa
		Permeate at $\Delta P$ (kPa) =				
		200	400	600		
flux (l/m <sup>2</sup> h)		16.7	35.0	52.0		32.8
Cond. ( $\mu$ S/cm) <sup>b</sup>	647	281 (57.4%)	215 (68.3%)	182 (73.9%)	1311	353 (73.1%)
Turb. (NTU) <sup>b</sup>	16.4	0.2 (98.8%)	0.5 (97.2%)	0.1 (99.5%)	4.8	0.2 (96.0%)
TOC (ppm) <sup>b</sup>	3.8	0.27 (93.1%)	0.38 (90.6%)	0.79 (80.9%)	8.82	0.23 (97.6%)
AOX (ppm) <sup>b</sup>	0.235	0.205 (13.1%)	0.125 (48.6%)	0.155 (36.4%)	0.556	0.175 (69.9%)
Cl <sup>-</sup> (ppm) <sup>b</sup>	110	85.2 (23.1%)	71.0 (37.1%)	58.6 (49.3%)	133	103 (23.7%)
HCO <sub>3</sub> <sup>-</sup> (ppm) <sup>b</sup>	135	62.8 (54.2%)	43.9 (68.9%)	37.8 (74.0%)	304	79.3 (75.2%)
SO <sub>4</sub> <sup>2-</sup> (ppm) <sup>b</sup>	143.3	4.26 (97.1%)	4.45 (97.1%)	4.80 (96.9%)	399	5.31 (98.8%)
Na <sup>+</sup> (mg/L) <sup>b</sup>	85.0	42.0 (51.4%)	33.5 (62.2%)	29.5 (67.6%)	95.5	54.0 (45.1%)
K <sup>+</sup> (mg/L) <sup>b</sup>	6.0	3.1 (49.2%)	2.5 (60.0%)	2.15 (66.5%)	10.5	4.38 (59.9%)
Ca <sup>2+</sup> (mg/L) <sup>b</sup>	55.5	16.3 (71.3%)	11.3 (80.8%)	8.4 (86.1%)	137.2	20.3 (86.0%)
Mg <sup>2+</sup> (mg/L) <sup>b</sup>	16.0	3.40 (79.3%)	2.08 (87.8%)	1.52 (91.4%)	42.0	3.45 (92.3%)

<sup>a)</sup> The surface water was collected on 11 July 2005 and was filtered through 1-micron cartridge filter. The Initial volume of water was 50 l and the recirculation flow rate was 650 l/h.

<sup>b)</sup> The rejection coefficient is displayed in brackets.

To anticipate if membrane scaling can occur under typical operating conditions it is important to predict the saturation indexes of precipitation reactions through the public-domain program PHREEQC. This computation method is more accurate than the computation of specific scale indexes (e.g. Langelier Saturation Index, Stiff & Davis Stability Index) to predict the scaling potential of a given water because it takes into account the detailed ion composition to identify all the potential insoluble salts. For both concentrates of Table 2 and 3, the saturation index of the calcite, CaCO<sub>3</sub>, and dolomite, CaMg(CO<sub>3</sub>)<sub>2</sub>, are positive, indicating that these salts are supersaturated and can precipitate in the membrane surface. This problem can, however, be solved by the reduction of pH. At pH 6.0 the predicted saturation indexes of both salts are sufficiently negative to avoid precipitation.

In relation to the permeate water obtained at high recovery rate, the saturation index of calcite is -0.72 for the NF permeate of Table 2 and -0.55 for the NF permeate of Table 3. These values would be much lower if the water pH would decrease to 6.0. For this reason, the NF permeate has a high corrosion potential and it is necessary to increase the SI of the NF permeate to improve the corrosion control. This problem will become more severe if a membrane with lower MWC is used to increase the AOX removal from the surface water.

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## 5. CONCLUSION REMARKS

The nanofiltration studies performed with surface water from Tagus River demonstrate that the rejection coefficient was in the range of 80-98% for bivalent ions and in the range of 20-70% for monovalent ions. The rejection coefficient increases with the transmembrane pressure and is not dependent on the water recovery rate, for the range of operating conditions investigated. The recirculation flow rate had almost no effect on the rejection coefficient of the ions, indicating that the concentration polarization was not severe.

The analysis of the saturation index indicates that the calcite and dolomite is supersaturated in the concentrated water at a recovery rate of ca. 65% and can cause membrane scaling. Scaling can, however, be prevented if the pH is reduced below 6.0. On another hand, the saturation index of calcite of the NF permeate is negative and its value must be made positive to reduce the corrosion potential of the treated water.

The NF process has a rejection of TOC higher than 80%, contributing to reduce strongly the formation potential of disinfection by-products in a final disinfection stage. The process is also adequate to remove partially the AOX from the surface water. Depending on the operating conditions, the rejection of AOX was in the range between 26% and 72% for transmembrane pressures higher than 400 kPa.

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