



# Membrane-free electrodeionization without electrode polarity reversal for high purity water production



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## HIGHLIGHTS

- MFEDI has been tested for high purity water production without electrode polarity reversal.
- Adding a thin strong base resin layer effectively prevented cation backward migration during regeneration.
- Effective regeneration was achieved without electrode polarity reversal.
- The effluent conductivity was 0.055–0.060  $\mu\text{S}/\text{cm}$  in 30 operational cycles.

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## ABSTRACT

Membrane-free electrodeionization (MFEDI) has been tested successfully for high purity water production in our previous work. However, in order to prevent the constant backward migration of cationic or anionic ions, to achieve equally effective regeneration for both the cationic and anionic resins, and to get neutral concentrate, the electrodes had to be reversed during regeneration. This not only complicated the regeneration process, but also decreased the water recovery and the electrode lifetime. In this work, we improved the resin layer structure and investigated the regeneration performance of MFEDI without electrode polarity reversal. A total of 30 operational cycles were examined. Effective regeneration was achieved. The effluent conductivity was 0.055–0.060  $\mu\text{S}/\text{cm}$  only; the average pH value of the concentrate was 7.2; the water recovery was 90%; and the energy consumption was 0.71  $\text{kWh}/\text{m}^3$  water.

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## 1. Introduction

High purity water is widely used in chemical and pharmaceutical production, laboratories, semi-conductor manufacturing, power generation and many other areas [1–3]. The conventional means of high purity water production is ion exchange in which the ionized and ionizable species contained in the solution are exchanged with the  $\text{H}^+$  or  $\text{OH}^-$  contained in the fixed matrix of ion exchange resin [4]. Ion exchange is a widely accepted purification technology that has been used for more than half a century due to the easiness of its operation and high ionized species removal efficiency. However, once the ion exchange bed is saturated, it must be regenerated with chemicals such as hydrochloric acid and sodium hydroxide. This produces a considerable amount of chemical waste that requires neutralization before it can be discharged [5,6]. In addition, regeneration process demands a plenty of auxiliary facilities for chemical storage, transportation, solution preparation, and metering.

Electrodeionization (EDI) has been considered as an alternative technique for producing high purity water without the need of periodic chemical regeneration of resins. It is composed of depleting chambers and concentrating chambers created by an alternating sequence of anion-exchange membranes and cation-exchange membranes between a pair of electrodes [7–10]. Usually, the depleting chambers are filled with ion exchange resins. Just like the resins working in the conventional ion exchange, the resins in the depleting chambers of EDI are responsible for ion entrapping and meanwhile serve as conducting media to step up the electric migration of the entrapped ions toward the membranes [11–14]. In such a process, the ion exchange resins can be continuously regenerated by  $\text{H}^+$  and  $\text{OH}^-$  ions generated through the electrically enhanced water split in the depleting chambers [15,16], making EDI environmentally friendly. EDI has been proven very effective. As a polishing means, it is now taking the place of chemically regenerated mixed beds to produce high purity water for a variety of applications, spanning from academic and clinical laboratories, through surface finishing, food and beverage producing, pharmaceutical, semi-conductor and chemical manufacturing, to power generation [8,17]. In addition, EDI has demonstrated its potentialities for use in wastewater

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treatment and chemical purification/recovery [18–21]. However, nowadays EDI still suffers from some hash problems due to the selective ion exchange membrane used. For example, the multiple expensive ion exchange membranes increase the investment of EDI and make repairing difficult. Because of the ion exchange membrane selectivity, only counter ions can pass through the membrane while the co-ions are repelled, giving rise to concentration polarity and water dissociation. In this case, calcium and magnesium ions are chemically inclined to precipitate on the membrane surface, leading to a decrease in deionization efficiency and an increase in the energy consumption [22,23]. Accordingly, thoroughly eliminating the ion exchange membranes from the EDI system is highly desired from both the technical and economic viewpoints.

In our previous work, a new process named as membrane-free EDI (MFEDI) has been proposed and tested successfully for high purity water production [24]. The effluent conductivity was 0.056–0.059  $\mu\text{S}/\text{cm}$  only; the water recovery was 89%; and the energy consumption was 0.68  $\text{kWh}/\text{m}^3$  water. Essentially, MFEDI is an electrically regenerated ion exchange process in which the mixed cation and anion resins are packed between a pair of reticular electrodes. This process is rather different from that invented by Davis [25] that used ion exchange fibers and had a complex configuration. It is also quite different from the one reported before by our group [19] that used a pair of ion exchange membranes. MFEDI is operated in batch, alternating with service and regeneration. During regeneration, the purity water passed through the resin layers, which were supplied with a constant current simultaneously. Under an electric field, most part of saline ions were desorbed from the resin phase to the aqueous phase, and then flushed away; and a small part of saline ions were driven out of the resin layer through the electrical migration inside the resin phase. Obviously, the ions migrating in the same direction as the water flow were easy to be regenerated; whereas the ions migrating against the water flow direction were difficult to be regenerated. The backward ions could migrate to the upper fresh resins and finally cause effluent quality deterioration. In order to deal with ion backward migration, we reversed electrodes during regeneration in our previous work. The electrode polarity reversal could help to prevent ion backward migration, to achieve equally effective regeneration for both the cationic and anionic resins, and to get neutral concentrates. However, a significant decrease of concentrate conductivity and a dramatic increase of voltage occurred after the electrode polarity was reversed. In addition, the process of electrode polarity reversal complicated the regeneration process and decreased the water recovery and the electrode lifetime.

The main objectives of this study are to examine the possibility of preventing the backward migration counter ions using a thin layer filled with a single type of resin, and to investigate the performance of the MFEDI system without polarity reversal.

## 2. Experimental section

### 2.1. Resin pretreatment

All the resins used in this work were the selected resins in our previous work. Their characteristics were listed in Table 1. Before use, the gel-type strong-acid cation resin (Monosphere 650C, Dow, Shanghai, China) and gel-type strong-base anion resin (Monosphere 550A, Dowex, MI)

were washed with deionized water until the conductivity of the resin solution is below 1  $\mu\text{S}/\text{cm}$ . Unlike 650C and 550A, the macroporous-type weak-acid cation resin (D116, Zhengguang Co., Hangzhou, China) was first soaked in deionized water at 80 °C for 2 h, then regenerated fully with a 5% hydrochloric acid solution, and finally washed with deionized water again.

### 2.2. Electrode preparation

Reticular titanium was used as the cathode, and reticular Ti/RuO<sub>2</sub>–Sb<sub>2</sub>O<sub>5</sub>–SnO<sub>2</sub> was used as the anode. The Ti/RuO<sub>2</sub>–Sb<sub>2</sub>O<sub>5</sub>–SnO<sub>2</sub> anode was fabricated using a thermal decomposition method. SnCl<sub>4</sub>·5H<sub>2</sub>O (99.9%, Aladdin, Shanghai, China), RuCl<sub>3</sub>·3H<sub>2</sub>O (38–42% Ru, Aladdin, Shanghai, China) and SbCl<sub>3</sub> (99.9%, Aladdin, Shanghai, China) solutions were all freshly prepared just before the electrode fabrication. The precursor solution was prepared by mixing the individual solutions of SnCl<sub>4</sub>·5H<sub>2</sub>O, RuCl<sub>3</sub>·3H<sub>2</sub>O and SbCl<sub>3</sub>. The molar ratio of Sb, Ru and Sn in the precursor solution was about 1:3:6. Prior to coating, the reticular titanium substrates underwent 10 min of ultrasonic cleaning in deionized water, 2 min of etching in boiling 37% hydrochloric acid, and another 10 min of ultrasonic cleaning in deionized water. After pretreatment, the titanium substrates were brushed at room temperature with the precursor solution first, dried at 80 °C for 5 min, then calcinated at 500 °C for 5 min. The procedure above was repeated for 20 times. Finally, the electrodes were annealed at 500 °C for an hour. More details of Ti/RuO<sub>2</sub>–Sb<sub>2</sub>O<sub>5</sub>–SnO<sub>2</sub> electrode preparation procedure can be found elsewhere [26].

### 2.3. Experimental setup for ion backward migration examination

The experimental setup for ion backward migration examination is shown in Fig. 1. It was used to examine the possibility of preventing the cation ion backward migration with a thin strong-base resin layer under an electric field. The resin layers, 200 mm high totally and 30 mm in diameter, were packed between the anode and cathode. Resin layers without the thin strong-base resin layer were shown in Fig. 1a. The top resin layer was 50 mm high and filled with strong-acid ion exchange resins; the bottom resin layer was 150 mm high and filled with mixed weak-acid and strong-base ion exchange resins (volume ratio = 1:2). Resin layers with a thin strong-base resin layer were shown in Fig. 1b. The thin strong-base resin layer, 10 mm high, was added between the top and bottom resin layers. Before the experiment, both bottom resin layers in Fig. 1a and 1b were saturated by sodium chloride in the Na<sup>+</sup> ion backward migration examination and by calcium chloride in the Ca<sup>2+</sup> ion backward migration examination, and the upper strong-acid cation resins were always H<sup>+</sup> form. To saturate the bottom resin layer, the sodium chloride or calcium chloride solution with a concentration of 100 mg/L was pumped through the resin layer at a flow rate of 14 L/h until the effluent conductivity was equal to the feed solution. The upper electrode worked as a cathode and the lower electrode worked as an anode. The purity water was pumped through the resin layers downward. DC power was supplied to the resin layers simultaneously for 5 h. After that, the strong-acid resin in the top resin layer was taken out and washed using 20 mL 5% hydrochloric acid. The concentration of Na<sup>+</sup> and Ca<sup>2+</sup> of the resin eluent, which reflected the backward migration situation of cation ions, was measured.

### 2.4. MFEDI system

The MFEDI system is shown in Fig. 2. The ion exchange column, 500 mm high and 30 mm in inner diameter, was filled with five mixed resin layers and four strong-base anion resin layers that were used to prevent the cation ion backward migration. The heights of the mixed resin layers and the anion resin layers were about 100 mm and 10 mm, respectively. The top mixed resin layer was filled with strong-acid and strong-base resins (volume ratio = 1:1), while the other

**Table 1**  
Properties of ion exchange resins<sup>a</sup>.

Designation	D116	MS 550A	MS 650C
Type	Weak-acid Cationic resin	Strong-base Anionic resin	Strong-acid Cationic resin
Matrix structure	Polyacrylic acid	Styrene-DVB	Styrene-DVB
Functional group	Carboxyl	Quaternary amine	Sulfo group
Porosity	Macro-porous	Gel	Gel
Exchange capacity (eq/L)	≥4.3	≥1.1	≥2.0

<sup>a</sup> Obtained from manufacturers.

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