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Membrane-free electrodeionization for high-velocity production of high-purity water

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ABSTRACT

Membrane-free electrodeionization (MFEDI) has been proven effective in desalination of water similar to the effluent of one-pass reverse osmosis (RO) in our previous work. In this work, we investigated MFEDI for use as a polishing desalination process after two-pass RO. In addition to the difference in simulated feedwater, we increased the flow velocity significantly from previous 15 to 70 m/h. An appropriate resin layer structure was selected to enhance resins regeneration, especially to obstruct the backward migration of cations in resin phase. Parameters of regeneration were adjusted to be more effective and energy efficient. The system was tested for over 60 operational cycles. After such a long-term service, the effluent conductivity still maintained below 0.060 μS/cm. Water recovery rate and energy consumption of the system were around 93% and 0.14 kWh/m^3 water, respectively. MFEDI was proven to be an economic and quality assured alternative for high-purity water production.

Keywords: High-purity water production; Ion-exchange resins; Electrical regeneration; Backward migration prevention; Barrier layers

1. Introduction

High-purity water is necessary in many areas such as electric power generation, chemical industry, and semiconductor manufactory [1,2]. With rapid industrial development, consumption of high-purity water is increasing. It is, therefore, significant to find a more economic but less polluting method to produce high-purity water.

As a core part of high-purity water preparation, desalination has been studied for a long time. Nowadays, reverse osmosis (RO) and ion exchange (IE) are widely used in water desalting processes [3]. RO is usually able to remove 95.0%–99.7% of the total dissolved solids in water [4–6]. Nonetheless, the conductivity of its effluent can hardly reach the standard of high-purity water yet. Because of this, RO is generally employed only for the primary deionization purpose. As a conventional technology in polishing desalination, IE has demonstrated its high purification efficiency and good reliability. Nonetheless, it needs chemical regeneration, which not only consumes large amounts of acid and alkali but also produces high-salinity wastewater, causing environmental pollution problems [7].

In recent years, electrodeionization (EDI) has been applied widely for high-purity water production. EDI is, in most cases, used as a polishing desalination process after RO and occasionally combined with other process [8,9]. It is a hybrid desalination technique combining electrodialysis and IE [10]. The resins in EDI are regenerated by H^+ and OH^- produced by water dissociation [11–13]. Because the regeneration and deionization occur simultaneously, EDI can produce high-purity water continuously. In spite that EDI has many advantages including environmental friendliness, easy operation, and small space demand [14,15], its disadvantages cannot be neglected, including high investment, difficult maintenance, and a series of membrane-associated problems such as membrane fouling and concentration polarization.

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In our previous works [16–21], we investigated a new desalination process. It is well known that the most important feature of EDI is that it only consumes electricity with no chemicals needed. The new desalination process that we reported also has the same feature as that of EDI. The main difference between EDI and the new process is that the former uses both IE resins and selective membranes whereas the latter uses IE resins only. Therefore, we named the new desalination process as membrane-free electrodeionization (MFEDI). Different from EDI, MFEDI is operated in a batch mode, switched between purification and regeneration. MFEDI purifies water like a multiple-layer mixed resin bed, but it is regenerated in a mode similar to that of EDI. MFEDI remains all advantages that EDI has, but overcomes the membrane-associated problems. In addition, profited from expensive membranes eliminating and hard membrane-associated problems disappearing, MFEDI is much lower in manufacturing and maintenance costs than the conventional EDI. Besides, because the resins are frequently regenerated, saturation of the resins changes within a quite small range, and therefore the resin breakage caused by internal pressure of resins is negligible.

In our previous works, we focused mainly on MFEDI performance for desalination of effluents from one-pass RO at a relatively low flow velocity, usually around 15 m/h. In this work, we investigated MFEDI for use as a polishing desalination process after two-pass RO at an elevated flow velocity, 70m/h, which could basically meet the demand of industrial application while the water head loss was acceptable. Although the situation where MFEDI desalinated effluents from one-pass RO at a low flow velocity was understood well, the performance of this new process for use after two-pass RO at a much higher flow velocity still remained unknown.

It is noticeable that with the flow velocity increased, obstruction of backward migration of cations should be enhanced. Backward migration refers to the migration of cations, the direction of which reverses the regeneration flow in the MFEDI system. Under an electric field, cations migrate toward the cathode whereas anions migrate toward the anode. Generally, there are three paths for ions to migrate electrically. Ions can migrate through resins only, through solution only or alternately through resins and solution, depending on the resistances of solution and resins [22]. It was reported that the strong acid resin (SAR) in Na^+ form has a conductivity of 30 mS/cm, which is equal to that of a 0.3 mol/L aqueous NaCl solution. Because the concentration of the solution in MFEDI is much lower, most of ions migrate in resin phase [23]. In the resin bed of an MFEDI system, the anions migrate toward the same direction with the flow in a regeneration process, which is beneficial for anions removal. Conversely, the electrical migratory direction of cations reverses the regeneration flow. This kind of migration, called backward migration, obviously impedes the regeneration. Increase in purification flow velocity leads to higher saturation of the top layer of the resin bed, which requires enhanced obstruction of the backward migration.

Therefore, further investigation is of significance, especially for potential industrial applications. The main objectives of this study are to investigate the purification performance, to examine the critical factors affecting regeneration, to evaluate the water recovery rate and the energy consumption, and to prove the function of the selected resin layer structure of the MFEDI system for production of high-purity water at a high flow velocity.

2. Materials and methods

2.1. MFEDI system

The MFEDI system is shown in Fig. 1. It mainly includes a pair of electrodes, several layers of resins, a column with four sampling ports, and a direct current (DC) power supply. Resins were filled in the column first, and then compressed by a spring. The total height of the resin bed was fixed to be 0.42 m. The selected resin layer structure in this work consisted of layers of mixed weak acid resins (WAR) and strong base resins (SBR) on the bottom and layers of mixed SAR and SBR resins on the top, divided by several thin anion resin layers, which were called barrier layers. The ratio of WAR and SBR was 1:2.5, and the ratio of SAR and SBR was 1:1.75.

The system was operated in a batch mode. In the water purification step, Valve 1 and Valve 2 were open while 3 and 4 were closed. The feedwater entered the column from Valve 1 and went up through resin layers. The purified water outflowed from Valve 2 and was collected in the reservoir. In the regeneration step, Valve 3 and Valve 4 were open while 1 and 2 were closed. High-purity water was pumped into the column through Valve 3. Simultaneously, DC electricity was supplied by an external power supply. Under the electric field, water was dissociated into H^+ and OH^-, which then displaced the ions adsorbed on resins. The concentrate flowed out of the system from Valve 4.

2.2. Resins and electrodes

Resins used in this work are listed in Table 1.

Before use, the resins were washed with high-purity water until the conductivity of the solution was below 2 μS/cm. Anion resins and cation resins were then mixed in proportions demanded and filled compactly in the column layer by layer. A reticular Ti/RuO_2-Sb_2O_5-SnO_2 electrode, with an effective area of 38.5 cm^2, was utilized in this MFEDI system as the anode [24,25], while reticular stainless steel serviced as the cathode.

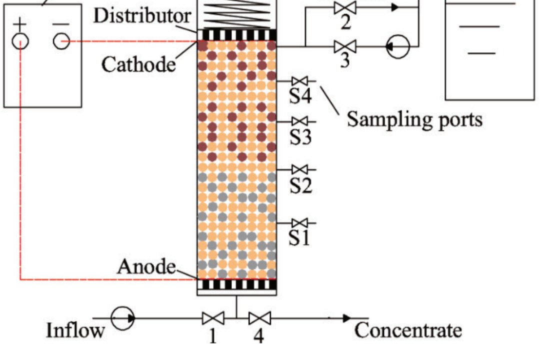


Fig. 1. MFEDI system.

2.3. Feedwater

The feedwater used in this work simulated the effluent from two-pass RO, mainly containing Na^+, Ca^{2+}, Mg^{2+}, $Cl^–$, $SO_4^{2–}$,and $HCO_3^–$. To test the effectiveness and stability of MFEDI with a more severe condition, proportions of divalent ions in the feedwater were higher than the actual proportions of effluent from two-pass RO, while the conductivity was similar to the actual conductivity. The molar ratios for all impurity ions were nearly equal, that is, $[Na^+]:[Ca^{2+}]:[Mg^{2+}] = 1:1:1$ and $[Cl^–]:[SO_4^{2-}]:[HCO_3^-] = 1:1:1$. The conductivity of the feedwater was 1–2 µS/cm.

2.4. Resins analysis

To understand the function of the selected resin bed structure better, we examined the ions distribution of the resin column using an elution method. The ions adsorbed on resins were eluted into solution first, and then analyzed using atomic absorption spectrometry, IE chromatography and titrimetry as shown in Table 2.

3. Results and discussion

3.1. Purification performance and stability

In order to know the desalination process of MFEDI at a high velocity in detail, conductivity variations of the effluents from different sampling ports were examined, and the results are shown in Fig. 2.

Table 1
Main properties of ion-exchange resins

Designation	D113	650C	550A
Type	Weak acid	Strong acid	Strong base
Function group	–COOH	$–SO_3H$	$–NR_3OH$
Matrix structure	Polyacrylic acid	Polystyrene	Polystyrene
Porosity	Macroporous	Gel	Gel
Size (mm)	0.40–0.70	0.59–0.66	0.54–0.64
Exchange capacity (eq/L)	≥4.4	≥2.0	≥1.1

Table 2
The eluent and analysis methods of the resin elution

Ion	Resin	Leacheate	Analysis method
Na^+	D113, 650C	KCl	Atomic absorption spectrometry
Ca^{2+}	D113, 650C	KCl	Atomic absorption spectrometry
Mg^{2+}	D113, 650C	KCl	Atomic absorption spectrometry
Cl^-	550A	$NaNO_3$	Ion-exchange chromatography
$SO_4^{2–}$	550A	$NaNO_3$	Ion-exchange chromatography
$HCO_3^–$	550A	$NaNO_3$	Stepwise titration by H_2SO_4

It can be seen from the figure that the effluent conductivity decreased along the resin layers slowly first, then quickly, and finally slowly again. This indicates that the adsorption abilities of different resin layers were different. Such difference was mainly caused by the difference from resin types, mixing ratios, as well as ion affinities. The affinities of different ions for the same kind of resins vary. Some ions are easy to be adsorbed but difficult to be desorbed; whereas some ions are difficult to be adsorbed but easy to be desorbed. Affinity of ions for the functional groups is a function of charge and size [26]. Ions with a larger ionic charge (Ca^{2+} vs. Na^+) have greater affinity, and therefore, the equilibrium will favor the higher charged ion on the solid phase. Relative affinities of common ions are given below [26–28]:

For SAR,
$Fe^{3+} > Al^{3+} > Ca^{2+} > Mg^{2+} > K^+ > Na^+ > H^+$

For WAR,
$H^+ > Fe^{3+} > Al^{3+} > Ca^{2+} > Mg^{2+} > K^+ > Na^+$

For SBR,
$SO_4^{2–} > NO_3^- > Cl^– > OH^– > HCO_3^- > HSiO_3^–$

The ions adsorbed on resins can be replaced by the ions with higher affinity. Thus, in the resin bed of MFEDI system, most of Ca^{2+} and Mg^{2+} ions were adsorbed onto WAR in the lower layers, remaining Na^+ going up with the flow and adsorbed onto the upper layers. Things went similarly with cations. Most $SO_4^{2–}$ ions and $HCO_3^–$ ions were adsorbed in the lower layers and in the upper layers, respectively. Because the adsorption rate of WAR was much lower than SBR, the effluents from sampling ports S1 and S2 were alkaline. It should be noted that the conductivity of $OH^–$ is much higher than saline anions. This explains why the conductivities of effluents from ports S1 and S2 were close to that of feedwater although the ion concentrations were much different.

It can also be seen from Fig. 2 that the conductivities of effluents were increased over time. This was attributed to the gradual saturation of resins. Resins in bottom layers were exhausted so that ions got into upper layers. That is, the working layer of the resin bed moved up. To prevent the

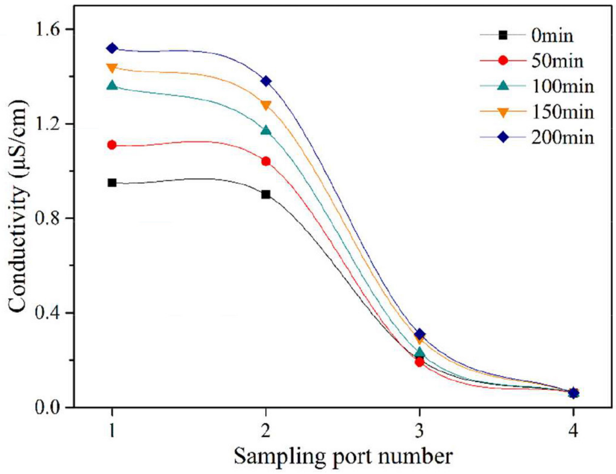


Fig. 2. Conductivity variations of effluents from different sampling ports at a purifying velocity of 70 m/h.

working layer from moving up unduly, timely regeneration is therefore needed.

To examine the performance stability, the MFEDI system ran for more than 60 cycles. Each operational cycle included a 200-min purification step and a 20-min regeneration step. As Fig. 3 shows, the conductivity of effluent remained below 0.060 μS/cm within 60 operational cycles, with no deterioration tendency in effluent quality being detected, indicating the stable purification performance of the MFEDI system.

In addition, to further demonstrate the effectiveness of regeneration, the average conductivities of the concentrates were compared with their theoretical values at a steady state. The theoretical average conductivity of concentrate was calculated according to the mass balance, as Eq. (1) expresses.

$$\left(\overline{C}_r - \overline{C}_0\right)V_r = \left(\overline{C}_l - \overline{C}_0\right)V_i \qquad (1)$$

where $\overline{C}_r$ is the theoretical average concentration of the concentrate, mol/L; $\overline{C}_i$ is the average concentration of the feedwater, mol/L; $\overline{C}_o$ is the average concentration of the high-purity water, mol/L; V_r is the volume of the concentrate; and V_i is the volume of the feedwater.

The equation could be transformed into $\overline{C}_r = \overline{C}_l V_i / V_r$, because $\overline{C}_o$ was negligible. In this work, the V_i/V_r ratio was 14.

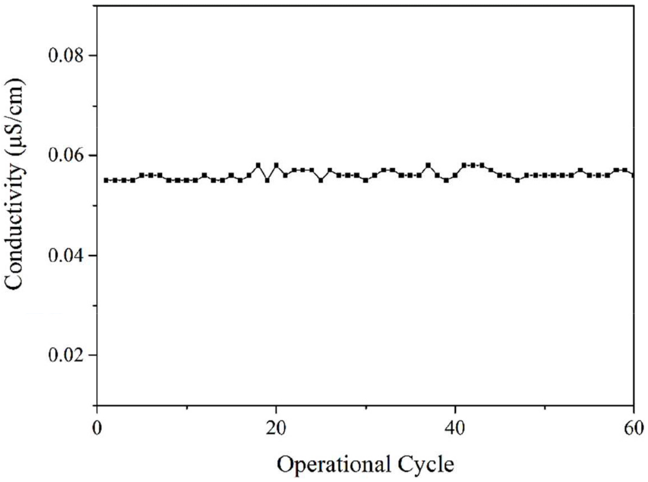


Fig. 3. Effluent conductivity variation during a long-term operation at a purifying velocity of 70 m/h.

Because proportions of ions in the concentrate were similar to those in the feedwater, concentrations of the feedwater and the concentrate were basically proportional to the conductivities of them. Therefore, theoretical average conductivity could be calculated according to the equation as well. The theoretical and the practical average conductivities of concentrates of the last five operational cycles are listed in Table 3.

As can be seen in Table 3, in each operational cycle, the practical average conductivity of concentrate is in a good agreement with the theoretical value, indicating effective regeneration was achieved. This explains why the purification performance could maintain stable after such a long-term service. In addition, the mass balance showed in the last five operational cycles also indicates that the high effluent quality and stability of the system was sustainable after such a long-term test and further proves the long service life of the system.

3.2. Critical factors affecting regeneration

3.2.1. Current density

In the regeneration process, current density is a key factor. To examine its influence, different current densities were applied, and the results are presented in Fig. 4. As shown in Fig. 4(a), the voltage required increased with current density, but deviated slightly from the linear relation downward. This is in a good agreement with that of EDI [20,29]. Such a phenomenon should be attributed to the gradual enhancement of water dissociation as the current density increased, which allowed more ions to be released from the resin phase to the regenerating solution, leading to a decrease in the solution resistance.

Theoretically, the effect of current density on regeneration is complicated. For anion resins, on one hand, high current density can enhance the water dissociation and, therefore, promote anions desorption. On the other hand, high current density can accelerate anions migration forward. Obviously, an increase in current density is beneficial for anion resins regeneration. However, the situation may be different for cation resins. Although high current density can also promote cations desorption, which is favorable for cation resins regeneration, an increase in current density can accelerate backward migration of cations, which is unfavorable for cation resins regeneration.

As demonstrated in Fig. 4(b), with the increase in current density, both the conductivity and pH value of the concentrate increased, indicating that an increase in current density

Table 3
The theoretical and the practical average conductivities of concentrates of the last five operational cycles

Operational cycle	Average conductivity of feedwater (μS/cm)	Theoretical average conductivity of concentrate (μS/cm)	Practical average conductivity of concentrate (μS/cm)
56	1.26	17.64	17.42
57	1.22	17.08	17.28
58	1.20	16.80	16.75
59	1.25	17.50	17.13
60	1.19	16.66	16.80

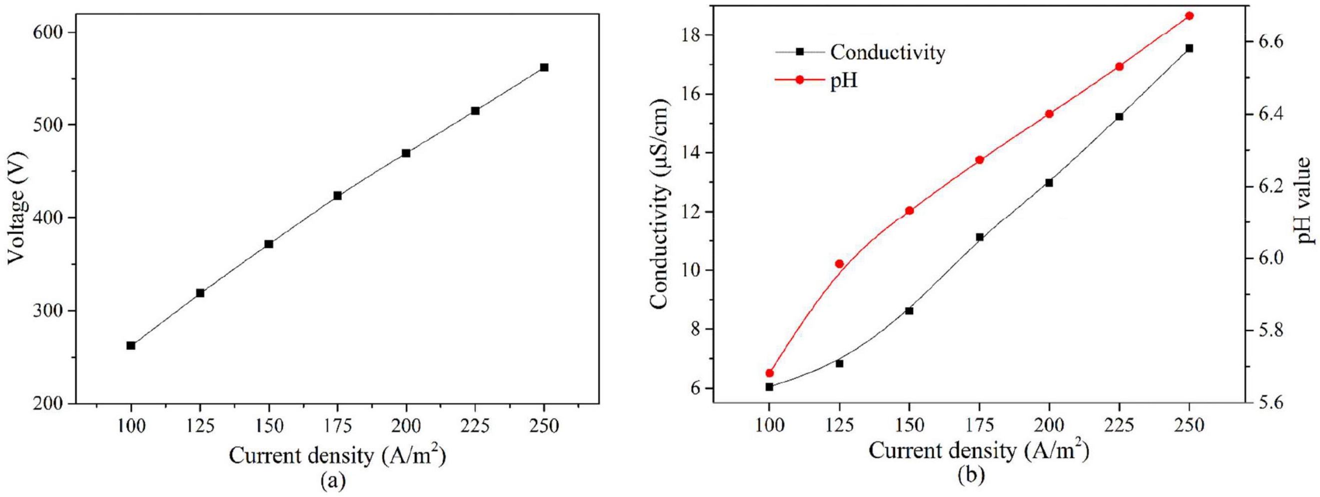


Fig. 4. Regeneration performance of MFEDI at a flow velocity of 50 m/h under different current densities: (a) voltage variation with current density and (b) conductivity and pH variations with current density.

could promote to regenerate both cation resins and anion resins. Such results reveal that the current density increase promotes the water dissociation more significantly than the backward migration of cations. It should be pointed out, however, that high current density means more energy consumption. Based on the consideration of the regeneration performance and energy consumption, the proper current density is about 200 A/m^2. The long-term test also proved that impurity ions adsorbed on the resins were desorbed effectively under 200 A/m^2, and the energy consumption was acceptable.

3.2.2. Regeneration time

Regeneration time is an essential factor influencing regeneration as well as energy and water consumption of the system. Fig. 5 shows the concentrate conductivity and pH value variations with regeneration time. It can be seen from the figure that the concentrate conductivity decreased as the regeneration proceeded. This is associated with the gradual decrease in resins saturation. Such a result means that the regeneration efficiency declines gradually with extension of regeneration time. From this viewpoint, shortening the regeneration step and the purification step proportionally should be helpful for energy saving because ions can be desorbed more at the beginning of a regeneration process. Nevertheless, overly shortening the regeneration time may result in a desorption inequality between cation resins and anion resins. As shown in Fig. 5, the concentrate was acidic at the beginning of regeneration, which means anion resins were easier to be regenerated than cation resins initially. As the regeneration proceeded, the pH value increased gradually, meaning that the proportion of saline cations in concentrate increased. This indicates that extending regeneration time is especially conducive to regeneration of cation resins. If the regeneration time is not long enough, the cations in resin phase would be more than anions, which is surely detrimental for the MFEDI system. Therefore, the regeneration time should be optimized in practical applications. In this work, the purification time, the purification flow velocity, and the regenerating flow velocity were fixed to be 200 min, 70 m/h, and 50 m/h, respectively, under which the proper regeneration time was about 20 min. The result of the long-term test further proved the applicability of 20-min regeneration, because desorption of impurity ions was sufficient, the average pH of the concentrate was close to that of the feedwater, and the water and energy consumption was relatively low.

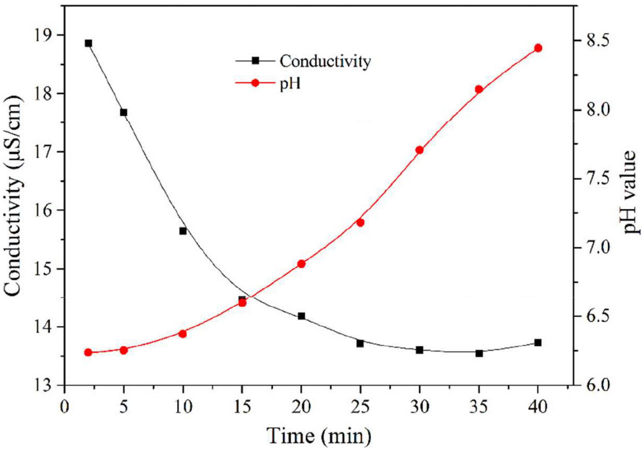
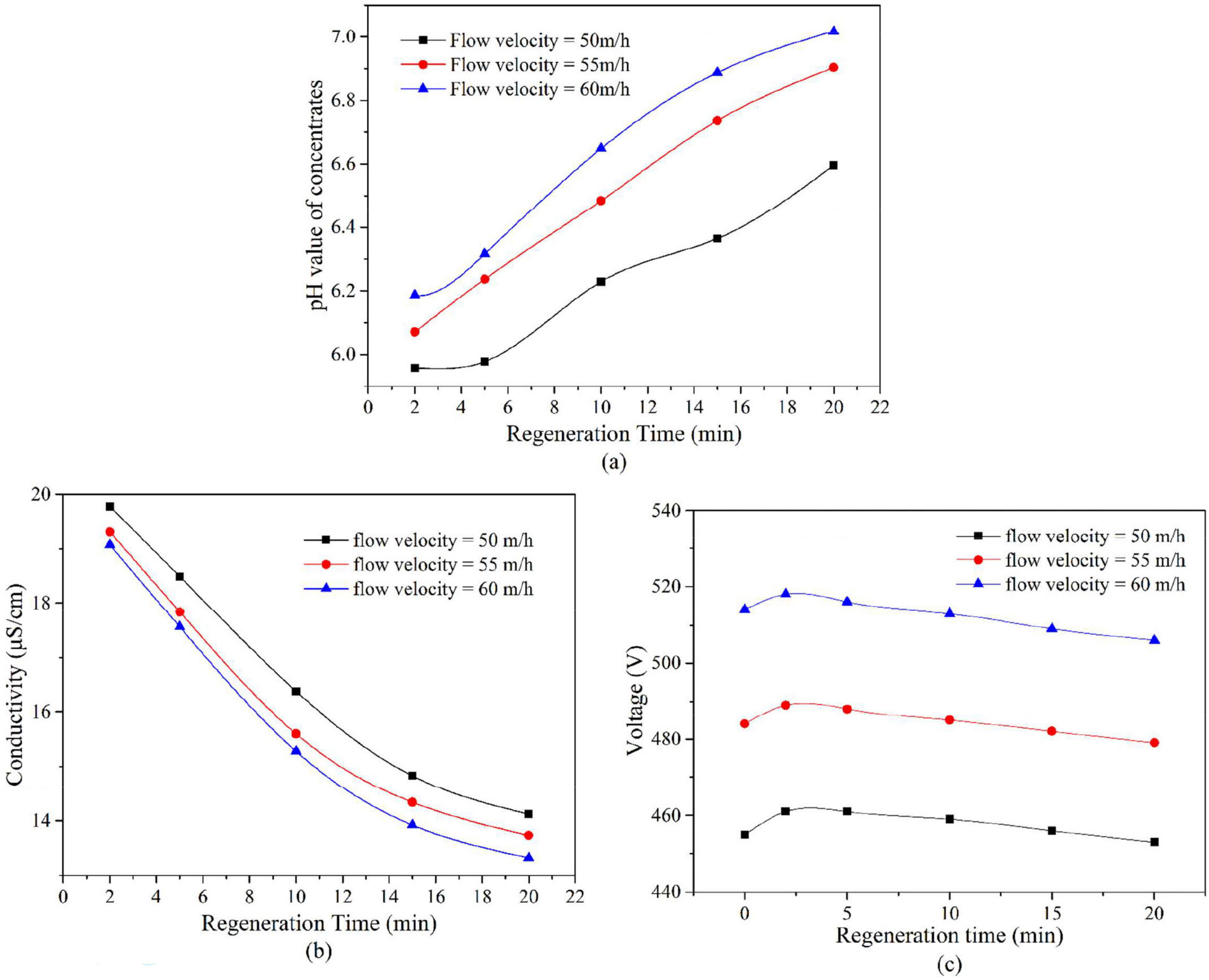


Fig. 5. Variations of conductivities and pH values of concentrates during a 40-min regeneration at a current density of 200 A/m^2 and a flow velocity of 50 m/h.

3.2.3. Regeneration flow velocity

The regeneration flow velocity is also a critical factor influencing the regeneration performance. Fig. 6 shows the variations of conductivity and pH value of the concentrates with regeneration flow velocities. As shown in Fig. 6(a), the concentrate pH value increased as the regeneration flow velocity increased, suggesting that high regeneration flow velocity facilitated cations desorption. This was related to a decrease in reabsorbed ions. In the regeneration process, the desorbed impurity ions were transferred from the resin phase to the aqueous solution. Some of them flowed



(a)

(b)

(c)

Fig. 6. Regeneration performance of MFEDI at a current density of 200 A/m^2 under different flow velocities: (a) concentrate pH variations, (b) concentrate conductivity variations, and (c) voltage variations.

out with concentrates, while the others were readsorbed by the lower layer resins. With the increase in regeneration flow velocity, the residence time of the regeneration flow was shortened, and accordingly there were less ions readsorbed by the resins in lower layers. The reduction of the readsorbing rate is very important for effective regeneration of resins, especially the cation resins. However, as shown in Figs. 6(b) and (c), with the velocity increasing, the concentrate conductivity decreased whereas the voltage required increased. This suggests that an excessively high velocity can lead to a waste of both the high-purity water and energy. The proper regeneration flow velocity is about 50 m/h, under which effective regeneration and relatively low consumption of high-purity water and energy can be achieved.

3.3. Water recovery and energy consumption

Water recovery and energy consumption of the MFEDI system can be calculated according to the following equations:

$$R = \frac{V_1 - V_2}{V_1} \times 100\% \tag{2}$$

$$E = \frac{I\bar{U}t}{V_i - V_r} \tag{3}$$

where R is the water recovery, %; V_i is the total purified water volume, L; V_r is the high-purity water volume consumed for regeneration, L; E is the energy consumption, kWh/m^3 water; I is the electrical current, A; $\bar{U}$ is the average voltage during the whole process of regeneration, V; and t is the regeneration time, h.

In this work, $V_1 = 898.3$ L; $V_2 = 64.2$ L; $\bar{U} = 450$ V; $I = 0.770$ A; $t = 1/3$ h. According to Eqs. (2) and (3), the water recovery rate and energy consumption were 93% and 0.14 kWh/m^3 water, respectively.

In comparison with EDI employed after RO [8,30,31], as Table 4 shows, the energy consumption of MFEDI, water recovery rate and effluent conductivity are all around the same level. Because the manufacturing and maintenance costs of MFEDI are much lower, we consider MFEDI as an economic substitute for EDI.

3.4. Resin layer structure for backward migration obstruction

Resin layer structure can influence the purification stability and regeneration performance dramatically. The resin layer structure employed in this work was selected based on our previous works [17,18,21]. To further prove the function of this structure, the resin bed was chemically analyzed after a long-term service. Fig. 7 shows the distribution of impurity ions right after a regeneration process.

It can be seen from the figure that a quantity of impurity ions, especially the cations, got into the upper layers of the resin bed. This is because the IE performance of the bottom resins was limited when the purifying velocity was up to 70 m/h, especially for the relatively low adsorption rate of WAR. If the backward migration of cations was not impeded effectively, the cations went up in the resin phase and accumulated in the upper layers, which could lead to a deterioration of purification performance. This explains why methods to impede the backward migration are necessary when MFEDI system is employed for purification with a relatively high velocity.

In this work, barrier layers were added as effective elements for preventing the backward migration of cations. The cation resin saturation difference between two sides of a barrier layer demonstrated that the barrier layers were effective in impeding the backward migration of cations. The function of barrier layers was even more significant in obstructing the backward migration of Na^+ ions. As shown in Fig. 7(a), the concentration of Na^+ ions within the resins under the top barrier layer was much higher than that within the resins above this layer. This means the backward migration of Na^+ ions was blocked by the barrier layer so that the ions accumulated more in the lower layers.

Table 4
Comparison of effluent conductivities, water recovery rates, and energy consumptions between EDI and MFEDI used after RO

Process	EDI	MFEDI
Effluent Conductivity (μS/cm)	0.056–0.067	0.056–0.060
Water recovery rate (%)	90–95	93
Energy consumption (kWh/m^3)	0.18–0.28	0.14

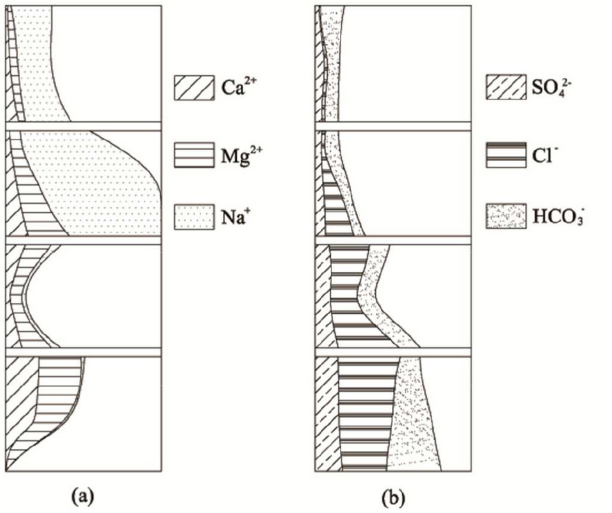


Fig. 7. Ions distribution within the resin bed after regenerated for 60 operational cycles: (a) distribution of Mg^{2+}, Ca^{2+}, and Na^+ within cation resins and (b)distribution of SO_4^{2-}, Cl^-, and HCO_3^- within anion resins.

4. Conclusions

MFEDI with barrier layers was proven to be effective and reliable for purifying low-conductivity water at a high velocity. After a long-term service at a velocity of 70 m/h, the effluent conductivity remained below 0.060 μS/cm, proving the high stability of the MFEDI system. It was demonstrated that regeneration performance was highly dependent on the operational conditions. Effective regeneration was achieved at a current density of 200 A/m^2, a regeneration time of 20 min, and a flow velocity of 50 m/h. The water recovery and energy consumption were around 93% and 0.14 kWh/m^3 water, respectively. In general, MFEDI is a good alternative to the conventional EDI for use in high-purity water production.

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