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Recovery of desalination brines: separation of calcium, magnesium and sulfate as a pre-treatment step

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ABSTRACT

Sustainable treatment of reverse osmosis (RO) desalination brines should be focused on achieving the highest global recovery of freshwater combined with the recovery of the valuable compounds. In this line of investigation, an integrated process where NaCl is transformed into chemical stuff based on the use of bipolar membrane electrodialysis was developed and reported previously. The efficiency of the membrane recovery step relies among other variables on the concentration and purity of the fed brines, making the application of different pre-treatment steps necessary. The objective of this study is to evaluate the viability of removing calcium, magnesium and sulfate from RO brines in order to obtain a purified solution rich in NaCl. The proposed system consists of a set of ion-exchange resins combining cationic- and anionic-exchange stages. Removal of calcium and magnesium using the weakly acid-resin Lewatit TP208 was evaluated. Whereas, the strong basic resin Lewatit Monoplus M600 was used for sulfate removal working in fixed bed columns.

Keywords: Brine softening; Ion-exchange resins; Hardness removal; Calcium; Magnesium

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1. Introduction

Desalination is an emerging and promising alternative for obtaining drinking water and water for urban, agricultural, and industrial use with a worldwide projected capacity of 98 million m^3/d by 2015 [1]. Currently, reverse osmosis (RO) is the leading technology for desalination with a global capacity of 39.4 million m^3/d, which is about twice the installed capacity of thermal desalination [2]. The major drawback of RO is the generation of brines as waste effluent. In desalination plants, generation of brine is about 55% of collected seawater [3]. Brine disposal in coastal desalination plants has been solved by direct discharge to seawater, a method that poses adverse environmental effects over marine ecosystems, as it has been deeply studied and demonstrated [4,5]. The alternatives for disposal of the concentrates from inland desalination plants have been, traditionally, deep-well injection, discharge to surface waters, and concentration of brines in evaporation ponds [6]. These management

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options have several drawbacks, as the requirement of large land areas, together with their adverse environmental impact, and so the development of better management technologies is a current demand [7].

The options for the treatment of RO brines should be focused on achieving the highest global recovery of freshwater combined with the recovery of the valuable compounds contained in the brines [8]. Thus, the purpose of this work is to develop an advanced treatment process for RO brines that enables to achieve two objectives: (1) to decrease the environmental concerns associated to the emission of these effluents, and (2) to recover the valuable compounds. RO brines are mainly composed by sodium chloride in a concentration range from 8 to 10 g/L NaCl in brines obtained from brackish water desalination to more than 70 g/L NaCl in brines generated in seawater desalination. In order to take advantage of the NaCl contained in brines, a process based on bipolar membrane electrodialysis (BMED) with the aim of recovering chemical commodities was developed. BMED was proved to be a technically feasible option for the production of 1.0 M acid and base solutions working with model seawater RO concentrates in a laboratory bench-scale set-up [9].

However, due to the complex composition of the fed brackish water and seawater, brines also contain other cations and anions that hinder the recovery of valuable compounds. In order to achieve the operation requirements of BMED and the improvement in quality of the recovered products, a brine pre-conditioning stage is required, with the aim of removing detrimental impurities before BMED application. According to the analysis of the chemical composition of brines [8], the major impurities that must be removed are calcium, magnesium and sulfate, due to the high concentration of these ions in brines and the associated problems to these compounds. The presence of calcium and magnesium salts cause the generation of scaling in membrane operations [10]. In RO, antiscalants are used to suppress the precipitation of mineral salts to a certain extent. However, the problem of scaling is not mitigated completely [11]. For example, in the zero-liquid discharge strategy, the primary RO is followed by the precipitation of calcium and magnesium, before the secondary RO achieves up to 90% water recovery rates. [12–15].

The usual methods for the removal of alkaline-earth metals from aqueous solution can be ordered as chemical precipitation, ion exchange (IEX), ultrafiltration, RO, electrodialysis and adsorption [16]. Softening of water using IEX resins has been deeply studied and this technology has applicability at industrial level for obtaining drinking water [17]. Cation exchange has been used for effective removal of Ca^{2+}, Mg^{2+} and other multivalent cations from spent regenerant NaCl solutions [18] and for production of high-purity electronic grade chemicals [19]. In the removal of hardness from seawater, previously to RO or electrodialysis desalination, lime softening is the most common process at industrial level [14,15,20,21]. The advantages of cation exchange over lime softening include complete removal of Ca^{2+} and Mg^{2+} since their removal is not controlled by mineral solubility, there is no need for pre- or post-treatment pH adjustment, better economics, and fewer precipitation problems [22].

The drawback of the presence of sulfate is related to its migration, together with chloride, towards the acid stack during BMED operation. To avoid the production of an acid mixture, sulfate should be removed from the RO brine. Technologies to treat sulfate include membrane technologies, processes based on ettringite formation, biological sulfate reduction, and precipitation with barium. Typically, these technologies have one or more technical and economical limitations [23]. Anion exchange can be a good option for the removal of sulfate due to the low capital cost compared to membrane processes and biological sulfate reduction [23,24].

The objective of this study is to remove divalent ions from desalination brines to provide a highly concentrated and purified NaCl solution that is fed to the membrane recovery step. The proposed system takes advantage of coupling cationic and anionic-exchange stages to remove the main impurities (calcium, magnesium, and sulfate) contained in RO brines (Fig. 1). Working in fixed bed columns, the calcium and magnesium removal capacity of Lewatit TP208 resin and the sulfate removal capacity of Lewatit Monoplus M600 were evaluated.

2. Materials and methods

2.1. Water source

Real RO brines were used to carry out the experiments. The source of these concentrates was the desalination plant located in Cuevas de Almanzora (Spain), where desalinated water is used for irrigation. About 50,000 m^3 of brackish groundwater are treated per day with an estimated conversion of 60%. Thus, 20,000 m^3 of RO brines are daily produced. The main characteristics of the RO brines used in the present study are depicted in Table 1. Accordingly, the concentrations of the main impurities to be retained in the IEX process are: $[Ca^{2+}] = 46$ mmol/L, $[Mg^{2+}] = 65$ mmol/L, and $[SO_4^{2-}] = 88$ mmol/L.

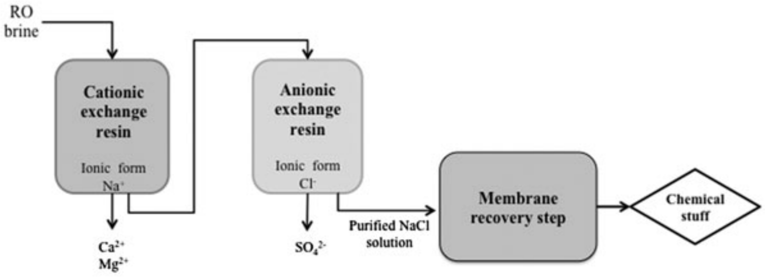

Fig. 1. Schematic treatment system based on IEX.

Table 1
Main characteristics of raw RO brine

Parameter	Raw RO brine	
pH	7.9 ± 0.1	
Conductivity (μS/cm)	34,300 ± 171.5	
TDS (mg NaCl/L)	20,700 ± 103.5	
Hardness (mg $CaCO_3$/L)	13,000 ± 577.4	
	Value (mg/L)	Value (mmol/L)
Chloride	7,279 ± 20.4	205 ± 0.6
Sulfate	8,465 ± 12.8	88 ± 0.1
Nitrate	447 ± 2.1	$7 \pm 3.4 \times 10^{-2}$
Sodium	5,176 ± 23.9	225 ± 1.0
Potassium	178 ± 5.9	5 ± 0.2
Magnesium	1,589 ± 46.8	65 ± 1.9
Calcium	1,828 ± 45.9	46 ± 1.1
Strontium	30 ± 1.1	$0.3 \pm 1.3 \times 10^{-2}$

2.2. IEX resins

The selection of IEX resins was based on previous studies reported in the literature [11,24,25] and recommendations of resins manufacturers. It has been reported that strong acid cation-exchange resins with sulfonic functional groups showed low efficiency in the treatment of seawater [25]. Therefore, Lewatit TP208, a weakly acidic cation-exchange resin was selected.

For the retention of sulfate the anion-exchange resin, Lewatit Monoplus M600, was chosen. Lewatit® MonoPlus M600 is a strongly basic anion-exchange resin (type II) designed for all demineralization applications.

Samples of Lewatit TP208 and Lewatit Monoplus M600 were kindly provided by the Spanish delegation of LANXESS Chemicals, S.L. The characteristics of both resins, as given by the manufacturer, are reported in Table 2 [26].

2.3. Experimental set-up

The scheme of the experimental set-up is showed in Fig. 2. Fixed bed experiments were carried out in a glass column having a diameter of 34 mm and a length of 300 mm. Experiments were performed at room temperature. The same scheme was used for the cation- and anion-exchange stages.

Raw RO concentrates were pumped using a Watson-Marlow 313S peristaltic pump. Approximately, 150 mL of wet resin were placed in the column and rinsed with deionized water. The breakthrough curves were obtained by analysis of successive samples of the effluent collected at regular time intervals. Breakthrough curves are represented using the bed volume (BV) parameter, which relates the operation flow rate, the resin volume, and the operation time:

$$BV = \frac{\text{flow rate} \times \text{time}}{\text{resin volume}} \tag{1}$$

This parameter represents the volumes of brine equivalent to resin volume that circulated through the IEX column for a time and a given flow rate. The detailed operation conditions for both cationic and anionic stages are shown in Figs. 3 and 4.

Table 2
Characteristics of selected resins: Lewatit TP208 and Lewatit Monoplus M600 [26]

Property	Lewatit TP208	Lewatit Monoplus M600
Matrix	Crosslinked Polystyrene	Crosslinked Polystyrene
Functional groups	Iminodiacetic acid	Quaternary amine, type II
Ionic form	Na^+	Cl^-
Density (approx. g/mL)	1.17	1.1
Total capacity, min (eq/L)	2.9	1.3
pH range	0–14	0–14

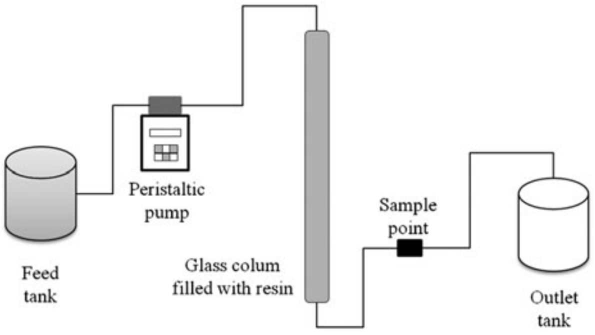

Fig. 2. Experimental set-up.

2.4. Analytical methods

The cation exchange was characterized by analyzing the hardness and the calcium concentration. The hardness was determined by EDTA titrimetric method according to Standard Methods 2340 C [27]. Calcium concentration was measured by EDTA titrimetric method according to Standard Methods 3500-Ca B [27], although it was observed that using eriochrome blue black R as indicator during titration the color changed from pink to purple, instead of the change from red through purple to a pure blue that is reported in Standard Methods. The difference was assigned to the high saline content of the RO brines used in the present study. A few selected samples were also measured by inductively coupled plasma-optical emission spectroscopy (ICP-OES) (Perkin Elmer ICP 400), to ensure that the titration measurement was correct. Magnesium concentration was determined as the difference between hardness and calcium as $CaCO_3$, according to Standard Methods 3500 Mg-B [27].

The concentration of sulfate and chloride were analyzed in a Dionex ICS 1100 ion chromatograph provided with an IonPac As-HC column, using a solution of 9 mM of Na_2CO_3 as eluent, with a flowrate of 1 mL/min and a pressure of around 2,000 psi, based on Standard Methods 4110B [27].

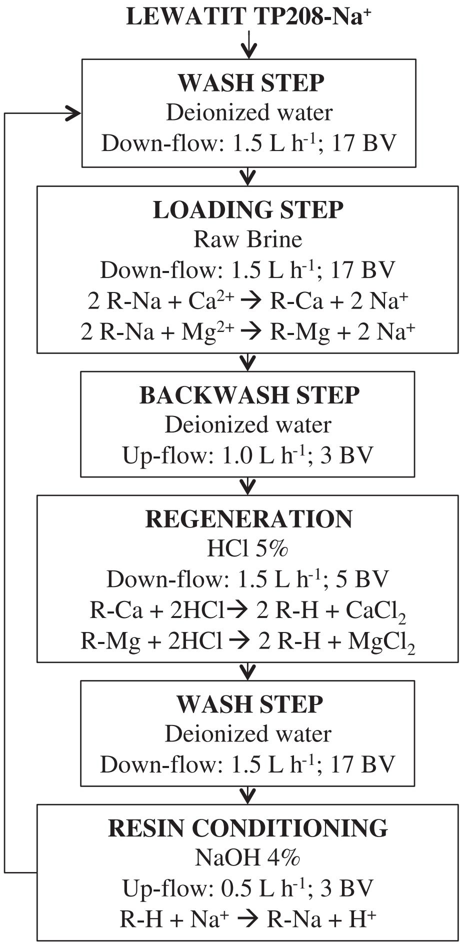

Fig. 3. Schematic diagram of the cation exchange stage (1 BV = 0.18 L).

3. Results and discussion

3.1. Calcium and magnesium removal

The breakthrough curves obtained in cation-exchange experiments are shown in Fig. 5, where the

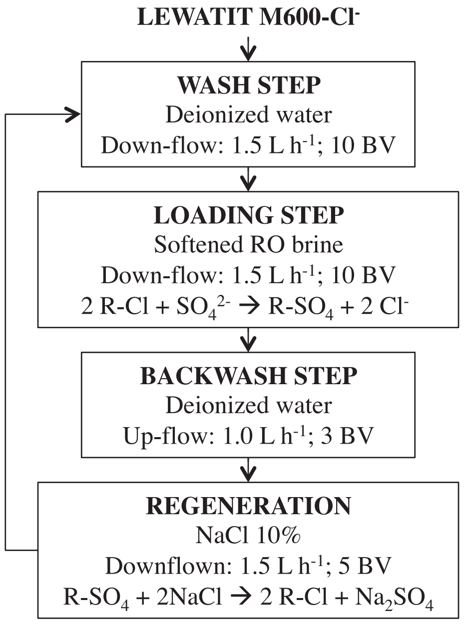

Fig. 4. Schematic diagram of the anion exchange stage (1 BV = 0.15 L).

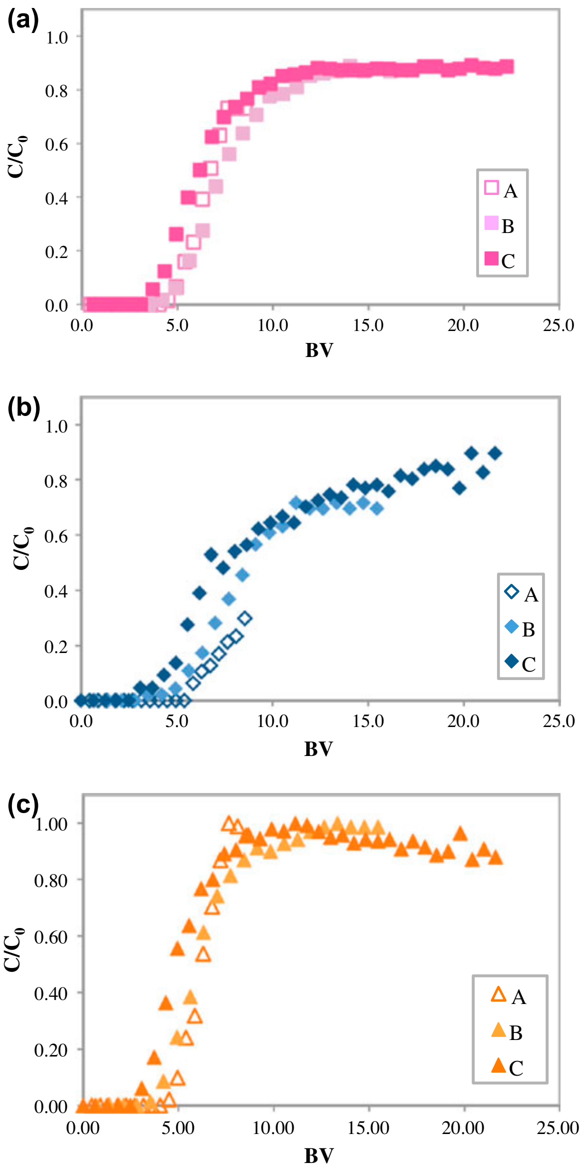

Fig. 5. Comparison of breakthrough curves obtained in the replications A, B and C with Lewatit TP208 for (a) hardness, (b) calcium, and (c) magnesium. Feed flowrate 1.5 L h^{-1} (flow velocity 4.33 × 10^{-4} m s^{-1}). Initial concentration C_0: 130 mmol $CaCO_3$/L hardness, 46 mmol/L calcium, 65 mmol/L magnesium.

dimensionless concentration (C/C_0) of species calculated over the initial concentration in the raw brine is depicted vs. BV. Three replicates were performed using the same resin bed regenerated according to the procedure detailed in Section 2.2. The total retention of cations, other than sodium, was confirmed until the break point that occurs after four BVs were flowed through the resin bed. Breakthrough curves show that the resin has more affinity for calcium than for magnesium, as indicates the affinity series given by the manufacturer (calcium > magnesium). Although the break point was reached at four BV for both cations, saturation of the resin with calcium occurred after the saturation of magnesium. To check if this hypothetical behavior was correct, the amount of calcium and magnesium absorbed onto the resin was calculated by a mass-balance relationship [28]:

$$q = \frac{v(C_0 - C)}{m} \tag{2}$$

where q is the amount of metal ions adsorbed onto the resin (mmol/g) at time t, V is the volume of the solution (L), m is the mass of the dry resin (g), and C_0 and C are ion concentrations in solution at beginning and time t (mmol/L). The adsorption curves for calcium and magnesium are shown in Fig. 6. The bed was not completely saturated at the end of the experiment, as the achieved value of q (2.2 meq/g resin) was still increasing after 22 BVs of the RO brine feed had been flowed through the column, although the value of the total capacity of the resin claimed by the manufacturer (2.47 meq/g resin) was being approached. The resin was saturated in magnesium before than in calcium. This behavior was also observed by Venkatesan and Wankat [11] working with IEX as pre-treatment for RO desalination of brackish and seawater. They

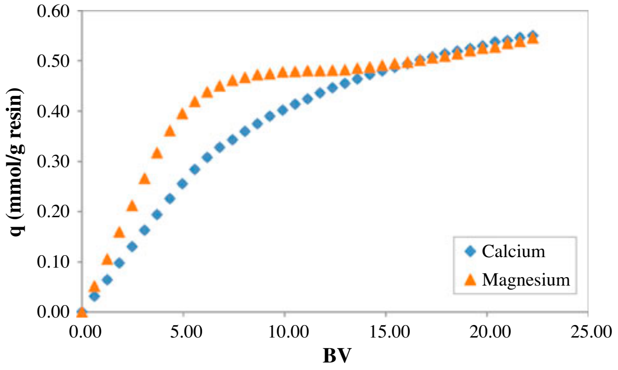

Fig. 6. Retention of calcium and magnesium onto Lewatit TP208 resin.

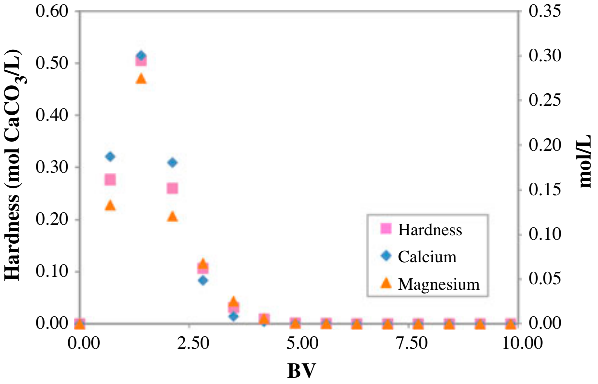

Fig. 7. Regeneration curve for Lewatit TP208 using HCl 5% as regenerant.

observed that Ca^{2+} breakthrough point occured after Mg^{2+} breakthrough point. According to the results achieved in this study, if the feed to the column is stopped when Mg^{2+} breakpoint concentration is reached (after approximately four BVs), the presence of calcium and magnesium salts in the next membrane stage for product recovery will be avoided, and thus membrane operation problems such as salts precipitation and membrane scaling can be prevented.

Regarding the effect of the number of cycles, the breakthrough curves are quite similar, which indicates that the regeneration process, performed as explained in Section 2.2, was effective to recover the cation-exchange capacity of the column. A total of eight complete consecutive loading-regeneration cycles were carried out, and the same results were obtained (break point at four BV).

The conditions of regeneration and the type of regeneration agent, hydrochloric acid, were selected according to manufacturer's recommendations. The use of sodium chloride brines for regeneration and conditioning is not impossible in theory but the capacity is expected to be very low. According to literature, the concentration of sodium chloride that is required to achieve effective resin regeneration should be very high, from 70 [29] to 300 g/L [30]. Moreover, the spent NaCl regenerant constitutes a waste stream that should be treated because the direct discharge of the spent regenerant causes environmental effects, even more adverse than desalination brines discharge. Treatment options for reusing this spent regenerant have been studied in the last few years [18,29,31–33].

As it is shown in Fig. 7, complete regeneration is achieved after four BVs of regenerant are flowed through the cationic resin bed. The efficiency of regeneration was calculated to be 93% (hardness as mg $CaCO_3$/L).

3.2. Sulfate removal

The objective of this stage was to remove sulfate while at the same time the effluent was enriched in chloride. The high concentration of sulfate in the RO concentrate, that is around 88 mmol/L, is a consequence of the particular geochemistry of the aquifers of the location of the desalination plant. According to the authors' knowledge, previous studies about sulfate exchange were performed with a much lower sulfate concentration than that found in RO brines [23,24,30], so, the feasibility of using this resin for high sulfate contents was evaluated in this study. Haghsheno et al. [24] worked with copper mine effluents with a sulfate concentration between 5.2 and 9.4 mmol/L by using the resin Lewatit K6362. Kratochvil et al. [23] used a solution based on groundwater composition with a sulfate average concentration of 15.6 mmol/L and chloride average concentration of 3.4 mmol/L. Comstock and Boyer [30] studied a combined IEX process in which anion-exchange resin and cation-exchange resin were used in the same vessel for simultaneous removal of DOC and hardness. The range of sulfate concentration varied between 0.1 and 8.6 mmol/L and 50% of sulfate removal was achieved. Sulfate concentration in the brine used in the present study is 10 times higher (88 mmol/L) than that in previous studies. Besides, this study also deals with high chloride concentrations (about 205 mmol/L).

The breakthrough curves for sulfate and nitrate are shown in Fig. 8. Although the objective of this stage was the removal of sulfate, experimental results show that nitrate was also removed due to the non-selective character of the resin [34]. The highest saturation point of sulfate removal for Lewatit Monoplus M600 resin was obtained before one BV had flowed through the column. Analyzing the adsorption capacity of the

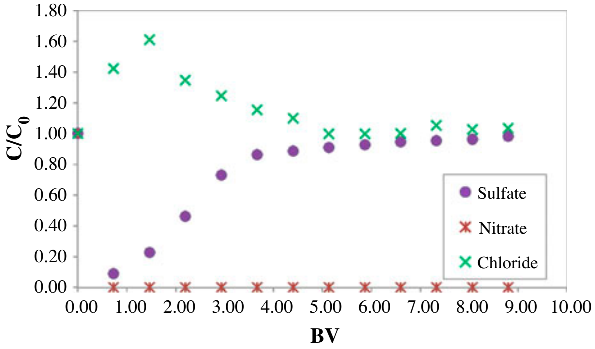

Fig. 8. Sulfate and nitrate breakthrough curves for Lewatit Monoplus M600; with feed flow was 1.5 L h^{-1} in all experiments (flow velocity 4.33 × 10^{-4} m s^{-1}) and evolution of chloride concentration during loading stage of Lewatit Monoplus M600. Initial composition: 88 mmol/L sulfate, 7 mmol/L nitrate, 205 mmol/L chloride.

resin (Fig. 9), it seems that resin has more affinity for nitrate, although the total adsorption of nitrate can also be explained because nitrate concentration is much lower than sulfate concentration. The affinity of the resin for the ions seems to be $NO_3^- > SO_4^{2-} > Cl^-$, as have been proven to be in nitrate specific resins [35].

Analyzing the evolution of the concentration of chloride (Fig. 8), the results show that chlorides initially increased in the effluent of the IEX bed as a result of their exchange by sulfate anions that were retained in the anionic resin, according to the expected behavior of the anion-exchange resin. The mass balance was calculated and it was found that the number of equivalents of chloride released by the resin was equal to the number of equivalents of nitrate and sulfate adsorbed. Therefore, by analyzing the evolution of the concentration of chloride it has been proved that the anion exchange has a correct development, but the resin volume seems to be not high enough to achieve the complete removal of sulfate.

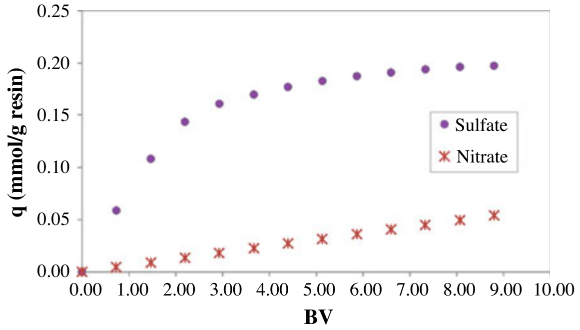

Fig. 9. Adsorption curves of sulfate and nitrate onto Lewatit Monoplus M600 resin.

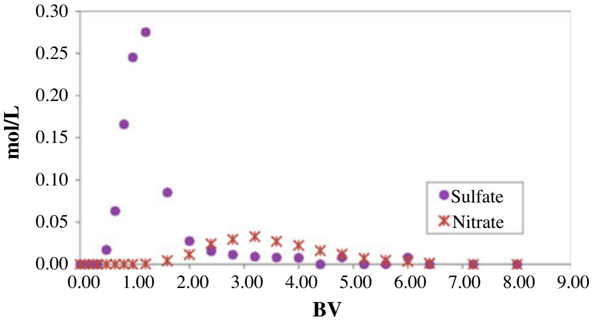

Fig. 10. Regeneration curve for Lewatit Monoplus M600 using NaCl 10% as regenerant at down-flow 0.72 L h^{-1} (flow velocity 2.33 × 10^{-4} m s^{-1}).

A solution of NaCl with a concentration of 10% was used as regeneration agent. The flow of NaCl stream was decreased to 0.72 L h^{-1} (flow velocity 2.33 × 10^{-4} m s^{-1}) in order to obtain a better representation of the regeneration curve (Fig. 10).

As it is shown in Fig. 10, sulfate removal was achieved after three BVs had flowed through the column and six BV of regenerant were required for complete regeneration taking nitrate release into account. The efficiency of regeneration was about 100% (both sulfate and nitrate concentration).

4. Conclusions

The purpose of this work is to study the removal of divalent ions from desalination brines to provide a highly concentrated and purified NaCl solution that is fed to the membrane recovery step.

The behavior of Lewatit TP208 cation-exchange resin in the removal of calcium and magnesium and Lewatit Monoplus M600 anion-exchange resin in the removal of sulfate from RO desalination brines was experimentally analyzed. Experiments were developed in fixed bed columns (150 mL BV) with feed flow of 1.5 L h^{-1} (flow velocity 4.33 × 10^{-4} m s^{-1}) according to the operation requirements established by the resins manufacturers.

Lewatit TP208 showed preferential exchange capacity for Ca^{2+} over Mg^{2+} so that the latter determined the maximum BVs that can be treated in the purification process. Eight complete consecutive loading-regeneration cycles were carried out, and reproducible results were obtained; so, it was concluded that the regeneration process was effective and the cation-exchange capacity of the column was satisfactorily recovered.

On the contrary, the anionic-exchange resin Lewatit Monoplus M600 showed poor retention of sulfate anions and the resin became almost saturated when <1 BV had flowed through the column. However, it was very effective in the retention of nitrate. Analyzing the evolution of the concentration of chloride it was proved that the anion exchange had a correct development, but the bed seemed to be not high enough to achieve the complete removal of sulfate.

Although the IEX resins showed adequate efficacy in the removal of calcium and magnesium from desalination brines, further studies aimed at the technical and economic assessment of the regeneration step that should be performed before application of the process.

Acknowledgements

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