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SEPARATION SCIENCE AND TECHNOLOGY
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Electrochemical Characterization of Ion-Exchange Resin Beds and Removal of Cobalt by Electrodeionization for High Purity Water Production

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ABSTRACT

The characterization of three cation exchange resins, IRN77, SKN1, and IR120, for the removal of cobalt ions from an aqueous solution by electrodeionization (EDI) was studied. The study includes investigation of resin beds in terms of their ion exchange capacity, electrical conductivity, and zeta potential. The conductivity of a resin bed was analyzed using a porous-plug model to understand the current path. From the experiments, more than a 97% cobalt ion removal was achieved under optimal conditions. Furthermore, production of ultrapure water with a two-stage EDI process was tested for the reuse of treated water.

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Key Words: Electrodeionization; Ion exchange resin; Electrochemical characterization; Cobalt removal; Porous-plug model.

INTRODUCTION

The primary coolant is an essential cooling medium used to control heat in nuclear power plants. The most commonly used primary coolant is high-purity water. During the operation of a nuclear power plant, the surfaces of the primary cooling circuits release corrosion products into the coolant system. These corrosion products may then enter into the core and become activated, leading to an activity build-up on the surfaces of the primary system. Among these corrosion products, the cobalt ion is the major radionuclide found dissolved in the primary coolant water.[1] Ion exchange is a convenient method for the removal of the cobalt ion from the aqueous solution. However, since the method is nonselective in removing radionuclides and nonradionuclides, a large volume of waste is generated and, yet, volume-reduction technology has not been developed for the management of such radioactive waste. To prevent the generation of solid wastes, an electrodeionization (EDI)-based process was studied for purifying the primary coolant using synthetic solutions.

EDI is a hybrid separation system of electrodialysis and ion exchange processes. This system does not require chemicals to regenerate the ion exchange resin or to concentrate the wastewater. In an EDI system, the ion exchange resin bed plays a major role in the reduction of the high electrical resistance in the diluate compartment, while the ion exchange membranes lead to depletion and concentration of the solutions in the diluate compartment and concentrate compartment, respectively.[2]

Ion exchange resins are polymers that have fixed ionic sites that can react with free ions of the opposite charge. The ionic groups, or counter ions, of the resin provide a place at which the ions in solution can be exchanged. Ion exchange resins in an electrolyte solution are electrically conductive, and counter ions can transfer across the polymer under an electric field, allowing mass transfer and associated current flow. The electrical conductivity of ion exchange resins varies with the mobility and affinity of the counter ions with which the resins are in contact.[3] When the concentration of counter ions in the resin phase is higher than the ionic concentration of the solution, the electrical conductivity in the resin bed is enhanced. Therefore, the main advantage of using ion exchange resins in the EDI system is that the resin phase in the diluate compartment can reduce the electrical resistance of the feed water.[4–9]

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Although ion exchange resins plays a significant role in the EDI process, the electrical characteristics of ion exchange resins have rarely been investigated. In this study, the electrical conductivity of ion exchange resins were examined with a flow cell and subsequent EDI experiments on the removal of cobalt ions were performed.

The current flow in ion exchange resin beds is complex, since the beds are a mixture of two phases of different properties, namely resin and interstitial liquid. Several theories have been proposed for the transport phenomena involved—such as conduction of heat or electrical current in mixtures.[10] However, most of the theories are based on the assumptions of either a regular lattice-type arrangement or a completely random distribution of the components. Wyllie suggested an approach for the ion exchange beds where the distributions of the resin particles constitute an irregular and discontinuous phase and the liquid is a continuous one.[11] The model estimates electrochemical properties from empirical and geometrical parameters. This "porous-plug" model is based on the assumption that the electrical current passes through three different paths within the bed. The first path is through alternating layers of particles and interstitial solution, the second is through particles in contact with each other, and the third is the channel through the interstitial solution (Fig. 1).[12] For the model, the conductivity of the resin bed depicted by

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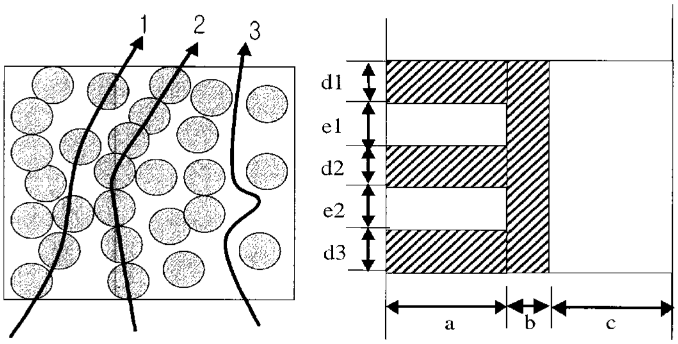

Figure 1. The "porous-plug" model. Left: schematic representation of the three paths which the current can take. Right: the simplified model consisting of three conductance elements in parallel. The geometrical parameters d and e are given by $d_1 + d_2 + d_3 =$ d, and $e_1 + e_2 = e$.

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Eqs. (1) and (2),

$$k_b = k_1 + k_2 + k_3 \tag{1}$$

$$k_1 = \frac{ak\bar{k}}{dk + e\bar{k}} \quad k_2 = b\bar{k} \quad k_3 = ck \tag{2}$$

k_b = conductivity of the resin bed
$\bar{k}$ = conductivity of the resin
k = conductivity of the interstitial solution
k_1 = conductivity through alternating layers of particles and interstitial solution
k_2 = conductivity through the resin
k_3 = conductivity through the solution
a = fractional cross section of conductance elements (first path)
b = fractional cross section of conductance elements (second path)
c = fractional cross section of conductance elements (third path)
d = contribution of the particles
e = contribution of the solution

The parameters of the model were estimated in this study to predict the electrochemical properties of the resins.

An EDI process consists of a mixture of cation and anion exchange resin beads to produce highly purified water. When separating metal ions by an EDI process, the metal precipitation occurs at a high pH as a result of a water-splitting reaction between the cation and anion exchange resin. Hence, both the compartments should be kept under neutral or acidic conditions to prevent formation of excess hydroxide ions. Precipitation on the ion exchange resin or membrane increases the electrical resistance of the system and eventually hinders the flow of water. To overcome this problem, a two-stage EDI process was tested in this study. In the first stage, the separation of cobalt ion is achieved using EDI with cation exchange resins to prevent precipitation in the diluate compartment. In the second stage, using a mixed ion exchange resin, production of pure water from the effluent of the first stage is attained. The concentrate compartment was fed continuously to make pure water while avoiding back migration of ions from the concentrate solution to the diluate solution.

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EXPERIMENTAL

Ion Exchange Capacity and Water Content

Five grams of cation exchange resin was converted into the H^+ form and repeatedly washed with ultrapure water. Exactly 1 g of this resin was transferred into a beaker. Two hundred milliliters of a standard 0.1 N NaOH in 5% NaCl solution was added into the beaker, and it was kept overnight. Then, 20 ml of the supernatant was back titrated with a standard 0.1 N HCl solution. The remaining 4 g of the resins were used for determining the water content.

Zeta Potential Measurement

The cation exchange resins were converted into the K^+ form and washed repeatedly with ultrapure water. For zeta potential measurements, a portion of the ion exchange resin was pulverized in a rock mill. Submicrometer-size fragments of the pulverized resin were obtained using a 1 μm filter. The zeta potential values of the ground resin were determined from electrophoretic mobility measurements made using a commercially available electrophoresis measurement apparatus (ELS-8000, Photal Otsuka Electronics, Japan) with a plate sample cell. The effect of the ionic strength on the zeta potential was demonstrated using various KCl solutions with different concentrations, ranging from 10^{-5} M to 0.1 M of KCl.[13]

Electrical Resistance Measurement

Electrical resistance of the resin bed was measured by using an LCZ meter (Model 2321, NF electronics Japan) and a flow cell. The feed solution was circulated through the flow cell until it reached a steady state with the packed resin in the flow cell. Figure 2 shows the configuration of the flow cell. The top pair of electrodes was used to measure the conductivity of solution by LCZ meter, and bottom pair of electrodes was used to measure the conductivity of solution and resin. The conductivity of the resin was obtained from the difference between them.

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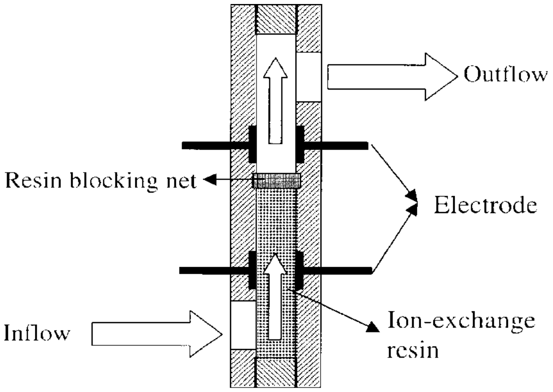

Figure 2. Configuration of the flow cell.

Cell Constant

The cell constant was calculated using a solution of known k value. An LCZ meter can measure the resistance R while k can be determined from Eq. (3):

$$R = \frac{1}{k}\frac{l}{A} \tag{3}$$

l = distance between the electrodes
A = area of the electrode

If "k" is the known value of the standard solution of a reference electrolyte, l/A (the cell constant) can be calculated from the observed resistance using the flow cell and the standard electrolyte. Once the cell constant is known, the conductivity of any electrolyte can be calculated from its measured resistance using Eq. (3).[14]

In this experiment, cobalt nitrate solutions were used as standards and the conductivity of these solutions were measured using a conductivity meter (Model 19820-10, Cole Parmer, Singapore). The cell constant of the flow cell was obtained for each electrode pair with a frequency of either 1 kHz or 100 kHz.

Removal of Cobalt Ion and Production of Pure Water Using Two-Stage EDI

The EDI used in this study was the modified TS-2-10 electrodialyzer (Tokuyama, Japan), which contains ion exchange resins (AMBERLITE IRN

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77 nuclear grade strong acid cation resin, DIAION SKN 1 nuclear grade strong acid cation resin, AMBERLITE IR 120 Na strongly acidic cation exchange resin). The effective area of each membrane was 200 cm^2.

In the first stage of the EDI system, a feed solution of 10 ppm cobalt (Merck, Germany) ion and a concentrate solution of 0.5 N hydrochloric acid (Merck, Germany) were used to prevent the precipitation of cobalt ions with hydroxide ion. The electrode rinse solution was 6% sodium sulfate. The flow rate of the diluate and concentrate solution was 100 mL/min and 400 mL/min, respectively. A current of 0.1 A was applied to the stack from a standard power supply (HP-IB 6674A,Hewlett Packard, USA).

In the second stage, the feed solution used was the effluent from the first stage of the EDI. A voltage of 20 V was applied to the stack. The concentrate solution was about 100 μs/cm acid solution, which is desirable for prevention of back migration. A nuclear grade cation/anion exchange resin mixture (AMBERLITE IRN 77 nuclear grade strong acid cation resin/AMBERLITE IRN 77 nuclear grade strong base anion resin) was used. The ratio of anion to cation exchange resin was adjusted to provide an equivalent value.

Stack Configuration

In the first stage, a bipolar membrane was placed at both ends of the electrodes to block the transfer of ions between the electrode rinse solution and concentrate solution. The bipolar membrane faces the cation exchange resin, making the diluate compartment solution acidic, which is favorable for the removal of heavy metals like the cobalt ion (Fig. 3). In the second stage, conventional EDI was used. Hence, the diluate compartment was packed with a mixed ion exchange resin bed (Fig. 4).

RESULTS AND DISCUSSION

Ion Exchange Capacity and Water Content

To determine the ion exchange capacity of the resins, experiments were carried out for IRN77, SKN1, and IR120. Table 1 shows the ion exchange capacity and water content of the three cation exchange resins. IRN 77 has a slightly higher ion exchange capacity than IR 120 and SKN 1. All the cation exchange resins investigated in this study show a capacity value of greater than 4 mEq/g. However, there was no significant difference in their ion

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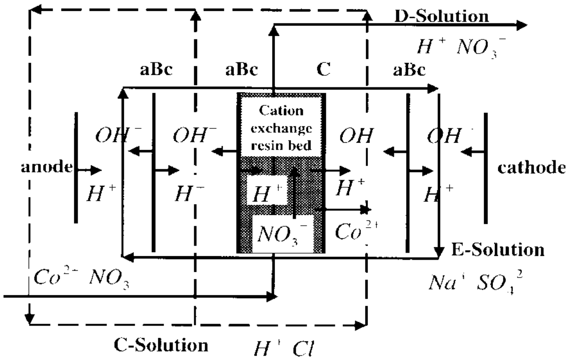

Figure 3. Schematic configuration of the EDI stack for the separation of cobalt ion.

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exchange capacities. The water content is in the order of IRN 77 > IR 120 > SKN 1 with all the three resins having values of greater than 40%. Usually, the water content is due to hydration of the functional groups present in the polymer structure. It was expected that IRN 77 would have the highest conductivity based on the capacity and water content.

Zeta Potential

The number of ionogenic groups in the material generally affects the effective surface charge of an ion exchange resin. This charge is an important

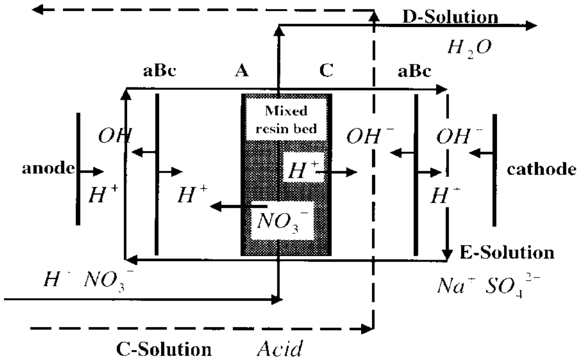

Figure 4. Schematic configuration of the EDI stack for the production of pure water.

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Table 1. Capacity and water content of the cation exchange resins: IRN77, SKN1, IR120.

Ion exchange resin	Exchange capacity (mEq/g)	Water content (%)
IRN77	4.4	50–53
SKN1	4.0	43–45
IR120	4.2	47–49

characteristic controlling the interaction between ionic solutes and the resin surface. The charge properties can be represented by the zeta potential, which is defined as the electrical potential value at the slipping plane (or slip layer) between the Stern layer and the diffuse layer.[15] In this study, an electrophoresis method was used for zeta potential determination, to examine the effective surface charge of the ion exchange resins. The zeta potentials of the three ion exchange resins showed similar results to normal colloidal particles when they were plotted in terms of their ionic strengths, as shown in Fig. 5. IRN77 has a slightly higher zeta potential than IR 120 and SKN 1. The zeta potential values were in the order of IRN 77 > IR 120 > SKN 1. Ion exchange resins contain a high charge density due to their high ion exchange capacities. Therefore, it is anticipated that IRN 77 will have a more negative zeta potential than the IR 120 and SKN 1 resins. It is believed that the zeta

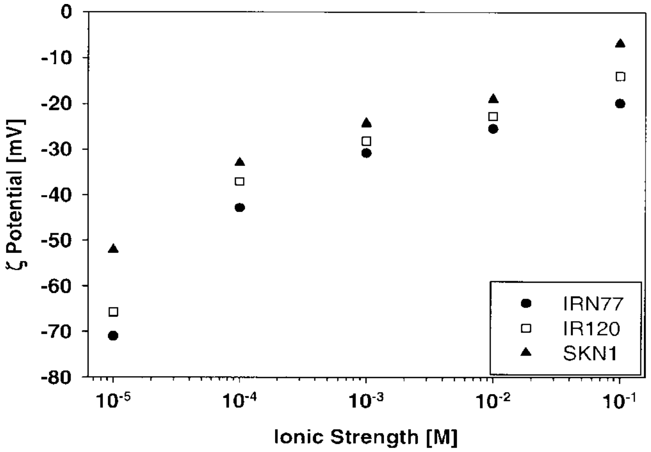

Figure 5. Zeta potential of ground resin as a function of ionic strength; IRN77, IR120, SKN 1.

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potential of IRN 77 mainly depends on the resin surface charge, since it has more dense functional groups.

Conductivity of the Ion Exchange Resin Bed

Figure 6 shows the conductivity of the cation exchange resin beds when in contact with the interstitial solution. At the equiconductance point, the bed conductivity is equal to the solution conductivity. This point represents the conductivity of the corresponding ion exchange resin bed. The IRN 77 resin bed had a higher conductivity than IR 120 and SKN 1 ion exchange resins. The conductivity of ion exchange resins was 7.5 ms/cm for IRN 77, 6.5 ms/cm for IR 120, and 4.2 ms/cm for SKN 1.

There was a significant difference in the conductivity even though only a little difference in the capacities was observed. IRN 77 appears to strongly associate with the cobalt ion, which increases the number of cobalt ions adsorbed on the resin. Therefore, its higher conductivity may lead to a higher ionic flux during electrodeionization. The order of conductivities of the beds is

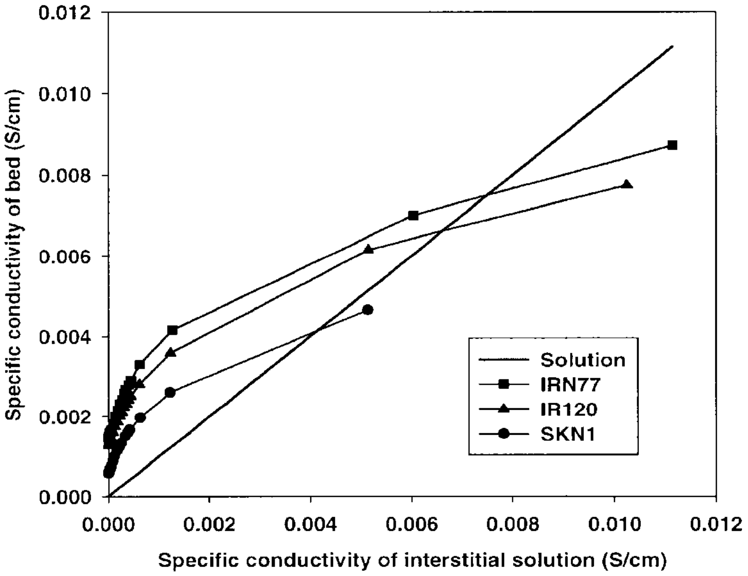

Figure 6. Specific conductivity of the bed as a function of the specific conductivity of the interstitial solution [$Co(NO_3)_2$]; concentration range: 0–1 M.

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shown in Fig. 6. Furthermore, the bed conductivity was measured with low cobalt concentrations as shown in Fig. 7. There was little variation in the conductivity of the ion exchange resin beds at concentrations lower than 1 ppm because the number of ions in the solution phase is insignificant. Operation of the electrodeionization process is desired where the conductivity of the ion exchange resin bed is greater than that of the interstitial solution because a greater conductivity expedites the transport of ions.

The conductivity of an ion exchange resin bed depends on the nature of the counter ion and the concentration of the solution, the conductivity being proportional to the mobility of the ion. As long as the solution is rather dilute, the conductivity of the ion exchange resin, which has the higher ionic concentration, is greater than that of the solution. When the solution concentration is increased, the concentration in the ion exchange resin also increases, and the conductivity of the solution increases faster than that of the ion exchange resin, as shown in Figs. 6 and 7. Thus, in a system consisting of a dilute ionic solution in contact with ion exchange resins, ionic transfer driven by an electrical potential will occur almost exclusively through the ion exchange resin, and not through the water. This ion exchange resin acts as a path for ion transfer and serves as an increased conductivity bridge between

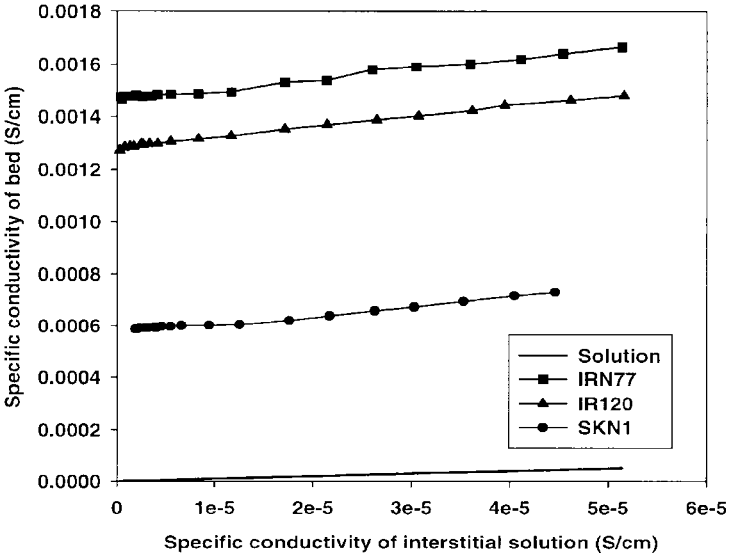

Figure 7. Specific conductivity of the bed as a function of the specific conductivity of the interstitial solution [$Co(NO_3)_2$]; concentration range: 0–10 ppm.

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the membranes for the movement of ions. When the solution concentration decreases, the function of resin is more effective due to the faster ion mobility through the conductivity bridge (ion exchange resin). The case of ion transfer, related to the affinity and mobility of the particular ions in the ion exchange resin, will significantly reduce the electrical resistivity of an electrodialysis system with ion exchange resin.

Porous-Plug Model

The equation for the porous-plug model is based on the geometrical configuration of a resin bed. Even though these parameters cannot be compared directly to the electrodeionization performance, it is possible to predict the portion of the current passing through the three different paths in an ion exchange medium. The parameters of the model were estimated in this study to predict the pattern of ionic flux in the ion exchange resin beds. From Fig. 6, the constants *a*, *b*, *c*, *d*, and *e* were calculated. Figure 8 shows the experimental results for the conductivity measurements for each path through

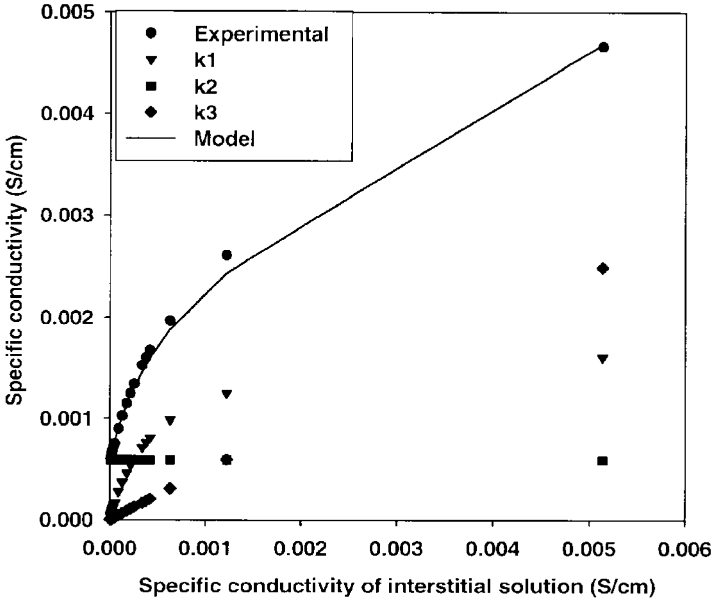

Figure 8. Specific conductivity of the resin bed and k_1 (solution and resin phase), k_2 (resin phase), k_3 (solution phase) with the porous-plug model; IRN77.

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the ion exchange resin beds. The solid line represents the model, which is the sum of k_1 (solution and resin phase), k_2 (resin phase), and k_3 (solution phase). With low interstitial solution concentrations, the current dominantly flows through the resin phase as shown in Fig. 8. Therefore, it is desirable to use a highly conductive resin during EDI operation.

Figure 9 schematically shows the ionic flux within the resin bed. It shows the ratio of the ion-conducting solid volume to solution volume for each resin bed, both horizontally and vertically. In this figure, current flows in the vertical direction. Gray-filled blocks represent the solid resin phase. White blocks correspond to the solution phase. From the left, they include transport through both the solution and resin phase, the solid phase, and the solution phase, respectively.

For SKN 1, in Fig. 9, a larger percentage of the current flows through the solution phase (38%) when compared to the results for IRN 77 and IR 120. Therefore, the ion exchange resins are more conductive than the solutions to be treated. These resins significantly reduce the device resistance and increase the available surface area for ion exchange, which is beneficial when performing a continuous electrodeionization operation with dilute solutions. Although the porous-plug model illustrates the pattern of the ionic flux, the conductivity of the resin bed is an important operating variable to maximize the total ionic flux.

Size Distribution

The size distribution analyses of the ion exchange resin beads were carried out using sieves. Since the ion exchange resin is used in water, sieving was performed with flowing tap water. Most of the IRN77 resin beads were in

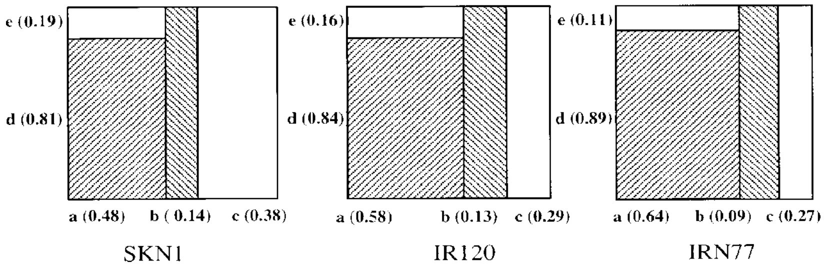

Figure 9. Configuration of the ratio between the solid and solution phases; gray blocks are solid phases and white blocks solution phases.

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the range 600–710 μm (Fig. 10). Uniform particle size and the absence of fine resin beads results in a lower pressure drop compared to IR 120 and SKN 1.

Compared to IRN 77, both SKN 1 and IR 120 have a large portion of resin beads that were smaller than the dominant size resin beads (850–1000 μm). These small beads can fill void volumes when the large resin beads are packed into the channel. Thus, an EDI system comprised of SKN 1 or IR 120 resins may have lower void fractions, as shown in Fig. 9.

Performance of EDI System for the Removal of Cobalt Ion (First Stage)

An EDI process was designed to remove cobalt ions. There are two types of mechanisms involved in the removal of the cobalt ion. One is ion migration forced by the electric potential, and the other one is exchange of the cobalt ion within the ion exchange resin with protons generated within the bipolar membrane (Fig. 11). More than 97% removal efficiency was achieved within 10 minutes after an electrical potential was applied to the stack. The cell voltage showed some variation, probably due to an increase in the electrical resistance and variations in the flow (Fig. 12).

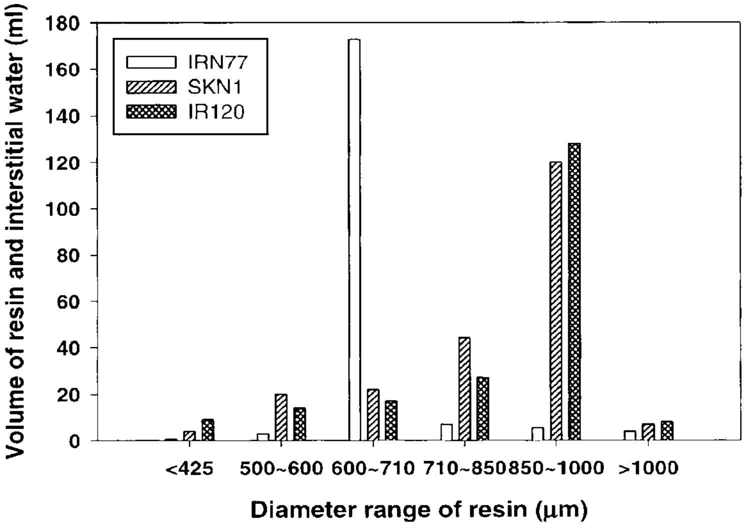

Figure 10. Size distribution of each ion exchange resin; IRN77, SKN1, and IR120; 200 mL of resin and interstitial solution was tested.

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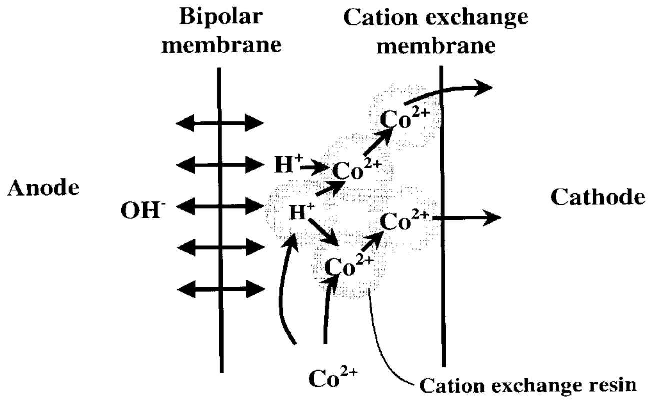

Figure 11. Two mechanisms for cobalt ion removal during EDI with the cation exchange resin and bipolar membrane facing together.

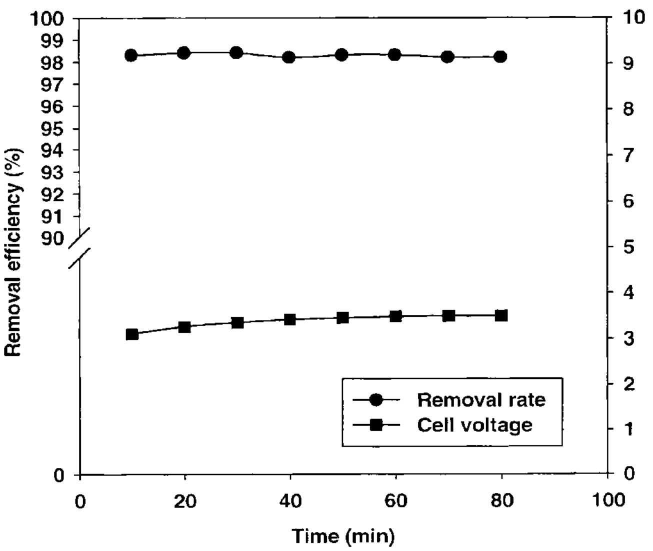

Figure 12. Removal efficiency of the cobalt ion and the voltage drop through the diluate compartment as a function of time; the ion exchange resin used was IR120. Current was 0.1 A.

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Effects of Various Ion Exchange Resins on the Performance of the EDI (First Stage)

EDI operations were performed under the same experimental conditions with the different ion exchange resins in order to compare the effects of each ion exchange resin. After a 30-minute operation, the concentration of the cobalt ion in the effluent of the diluate solution was measured. The experiments were triplicated for each ion exchange resin to ensure reproducibility.

The removal efficiencies and voltage drops across the diluate compartment with the different ion exchange resins were examined. A similar range of removal was achieved although the conductivity of the ion exchange resins was different (Fig. 13). The removal efficiency for all resins was more than 97%. Therefore, the conductivity of the ion exchange resin tested in this pilot scale system did not affect the EDI performance significantly under the experimental conditions employed in this study.

The conductivity and pH of the diluate solution was also similar and favorable for EDI operation with all the ion exchange resin beds (Fig. 14). The conductivity of the diluate solution was 140–200 μs/cm and the pH was in the range of 3–4. The results show that all the three resins are effective in removing cobalt ions during EDI operation.

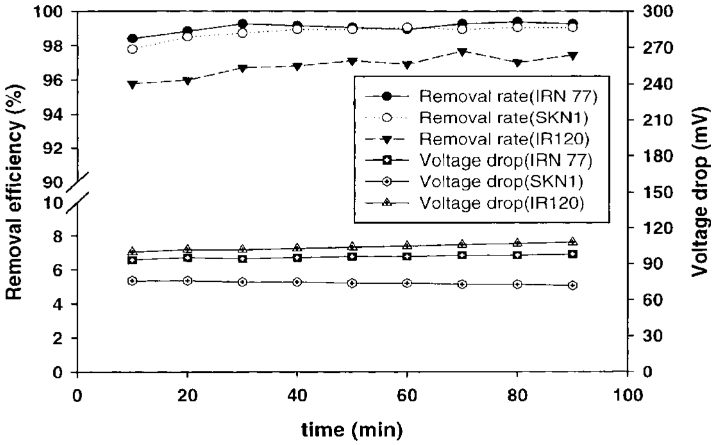

Figure 13. Removal efficiency of the cobalt ion and the voltage drop through the diluate compartment as a function of the ion exchange resin bed; the ion exchange resin used was IR120. Current was 0.1 A.

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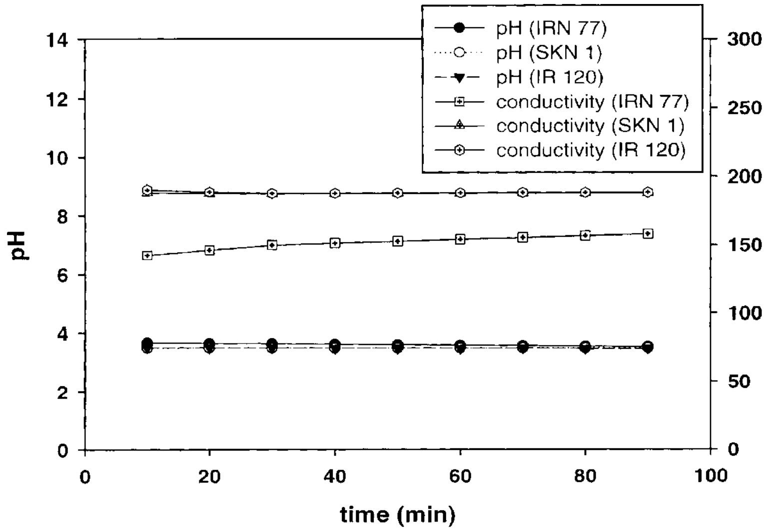

Figure 14. Conductivity and pH of the diluate solution passed through the EDI stack as a function of the ion exchange resin bed.

Performance of EDI Designed to Make Pure Water (Second Stage)

The first-stage EDI was designed to remove cations (in this case, the cobalt ion) only. In the second stage, though, the EDI should remove both cation and anion to produce pure water. While a bipolar membrane, a cation exchange resin was used for the first-stage EDI, an anion exchange membrane was used to remove anions via incorporation of a mixed ion exchange resin bed in the second-stage EDI.

The feed solution used for the second-stage EDI was the effluent from the diluate compartment of the first stage of the EDI, which included nitric acid and hydrochloric acid. Figure 15 shows that the steady production of pure water at a constant pH of 5–6 is possible. The current through the stack was proportional to the conductivity of the diluate solution. Within 60 minutes, the operation of the EDI reached a steady state.

To supply a continuous concentrate solution, another EDI system should be included to desalt the concentrate solution of the second stage of the EDI. Figure 16 conceptually shows the flow diagram of the three-stage EDI. The concentrate solution of the second stage is fed as the diluate solution while the concentrate solution of the first stage is fed as a concentrate solution to another EDI. It is expected that the three-stage EDI configuration would maintain

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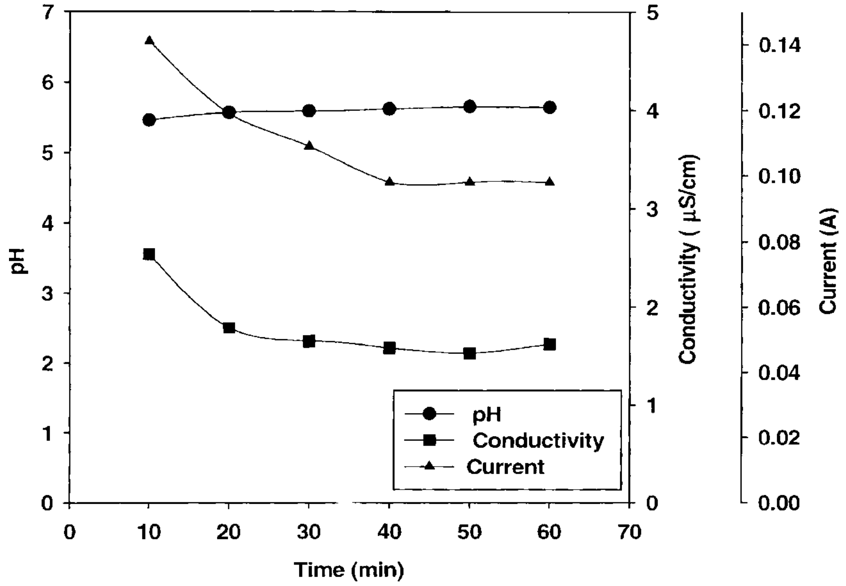

Figure 15. Conductivity, current, and pH of the diluate solution as a function of time; continuous concentrate solution (100 μs/cm), voltage was 20 V.

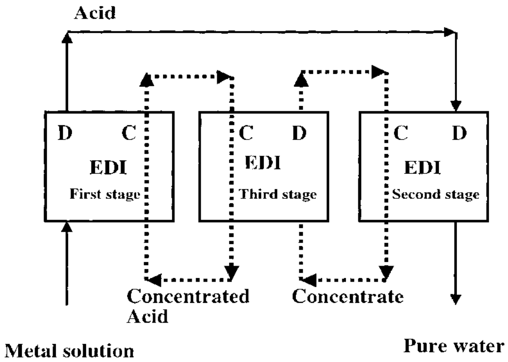

Figure 16. Configuration of the three-stage EDI used to provide pure water steadily from a metal solution.

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the concentration in the concentrate compartment at a reasonable level so that back diffusion into the diluate compartment may be avoided.

CONCLUSION

For selection of the ion exchange resin to be used in an EDI system, the electrical characteristics of several resins were studied using a flow cell. It was found that IRN 77 is the most conductive resin of the resins investigated in this study. From theoretical analyses, the current profile and the geometric configuration of the resin bed was obtained. For the removal of cobalt ions and the production of pure water, a two-stage EDI system was tested. It was found that a removal of more than 97% of the cobalt ions was possible. Comparing the removal efficiencies and cell voltage drops for ion exchange resins packed in the EDI, no significant differences were observed, although the conductivity of the ion exchange resins was different. The effluent of the diluate compartment in the first stage of the EDI system was the purified in the second stage of the EDI system, which was packed with a mixed-bed ion exchange resin. Although our study provides promising results, further studies are needed to investigate the optimum hydrodynamic conditions of the resin bed, the resin fouling, and the long-term stability of the resins.

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