An innovative compound bed of EDI device with enhancing ion-exchange resins regeneration efficiency
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ABSTRACT

Electrodeionization (EDI) technology is limited by low regeneration efficiency of ion exchange resins, requirements of high-quality influent water, fouling of the ion exchange membrane and electrode, etc. In this work, a novel bed type called a compound bed in which cation and anion exchange resins were near the cation and anion exchange membrane and placed in layers, was proposed to implement high-efficiency regeneration of ion exchange resins. The influence of different operating conditions on the regeneration efficiency of ion exchange resins was elucidated as well. The regeneration efficiency of ion exchange resins could reach 73.1%, when the device was operated for 5 h under current density of 9 mA/cm², with a cation and anion exchange resins ratio of 2:3, influent water conductivity of 1,360 μS/cm and hardness of 400 mg/L. Therefore, the proposed compound bed structure not only widened the inlet water conditions, but also achieved the high-efficiency regeneration of ion exchange resins and anti-fouling of membranes and electrodes.

Key words | compound bed, electrodeionization, ion exchange resins, regeneration

HIGHLIGHTS

- A novel bed type called a compound bed (CB) was proposed.
- CB structure facilitated water dissociation reaction and shortened the path of ion migration.
- Optimized CB structure possessed high regeneration efficiency of ion exchange resins.
- Optimized CB structure conferred anti-fouling performance on this device.

GRAPHICAL ABSTRACT

INTRODUCTION

Electrodeionization (EDI) combines the ion exchange resins (IER) and ion exchange membrane (IEM) to produce hydrogen ions (H⁺) and hydroxide ions (OH⁻) in the water
dissociation reaction under the action of a direct current electric field. The continuous regeneration of IER is implemented by electrochemical method without chemical regeneration (Arar et al. 2014). Thus, this novel and friendly separation process has been widely applied to industrial production, such as heavy metal recovery (Dermontzis et al. 2010; Lu et al. 2010), microelectronics, electric power, chemical industry (Strathmann 2010), and desalination (Wardani et al. 2017). But further research is still in progress for reducing energy consumption (Lee et al. 2007), preventing membrane and electrode surface scaling (Song et al. 2005; Aguilera et al. 2021), continuous operation (Li et al. 2017), etc.

High energy consumption of the EDI process is a factor restricting its large-scale industrial application, because the acid and base generated by the regeneration process is not utilized effectively, which enormously impacts the regeneration efficiency of IER. Therefore, many efforts have been concentrated on regenerating IER efficiently (Jordan et al. 2020), in which reducing the overall stack resistance and enhancing water dissociation reaction are the main purpose (Hakim et al. 2020). For example, an IEM with high conductivity and high selectivity can reduce energy consumption as higher ionic transfer and lower stack resistance (Zhao et al. 2018). A narrow particle size distribution of IER exhibits a lower stack resistance and better ion transport than standard IER distribution (Lu et al. 2021). Besides, the water dissociation reaction in the separated-bed configuration can be enhanced by inserting a bipolar membrane between the two beds (Strathmann 2010; Lakehal & Bouhidel 2017).

Although these methods have successfully enhanced the regeneration of IER, it is considered to be a more economical and effective method to enhance the regeneration of IER by changing the bed type (Jin et al. 2018). Notably, the regeneration efficiency of IER can be promoted by placing the same type of IER together (Golubenko et al. 2015). Consistent with the traditional bipolar membrane process, this method enhances water dissociation reaction, thus improving the regeneration efficiency of IER. Different from the traditional bipolar membrane process, this method is also favorable for formation of a great many channels, rapid migration of ions and enhancement of ion conduction efficiency and current efficiency, because the same type of IER relatively concentrates.

In this work, a special IER bed type called a compound bed was proposed to increase the regeneration efficiency of IER. The effects of bed type, current density, regeneration time and membrane spacing on the regeneration process of IER were investigated in the calcium magnesium simulation solution. At the same time, the anti-scale property was explored by changing the proportion of IER.

**METHODS**

**Materials and experimental instruments**

Sodium hydroxide (NaOH), silver nitrate (AgNO₃) and potassium dichromate (K₂Cr₂O₇) were obtained from Sinopharm Chemical Reagent Co., Ltd China. Calcium chloride (CaCl₂), hydrochloric acid (HCl), magnesium chloride (MgCl₂), sodium bicarbonate (NaHCO₃) and chromium black T were obtained from Tianjin Kernel Chemical Reagent Co., Ltd China. Ethylenediamine tetracetic acid disodium (EDTA) was obtained from Tianjin North Tianyi Chemical Reagent Factory, China. All reagents were analytical grade. The key performance parameters of heterogeneous polypropylene IEM (Hangzhou Lyhe Environmental Technology co. Ltd) are shown in Table 1. The simulated solution contained 3.33 M CaCl₂, 1.17 M MgCl₂ and 6.03 M NaHCO₃, and the total hardness and conductivity were 400 mg/L (calculated as CaCO₃) and 1,360 μS/cm, respectively. Besides, the necessary instruments are shown in Table 2. The key performance parameters of IER (Sinopharma Chemical Reagent Co. Ltd), including Na⁺ type 732 cation exchange resins (CR) and Cl⁻ type 717 anion exchange resins (AR) are listed in Table 3.

**Description of EDI device**

The EDI experimental setup, made by acrylic plate, was divided into three chambers, as shown in Figure 1. The cathode and anode chamber (concentrated chambers) approached cation exchange membrane (CM) and anion exchange membrane (AM), respectively. The solution in the cathode and anode chambers was pumped under the flow rate of 600 mL/min. The regeneration chamber (diluted chamber) was formed between CM and AM, in which 30 g IER was filled in different methods and proportions. The

**Table 1 | Key performance parameters of ion exchange membrane**

| Performance parameters                  | Cation exchange membranes | Anion exchange membranes |
|----------------------------------------|---------------------------|--------------------------|
| Membrane resistance (Ω cm²)            | 11                        | 13                       |
| Membrane thickness (mm)                | 0.42                      | 0.42                     |
| Selective transmittance (%)            | 93                        | 95                       |
| Permeability of water (mL h⁻¹ cm⁻² MPa⁻¹)| 10                        | 10                       |
| Ion exchange capacity (mol kg⁻¹)       | 2.0                       | 1.8                      |
| Heat stability (°C)                    | <42                       | <42                      |
The distance between AM and CM was 10 mm, and the effective area of the IEM was about 66 cm². The distance between anode (cathode) and AM (CM) was 30 mm. Additionally, the cathode and anode were Ti/RuO₂, and the effective area of each electrode was 42 cm². The compound bed (CB), in which AR and CR are near the AM and CM and are placed in layers, is different from the mixed bed (MB), a relatively mature and traditional bed type where CR and AR are mixed uniformly in a certain ratio. In the experiments, the ratio of CR and AR was 2:3.

Regeneration experiment

The regeneration experiments of IER were carried out in complete failure state. The AR and CR were pretreated by ultrasound in the simulated solution, respectively. The conductivity, and pH value of the solution were measured and recorded every half an hour until they were stable. Then, the IER was washed by ultrapure water until its conductivity was below 10 μS/cm. Finally, the IER was dried for regeneration experiment. Before each experiment, the IER was in complete failure state.

Analysis methods

Regeneration efficiency of ion exchange resins

The washed and dehydrated IER reacted with excess NaCl solution, then the obtained H⁺ and OH⁻ reacted with NaOH and HCl, respectively. On the basis of the amount of NaOH and HCl, the ion exchange efficiency of CR and AR was calculated. For EDI device with CB structure, the regeneration efficiency of IER (η₀) was calculated, according to Formula 1.

\[
\eta_0 = \frac{m_c \eta_c + m_a \eta_a}{m_c + m_a}
\] (1)
where \( m_c \) and \( m_a \) were the masses of CR and AR, respectively. \( \eta_c \) and \( \eta_a \) were the regeneration efficiencies of CR and AR, respectively. The mixed IER was treated by simulated solution. For EDI device with MB structure, the regeneration efficiency of IER (\( \eta_1 \)) was calculated, according to Formula 2.

\[
\eta_1 = \frac{\eta_0 V_1}{V_0}
\]

where \( V_0, V_1 \) were the treatment capacity of 10 g IER with the same mixing ratio, of which regeneration efficiencies were \( \eta_0, \eta_1 \). (Solution conductivity \( \geq 10 \mu S/cm \) was the end point).

**Analysis and monitoring of water quality**

Conductivity and pH value in the concentrated chamber were measured using a conductivity meter (Five Easy FE30, Mettler Toledo, resolution: 0.01 \( \mu S/cm \)) and a pH/ISE meter (PXSJ-216, Inesa, China). Since the exchanged ions eventually migrated to the concentration chamber, the change trend of the conductivity in the concentration chamber depended on the exchange capacity of IER and the efficiency of ion migration. Therefore, the \( Ca^{2+}, Mg^{2+} \) and \( Cl^- \) in the concentrated chamber were measured. The \( Ca^{2+} \) and \( Mg^{2+} \) were complexed with EDTA solution by using chromium black T as indicator at the pH of 10. At the beginning of titration, chromium black T complexed with \( Ca^{2+} \) and \( Mg^{2+} \) to make the solution purple. With the titration going on, EDTA first complexed with free \( Ca^{2+} \) and \( Mg^{2+} \). Then EDTA reacted with \( Ca^{2+} \) and \( Mg^{2+} \). At the end point, the color of the solution turned sky blue. AgNO\(_3\) interacted with \( Cl^- \) to form white AgCl precipitation. Excessive AgNO\(_3\) reacted with K\(_2\)CrO\(_4\) indicator to form brick-red Ag\(_2\)CrO\(_4\), indicating that the reaction had reached the end point.

**RESULTS AND DISCUSSION**

**Determination of limiting current density**

According to the definition of the limit current density (Strathmann 2016), with current gradually increasing, the ions in the diluted chamber were continuously transferred to the concentrated chamber, which caused the ions existing at the membrane/solution interface to be insufficient to carry an appropriate current flow. Severe water dissociation reaction occurred at the membrane, solution and IER interfaces due to regional ion depletion, producing an amount of \( H^+ \) and \( OH^- \) (Aguilera et al. 2021). Hence, the \( H^+ \) and \( OH^- \) were not only used for regenerating IER, but also for conducting electrical current. As seen in Figure 2, the current rapidly increased at the voltage of 60 V, so the limit current density was determined to be 9 mA/cm\(^2\) (0.381 A).

**Effects of different bed types on regeneration process**

**Regeneration efficiency**

In order to compare the performance of EDI based on MB and CB structure, the effects of regeneration time and current density on the regeneration efficiency of IER were explored (Figure 3). For MB structure, the regeneration efficiency increased slowly, and it was only 23% at the regeneration time of 3 h. While for CB structure, the regeneration efficiency increased rapidly, and reached 39% at the regeneration time of 1 h, which was superior to the recent reports (Hu et al. 2015, 2016). After the regeneration time of 3 h, regeneration efficiency increased slowly, proving that it had reached the reaction limit of the regeneration process. For MB structure, the regeneration efficiency increased slowly with increasing current density. However, for CB structure, the regeneration efficiency increased rapidly with increasing current density. But when the current density reached 11 mA/cm\(^2\), the regeneration efficiency didn’t increase obviously. The possible reasons were that the degree of water dissociation reaction didn’t increase obviously. The possible reasons were that the degree of water dissociation reaction didn’t increase obviously and the regeneration process was close to the limit. The regeneration efficiency of the traditional chemical regeneration process using 4% NaOH and 2% NaCl could reach 80% (Martin et al. 2009). Therefore, EDI device with

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**Figure 2** | Volt-ampere curve for EDI device. (CR: AR – 2:3, membrane space – 10 mm).
CB structure was close to the efficiency of acid-base chemical regeneration at the current density of 11 mA/cm². At the limit current density of 9 mA/cm² and the regeneration time of 3 h, the IER had basically reached the limit of regeneration. Therefore, the regeneration time of 3 h was taken in the following research conditions.

**Ion migration**

The desalination capacity depended on the actual exchange capacity of IER and the ability for rapid ion transfer to concentrated chamber. As shown in Figure 4(a), the water conductivity for EDI with the different bed types of structure changed in the same trend. In addition, compared with the MB structure, the water conductivity for EDI with CB structure was significantly higher, indicating that more ions were migrating from the diluted chamber to the concentration chamber.

The regeneration of saturated IER depended on ion-exchanging with H⁺ and OH⁻ generated by water dissociation reaction. Equations (3)–(5) are the reaction for regeneration process. Most of the anions and cations desorbed from the IER were transferred to the concentration chamber, and the hardness, Cl⁻ concentration and pH value in the concentration chamber were further tested (Figure 4). The variation trends of the hardness, Cl⁻ concentration and pH value were generally identical. In addition, it can be seen from Figure 4(b) and 4(c) that both the hardness and Cl⁻ concentrations were higher compared with MB structure, indicating that more ions were desorbed from saturated IER and quickly migrated to the concentrated chamber. Notably, the conductivity and H⁺ concentration decreased after 3 h, as shown in Figure 4. After the regeneration time of 3 h, the change of Cl⁻ concentration was not obvious, which proved that the regeneration limit of AR was reached (the regeneration process of the IER was a reversible reaction). However, the hardness was still increasing, so H⁺ was used to regenerate CR, and the generated OH⁻ migrated to the concentration chamber for neutralization reaction.

$$R \left\{ \frac{1}{2} \text{Ca}^{2+} + \frac{1}{2} \text{Mg}^{2+} + \text{Na}^+ \right\} + \text{H}^+ \rightleftharpoons \frac{1}{2} \text{Ca}^{2+} + \frac{1}{2} \text{Mg}^{2+} + \text{Na}^+$$

$$R \left\{ \frac{1}{2} \text{SO}_4^{2-} + \text{HCO}_3^- \right\} + \text{OH}^- \rightleftharpoons \frac{1}{2} \text{SO}_4^{2-} + \text{HCO}_3^-$$

$$\text{H}_2\text{O} \rightleftharpoons \text{H}^+ + \text{OH}^-$$

**Energy consumption**

It is shown from Figure 5(a) that the EDI device with CB structure had a lower energy consumption than that with MB structure, due to its lesser stack resistance. Figure 5(b) shows energy consumption at a regeneration efficiency of 39% per gram of IER. The energy consumption of the CB structure was much lower than that of the MB structure, which proved that the CB structure had a higher current efficiency and was conducive to the efficient and rapid regeneration of IER. As shown in Figure 5(c), the ions in the diluted chamber migrated to the concentrated chamber at the beginning of the reaction, and the conductivity in
the diluted chamber gradually decreased and the bed temperature gradually increased. Generally, the bed temperature gradually increased as the reaction proceeded. For the MB structure, the bed temperature increased rapidly and then maintained at 70°C, proving that the heating process consumed a lot of electrical energy. For the CB structure, the bed temperature increased gradually after 1 h and maintained at 50°C, indicating that the electrical energy was mainly used for the regeneration process afterwards. And the lower bed temperature led to higher current efficiency and regeneration efficiency.

**Regeneration mechanism**

The literature (Alvarado & Chen 2014) showed conductive counterions adsorbed on the IER surface due to electrostatic attraction, and underwent electro migration under an applied electric field. As shown in Figure 6, with the ion concentration on the surface of IER gradually reducing, the potential difference between the IER and the solution was higher than the thermodynamic potential of the water. Then, an intense water dissociation reaction occurred at the surface of IER and IEM, producing a large number of $H^+$ and $OH^-$, which ion-exchanged with the corresponding IER to implement regeneration of IER.

As shown in Figure 7, the regeneration mechanism of IER with different bed type structures was explored based on the above theory. Figure 7(a) describes the AR regeneration process in MB structure. In general, most desorbed cations gradually migrated to the concentrated chamber through an ion transport channel consisting of adjacent AR layers. However, ion transmission efficiency was lower because in the uniform IER it was difficult to form continuous ion channels in the MB structure. At the same time, the uniform IER enabled $H^+$ to interact with $OH^-$, so they were not used for regeneration of IER effectively. Therefore, ion...
transmission efficiency and the $H^+$ and $OH^−$ utilization efficiency were reduced, which finally affected desalination performance. As shown in Figure 7(b), it was an example of the CR regeneration in the CB structure. First of all, the desorbed cation could quickly pass through the ion transport channel, which consisted of adjacent CR layers, and enter the concentrated chamber. Then, the remaining $H^+$ was transferred to the surface of CR under the strong electric field, so the CR could be effectively regenerated. In the CB structure, the path and direction of conductive ion migration was more directional because of the layering of stacked IER. And the directional migration of ions increased concentration polarization, so the enhancement of the bipolar interface between IER and IEM promoted the water dissociation reaction. The strong electric field direction generated by the IER layer was consistent with the direction of ion migration, so the strong electric field was enhanced to separate anion from cation. At the same time, it effectively reduced the probability of collision of $H^+$ and $OH^−$ again, thus preventing a neutralization reaction and improving current efficiency. So, the CB structure significantly improved the regeneration efficiency of IER as compared to the MB structure.

Electrochemical regeneration performance for EDI device with compound bed structure

Membrane spacing

Generally, membrane spacing exerted influences on the regeneration efficiency of IER with CB structure, as shown in Figure 8. With the increase of membrane spacing, the
regeneration efficiency of IER presented an overall downward trend. In addition, as the membrane spacing exceeded 10 mm, the regeneration efficiency of IER decreased rapidly. With the membrane spacing increasing, the distance of desorbed salt ions to the concentration chamber increased, which increased the probability of the desorbed salt ions’ readsoption, and led to the reduction of ion transport efficiency. In addition, the contact surface area between IER and IEM decreased, and the rate of water dissociation reaction decreased. Although the amount of IER filled could be increased to improve the water quantity handled, the stack resistance also increased at the same time. So, the energy consumption and temperature gradually increased, which affected long-term stable

Figure 6 | Schematic diagram of IER regeneration.

Figure 7 | Schematic diagram of different bed type structures.
operation. Therefore, the membrane spacing was determined to be 10 mm.

**Anti-scaling performance**

After the long-term operation of the EDI device, calcareous deposits (CaCO$_3$ and Mg(OH)$_2$) were more likely to deposit on the membrane surface and electrode surface, which affected the long-term stable operation of the device. Therefore, the effect of the IER ratio on the scale resistance of CB was investigated. As shown in Figure 9(a), the pH value of the solution varied with different IER ratios. As the proportion of the anion exchange resins increased, the solution gradually changed from alkaline to acidic, because more OH$^-$ ion-exchanged with AR, and the remaining H$^+$ was conductive to form an acid environment. Besides, it is worth noting that the pH value rapidly decreased from neutral to 2–3 after 1 h under a CR and AR ratio of 2: 3, and then it tended to be stable. Considering the acid-base tolerance range of the heterogeneous IEM and the precipitation of Ca$^{2+}$ and Mg$^{2+}$, the IER ratio was finally determined as CR: AR = 2: 3. In addition, the current density was also a factor that influenced the pH value. With the current density increasing, different amounts of H$^+$ and OH$^-$ migrated to the concentration chamber, because the discrepancy of water dissociation degree between the surface of AR and CR increased. Otherwise, the current density also affected the electrode reaction. The H$^+$ concentration at different current densities is shown in Figure 9(b). With the current density increasing, the pH value was lower, and the anti-scaling performance was better.

As shown in Figure 10, the scale formation on the surface of the electrode and CM with different IER ratios was measured. Obvious precipitates, of which the main components were Mg(OH)$_2$ and CaCO$_3$, appeared on the surface of the electrode and CM under the CR and AR ratio of 2:1, because the solution in the concentration chamber was alkaline. However, little precipitation appeared on the surface of the electrode and CM under the CR and AR ratio of 2:3. This was consistent with the previous conclusion that more AR was conducive to forming an acidic environment to prevent scaling.

**Figure 9** Effects of (a) IER ratios (membrane spacing = 10 mm, current density = 9 mA/cm$^2$) and (b) current density (CR: AR = 2:3, membrane spacing = 10 mm) on the pH value of the solution in the concentrated chamber.
In general, the EDI device with CB structure possessed excellent regeneration performance by optimizing the arrangement of IER. The proposed CB structure not only enhanced the water dissociation reaction, but also shortened the path of ion migration, thus improving the regeneration efficiency of IER and reducing the energy consumption. Therefore, the proposed EDI device was superior to traditional EDI devices in terms of energy utilization. The EDI device with CB structure could realize continuous operation, so further work would need to be done. By changing the structure of the device and the type of IER, the device with rapid regeneration of IER would be designed under the condition of ensuring the quality of effluent water. It was believed that these studies would contribute to the industrial application for operating continuously and solve the problem of scale formation for the EDI device.

![Figure 10](http://iwaponline.com/wst/article-pdf/doi/10.2166/wst.2021.161/882651/wst2021161.pdf)

**Figure 10** The surface of the anode at the CR and AR ratio of (a) 2:1 and (b) 2:3. The SEM of the cation exchange membrane surface at the CR and AR ratio of (c) 2:1 and (d) 2:3. The EDS of cation exchange membrane surface at the CR and AR ratio of (e) 2:1 and (f) 2:3. (membrane spacing – 10 mm, current density – 9 mA/cm²).
CONCLUSIONS

The proposed EDI device could achieve high efficiency regeneration of IER together with anti-scaling performance. The designed CB could enhance the water dissociation reaction and reduce ion collisions, providing the possibility to replace the traditional chemical regeneration method. In addition, the ratio of the IER, current density, regeneration time and membrane spacing were optimized in the case of concentrated water circulation. More AR was conducive to forming an acidic environment, effectively preventing the formation of precipitation. The regeneration efficiency of IER could reach 73.1%, when the device was operated for 5 h under a current density of 9 mA/cm², cation and anion exchange resins ratio of 2: 3, influent water conductivity of 1,360 μS/cm and hardness of 400 mg/L. Therefore, the proposed EDI device could break the limitation of EDI influent water conditions and increase regeneration efficiency of IER, which was beneficial to the industrial application of EDI technology.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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