# 이온교환 작용기를 갖는 Poly(ether ether ketone): 제조 및 축전식 탈염 효율을 향상시키기 위한 탄소전극 바인더로서의 활용

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# Ion-Exchange Functional Group-Containing Poly(ether ether ketone)s: Preparation and Use as Binder in Fabrication of Carbon Electrodes to Improve Capacitive Deionization Efficiency

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초록: 본 연구에서는 4차 암모늄염기를 갖는 poly(ether ether ketone)(PEEK)와 술폰산기를 갖는 PEEK를 제조하고, 축전식 탈염(capacitive deionization, CDI) 셀에 필요한 탄소전극의 바인더로서 사용하였다. 이온교환 고분자 바인더를 사용하여 제조한 탄소전극으로 조립된 CDI 셀은(500 ppm NaCl 수용액의 투입 속도: 20 mL/min, 흡착과 탈착: 각각 1.0 V와 0.0 V), styrene-butadiene 바인더로 제조한 탄소전극에 비하여 크게 향상된 염 흡착 능(salt adsorption capacity, SAC)과 전하효율(charge efficiency, CE)을 나타냈다(SAC: 10.1 w 6.8 mg/g, CE: 79.8 w 37.7%). CDI 셀의 성능이 크게 향상된 주된 이유는 흡착단계에서 하전된 탄소전극에 이온들이 선택적으로 흡착하도록 하는 이온교환 고분자 바인더 때문인 것으로 이해된다.

**Abstract:** In order to develop polymer binder for use in the fabrication of activated carbon (AC) for capacitive deionization (CDI), we prepared quaternary ammonium group-bearing poly(ether ether ketone) (PEEK) and SO<sub>3</sub>H-bearing PEEK, and used them as binder in the preparation of AC electrodes. A CDI cell assembled using the electrodes (feed concentration: 500 ppm NaCl, flow rate: 20 mL/min, adsorption: 1.0 V, desorption: 0.0 V), exhibited greatly improved salt removal performance, compared to a cell assembled using the carbon electrodes fabricated from styrene-butadiene rubber as a binder (salt adsorption capacity: 10.1 vs 6.8 mg/g, charge efficiency: 79.8 vs 37.7%). The greatly improved cell performance was attributed to the ion-exchange binders that were well distributed throughout the AC electrodes, leading to selective ion adsorption onto the carbon electrodes during the adsorption steps.

**Keywords:** ion-exchange polymer, poly(ether ether ketone), polymer binder, capacitive deionization cell, carbon electrode.

#### Introduction

Capacitive deionization (CDI) can be employed to remove dissolved salts from brackish water with much less energy, compared to electrodialysis and reverse osmosis.<sup>1,2</sup> In CDI

technology, during the adsorption step, counter-ions are adsorbed onto the electrode surface, while co-ions are repelled. This means that ions can adsorb and desorb concurrently and repeatedly in the electrode pores, resulting in a decrease in charge efficiency (CE). When ion-exchange membranes are placed onto the surface of the carbon electrodes, to form a membrane capacitive deionization (MCDI) cell, the salt removal performance can be largely improved.<sup>3</sup> However, ion-exchange membranes are expensive, and limit the commercialization of MCDI system in the actual desalination process.

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One of the approaches to improve CDI cell performance without using ion-exchange membranes is to develop high-performance carbon materials. Thus, various types of carbon materials can be employed as electrodes, including activated carbon (AC),<sup>4-6</sup> carbon cloth,<sup>7,8</sup> carbon fiber,<sup>9,10</sup> carbon aerogel,<sup>11,12</sup> carbon nanotube (CNT),<sup>13-15</sup> and graphene.<sup>16,17</sup> CNT and graphene have some limitations because of insufficient porosity,<sup>18,19</sup> and carbon fibers are expensive. In contrast, AC may be one of the best choices, because of its good electrical conductivity and high chemical stability, as well as high specific surface area.

The second approach may be a chemical modification of the AC electrode surface to incorporate appropriate functional groups. For example, Min and coworkers synthesized the SO<sub>3</sub>H-containing AC from TiO<sub>2</sub>-coated carbon.<sup>20</sup> The modified electrode-based CDI cell exhibited higher efficiency than the unmodified electrode-based cell, due to the modified electrode with high ion selectivity and wettability. Haq *et al.* prepared NH<sub>2</sub>- and SO<sub>3</sub>H-containing carbon electrodes via chemical treatments of AC powders. The salt adsorption capacity (SAC) of the functionalized electrodes-based CDI cell (14.7 mg/g) was comparable to that of MCDI cells.<sup>21</sup> They claimed that the highly enhanced cell's performance resulted from the functional groups that make the electrodes more easily wet and ion-selective.

The third approach may be the chemical modification of polymer binders for AC electrodes because ion-exchange group-containing binders may improve ion selectivity, as well as wettability, of the electrodes. Poly(vinyl alcohol) (PVA) was

employed as AC electrode binder to improve the wettability.<sup>22,23</sup> The specific capacitance of the hydrophilic binder-based electrode was 1.3 times higher than that of the hydrophobic poly(vinylidene fluoride)-based electrode. Furthermore, the resulting CDI cell exhibited the SAC of 9.96 mg/g (200 ppm NaCl solution, 1.2 V). Poly(arylene ether sulfone) copolymers were also tested as a binder of AC electrode, but their effect on the desalination performance was not reported.<sup>24</sup>

In this work, we prepared quaternary ammonium group-bearing poly(ether ether ketone) (APEEK) and sulfonic acid group-bearing poly(ether ether ketone) (SPEEK) from commercial poly(ether ether ketone) (PEEK), one of the engineering polymers. The ion-exchange polymers were used as binder to prepare AC electrodes. We fabricated CDI cells using the electrodes, and tested their desalination performance for 500 ppm NaCl solution. For comparison, styrene-butadiene rubber (SBR), which is hydrophobic and has no functional group, was also used as binder to fabricate the AC electrodes. A CDI cell was prepared from the SBR-based AC electrodes, and tested under the same conditions.

## Experimental

Materials. Poly(ether ether ketone) (PEEK) and trimethylamine (30%) were purchased from Victrex® USA Inc. and JUNSEI Chem. Co. Ltd., Japan, respectively. Methanesulfonic acid (99%), sulfuric acid (95%), *N,N*-dimethylformamide (DMF, 99%), diethyl ether (99%), sodium chloride (NaCl, 99.5%), sodium hydroxide (NaOH, bead, 98%) and hydro-

Scheme 1. Schematic routes to APEEK and SPEEK.

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Table 1. Fabrication Conditions for the Various AC Electrodes

(unit: g)

Electrode	Activated carbon	Polymer binder	Solvent	CMC
3A–AC	4.0	0.12 (APEEK)	9.0 (DMF)	0
3S–AC	4.0	0.12 (SPEEK)	9.0 (DMF)	
5A–AC	4.0	0.20 (APEEK)	9.0 (DMF)	0
5S–AC	4.0	0.20 (SPEEK)	9.0 (DMF)	0
7A–AC	4.0	0.28 (APEEK)	9.0 (DMF)	0
7S–AC	4.0	0.28 (SPEEK)	9.0 (DMF)	0
SBR-AC	4.5	0.50 (SBR)	11.0 (water)	0.05

chloric acid (35-37%) were purchased from SAMCHUN Chemical Co. Ltd., Korea. Paraformaldehyde (powder, 95%), tin(IV) chloride (98%), and sodium carboxymethyl cellulose (CMC) were purchased from Sigma-Aldrich. Trimethylchlorosilane (98%) and phenolphthalein solution (1%) were purchased from Daejung Chemicals, Korea. AC powders (CEP-21K) were purchased from Power Carbon Technology Co., Korea. Graphite sheets (F02511, thickness: 300 μm) were purchased from Dongbang Carbon Co., Korea. SBR emulsion (EQ-Lib-SBR) was purchased from MTI Co. Ltd., Korea. All the chemicals were used as received, without any further purification.

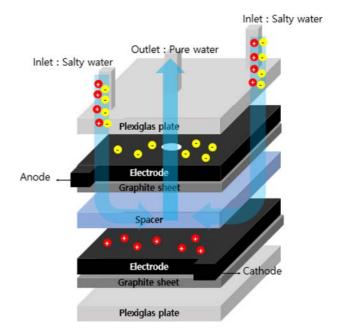
Preparation of APEEK and SPEEK. The ion-exchange polymers were prepared from PEEK by following reported procedures, <sup>25,26</sup> as shown in Scheme 1 (See the Supporting Information Section 1 and 2 for the experimental procedures).

Preparation of AC Electrodes. APEEK or SPEEK was dissolved in DMF, followed by the addition of AC powders. The weight ratios of the polymer binders to the AC powders were varied to be 1:0.03, 1:0.05, and 1:0.07 (Table 1). Each slurry was spread on a graphite sheet using a doctor blade (coating thickness: 350 µm), and the electrodes were dried (60 °C, 1 h). The electrodes prepared from APEEK were designated 3A-AC, 5A-AC, and 7A-AC, respectively. The numbers correspond to the weight percentages of APEEK over the total weights of binder and AC, and the capital A means APEEK. Similarly, the electrodes prepared from SPEEK were designated 3S-AC, 5S-AC, and 7S-AC, respectively. The numbers correspond to the weight percentages of SPEEK over the total weights, and the capital S means SPEEK. For comparison, SBR was also used to prepare an AC electrode (SBR-AC) as a reference binder where the SBR content was 10 wt%, and CMC was used as a dispersing agent (1 wt%).

Instrumental Analysis. Fourier-transform infrared (FTIR) spectrometry (4100E, JASCO Deutschland GmbH, Pfungstadt,

Germany) was employed to obtain FTIR spectra. Proton nuclear magnetic resonance (NMR) spectra were obtained on a JEOL FT-NMR (400 MHz, JEOL Ltd., Japan) spectrophotometer. The presence of either sulfur or nitrogen element in the prepared AC electrodes was confirmed by elemental mapping using field emission scanning electron microscope (FE-SEM, Hitachi SU-70, Japan). The electrical resistances were measured using a four-point probe system (CMT-SR2000N, AIT Co., Korea), where three different places were chosen for measurements, and the average value was taken.

Desalination Experiment. Each cell consisted of a graphite sheet (current collector), carbon electrode, and a spacer (180 μm thickness for water flow channel) (Scheme 2).<sup>27</sup> The area of each electrode was 70 cm<sup>2</sup>. The experimental details were explained in the literatutre.<sup>21</sup> A 500 ppm NaCl feed solution was fed to the CDI cell (20 mL/min). 1.0 V was provided to



Scheme 2. Schematic representation of CDI cell.

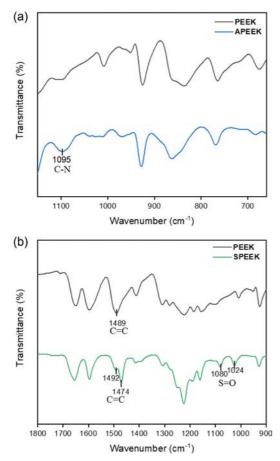
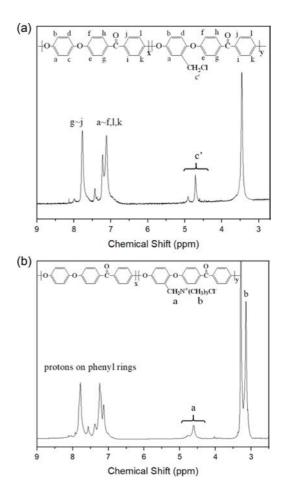


Figure 1. FTIR spectra: (a) APEEK; (b) SPEEK.

the CDI cell for 5 min for adsorption, and then the cell was discharged for 5 min for desorption, using a potentiostat (WPG100, WonA Tech Co., Korea). The effluent electrical conductivity was determined with 3 s intervals (Labquest, Vernier Software & Technology, USA).

#### Results and Discussion

FTIR, NMR Spectra, and IEC of the Prepared Polymers. APPEK was prepared from PEEK by following a reported procedure, as shown in Scheme 1.<sup>25</sup> The FTIR spectrum of APEEK in Figure 1(a) reveals a new peak for C–N bond at 1095 cm<sup>-1</sup>. This result indicates that APEEK was successfully prepared from PEEK. We used concentrated sulfuric acid to prepare SPEEK from PEEK, whose procedure has been well-known in the literature.<sup>26</sup> SPEEK in Figure 1(b) shows two new peaks for sulfonic acid group at 1024 and 1080 cm<sup>-1</sup>, whose absorption peaks are not seen in the IR spectrum of PEEK. This result indicates that under the experimental conditions, sulfonic acid groups were successfully introduced into



**Figure 2.** <sup>1</sup>H NMR spectra: (a) CMPEEK; (b) APEEK (DMSO-*d*<sub>6</sub>).

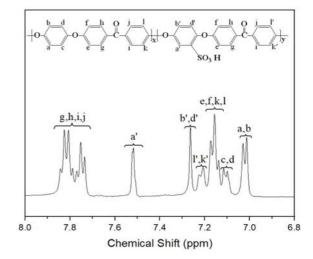


Figure 3. <sup>1</sup>H NMR spectrum of SPEEK (DMSO-d<sub>6</sub>).

#### PEEK.

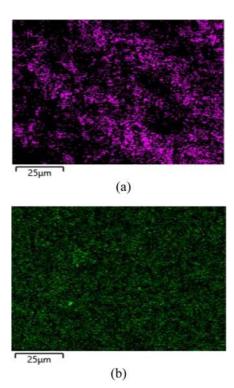
The chloromethylated PEEK (CMPEEK) proton NMR spectrum in Figure 2 shows the resonance peaks at 4.91 and 4.71 ppm, corresponding to CH<sub>2</sub>Cl protons. The peaks in the range

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7 to 8 ppm correspond to the aromatic ring protons. Based on the integrals of these peaks, the degree of chloromethylation was calculated to be 90%.<sup>25</sup> Figure 3 shows the proton NMR spectrum of SPEEK, where the resonance peak a' at 7.5 ppm corresponds to the protons that are *ortho* to the SO<sub>3</sub>H group. Based on the spectrum, the degree of sulfonation (DS) was calculated to be 77%, using eq. (1).

$$DS(\%) = \frac{12 \times Area \ H(a')}{\sum_{n \neq a} Area \ H(n) - 9 \times Area \ H(a')} \times 100 \tag{1}$$

The ion-exchange capacity (IEC) is the number of equivalents of ion-exchange functional groups per 1 g of sample. As the IEC value increases, the electrical conductivity increases; but when the value is too high, the dimensional stability and mechanical strength of the sample decrease significantly, because of excessive water uptake. The IEC values of APEEK and SPEEK were determined to be 2.2 and 2.0 meq/g, respectively<sup>28</sup> (see the Supporting Information Section 3). The water uptakes of APEEK and SPEEK were estimated to be 31 and 27%, respectively (see the Supporting Information Section 4). Based on these results, both APEEK and SPEEK have appropriate IEC values and water uptakes for their use as binders of AC powders.

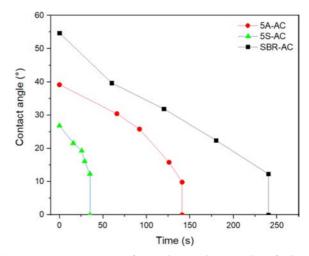


**Figure 4.** FE-SEM elemental mapping images of (a) 5A–AC (N: purple); (b) 5S–AC (S: green).

Characterization of Carbon Electrodes. Three different types of AC electrodes were prepared using APEEK, SPEEK, and SBR as polymer binders. The electrodes prepared using 3 wt% of the ion-exchange polymer (either APEEK or SPEEK) were not mechanically stable enough, because they easily lost their mass in water, indicating that the content of the ion-exchange binders was not high enough. However, when the content was 5 wt%, there was no apparent loss in the weights of the electrodes in water. This observation suggests that the polymer content of 5 wt% might be the appropriate amount for preparing the electrodes.

Figure 4 presents the elemental mapping results of 5A–AC and 5S–AC. In Figure 4(a) (5A–AC), purple dots are well dispersed over the entire surface, suggesting that trimethylammonium groups may be well distributed in the 5A–AC electrode. Similarly, Figure 4(b) shows that green dots are well dispersed over the surface, suggesting that sulfonic acid groups may be well distributed in the 5A–AC electrode. These observations reveal that the ion-exchange polymers (either APEEK or SPEEK) are well-mixed with the AC powders in the carbon electrode.

The wettability of the prepared electrodes was estimated, and the result is shown in Figure 5. The water on the SBR electrode showed the initial contact angle of 55°, but was completely absorbed into the electrode at 240 s. The reason for the slow but complete water absorption resulted from the presence of hydrophilic CMC (1 wt%). The water droplets on the 5A–AC and 5S–AC electrodes showed the initial contact angles of 39 and 27°, respectively, and were completely absorbed in 141 and 35 s, respectively. These observations indicate that these

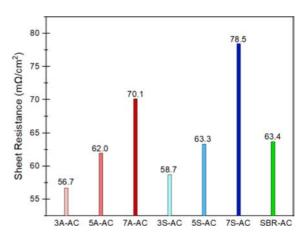


**Figure 5.** Water contact angles on SBR-AC, 5A-AC, and 5S-AC electrodes at room temperature.

ion-exchange polymers-based electrodes are much more hydrophilic than the SBR-AC electrode.

The electrical resistance of carbon electrode is an important characteristic for determining electrode performance. The electrical resistances of the prepared electrodes were measured, and Figure 6 shows the results. As the APEEK content increases from 3, to 5 to 7%, the surface electrical resistance of the A-AC series increases from 56.7, to 62.0 to 70.1 m $\Omega$ /cm<sup>2</sup>, while as the SPEEK content increases from 3, to 5 to 7%, the surface electrical resistance of the S-AC series increases from 58.7, to 63.3 to 78.5 m $\Omega$ /cm<sup>2</sup>. Obviously, as the binder content increased, the surface electrical resistance increased because the polymer binders are intrinsically insulators. Taking these results of the mechanical property and the electrical resistance into account, 5A-AC and 5S-AC were chosen as the electrodes for CDI cells, because the 5 wt% of the binders may be an appropriate content, as below the value, the electrodes are not mechanically stable, while above the value, the electrical resistance becomes too high. The SBR-AC electrode exhibited the surface electrical resistance of  $63.4 \,\mathrm{m}\Omega/\mathrm{cm}^2$ , even though the SBR content was as high as 10 wt%. In fact, such a high content of SBR binder is required to prepare the mechanically stable AC electrode. The lower resistance of SBR-AC is probably due to hydrophobic SBR that may bind more strongly, making the hydrophobic AC powders more closely packed. Consequently, the AC particles become better contacted, due to the absence of the sterically large and hydrophilic functional groups, such as the trimethylammoium methylene groups in the A-AC series, and sulfonic acid groups in the S-AC series.

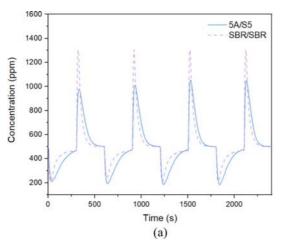
Desalination Performance. Figure 7(a) shows the results

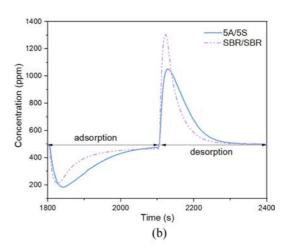


**Figure 6.** Sheet Resistances of the prepared electrodes of the A–AC series, S–AC series, and SBR–AC.

on the effluent NaCl concentration (ppm) *versus* operation (s) time during the four adsorption-desorption cycles. The fourth cycle is shown in Figure 7(b) for closer comparison between the prepared CDI cells. The 5A/5S CDI cell shows a lower minimum concentration than the SBR/SBR cell (182 *vs* 211 ppm), suggesting that the 5A/5S cell may be more efficient in adsorption than the SBR/SBR cell.

The SAC and CE are used as a standard metric for comparing the desalination performance of the CDI system. The SAC, which represents the adsorption amount of the CDI cell, is the mass of ions adsorbed on the carbon electrode divided by the mass of the carbon electrode. The CE represents the ratio between the total amount of charge supplied to the cell and the amount of ions adsorbed. The SAC of CDI cells was calculated using eq. (2).





**Figure 7.** Effluent concentration *versus* operation time of CDI cells: (a) during four adsorption-desorption (1.0 V/0.0 V) cycles; (b) the fourth cycle (feed concentration: 500 ppm, flow rate: 20 mL/min).

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$$SAC(mg/g) = \frac{(C_o - C_{avg}) \times V}{m}$$
 (2)

where,  $C_0$  is the initial NaCl concentration,  $C_{\text{avg}}$  is the average NaCl concentration of the effluent solution, V is the total volume of NaCl solution that passes through the CDI cell during the adsorption step, and m is the mass of the carbon electrodes.<sup>27</sup> The CE of the CDI cells can be calculated using eq. (3).

$$CE(\%) = \frac{(C_o - C_{avg}) \times V \times F}{\int I dt} \times 100$$
 (3)

where, F is Faraday's constant, and I is the supplied current. The SAC and CE of the 5A/5S cell were 10.1 mg/g and 79.8%, respectively, while those of the SBR/SBR cell were only 6.8 mg/g and 37.7%, respectively (Table 2). This result indicates that the 5A/5S cell is much more efficient than the SBR/SBR cell in the removal of NaCl from the solution. According to the water contact angle experiment, all the AC electrodes (5A-AC, 5S-AC, and 10SBR-AC) might be almost fully wetted under the desalination conditions, because before assembling the CDI cell, the electrodes were immersed in the feed solution for 1 day. In the 5A/5S cell, the cathode is covered with -SO<sub>3</sub> groups, while the anode is covered with -CH<sub>2</sub>N(CH<sub>3</sub>)<sub>3</sub><sup>+</sup> groups. Thus on adsorption, Na<sup>+</sup> and Cl<sup>-</sup> ions may move much faster toward the electrodes (because of additional attraction of the ions toward the binder, as well as attraction of counter-ions toward the charged electrodes), than those ions in the SBR/SBR cell, where the only electrostatic attraction operates between counter-ions and the charged electrodes. Furthermore, in the 5A/5S cell, much less co-ions could stay in the electrode pores, due to repulsion between the co-ions and the functional groups of the polymer binders. These considerations led us to speculate that the prepared polymer binders may serve partly as ion-exchange membranes used in the

As mentioned in the introduction, the CDI cell assembled using the crosslinked PVA binder-based AC electrodes showed the SAC of 9.96 mg/g, but direct comparison between our ion-exchange polymers and the crosslinked PVA is not possible,

Table 2. SAC and CE of the CDI Cells Assembled Using Carbon Electrodes Prepared from the SBR and Ion-exchange Polymers as Binder

CDI cell	SBR/SBR	5A/5S
SAC (mg/g)	6.8	10.1
CE (%)	37.7	79.8

because the experimental conditions are different (adsorption conditions: 1.2 V and 200 ppm NaCl vs 1.0 V and 500 ppm NaCl).<sup>23</sup> The degree of crosslinking of PVA binder in the AC electrode should depend on the uniformity of the binder dispersion throughout the AC powders, and the results may not be consistent. Our strategy also has an advantage over the chemical modification of AC itself, because the polymer binders can be readily prepared in large quantities, but very small amounts of them (5 wt%) are used in preparing AC electrodes, and thus the overall process of the preparation of CDI cells is much less costly, compared to the strategy where the whole mass of AC electrodes should be synthesized via complicated procedures.

#### Conclusions

Anion- and cation-exchange polymer binders (APEEK, SPEEK) were successfully prepared from PEEK. The degrees of amination of APEEK and sulfonation of SPEEK were 90 and 77%, and their IEC were 1.22 and 1.20 meg/g, respectively. The water uptakes of APEEK and SPEEK were 31 and 27%, respectively, which with respect to dimensional stability are within the usable range. The surfaces of 5A-AC and 5S-AC were more easily wetted than that of the SBR-AC, but the SBR-AC electrode also completely absorbed a water droplet (141 s and 35 s vs 240 s), due to the presence of hydrophilic CMC. The 5A-AC and 5S-AC electrodes exhibited the surface electrical resistances that are close to that of SBR-AC. The 5A/5S CDI cell showed much higher SAC and CE (10.1 mg/g and 79.8%), compared to the SBR/SBR cell (6.8 mg/g and 37.7%). The largely improved performance of the 5A/5S cell is due to the polymer binders that can act partly as ionexchange membranes of MCDI cells. This research clearly reveals that ion-exchange polymers can be used as a binder of AC electrodes to improve CDI performance.

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Supporting Information: Information is available regarding the experimental procedures for the preparation and measurement of IEC/water uptake of APEEK and SPEEK. The materials are available *via* the Internet at http://journal.polymer-korea.or.kr.

MCDI cells.

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