



## Treatment of reverse osmosis brine by direct contact membrane distillation: Chemical pretreatment approach



J.A. Sanmartino<sup>a</sup>, M. Khayet<sup>a,b,\*</sup>, M.C. García-Payo<sup>a</sup>, H. El-Bakouri<sup>c</sup>, A. Riaza<sup>c</sup>

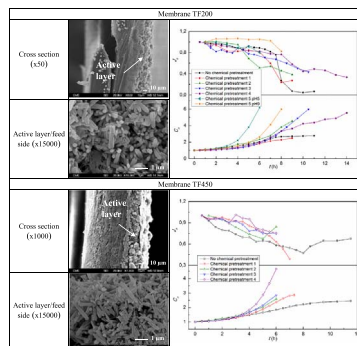
<sup>a</sup> Department of Applied Physics I, Faculty of Physics, University Complutense of Madrid, Avda. Complutense s/n, 28040, Madrid, Spain

<sup>b</sup> Madrid Institute of Advances Studies of Water (IMDEA Water Institute), Avda. Punto Com no. 2, 28805 Alcalá de Henares, Madrid, Spain

<sup>c</sup> Abengoa S.L., Research Development Center, Prolongación c/ Don Remondo s/n, Barriada Fuente del Rey, 41703 Dos Hermanas, Spain

### GRAPHICAL ABSTRACT

RO brine crystals fouling on the PTFE layer of the membranes, the normalized DCMD permeate flux ( $J_n$ ) and the normalized feed concentration ( $C_n$ ) evolution obtained at different chemical pretreatments.



### ARTICLE INFO

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### ABSTRACT

Different chemical pretreatment strategies followed by direct contact membrane distillation (DCMD) process were adopted for the processing of reverse osmosis (RO) brine. CPT with  $\text{Na}_2\text{CO}_3 + \text{NaOH}$  allowed scale reduction, removing both permanent calcium hardness and temporary calcium hardness whereas the chemical pretreatment with  $\text{BaCl}_2$  permitted to remove sulfate ions from RO brine. This last chemical pretreatment was found to be the most efficient pretreatment improving the DCMD performance because of the corresponding highest permeate flux, lowest permeate flux decline and best permeate quality. However, it is relatively an expensive pretreatment not recommended for consumption because of the toxic residual barium. The brine was concentrated up to 37 wt% of salts in water, which is above the limiting salt ( $\text{NaCl}$ ) saturation concentration. Therefore, the volume of discharged RO brine can be reduced considerably facilitating its efficient management.

### 1. Introduction

Due to the exponential growth of the world population, seawater desalination has become a necessity to supply drinking water to certain areas. Reverse osmosis (RO) is currently the most used seawater desalination technology all over the world [1–3]. More than 50% of the

worldwide installed desalination plants use RO technology because of its simplicity, a relatively low energy cost compared to other processes and its continuous advancement attributed to the use of novel membrane materials, design of improved membranes and modules, coupling with other processes and optimization studies reducing further the specific energy consumption and the cost of water production [3].

\* Corresponding author at: Department of Applied Physics I, Faculty of Physics, University Complutense of Madrid, Avda. Complutense s/n, 28040, Madrid, Spain.  
E-mail address: [khaytm@fis.ucm.es](mailto:khaytm@fis.ucm.es) (M. Khayet).

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Nevertheless, RO cannot be used for the treatment of high saline concentration effluents ( $> 65$  g/L) since the osmotic pressure increases considerably with the salt concentration (above  $80 \cdot 10^5$  Pa) leading to a significant enhancement of the specific energy consumption (i.e. operation and maintenance costs due to the risk of crystallization fouling).

However, the major drawback of RO is the management of the high volume of the produced brine. Untreated or improperly managed brines can result in adverse environmental effects, due to their high salinity, organic and inorganic contaminants [4]. In fact, RO desalination plants usually discharge the concentrated brine to the sea, contributing to the pollution of the marine environment. In this sense, two new lines for the management of brines, zero-liquid discharge (ZLD) [5] or near-ZLD (feed water recovery of 95–98%) and zero desalination discharge (ZDD) [6] can be considered. ZLD aims to avoid the liquid waste while it generates saline solid products. In contrast, the ZDD usually reintroduces the liquid waste to the process or converts it into saline solids for raw material. The final objective of some designed processes such as the evaporative ones is to minimize as much as possible the volume of the discharged brine whereas other more complex processes are designed to selectively recover valuable salts.

Research studies dealing with the treatment of emerging RO concentrate are needed in order to develop cost-effective methods to minimize the potential impacts on the environment as well as alternative strategies to extract available salts and to recover purified water. Several strategies have been adopted for the treatment technologies of RO brines and some review papers have been published in the last 7 years [4,7–10]. Traditional disposal options for RO concentrate are surface water discharge, deep well injection and evaporation ponds [4,11]. Membrane-based, thermal-based or emerging technologies are capable not only to reduce RO concentrate volume, but when they are combined ZLD can be achieved [12]. The selection of the best available technology for concentrate volume minimization depends mainly on the characteristics of RO concentrate, the treated water quality, energy consumption and costs [4,9,13]. Emerging technologies, such as electrodialysis (ED) [14,15], forward osmosis (FO) [16–18], membrane distillation (MD) [19–23] or even its coupling with crystallization (MDC) [24,25], and eutectic freeze crystallization (EFC) [26–28], have been developed recently to reduce RO concentrate volume with the objective to achieve ZLD and to recover some valuable compounds present in RO concentrate. However, these technologies are still under development and operational data on large-scale facilities are limited.

MD is considered an efficient alternative, either alone or combined with FO or MDC, in terms of increasing water production whilst minimizing the volume of the generated brines as much as possible [18,19,22,25]. However, as it is well known, among other drawbacks of MD technology such as membrane pore wetting, it is limited by fouling and scaling phenomena [29–31]. Zhang, et al. [32] observed by means of scanning electron microscopy coupled with an energy dispersion spectroscopy (SEM-EDS) and inductively coupled plasma (ICP) analysis that permeate flux reduction during processing of seawater RO brine was firstly caused by precipitation of calcium carbonate ( $\text{CaCO}_3$ ) and calcium sulfate ( $\text{CaSO}_4$ ) due to their low solubility forming mixed crystal deposits on the membrane surface. The concentration of brine above the saturation point of salts resulted in the formation of crystals and the subsequent damage of the membrane [33,34]. These crystals could modify the membrane structure breaking the membrane fibrils and allowing the passage of liquid brine through the membrane pores (i.e. wetting of the membrane pores) [31]. Therefore, it would be necessary to find an appropriate procedure for the pretreatment of RO brines avoiding high energy consumption and/or obstruction or deterioration of the MD membranes.

Chemical treatments have been demonstrated to be a viable method for salt recovery from RO brines by using different reagents as sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) [7,35], sodium hydroxide (NaOH) [36], or calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) [25]. Drioli, et al. [37] performed a research study to recover  $\text{CaCO}_3$ , NaCl and  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  from nanofiltration

(NF) retentate using reactive precipitation by adding  $\text{NaHCO}_3/\text{Na}_2\text{CO}_3$  aqueous solutions to the NF retentate.  $\text{Ca}^{2+}$  ions were precipitated as calcium sulfate, which causes the reduction of  $\text{SO}_4^{2-}$  content in the solution, limiting the recovery of magnesium sulfate. Calcium and magnesium by-products recovery from an RO brine was carried out by Casas, et al. [36] using  $\text{Na}_2\text{CO}_3$  and NaOH reagents at  $25^\circ\text{C}$  and  $65^\circ\text{C}$ . Ji, et al. [25] used lime/soda ash ( $\text{Ca}(\text{OH})_2/\text{Na}_2\text{CO}_3$ ) softening to the RO concentrate in order to reduce calcium and magnesium hardness and limit scaling problems in MDC process. Qu, et al. [38] could control calcium scaling by accelerated precipitation softening (APS) prior direct contact membrane distillation (DCMD) process of the RO brine. The APS process involved pH adjustment with sodium hydroxide along with calcite seeding, followed by microfiltration (MF) to avoid seeds clogging of the DCMD module.

In this paper, different chemical pretreatments of seawater RO brine have been considered prior MD application in order to increase the water production rate and minimize the volume of RO brine. The treated RO brine has a concentration of 55 g/L total dissolved solids (TDS) being the most abundant salt the NaCl. The first step consisted on chemical pretreatments of RO brine in order to remove permanent calcium hardness, temporary calcium hardness and/or to reduce sulfates. These chemical pretreatments do not require solvents and/or inert dispersion agents, which prevent nucleation and crystal growth and minimize, as far as possible, the risk of incrustation formation on the membrane surface. Once the RO brine has been treated, the precipitates were removed by filtration techniques. The second step consisted on the treatment of the chemically pretreated RO brine by DCMD evaluating its performance in terms of the permeate flux and the brine rejection factor. The objectives were the concentration of the RO brine and the production of a good quality distillate. For these purposes, two polytetrafluoroethylene (PTFE) membranes with different mean pore sizes (TF200 and TF450) were employed.

## 2. Material and methods

### 2.1. RO brine composition

The treated RO brine was supplied by the company Abengoa Water S.L.U. and corresponds to the brine discharged by a RO desalination plant located in Almería (Spain). The RO brine composition was determined in the *Geochemical and Environmental Analysis Lab* of the University Complutense of Madrid (UCM), by using different techniques: ionic Chromatography (IC) for the determination of chlorides and sulfates; potentiometry to measure the alkalinity; Atomic Emission Spectrometry with Inductively Coupled Plasma (ICP-AES) to determine the concentration of Ca, Mg, Na, Si ions; and UV-Vis Spectrometry to determine the anionic detergents following the methylene blue active substances (MBAS) method. The obtained concentrations of the different components present in the RO brine are summarized in Table 1.

In order to characterize the non-volatile solutes of the RO brine, a semi-quantitative X-Ray diffraction analysis (XRD) was employed using a diffractometer X'Pert-MPD (Philips) at a wavelength of the Cu K $\alpha$  ( $\lambda = 1.54 \text{ \AA}$ ). The scanning range was varied from  $5^\circ$  to  $70^\circ$  in steps of  $0.4^\circ$ , with a scanning speed of 1 step/s. The operating conditions were 45 kV and 40 mA. The characterized solids were obtained once the liquid brine was evaporated. The results are presented in Table 2. A majority of NaCl is observed (Halite, 66.7%), followed by magnesium chloride bi hydrate (Bischofite, 15.2%) and calcium sulfate (10.1%).  $\text{CaCO}_3$  is associated with magnesium forming Dolomite ( $\text{CaMg}(\text{CO}_3)_2$ ) in less proportion. As it can be seen, in the used RO brine it was detected a major presence of sulfates than carbonates.

Among all salts present in the RO brines, both  $\text{CaSO}_4$  and  $\text{CaCO}_3$  are the less soluble salts. In addition, the solubility of these salts decreases with the increase of temperature, making it difficult for the treatment by DCMD. As it can be seen in Fig. 1, the solubility of  $\text{CaSO}_4$  has a

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