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Optimization of the electrodeionization process: comparison of different resin bed configurations

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ABSTRACT

Optimization of the EDI resin bed poses a serious challenge for the design. This research work deals with this optimization problem. It is based on the comparison of five different configurations (homogenous/mixed, cationic, anionic, inert and two layers ordered bipolar beds). The performance of the EDI systems was evaluated in terms of removal efficiency (RE), current efficiency (CE), power consumption (P) and was evaluated by the comparison of I–V curves. The ionic mobility was also calculated using Nernst–Planck equation. Our results are summarized by this efficiency order: two layers ordered bipolar bed > homogenous mixed bed > cationic bed > anionic bed > inert bed. This is particularly due to the bipolar effect between resin layers and the polarization decrease at the cation-exchange membrane and anion-exchange membrane surfaces.

Keywords: Electrodeionization; Efficiency; Ion exchange; Electrodialysis

1. Introduction

Electrodeionization (EDI) is a separation process combining electrodialysis (ED) and ion-exchange (IEX); the resulting hybrid process gained increasing attention for removal/recovery of ions from water [1].

Ion-exchange mixed bed is the primary deionization technology associated with ultra-pure water production. The discontinuity of the process and the need for high-purity regenerating chemicals constitute serious disadvantages. Likewise, ED is not an economical process for treating diluted solutions. High resistances, energy consumption and polarization (limiting current and precipitation) are the main drawbacks [2]. The EDI process eliminates these problems and can be considered, in principle, as an electrically regenerated IEX process or as an ED system whose conductivity and selectivity are aided by the presence of IEX particles in the dilute compartment. A typical EDI cell contains alternating permselective anion- and cation-exchange membranes, IEX resins and a pair of electrodes with direct current (DC) as a driving force. As the solution to be treated flows through the cell, the charged ions are captured by the IEX resin. When a voltage is applied across the cell, the captured cationic and anionic impurities begin to migrate across the resin bed in the direction of the appropriate electrode. This migration is controlled by ionic membranes. A cationic membrane will only allow positively charged ions to pass, while blocking the passage of negatively charged ions. An anionic membrane does just the reverse. Therefore, two types of chambers are created, one contains high ionic concentration, called concentrate chamber, and the other contains very low ionic concentration, called dilute chamber. The IEX resins enhance the transport of ions and can also participate as a substrate for electrochemical reactions, such as splitting of water into H^+ and OH^- ions [1,3]. Produced H^+ and OH^- ions continuously regenerate the IEX resins electrochemically without using regenerating chemicals. Besides, EDI can be considered as an environmentally friendly technology.

The EDI process is not a new idea; its concept has been extensively recognized since the mid-1950s. Walters et al. [4]

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investigated a batch electrodeionization process for the concentration of radioactive wastewater. Glueckauf [5] proposed a theoretical discussion of the technology and suggested an ion removal mechanism that involved two stages: (i) diffusion of ions into the resin and (ii) ionic transfer through the resin bed. Sammons and Watts (1960) used multi-cell EDI modules for the deionization of sodium salt solutions and quantified the relationships between solution concentration, flow rate and applied current [6]. An extended investigation of operating conditions and performance of the EDI process was conducted by Matejka [7] in the 1970s for high-purity water production. In 1998, Verbeek et al. [8] proposed a model to predict IEX bed behavior via digital simulations. Spoor et al. [9–11] studied the migration of nickel ions in an electrodeionization system. They determined the flux of the ion by the Nernst–Planck equation, taking into account only mass transport via migration. In addition, there are some literatures on water dissociation in EDI system [12–15].

Since the initial commercialization in 1987, EDI systems have found worldwide application in industries requiring high-purity water such as in the manufacture of pharmaceuticals and semiconductors, in power plants, surface finishing, and others [1,3,16–18]. Also, the EDI technique has received great attention in wastewater treatment and chemical purification/recovery [1,18–21].

The equipment used for most EDI studies comprised a number of test cells of plate and frame design, spiral wound design, rectangular and cylindrical shape and a variety of sizes. A number of these EDI cells possess dilute compartments filled with IEX resin either as a mixed bed, as clustered beds, as layered beds or as separated beds [22–26]. The results of the operation may vary depending on the method of resin filling between the IEX membranes. Other configurations using IEX textiles material instead of resins in the feed compartment were also tested [27–30].

Various modifications of the original EDI stack design have been proposed in recent years to improve the efficiency of the process [31,32]. However, increasing research efforts on elucidation of detailed operational mechanisms is still necessary for a better choice of these systems.

Thus, in order to determine optimal operational conditions, process parameters and resin bed configuration have been investigated by a number of researchers [33–36].

However, comparisons of five different configurations (homogenous/mixed, cationic, anionic, inert and two layers ordered bipolar beds) can only be found in this study. The present research endeavors to examine the influence of configuration on the process efficiency under similar experimental conditions and measures the ionic mobility in the bed of an ion exchanger by using Nernst–Planck equation. The experiments have been carried out with a laboratory-scale pilot with 0.006 M NaCl solution.

2. Experimental

2.1. Description of the cell

The EDI cell used in this study was machined out of plexiglass. This cell consisted of three vertical compartments (Fig. 1).

The central compartment is located between two ion-selective membranes positioned at the distance of 2 mm. The effective area of the membranes inside the cell is 2.88 cm^2 (3.2 cm × 0.9 cm). This compartment contains the IEX resin bed. It is percolated by a salty solution from the feed tank.

The cathodic left compartment is separated from the central compartment by the cation-exchange membrane (CEM). It contains a rectangular carbon electrode embedded in the plexiglass. The compartment volume is 25 cm^3 filled with a buffer solution at pH 7. The solution is in free convection.

The anodic right compartment is symmetrical to the cathodic compartment (it has an identical electrode, solution and dimensions). It is separated from the central compartment by an anion-exchange membrane (AEM).

2.2. Materials

The IEX resins used in the dilute compartment were obtained from Merck (Germany).

The anion- and cation-exchange membranes were Selemion AMV and CMV membranes (Asahi Glass, Japan).

The properties of the IEX resins used are listed in Table 1 and IEX membranes used in Table 2.

All experiments were carried out using the following chemicals (high-purity grade) from Merck (Germany): NaCl, Na_2HPO_4, H_2O and NaH_2PO_4, H_2O and pure water (doubly distilled).

2.3. Procedure

We have used a four electrodes cell with three compartments. The cathode and anode compartments of the cell were separated from the central compartment with the Selemion CMV cation-exchange and Selemion AMV anion-exchange membranes. The central compartment is the dilute compartment. It was filled with the IEX resin. Different configurations were tested: homogenous/mixed, cationic, anionic, inert and two layers ordered bipolar beds.

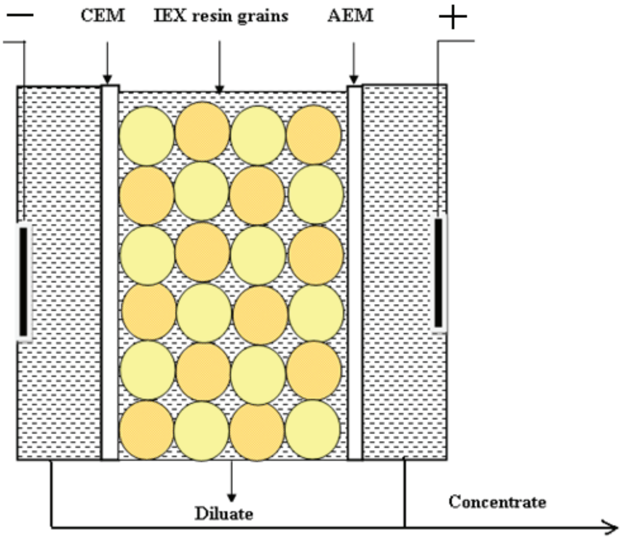

Fig. 1. Experimental electrodeionization pilot.

Table 1
Ion-exchange resin properties [2]

	Strongly acidic cation-exchange resin	Weakly basic anion-exchange resin
Type	Merck/Art. 4765 (LAB)	Merck/Art. 4766 (LAB)
Apparent volumic mass, g mL^{-1}	0.8	0.5
Ion-exchange capacity, meq g^{-1}	4.5	5
Water content, %	45–55	35–45
Granulometry, mesh (ASTM)	20–50 or 0.3–0.9 mm	20–50 or 0.3–0.9 mm

Table 2
Ion-exchange membrane properties [2]

	Cation-exchange membrane (CEM)	Anion-exchange membrane (AEM)
Type	Selemion CMV	Selemion AMV
Efficient area, cm^2	2.88	2.88
Thickness, mm	0.15	0.14
Ion-exchange capacity, meq g^{-1}	2.4	1.9
Resistance, Ωcm^2	2–3.5	2–3.5

Strong cation-exchange resins alone were used in cationic bed, while weak anion-exchange resins were used in anionic bed and inert particles of similar size were used in inert bed configuration. In the mixed bed configuration, the central compartment was filled with an intimate mixture of both cation-exchange resins and anion-exchange resins in a 1:1 ratio. The ordered bipolar bed is composed of two juxtaposed IEX layers of opposite charges: the anion-exchange resins in contact with the anion-exchange membrane and the cation-exchange resins in contact with the cation-exchange membrane.

Carbon electrodes were used as anode and cathode and the potential difference across the resin bed was measured by using two references golden electrodes.

The EDI performance was evaluated in terms of removal efficiency (RE), current efficiency (CE), power consumption (P) and by the comparison of I–V curves.

The characteristics of I–V (intensity of the current according to the transmembrane voltage) are obtained by taking measurements in potentiostatic mode using an apparatus Autolab (PGSTAT 30 METROHM). Measurements are taken with a speed equalizes of 0.01 V s^{-1} and a step of 0.05 V. We have applied a similar procedure of demineralization described in previous study [2].

The solution treated was a NaCl solution of 6×10^{-3} M. This solution flowed into the dilute compartment at a constant flow rate of 1.5 mL min^{-1}. Samples (15 mL) were collected from the central compartment by regular time intervals for analysis.

The solutions in the cathode and anode compartments of the cell are pH buffered by hydrogen/dihydrogen phosphate. The pH was kept stable (near 7) during the run. This eliminates H$^+$ and OH$^-$ interference in the dilute compartment.

The pH of the treated solution was measured with a Radiometer pH/ion meter apparatus (240 M). The pH remained almost constant until polarization where pH variations were then observed.

All experiments were carried out at similar room temperatures (≈25°C). The initial form of the IEX resins was Na$^+$ for cationic and Cl$^-$ for anionic resins (saturation step with NaCl followed by a long rinsing operation).

To calculate the removal efficiency (RE), we measured the inlet and outlet conductivities of the percolating solutions with a Tacussel apparatus. A calibrating curve converts conductivities to concentrations.

The removal efficiency RE is defined by:

$$\mathrm{RE}(\%) = 100\frac{\Delta C}{C_i} \tag{1}$$

In this equation, $\Delta C = C_i - C_f$, C_i represents the initial concentration; C_f represents the final concentration, in mmol L^{-1}.

Power consumption (P in Wh eq^{-1}) during EDI process represents the amount of energy needed to transport one gram equivalent of NaCl from dilute compartment to concentrate compartment. P has been calculated using the following equation [17]:

$$P = \int_0^t VIdt \,/\, w \tag{2}$$

where V is the applied voltage; I is the current (amp); dt is the time (h) allowed for the EDI process and w is the weight of salt (eq) removed.

The current efficiency (CE) was defined as the ratio of the current that transfers salt to the total amount of applied current [34]. CE has been calculated using the following equation:

$$\mathrm{CE} = \frac{\mathrm{QF}(Nf - Np)}{nI} \tag{3}$$

Q: the module dilute flow rate (L s^{-1}); F: the Faraday constant (C eq^{-1}); Nf: the feed water normality (eq L^{-1}); Np: the product water normality (eq L^{-1}); I: the DC current (amps); n: number of compartments.

3. Results and discussion

3.1. Current–voltage curves

A comparative study was performed using five different configurations: homogenous/mixed, cationic, anionic, inert and two layers ordered bipolar beds. The results are given in Figs. 2–4. The curves were obtained at the same average linear velocity of solution.

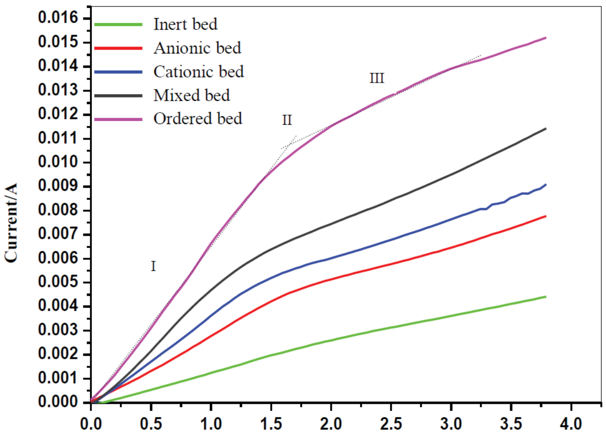

Fig. 2. Current–transmembrane voltage curve of continuous EDI.

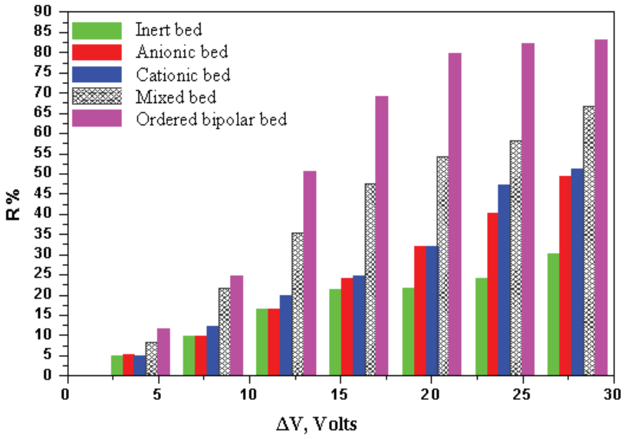

Fig. 3. Influence of operated voltage on EDI efficiency for different configurations.

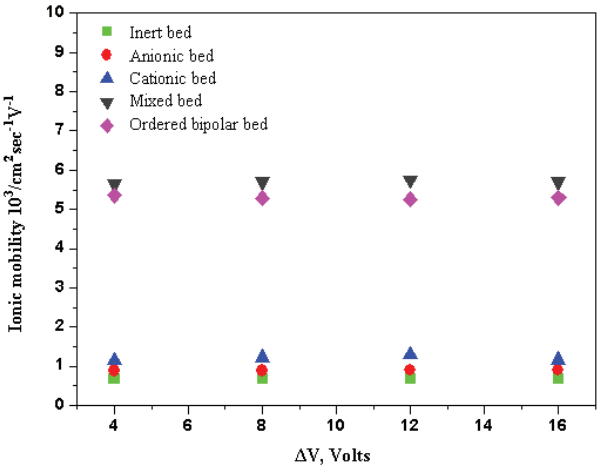

Fig. 4. Influence of operated voltage on ionic mobility for different configurations.

Fig. 2 shows typical current–transmembrane voltage curves for the EDI configurations. For all the studied configurations, three main regions appeared in the I–V relations. In the first region, the intensity of the current increases linearly with the transmembrane potential and the slope is higher than that of the third region where we also attend a progressive increase in the intensity of the current. Also, curvature region (region II) existed between those two linear regions (regions I and III). The value of the limiting current (I_{lim}) is in the curvature region (region II) determined by tracing resistance (U/I) vs. the reverse of the current (1/I). This value is at the intersection of the tangential lines (presentation of Cowan–Brown).

I_{lim} thus determined, present the following ascending order (Table 3):

Inert bed < anionic bed < cationic bed < mixed bed < ordered bed.

The current flowing through the dilute compartment in EDI system is complex since it is composed of two phases with different properties: ion conducting media and interstitial liquid. There are three possible transport paths of an ion through a bed of IEX particles. The first path leads through alternating layers of resins and interstitial solution, the second solely through the resin particles which are in contact with one another and the third exclusively through the interstitial solution. The EDI system is usually operated with a low concentration solution. Thus, more current is expected to flow through the second paths because the resistance of the interstitial solution is higher than the IEX media. The use of an inert bed allows us to estimate the resistance of the interstitial solution and compare its importance relative to other configurations with resin (Table 3), The values of the limit resistance calculated from the limiting current and limit voltage show the following ascending order:

Ordered bed < mixed bed < cationic bed < anion bed < inert bed.

From these results, we can see that the introduction of resins in the demineralizing compartment (dilute compartment) significantly reduced the resistance of the system, indicating that the preferred path of the ions is the resin that is virtually the only conductive region in the case of solutions very dilute. It also appears that the concentration polarization is more significant with the anionic and cationic beds than the mixed bed or ordered bed in two juxtaposed layers. This observation can be explained by the difference in mobility of ions between the resin and the solution, creating a charge accumulation of the same sign at the interfaces of ion exchangers.

Table 3
The values of limiting current, resistance and limit voltage of different configurations considered

Configuration	Inert	Anionic	Cationic	Mixed	Ordered
I_{lim} (mA) Cowan–Brown	2.50	4.61	5.06	5.8	10.126
R_{lim} (ohm)	744.37	348.77	272.16	202.91	147.79
V_{lim} (V)	1.86	1.60	1.376	1.17	1.51

3.2. Removal efficiency, current efficiency and power consumption

The RE, CE and P value are the crucial parameters of EDI process. For an efficient EDI process the *P* value should be lower, and RE and CE values will be higher.

Fig. 3 shows the influence of operated voltage on EDI removal efficiency for different configurations. As can be seen from Fig. 3, the removal efficiency of anionic and cationic bed configurations are comparable and relatively low compared with the mixed or ordered bed, because the resin in this case facilitates the passage of only one type of ion. The removal efficiency is becoming more important with the use of a mixed bed. However, at high values of potential, this remarkable increase in RE is limited due to the phenomenon of concentration polarization that persists despite the presence of resins. It is now well known that a phenomenon such as the dissociation of water is increased in the contact areas of the ion exchangers of opposite signs.

Moreover, the system of EDI with a resin bed ordered in two bipolar layers has the best performance in terms of removal efficiency (RE) on all the range of potential considered. These results show also a limit value of potential, from which an increase in electrical potential difference does not affect significantly RE. This value is considered as an optimum, and its exceeding only leads to additional energy consumption. This proves that the increase in the effective area of IEX membranes in the ordered bed configuration is not enough to compensate the catalytic activity of water dissociation.

Also using the experimental data, the performance of the EDI system was evaluated in terms of current efficiency (CE) and power consumption (P).

P and CE values have been calculated for all the configurations at different applied potential during EDI operation. These results are summarized in Table 4.

It is observed that CE decreased while P increased with an increase in the applied potential from 8 to 20 V for all type of configurations. The CE and P values of ordered bipolar bed are close to mixed bed. Globally the EDI process with a layered bed appears to allow the best results.

3.3. Ionic mobility

The calculation of ionic mobility through the resin beds was also examined by using the method developed by Spoor et al. [9]. This technique is based on investigation of ion transport through the system, which involves ion-exchanger bed and membrane, under the influence of applied potential.

Table 4
Comparative value of CE and *P* of different configurations during EDI process

	Voltage (V)	8	12	16	20
Bipolar bed	CE (%)	88.6	70.3	60.5	37.6
	P (Wh eq^{-1})	241	457	708	1,423
Mixed bed	CE (%)	80.8	73.4	66	38.8
	P (Wh eq^{-1})	265	438	648	1,417
Cationic bed	CE (%)	60.7	58.9	48.4	40.2
	P (Wh eq^{-1})	353	546	885	1,332
Anionic bed	CE (%)	57	56.7	53.2	53
	P (Wh eq^{-1})	372	556	741	1,332
Inert bed	CE (%)	60.9	59.9	55.4	40.9
	P (Wh eq^{-1})	351	537	773	1,308

It is well known that the conductivity of an IEX resin is considerably higher than that of liquid especially when the ionic concentration of the solution is quite low. It is reasonable to assume that the ion-conduction path is predominantly through the resin phase. The transport of ions through a homogenous bed of IEX resin can be described by Nernst–Planck relationship [9,37,38]:

$$J_i = D_i \frac{dci}{dx} + z_i C_i U_i \text{grad}\varphi + \nu Ci \tag{4}$$

The first term describes the diffusion of ion with concentration C_i and diffusion coefficient D_i. The second term accounts for migration of ion *i* under a potential gradient grad ϕ, z_i and U_i being its valence and mobility, respectively. The third term describes the convection flux due to the movement of the liquid phase within the IEX materiel. In this paper, the diffusion and convection terms will be neglected and the Nernst–Planck equation reduces to the migration term as assumed previously by Spoor et al. [9]:

$$J_i = U_i z_i C_i \text{grad}\varphi \tag{5}$$

The mobility values calculated using the Nernst–Planck simplified equation for different configurations are illustrated by Fig. 4.

As can be seen from Fig. 4, the ionic mobility is more or less independent of the applied voltage, and this regardless of the configuration considered. The values of calculated mobilities are about 6.6 × 10^{-4}, 8.8 × 10^{-4}, 1.2 × 10^{-3}, 5.7 × 10^{-3} and 5.3 × 10^{-3}, respectively, for the configurations inert, anion, cationic, mixed and ordered. According to these results, we thus see clearly the electric conducting role of the resin. It is remarkable to note that ordered and mixed bed configurations allow the ions to be more mobile than in the solution.

4. Conclusion

Optimization of the EDI resin bed was investigated. It is based on the comparison of five different configurations.

The results of this study showed that the introduction of the resins in the desalting compartment has significantly reduced the resistance of the system particularly in the ordered bed.

The application of the Nernst–Planck migration equation gives directly the numerical value of the average ion mobility. It can be seen that ionic mobility in mixed and ordered bipolar bed configurations is accelerated up to eight times compared with the ionic mobility in aqueous solutions.

There is a threshold at which an increase in applied voltage does not affect significantly the EDI efficiency. This value is considered as an optimum, and must not be exceeded to reduce polarization problems and to improve the lifetime of the membranes.

The EDI process, which uses ordered bipolar configuration, is promising for the production of high-quality water. A high efficiency is obtained in comparison with the classical EDI process using mixed bed.

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