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Review Article

Polymeric Ion Exchangers for the Recovery of Ions from Brine and Seawater

Mayyada M.H. El-Sayed[1,2]*, Heba A. Hani[1] and Mohamed H. Sorour[1]

[1]ChemicalEngineering and Pilot Plant Department, National Research Center, Egypt
[2]ChemistryDepartment, American University in Cairo, Egypt

***Corresponding author**
Mayyada M.H. El-Sayed, Chemical, Biochemical and Environmental Engineering Department, University of Maryland, Baltimore County (UMBC), MD, USA, Tel: 443 310 1321; E-mail: Mayyada@aucegypt.edu

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Abstract

This paper reviews the different types of ion-exchangers used for the removal/recovery of ions from brine and seawater. The applications of these exchangers in the fields of water softening, light and heavy metal removal as well as others are discussed. Exchangers including cationic, anionic and chelating agents are compared in terms of their structure, capacity and function. For water softening, most of the adsorbents are cationic of a gel-type structure where the polystyrene-based matrix is functionalized with sulfonic groups. Metal removal/recovery exchangers, on the other hand, are either cationic or chelating and are of a macroporous structure. The crosslinked polystyrene matrix could be functionalized with thiol, sulfonic or iminodiacetic groups for the cationic exchangers, and is conjugated with N-methyl glucamine functional groups for all the chelating ones. In addition, the ion-exchange processes employed for each application are reviewed with regard to the type of process, operating conditions as well as obtained recoveries. Ion exchange chelation proves to be the most specific for the removal of metals such as boron and uranium. This review will potentially help in selecting the ion-exchange material and process suitable for a certain application.

ABBREVIATIONS

RO: Reverse Osmosis; EDR: Electrodialysis Reversal; IEX: Ion Exchange; CEX: Cation exchange; AEX: Anion Exchange; R: Recovery; C: Column mode; b: batch mode

INTRODUCTION

Seawater is an almost inexhaustible source of raw materials. It is composed of 96.7% water and 3.3% dissolved salts. Seven elements (Na, K, Mg, Ca, Br, S, and Cl) account for 93.5% of the dissolved salts [1], the concentration thereof ranges between 33-37 g/l.

Desalination of seawater is a common method for providing fresh drinking water around the world. The use of reverse osmosis (RO) membranes is becoming an increasingly practical method of desalination, as a result of significant improvements in energy recovery systems and pre-treatment processes over the past couple of decades. However, disposal of the concentrated brine produced by the desalination process poses significant environmental issues due to the high concentrations of metals and salts.

The reject brine is more concentrated than seawater and has inorganic salts as its major constituents. It also contains small quantities of anti-scale additives, washing solutions and other reaction products. Recovery of selected elements from RO concentrate would provide environmental benefits, both in reducing the magnitude and environmental impact of disposal, as well as economic benefits in production of valuable metals [2,3].

Presented in (Table 1) are the ranges of concentrations of the major cations (Table 1A) and the average concentrations of the minor ions (Table 1B) present in either seawater or the brine that is produced from RO desalination plants.

The cations mentioned in (Table 1) have numerous applications in different fields. The monovalent cations Na and K are used in the fertilizers industry, while Rb is important in laser applications. The divalent cations, on the other hand, have various industrial applications. Thus, Mg finds use in the manufacturing of alloys, whereas Sr is used in electronics and colorants industries. In addition, Ca has applications in cement, glass and steel industries. The trivalent boron is also implicated in the manufacture of glass and porcelain, and uranium is employed in the nuclear fuel cycle.

The recovery of ions from brine could be performed by means of different physical and chemical techniques. The former includes electrodialysis, electrolysis, membrane distillation, membrane

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Table 1a: Composition range of seawater and brine with respect to major cations.

Target ion	Seawater concentration (g/l)	Brine concentration (g/l)
Na^+	10.77-26.70	0.085-72.10
Ca^{2+}	0.23-0.50	0.173-3.020
K^+	0.21-0.50	0.013-1.850
Sr^{2+}	0.008-0.133	0.007-0.028
Mg^{2+}	1.29-1.77	0.260-3.600

Table 1b: Average composition of seawater and brine with respect to minor ions.

Target ion	Seawater concentration (mg/l)	Brine concentration (mg/l)
Li^+	0.17	0.27
Rb^+	0.12	0.19
Ba^{2+}	0.03	0.2
Cu^{2+}	0.003	0.05
B^{3+}	5.0	2.0
$UO_2(CO_3)_3^{4-}$	0.0033	0.0039

separation, evaporation, and ion exchange adsorption; whereas the latter includes complexation, chelation ion exchange, precipitation and solvent extraction. Criteria for selection of the appropriate recovery technique include concentration of the feed water, physico-chemical properties of the target ion such as formulation, reactivity and concentration in water, as well as other economic and logistic factors. The current commercially-available methods for brine recovery as reverse osmosis (RO), electrodialysis reversal (EDR) and thermal evaporation are limited. Ion exchange is becoming more competitive with these methods due to the growing industrial demand for recovering specific target ions from seawater and brines containing low concentrations thereof. Ion exchangers used for brine recovery could be either inorganic or organic polymeric sorbents. Examples of the former are natural clays, natural and synthetic zeolites and other low-cost sorbents like fly ash and amorphous zirconium hydroxide. Polymeric organic adsorbents, on the other hand, are generally composed of solid polymeric matrices covalently attached to positively and negatively-charged functional groups such as sulfonic (RSO_3^-) in case of cation exchangers, and quaternary amine (R_4N^+) in case of anion exchangers. These polymers have the merit of providing selective removal of ions, in addition to their ability to overcome the economic and environmental limitations associated with other removal methods. They also allow for potential regeneration and reuse of the removed ions.

This paper focuses on the use of polymeric organic ion exchangers in the recovery of ions from seawater and brine. In what follows, the ion exchange processes employed for the applications of water softening, light and heavy metal removal/recovery as well as anion removal will be reviewed. In addition, the general characteristics of the commercially-available adsorbents used for each application will be compared. Cationic, anionic and chelating exchangers will be covered.

Water softening

The two main cations responsible for water hardness are calcium (Ca^{2+}) and magnesium (Mg^{2+}) and these are generally removed using cation exchangers having a divinyl benzene copolymeric matrix functionalized with sulfonic acid groups. Muraviev et al. used commercial Lewatit®cation exchangers to adsorb 0.02 g/l of Ca and 0.11 g/l of Mg from synthetic and natural seawater [4]. This adsorption was performed on a 1.4-cm diameter column at a flow rate of 3 ml/min and temperatures ranging from 10°C to 80°C. Entezariand Tahmasbi studied the adsorption equilibrium and kinetics of synthetic solutions of Ca and Mg salts using a combination of ion exchange and ultrasound [5]. The cation-exchange resin used for that purpose was a synthesized styrene-divinyl benzene copolymer with sulfonic acid group. Initial concentrations of 0.045 g/l of each of Ca and Mg ions were employed and batch adsorption took place at temperatures ranging from 10 to 80°C. In addition, a lab-prepared anion-exchange resin containing $MgX_2.2Al(OH)_3$ was synthesized by Lee and Bauman and used to adsorb Mg from synthetic brine [6]. Column adsorption was performed at pH 6, flow rate 21 ml/min and a temperature of 90°C. The general features of commercial polymeric adsorbents available for water softening are given in (Table 2).

Most of these adsorbents are cationic of gel-type structure, and consisting of a polystyrene-based matrix functionalized with sulfonic acid groups. A couple of Amberlite chelating resins are also commercially available for the removal of hardness from brine. These are characterized by a macroporous structure and a polystyrene matrix that is crosslinked with divinyl benzene and functionalized with either aminophosphonic or iminodiacetic groups.

Light metal removal/recovery

Light metals commonly found in seawater and brine are Na, Ca, Mg, K, Li, Sr and Rb. Apart from Ca, Mg and Sr which are alkaline earth metals, these metals all belong to the alkali metal group. In the previous section, the removal of Ca and Mg was discussed. Following is a review of the work that has been done for the removal of other light metals.

For monovalent ions, Steinberg and Dang recovered Li from seawater using a cation-exchange DOWEX 50-X16 column where Li was eluted using 0.2-0.5 M HCl [7]. Furthermore, Lee and Bauman employed a lab-prepared anion exchanger to remove 69% of the same ion from synthetic brine, along with other ions as Na, Ca, Mg, K, and B. Packed-bed adsorption was performed at pH 6, a flow rate of 6.4 ml/min and an average temperature of 87.5± 2.5°C [8].

The Na ion was removed by Yalala et al. from synthetic brine wastewater with the cation-exchanger Amberlite 252 RFH adsorbent and poly (amidoxime) fibers. The ion was adsorbed in a batch mode from synthetic NaCl solutions of concentrations ranging from 0.1-1.0 mg/l, where maximum adsorption capacities (q_{max}) of 25.03 mg/g and 16.40 mg/g were obtained in case of Amberlite 252 RFH and poly (amidoxime) fibers, respectively [9]. The same ion was also separated by Tanaka et al. from reverse osmosis (RO) desalination brine by means of an

Table 2: Water softening commercial polymeric ion exchangers.

Manufacturer	Product name	Type	Capacity (min) eq/L	Functional group
Lanxess Deutschland GmbH	Lewatit®S1467	gel	2 (H^+)	sulfonic
Lanxess Deutschland GmbH	Lewatit®MonoPlusS100	gel	2 (H^+)	sulfonic
Sybron Chemicals Inc. (Bayer)	Ionac C249 NS	gel	1.9 (Na^+)	sulfonic
DOWEX	DOWEX HCR-S	gel	2 (Na^+)	sulfonic
DOWEX	DOWEX Marathon C	gel	2 (Na^+)	sulfonic
Purolite	Pur®C100DK	gel	1.9 (Na^+)	sulfonic
Rohm & Haas	Amberlite™ IRC-747	macro-porous	1.75 (Na^+)	amino-phosphonic
Rohm & Haas	Amberlite™ IRC-748	macro-porous	1.35 (Na^+)	imino-diacetic

ion exchange membrane electrodialyzer with Ag-AgCl electrodes. An equivalent Na concentration of 1.5 eq/dm^3 was adsorbed on a 2 m^2 membrane at a feed velocity of 5 cm/s [10].

In addition, Rb with initial concentrations of 50-110 µg/l was selectively adsorbed from reverse osmosis (RO) concentrate on a macroporous sulfonated phenol formaldehyde cation-exchanger [3]. The divalent Sr ion, on the other hand, was removed with high efficiency from synthetic brine using a countercurrent cation exchange column of the polyacrylic acid resin KB-4 at pH 7 [11].

Table 3 gives a review summary of the work done on the removal and recovery of light metals. The table compiles the employed ion exchange material and method, as well as % removal for each target ion and the source of water from which this ion was removed. Table 4 depicts the main characteristics of the commercially available adsorbents that are used in this regard and these include their minimum capacity and functional groups. From the physical stability point of view and according to the manufacturers' documentation, the Amberlite and Lewatit Mono Plus resins are resistant to osmotic pressures and mechanical attrition and are stable over a wide pH range.

Heavy metal removal/recovery

From a strictly chemical viewpoint, heavy metals constitute transition and post-transition elements along with metalloids. They have higher specific gravities relative to light metals, often exist in different oxidation states, and are known to be toxic [12]. Gonzalez-Davilla et al. performed batch ion exchange experiments to remove divalent Cu from synthetic seawater using Chitin resin at a pH range of 2-9 [13]. Hg was also recovered from seawater by Sugil et al. They performed packed-bed adsorption on the cation-exchanger thiazoline at pH 1, and eluted the column with 0.1 M HCl/5% thiourea where an adsorption capacity of 2.8 mmol/g was obtained [14].

Some elements were selectively adsorbed using chelating resins. For example boron, where borate anion is known for its low retention on the normal anion exchangers, was recovered from brines both as boron ion or boric acid. In aqueous environments, dissolved boron is present as several species, depending on the concentration of boron. At the 5 mg/l concentration in seawater, boron is found in the form of the mononuclear species $B(OH)_3$ and $B(OH)_4$ [15]. Specific chelating adsorbents such as Amberlites were used for boron removal. The ion was removed from refined brine by Yan et al., both in a batch and column mode. Brine with boron concentrations of 40, 50 and 100 mg/l was adsorbed on the chelating resin XSC-800 at a pH range of 4-12, flow rates of 5, 16, and 30 BV (bed volume)/h and temperatures ranging from 20-50°C [16]. Duyvesteyn et al. also removed the same ion from geothermal brine using Amberlite and WOFATIT resins at an initial boron concentration above 0.1 g/l, pH of 4.0-5.5, and temperatures above 90°C [17]. Furthermore, boron at a concentration of 30 mg/l was removed from a synthetically-prepared geothermal waste brine solution using Amberlite IRA-743 [18], where it was possible to recover boron using single-stage regeneration with either 5% HCl or 5% H_2SO_4. Kabay et al. tested the performance of Diaion CRB02 and DOWEX XUS43594.00 for boron removal from model seawater. Batch experiments were conducted at different resin particle sizes and a maximum removal of about 96% was attained. During column experiments, both resins manifested good sorption performance [19]. In addition, the packed-bed adsorption of boron in the form of boric acid was investigated by Schilde and Uhlemann at an initial concentration of 196 mg/l, pH of 7.4-8.5, and flow rate of 2 ml/min. Boron was also recovered from brine containing alkali and alkaline earth salts using a 0.5 M HCl eluent, and a 3.5 g/l adsorption capacity was achieved [20]. Moreover, Mahdy et al. recovered boric acid from bittern on an Amberlite IRA-743 resin at an initial boron concentration of 38 mg/l, pH 3.9-6.3, flow rates of 0.5-1.0 ml/min and a 5% HCl eluent [21]. As revealed by the above review, the best performance for boron removal was manifested by the boron-specific adsorbents that are based on macroporous polystyrene matrices with N-methylglucamine ligands.

For the extraction of uranium (U) from seawater and brines, a number of chelating polymeric adsorbents were employed. Micro and macroporous polymers having dihydroxamic acid groups were studied for the uranium uptake from seawater, and it was found that introducing N, N-dimetholacrylamide into the hydroxamic acid polymer significantly increases the rate of adsorption [22]. Sodaye et al. proposed a polyamidoxime chelating resin grafted with calixarene for the extraction of uranium from the concentrated brine rejected by integrated nuclear desalination plants. The resin was selected due to its high selectivity for uranium, and the resin capacity was reported to be 5.2×10^{-4} mol/g when a column of 2.5 m height and 0.25 m internal diameter was filled with 66% volume of the resin [23]. Furthermore, Gibert et al. successfully extracted U (VI) from brine rejected by a seawater RO plant using the commercially available Purolite S910 chelating resin, and a capacity of 41.2 mg/g was achieved. In general, sorbents containing amidoxime

Table 3: Review of the metal removal/recovery methods performed on polymeric adsorbents.

Ion	Source	Method	Material	%R	Ref.
Rb	RO concentrate	CEX (b)	sulfonated phenol formaldehyde		[3]
Sr	synthetic brine	CEX (C)	polyacrylic acid KB-4	98	[11]
Li	seawater	CEX (C)	DOWEX 50-X16		[7]
Li	synthetic brine	AEX (C)	resin containing $LiX.2Al(OH)_3$	69	[6]
Na	synthetic brine wastewater	CEX (b)	Amberlite 252 RFH Poly(amidoxime) fibers	51.5 33.8	[9]
Na	RO brine	IEX electrodialyzer	Ag-AgCl electrodes		[10]
B	geothermal brine	chelation (b)	Amberlite IRA-743 WOFATIT MK51	80	[17]
B	refined brine	chelation (b, C)	XSC-800		[16]
B	brine	chelation (C)	WOFATIT MSK1		[20]
B	bittern	chelation (C)	Amberlite IRA-743	84	[21]
B	synthetic geothermal brine	chelation (C)	Amberlite IRA-743		[18]
B	model seawater	chelation (b, C)	DiaionCRB02 DOWEX XUS43594.00	96	[19]
Cu	synthetic seawater	IEX (b)	chitin		[13]
Hg	seawater	CEX (C)	thiazoline		[14]
U	seawater	chelation (b)	hydroxamic acid polymer		[22]
U	brine reject	chelation (b)	poly(amidoximie)-calixarene		[23]
U	brine reject	chelation (b)	Purolite S910		[24]

Abbreviations: IEX: ion exchange, CEX: cation exchange, AEX: anion exchange R: recovery, C: column mode, b: batch mode

Table 4: Metal removal commercial polymeric ion exchangers.

Manufacturer	Product name	Capacity (min) (eq/L)	Target ion	Functional group
Lanxess Deutschland GmbH	Lewatit®Mono Plus TP207 XL	2 (H^+)	Cu, U, Pb, Ni, Zn, Cd, Ba, Sr	imino-diacetic
Rohm & Haas	Duolite GT 73	1.4 (H^+)	Pb, Hg, Cu, Cd	thiol
Purolite	FerrLX™A33E		As	
Purolite	Purolite S910		U, Cu, Fe	aminoxime
Purolite	Purolite SST60		Ba, Ra, Sr, Mn, Fe	sulfonic
Rohm & Haas	Ambersep™ GT74	1.3 (H^+)	Hg, Rh	thiol
Rohm & Haas	Amberlite™ IRA-743	0.7 (Na^+)	B	N-methyl glucamine
Rohm & Haas	Amberlite™ PWA10	0.7 (Na^+)	B	N-methyl glucamine
Mitsubishi Chemicals	DiaionCRB01	1.2 (Na^+)	B	N-methyl glucamine
Mitsubishi Chemicals	DiaionCRB02	0.9 (Na^+)	B	N-methyl glucamine
DOWEX	Dowex™ BSR-1	0.7(Na^+)	B	N-methyl glucamine
Purolite	Purolite S108	0.6(Na^+)	B	N-methyl glucamine
Purolite	Purolite S110	0.8(Na^+)	B	N-methyl glucamine

groups were found to show uranophile properties that render them suitable for the selective recovery of uranium from aqueous solutions, including seawater [24]. The mechanisms responsible for the binding of U (VI) to amidoxime groups were acknowledged as complicated. It is widely believed that the chemical reaction involves decomplexation of $[UO_2 (CO_3)_3]^{4-}$ to UO_2^{2+}, followed by complexation of UO_2^{2+} with four amidoxime groups [23,24].

A review of the work done on heavy metal removal and recovery is presented in (Table 3). The relevant commercial polymeric ion exchangers and their main characteristics are given in (Table 4).

All of the metal removal exchangers mentioned in these tables have a macroporous structure and are based on a polystyrene matrix crosslinked with divinyl benzene. However, Amberlite™ IRA743 is based on a non-crosslinked polystyrene matrix, whereas Ambersep™ GT74 is based on a macroretinal matrix.

Table 5: Commercial polymeric adsorbents used for anion removal.

Manufacturer	Product name	Capacity (min) (eq/L)	Target ion	Functional group
Lanxess Deutschland GmbH	Lewatit®Mono Plus SR7	0.6 (Cl^-)	NO_3^-, NO_2^-, Br^-	quaternary amine
Lanxess Deutschland GmbH	Lewatit®Mono Plus MK51	1.3 (Cl^-)	BO_3^{3-}	polyalcohol
Lanxess Deutschland GmbH	Lewatit®Mono Plus FO36		HPO_4^{2-}, SCN^-, $HSbO_4^{2-}$	FeO(OH)
Sybron Chemicals Inc. (Bayer)	Ionac SR-7	0.8 (Cl^-)	NO_3^-	
Sybron Chemicals Inc. (Bayer)	Ionac A-554	1.4 (Cl^-)	NO_3^-	
Rohm & Haas	IMAC HP555	0.9 (Cl^-)	NO_3^-	quaternary amine
Purolite	Purolite®A520E	0.9 (Cl^-)	NO_3^-	quaternary amine

As seen in the table, cation-exchangers could be functionalized with thiol, sulfonic or iminodiacetic groups, whereas chelating adsorbents are all functionalized with N-methyl glucamine.

Anion removal

Monovalent anions of NO_3^-, NO_2^- and Br^- were recovered from synthetic brine using a hydroxylated polyacrylate matrix functionalized with quaternary NH_4^+ group. Adsorption was conducted on anion exchange column at a pH range of 2.0-11.8; while elution was carried out at a flow rate of 1.5 ml/min using KCl solution [25]. Table 5 gives a list of the commercial anion exchangers used for the removal of nitrates, nitrites, bromides, borates and oxoanions.

All of the aforementioned commercial anion exchangers are characterized by a macroporous structure, apart from the Ionac A-554 which has a gel structure. These adsorbents all have a polystyrene-divinyl benzene copolymer matrix and most of them are functionalized by quaternary amine groups.

CONCLUSION

In conclusion, the polymeric ion exchangers used for the removal/recovery of ions from seawater and brine are generally comprised of polystyrene or polystyrene-divinyl benzene polymeric matrices conjugated with functional groups, such as sulfonic, quaternary amine and thiol, depending on the application at hand. Most of the adsorbents suitable for water softening have a gel-type structure, while those employed for metal and anion removal possess a macroporous structure.

For metal removal, ion exchange chelation technology demonstrates significant benefits with regard to its specifity in removing elements as boron and uranium. Owing to its complexing and high selectivity nature, the chelating exchanger performance is not substantially affected by variation in operating conditions such as temperature, pH or salinity.

The commercial applications of ion exchange processes in the recovery of ions from seawater and brine are limited. This is because ion exchange becomes less economically viable as the concentration of the target ion in the feed water increases due to frequent exhaustion and hence the need for regenerating the ion exchanger. Ion exchange becomes, however, particularly competitive with other recovery methods at low feed concentrations of the target ion. It is therefore best used as a selective polishing step to ensure high quality of the treated water. Nevertheless, innovative ion exchange process design may help accomplish removal and recovery of fairly high concentrations of ions. More research work should hence be targeted toward investigating the regeneration of ion exchangers in order to develop the optimum process for ion recovery.

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