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Preparation and Characterization of Immobilized Ion Exchange Polyurethanes (IEPU) and Their Applications for Continuous Electrodeionization (CEDI)

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Abstract−Immobilized ion-exchange polyurethanes (IEPU) were prepared as ion conducting spacers in continuous electrodeionization (CEDI) for the treatment of a synthetic primary coolant. Ion exchange resins were immobilized by using allophanate/biuret cross-linking in preparation of polyurethane. Synthesized IEPU was characterized in terms of mechanical strength, ion exchange capacity (IEC), electrical conductivity and the porous plug model, which schematically represents the transport pattern through the IEPU. CEDI was carried out in a laboratory scale with an effective area of 20 cm^2. The CEDI operation with a layered bed configuration showed the main removal mechanism of cobalt ion was dependent on the active surface area between ion conducting materials. The performance of the CEDI operation showed over 98% removal of cobalt ions, suggesting the feasibility of IEPU as ion conducting spacers in a CEDI system.

Key words: Ion-exchange Polyurethane, Allophanate/Biuret Cross-linking, Continuous Electrodeionization, Porous Plug Model, Ion-conducting Spacer

INTRODUCTION

Continuous electrodeionization (CEDI) is a novel hybrid separation process of electrodialysis (ED) and ion exchange (IX). The principle of CEDI is illustrated in Fig. 1 and the process operates in two regimes. In the first (enhanced transfer) regime, at a high salinity, the resins in the depleting streams remain in the salt forms, and efficiencies are derived from the resin-enhanced electrical conductivity of the ion-depleting compartments. In the second (electroregeneration) regime, at a low salinity, the resins are electrochemically converted to the hydrogen and hydroxide forms, and deionization is consistent with the model of a continuously regenerated mixed bed ion-exchange column [Kim, 1995; Lee et al., 2002, 2003; Choi et al., 2002]. Therefore, the ion exchange resins packed in CEDI system reduce the electrical resistance and polarization phenomena, accelerating continuous transport of ions by ionic and electronic substitution mechanisms. Since ion exchange resins are also continuously regenerated electrochemically by hydrogen and hydroxyl ions, produced by a water dissociation reaction in an applied electric field, chemical regeneration is not required in CEDI system [Walters et al., 1995; Ganzi et al., 1987, 1992; Thate et al., 1999; Yeon et al., 2004]. It is usually known that CEDI has excellent performance for the ultrapurification of water, producing high purity water over 15.0 MΩ·cm. As a result, a resin-packed CEDI system has recently been developed and available as industrial units [Hernon et al., 1994; Goffin et al., 2000; Giuffrida et al., 1990].

In spite of many advantages of a resin-packed CEDI system, it has shown some problems such as packing difficulty of the resin beads, decreasing free flow with the operation, sinking of the resin beads due to the gravity, irreversible surface degradation of the resin beads by erosion, *etc*. Also, movement of the resins during CEDI operation reduces the active surface area, which is related to the main removal mechanism in the CEDI system. Therefore, CEDI performance becomes reduced with operation, inducing degraded water quality [Kongold et al., 1998; Laktionov et al., 1999].

In this study, IEPU, which can overcome these drawbacks of the resin packed CEDI, were prepared by using a blending method that produces the immobilized resin beads through allophanate/biuret cross-linking [Lee, 2001; Yim et al., 2002; Kim et al., 2003]. To use the synthesized ion exchange media in a CEDI system, the performance of IEPU was investigated for the production of high purity water from the synthetic primary coolant of a nuclear power plant. IEPU may have a high permeability due to the reduced pressure drops by structural geometry. Furthermore, IEPU enables convenient assembly of the stack with their rectangular geometry as shown in Fig. 2.

EXPERIMENTAL

1. Preparation of Immobilized Ion-exchange Resins with Polyurethane

Fig. 3 shows the schematic illustration for the synthesis of ion exchange polyurethane (IEPU). IRN77 and IRN78 (Rohm and Haas, France) were selected as cation exchange resins and anion exchange resins, respectively. Toluene diisocyanate (2,4=80%, 2,6=20%) in a four-neck flask reacted with polypropylene glycol (PPG, M_n=1,000) at 110 °C under nitrogen atmosphere until the theoretical isocyanate content, 10%, was reached. Polyurethane prepolymer (200 g) was mixed with deionized water (19.5 g), ion exchange resin, glycerol (32.5 g) and L-5614 silicone surfactant (15 g) at room temperature. Prepared polyurethane prepolymer was placed in the mold at 40 °C for 24 hr. Then, IEPU were soaked in distilled water for 30 minutes at 40 °C to rearrange the polymeric structure.

2. Characterization of Ion-conducting Media

A Universal Test Machine (INSTRON) was used to determine

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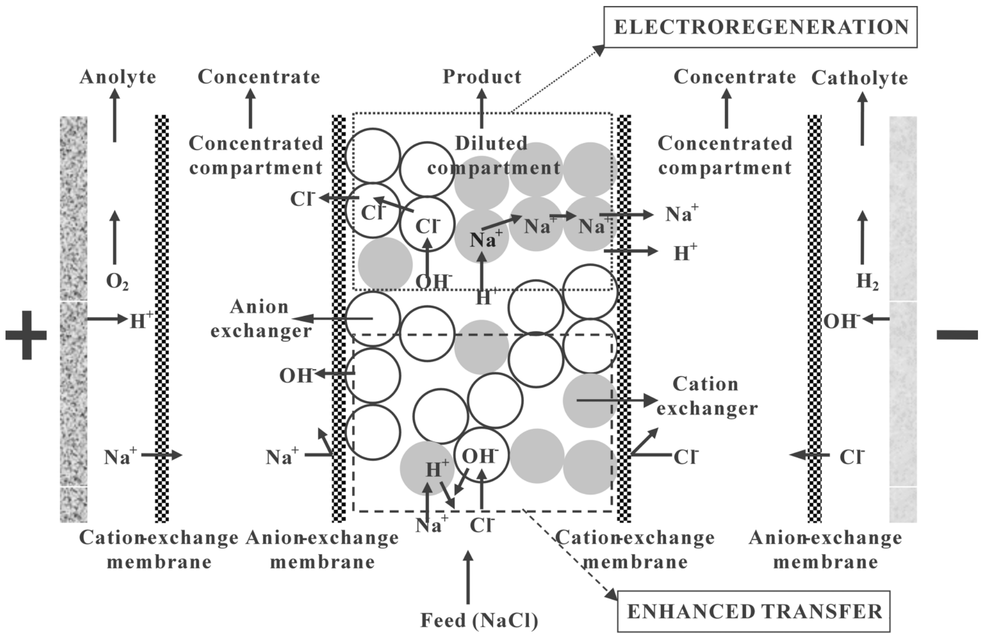


Fig. 1. Principles of CEDI system.

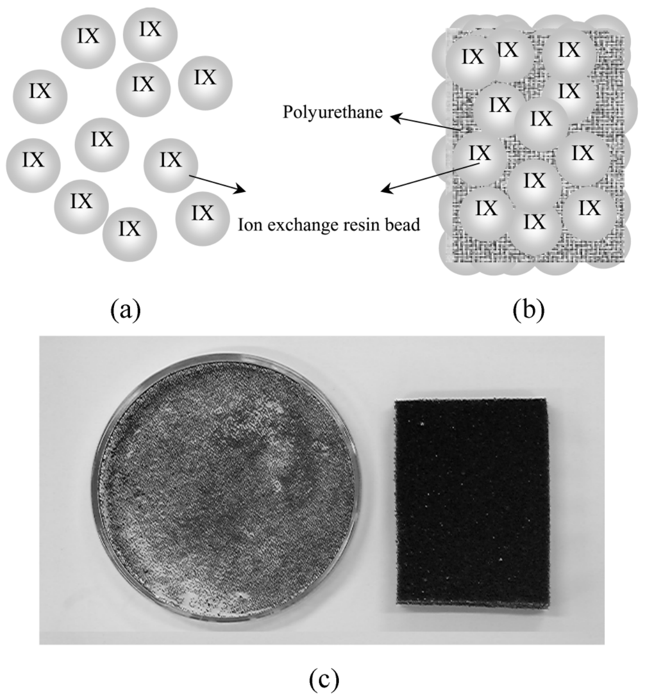


Fig. 2. Schematic illustration of ion exchange resin (IX) (a) and ion exchange poly urethane (IEPU) (b), and digital pictures of the ion-exchange resin (left) and ion exchange polyurethane (right) (c).

the mechanical strength of IEPU by using ASTM-1822-L. The thickness of specimen was 0.5 cm. Modulus, strain and stress were determined at room temperature with a crosshead speed of 25.4 mm min^{-1}

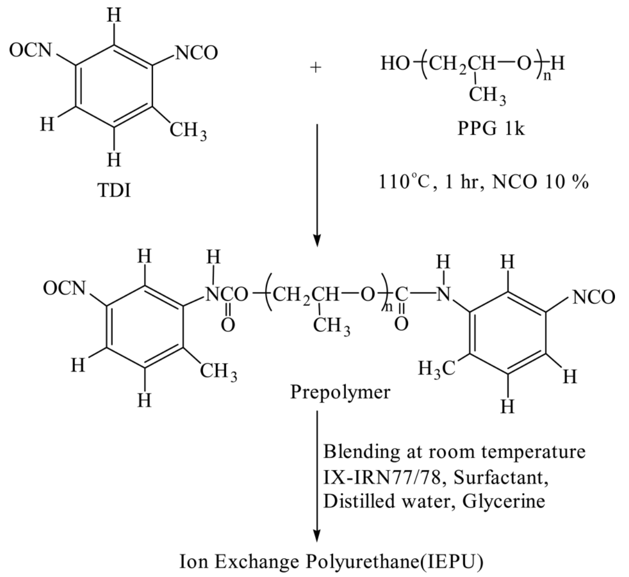


Fig. 3. Synthesis of the ion exchange polyurethane.

[Yeon et al., 2002].

To characterize the water swelling properties of IEPU, the samples were soaked in distilled water for more than 24 hrs. Then, they were wiped with filter paper and weighed. The samples were then dried at 60 °C under a vacuum condition until a constant weight was obtained. The swelling ratio was determined by the following equation:

$$\text{Swelling ratio (\%)} = \frac{W_{wet} - W_{dry}}{W_{dry}}$$

where W_{wet} and W_{dry} are wet and dried sample weights, respectively.

Prepared cation exchange polyurethanes (CIEPU), as an ion conducting medium, were characterized with their removal efficiency of cobalt ion. Predetermined amounts of ion conducting media were added to one hundred milliliters of 10-150 mg dm^{-3} of Co(II) solution. The solutions were agitated at 25±1 °C in a shaking incubator (Model-Jeio Tech SI-900R, Korea). The medium was then separated and the filtrate was analyzed to determine its cobalt content by using an Inductively Coupled Plasma Spectrophotometer (Model-Thermo Jarrel ash IRIS/AP, ICP-AES, UK). All mixing bottles were kept sealed throughout the duration of each isotherm test to minimize volume loss due to evaporation [Rengaraj et al., 2002].

A flow cell was used for the measurement of the electrical conductivity of the media when saturated with a solution of cobalt nitrate [Yeon et al., 2003]. Ion conducting media were placed between a pair of perforated platinum electrodes. The feed solution was circulated through the flow cell until it reached a steady state with the packed resin. The electrical conductivity of ion conducting media was measured with an LCZ meter (Model 2821, NF Electronics, Japan) with the flow cell set at 1.5 cm sec^{-1}. The cell constant was calculated by using a solution having a known k value, while the LCZ meter provided the resistance; R. Therefore, k was determined from the equation:

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

where, l is distance between the electrodes, A, surface area of the electrode.

The permeability test was used to measure the hydraulic resistance of the media in the CEDI module under the presence of ion-exchange membranes. Each ion conducting media was packed in the CEDI system and the pressure drop in the diluted compartment was measured for various flow rates. The permeability of the media in the CEDI module was calculated from the equation [Yeon et al., 2003]:

$$\text{Permeability} = \frac{J \times L}{\Delta P}$$

where, J is flux, L, thickness of the media, ΔP, difference in pressure across the media

3. CEDI Operation

A one-cell laboratory CEDI unit with an effective membrane area of 20 cm^2 was operated for the purification of synthetic primary coolant of a nuclear power plant. The thickness of diluted compartment packed with ion exchange media was 0.95 cm. The CEDI unit was operated with layered bed configuration as shown in Fig. 4. This layered bed configuration was developed to prevent metal precipitation on the ion exchangers [Yeon et al., 2002, 2003, 2004]. The ratio of anion to cation exchange resins was adjusted to be an equivalent value. Prior to CEDI operations, the counter ions in the media were substituted into cobalt and nitrate forms to prevent the removal effect by ion exchange alone. A 0.17 mM cobalt ion was pumped through the diluted compartment at a flow rate of 5 mL min^{-1}. The concentrated solution (1.89 mM Na_2SO_4) and electrode rinse solutions (4.06 mM Na_2SO_4) were circulated at flow rates of 5 mL min^{-1} and 10 mL min^{-1}, respectively. The solution temperature was maintained at 25 °C in the water bath. The cobalt concentration in CEDI effluent was analyzed by ICP-AES (Sci-Tek Instruments, Thermo

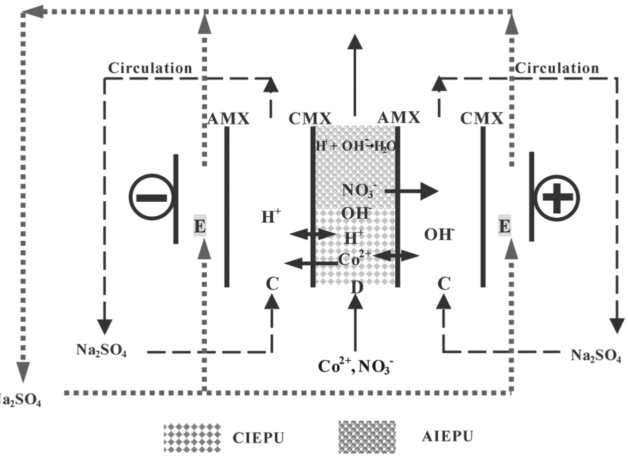


Fig. 4. Layered bed configuration in the diluted compartment. (CMX: cation exchange membrane, AMX: anion exchange membrane, IEPU: ion-exchange polyurethane, CIEPU: cation-exchange polyurethane, AIEPU: anion-exchange polyurethane, C: concentrate, D: diluted, E: electrode rinse solution, Volume ratio: CIEPU : AIEPU=3 : 2).

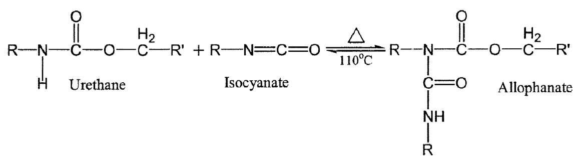
Jarrell Ash IRIS/AP, UK). A conductivity meter (Model 19820-10, Cole-Parmer, Singapore) and pH meter (Orion, USA) were used to measure conductivity and pH during the CEDI operation.

RESULTS AND DISCUSSIONS

1. Crosslinking Mechanisms of IEPU

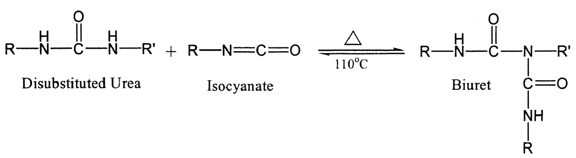
Synthesized IEPU must have high mechanical strength to be used in a CEDI module. However, crosslinking with main reaction in urethane preparation provides weak coherence to cross-linked ion exchange resins and polyurethane, inducing poor mechanical strength. To overcome the weakness, the side reaction of urethane preparation was introduced, leading to the crosslinking reaction between polymer and ion exchange resins with allophanate/biuret groups.

When the reaction temperature is 110±5 °C during the preparation process of urethane foam, the hydrogen on the nitrogen atom of the urethane group reacts with additional isocyanate to form allophante group as follows (1):



(1)

Also, amines produced from the foaming process also react with additional isocyanate, producing a disubstituted urea. Distributed urea generates the biuret linkage as following reaction (2):



(2)

The produced allophanate and biuret linkages from Eq. (1) and (2)

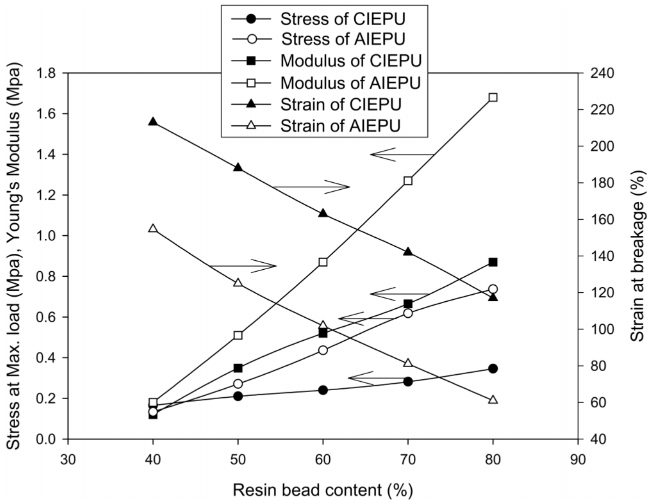


Fig. 5. Mechanical properties of the ion-exchange polyurethane.

can form cross-linking between the polymer and ion exchange resins in IEPU, inducing high mechanical strength [Lee, 2001; Yeon et al., 2002], as illustrated in Fig. 3.

2. Characterization of IEPU as Ion Conducting Spacers

2-1. Effect of Mechanical Properties According to Resin-bead Loading

Fig. 5 presents the effects of the resin content on the mechanical properties of the cation-exchange polyurethane (CIEPU) and anion-exchange polyurethane (AIEPU). In the IEPU, the ion-exchange resins and polyurethane offer the ion exchange capacity (IEC) and mechanical properties, respectively. To be used in a CEDI system, IEPU must have a high mechanical strength and a high ion exchange capacity. These properties of IEPU were greatly influenced by the ratio of ion exchange resin to polyurethane. Fig. 5 shows the stress at the maximum load and modulus of IEPU increased but strain decreased with the resin bead contents. According to the mechanical properties, IEPU with high resin contents up to 80% showed the characteristics of suitable ion-conducting spacer under the influent pressure in a CEDI system.

2-2. Effect of Electric Conductivity with Resin-bead Loading

Fig. 6 presents the effects of the resin content on the specific conductivity of CIEPU and AIEPU. Electrically active media, like IEPU, in CEDI devices may function to alternately collect and discharge ionizable species, or to facilitate the transport of ions continuously by ionic or electronic substitution mechanisms. From Fig. 6, it is evident that the electric conductivity of IEPU increases with the IX bead content. Fig. 6 also shows the conductivity patterns of the IEPU with the resin content in the corresponding solution. For all cases, IEPU showed the higher conductivity than solution and their conductivities increased rapidly with the resin beads content. This result implies that IEPU induces a higher current density than the corresponding solution under the influence of an electric field, showing its feasibility of exchanging counter ions in the CEDI unit as ion conducting spacers.

2-3. Effect of Resin-bead Loading on the Swelling Ratio

Ion conducting spacers must have a high swelling ratio to increase the sorption effect for solvent and a high ion exchange capacity to increase the removal efficiency of CEDI unit. However, it is difficult to optimize the properties of ion conducting spacers because opposite trends are often observed. For instance, a high swelling ratio can be achieved by a low degree of crosslinking in the IEPU, but this also leads to poor mechanical stability and low ion exchange capacity. Thus, an optimized content of polyurethane and resin beads was determined in consideration of swelling ratio and ion exchange capacity, and mechanical strength (Figs. 5, 6, and 7).

Fig. 7 presents the swelling ratio of CIEPU and AIEPU according to the resin-bead content. The swelling ratio of IEPU decreases with a resin content and is inversely proportional to the ion-exchange capacity. This tendency is mainly due to the swelling properties of polyurethane foam. To determine the suitable ratio of prepolymer to resins, they were soaked in water for 24 hours. After the soaking, most IEPU was structurally broken due to different swelling properties of resins and polyurethane. However, the structural change in IEPU was negligible when the fraction of prepolymer to resins was over 0.7 as a weight ratio. From these results, the ratio of prepolymer to resin beads, 3 : 7, was found to be the most suitable. In the subsequent experiments, the ratio of prepolymer to resin beads

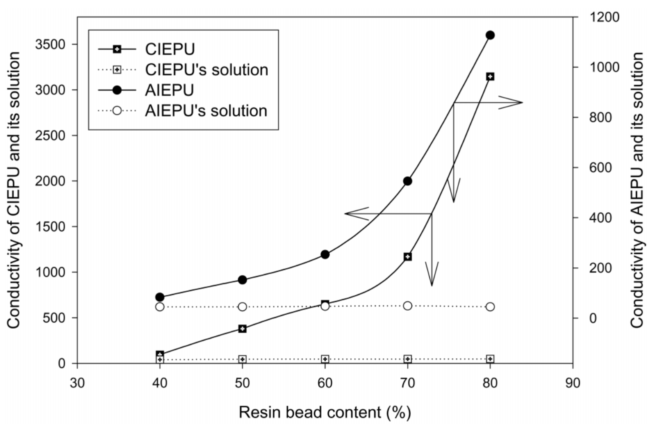


Fig. 6. Specific conductivities of the ion-exchange polyurethanes and their solutions dependent upon the resin bead content. Initial concentration of NaCl=21.8 mg l^{-1}, 46.7 μs cm^{-1}; temperature of test solution=25 °C.

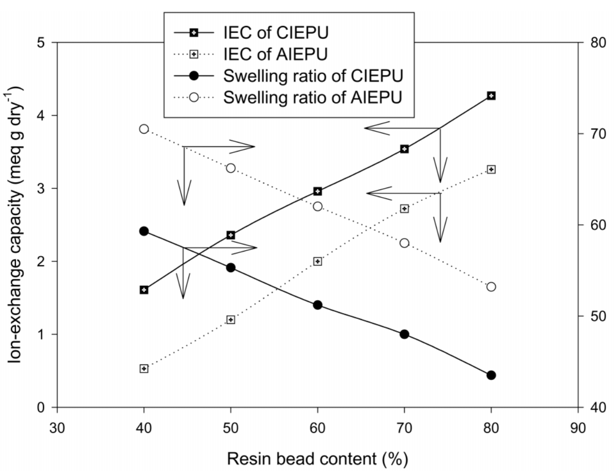


Fig. 7. Correlation between the ion-exchange capacity and swelling ratio according to the resin bead content.

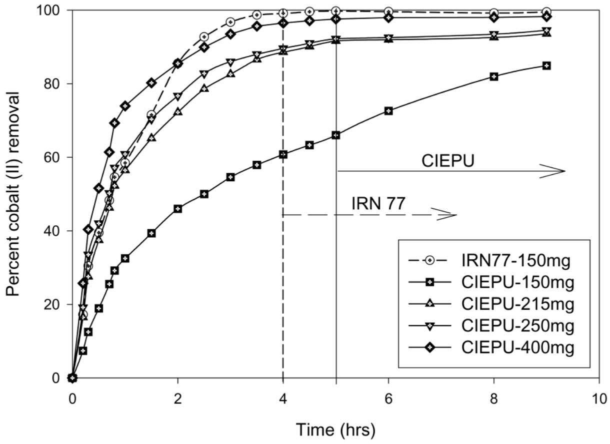


Fig. 8. The effect of contact time on the removal of Co(II) by IRN77 or CIEPU (Conditions: initial Co(II) concentration= 100 mgl^{-1}, pH 5.3).

was fixed to 3 : 7.

2-4. Effects of the Contact Time on the Removal of Co(II)

Fig. 8 shows the effects of the contact time on the removal of Co(II) by IRN77 and CIEPU. The removal rate increases with time and reaches equilibrium within 4 hours for IRN77 and 5 hours for CIEPU at an initial Co(II) concentration of 100 mg dm^{-3}. The metal uptake versus time curves are single, smooth and continuous, leading to saturation. This suggests a possible mono layer coverage of the metal ions on the surface of the adsorbent [Namasivayam et al., 1995; Battacharya et al., 1984].

2-5. Electrical Characteristics

To examine the electrical characteristics of the prepared CIEPU, ion exchange capacity and electrical conductivity were measured. Ion exchange capacity of the media is the maximum concentration for taking up the counter ions during the ion exchange operation. The ion exchange capacity for the CIEPU was found to be 3.7 meq g^{-1}, which was lower than that (4.2 meq g^{-1}) of IRN 77 [Yeon et al., 2003].

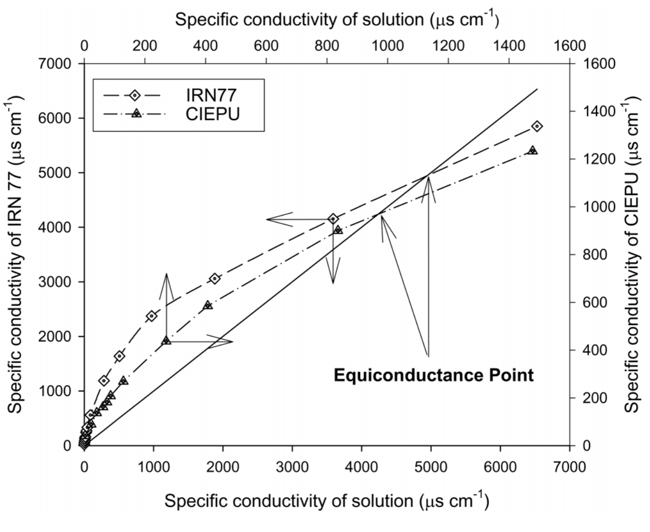


Fig. 9. Specific conductivity of the ion conducting media in versus that of the solution when in equilibrium with $Co(NO_3)_2$.

Fig. 9 shows the electrical conductivities of CIEPU and IRN 77. Operating regions of CEDI can be determined from the equiconductance point. Below the equiconductance point, ion exchange resins help to induce the high current of the system with their conductivities. From Fig. 9, the specific conductivity at equiconductance point, $\bar{k}$, of the media was determined as 1,127 and 4,900 μs cm^{-1} for CIEPU and IRN77, respectively. These results show that CIEPU can be operated under the high feed conductivity, 1,127 μs cm^{-1}, in the CEDI system. Electrical conductivities of the CIEPU indicate the feasibility of the CIEPU as the ion conducting media for the treatment of the highly conductive feed water [Helfferich, 1962].

2-6. Porous Plug Model

The "porous-plug" model is based on the principle that the electrical current passes through three different paths within the media. The first path is the alternating layers of the foam and interstitial solution, the second is active conducting surface areas which are in contact with each other, and the third is the channel through the interstitial solution [Helfferich, 1962; Wyllie et al., 1955]. From this model, the electrochemical properties can be estimated by using empirical and geometrical parameters.

The parameters of the model were estimated, in this study, to predict the electrochemical properties of the media [Yeon et al., 2002, 2003]. From electrical conductivity of media (Fig. 9), the geometrical parameters a, b, c, d and e were determined (Fig. 10). Even though these parameters cannot be compared directly to the electrodeionization performance, it is possible to predict the portion of electrical current passing through the three different paths in an ion-exchange medium. Fig. 10 shows the rectangular configuration of the media. It illustrates the configuration of the ratio between the solid and solution portions of the media horizontally and vertically. Fig. 10 shows most of the current passes through a mixture of the media and solution phases in the IEPU conducting spacer. This result indicates that IEPU is electrically more conductive than the solutions, suggesting that IEPU, as ion conducting medium, is desirable for the treatment of diluted solutions with a CEDI operation. Fig. 10 also indicates the effect of geometrical shapes of ion exchange media. A higher value of b in CIEPU than that in IRN 77 suggests that an active conducting pathway in CIEPU is well developed due to the immobilization of ion exchange resin bead in the polyurethane foam. Also, CIEPU has larger current pathway than IRN 77 through a mixture of the media. This may be caused by the geometrical shapes

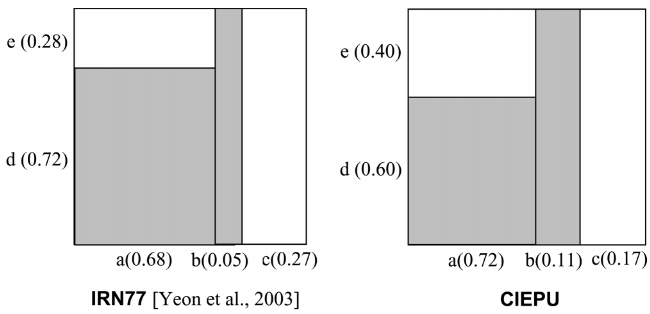


Fig. 10. Ratio configuration for conductance through the solid phase and the solution phase based on the "porous-plug" model; Gray blocks are solid phases and white blocks are solution phases.

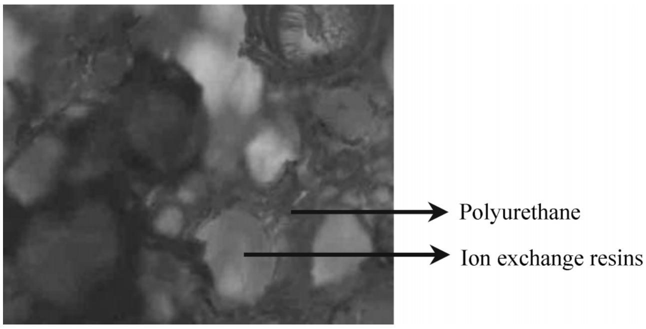


Fig. 11. Photograph of IEPU (10-fold magnification).

of IEPU as shown in Fig. 11. The morphology of CIEPU shows resin beads are enclosed by polyurethane. The geometrical configuration can induce a current path through a mixture of the solid and solution phases. Thus, CIEPU has a larger effective area than IRN 77 in conducting pathway through the mixtures of media (“a” in Fig. 10).

3. CEDI Applications

3-1. Permeability of the Media through CEDI Module

When the diluted compartment is packed with ion conducting media, the feed solution is driven upward in the compartment and attaining a freely flowing solution is difficult. Therefore, a permeability test was carried out to measure the hydraulic resistance of the media in the CEDI module in the presence of ion-exchange membranes and media.

The permeability of the media is a function of the pore size, the specific void volume or the porosity, and the thickness of diluted compartment in CEDI system. The media permeability has significant effects on the design of CEDI modules and systems as well as on process economics [Winston Ho et al., 1992; Mulder, 1996]. Permeability was determined by using the CEDI module with IEPU at the same thickness as diluted compartment with various IEPU spacers and the results are summarized in Table 1. The density of the CIEPU was in the range of 0.39-0.60 g cm^{-3}. Permeability was inversely proportional to the contents of ion exchange resins, but all the cases showed the higher permeability than that of IRN 77, indicating the prepared IEPU is suitable as an ion conducting spacer in the CEDI system.

3-2. CEDI Operation with Vertically Layered Bed

CEDI with mixed-bed ion exchange resins has been used for high-purity water production in previous studies [Ganzi et al., 1987, 1992; Thate et al., 1999]. However, such a CEDI configuration cannot treat solutions containing metal ions because of the precipitation of metal hydroxides. In order to improve the performance of a CEDI device, a CEDI system packed with a layered bed configuration

Table 1. Permeability for the removal of Co(II) by IRN77 and CIEPU system

	CIEPU 1	CIEPU 2	CIEPU 3	IX* (IRN77)
Contents of IX (%)	40	50	60	100
Density (g cm^{-3})	0.39	0.49	0.60	0.87
Permeability ($l \cdot m^{-1} \cdot hr^{-1} \cdot bar^{-1}$)	16,000	14,700	13,300	9,000

*Yeon et al., 2003

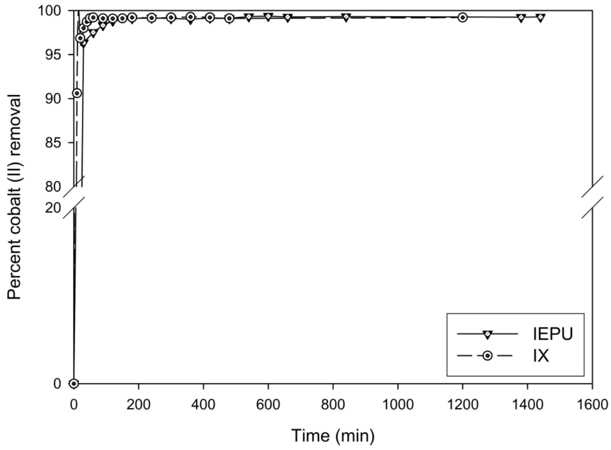


Fig. 12. Percent Co(II) removal over time dependent upon the spacer used.

was employed as shown in Fig. 4.

Fig. 12 shows the result of CEDI operation with ion exchange resins and IEPU. Removal of the cobalt ions reached 99% and the pH of the diluted compartment was between 6.5 and 7.5. There was no significant difference between CIEPU and IRN 77 for the removal of cobalt ion, showing the removal rates of 98-99%. The treated water with the two different media in the CEDI unit showed a resistivity over 1.0 M$\Omega \cdot$cm. The high removal rate with both conducting media in spite of their different ion exchange capacity indicates that the removal mechanism is not mainly dependent on the ion exchange capacity of media but other properties [Yeon et al., 2003]. In this case, the high removal rate of IEPU may be due to a higher active conducting surface area (“b” in Fig. 10) than that of IRN 77. The increase in the active conducting surface area by the immobilization of ion exchange resins in the CIEPU structure causes the effective conduction of ions in the CEDI system.

CONCLUSIONS

Ion conducting spacers for the removal of cobalt ions in CEDI system were prepared by using flexible polyurethane foams. Characterization of IEPU proved the feasibility of synthesized IEPU as ion conducting spacers in consideration of mechanical strength, permeability, and removal rate. The porous plug model suggested the transport patterns of ions through the ion exchange media. The performances of the CEDI operation showed a removal rate of cobalt ion over 98%, producing high purity water. Comparing the removal efficiencies for IX and IEPU packed in the CEDI, no significant differences were observed although the time required for a steady state was different. The high removal rate of IEPU was due to increased active conducting surface area by immobilization of ion exchange resins, not by their ion exchange capacities. IEPU spacer was proved to be an effective ion-conducting spacer with advantages of elastic mechanical properties, ease of handling and uniform distribution of feedwater.

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