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Improvement of desalination efficiency in capacitive deionization using a carbon electrode coated with an ion-exchange polymer

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ABSTRACT

A composite carbon electrode coated with a cation-exchange polymer, crosslinked poly(vinyl alcohol) with sulfosuccinic acid, was fabricated to enhance the desalination performance of a capacitive deionization (CDI) system. The electrochemical properties of the prepared electrode were characterized by impedance spectroscopy, and desalination experiments were carried out at various operating conditions using a CDI cell with carbon electrodes only, and a membrane-capacitive-deionization (MCDI) cell including a coated-carbon electrode, to evaluate the effect of the coated-carbon electrode on desalination performance. The electrical resistance of the coated electrode was increased by a small amount over the uncoated electrode, but the capacitance was improved by the coating. In the CDI cell, the salt-removal efficiencies were in the range of 50–67%, while the efficiencies increased to 75–85% for the MCDI cell. Depending on the operating conditions, the salt-removal and current efficiencies of the MCDI cell were enhanced by 27–56% and 69–95%, respectively, compared to the CDI cell. The enhanced efficiency for the MCDI cell was attributed to the selective transport of cations between the electrode surface and bulk solution due to the cation-exchange coating layer.

1. Introduction

The capacitive deionization (CDI) process is defined as a potential-driven adsorption of ions onto a charged electrode surface. When an electric potential is applied to the electrode, charged ions migrate to the electrode and are held in the electric double layer (EDL); once the potential is removed, adsorbed ions are quickly released back into the bulk solution (Ying et al., 2002; Xu et al., 2008; Zou et al., 2008a,b).

CDI technology has attracted interest as a desalination technology since the 1960s, when the concept of CDI was first introduced by Murphy and Caudle (1967). Later, Johnson and Newman (1971) developed a porous-electrode model for ionic adsorption on porous carbon. In recent years, interest in CDI technology has increased significantly because it is regarded as a promising method which exhibits several advantages over conventional desalination processes, e.g., evaporation, reverse osmosis, electrodialysis, or ion exchange. It is an energy-efficient desalination process because it operates at a lower electrode potential at which no electrolysis reactions occur. In addition, this process is environmentally friendly because it requires no chemicals (Park et al., 2007; Gabelich et al., 2002; Li et al., 2009). Because of these benefits, CDI is regarded as an emerging desalination technology.

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The basic operating principle of CDI is similar to the electric double-layer capacitor (EDLC); both CDI and the EDLC use adsorption and desorption processes in an EDL. In EDLCs, the fluid electrolyte is held static, whereas the brine solution flows between the electrodes in a CDI process. Thus, a CDI cell acts as a flow-through capacitor (Lee et al., 2006).

Many studies have been conducted with the goal of increasing the performance of the CDI process, especially by exploring ways to increase the adsorption capacity of the electrodes. Porous carbon materials constitute very attractive electrodes for CDI processes because of their high specific surface areas and good electrical conductivities (Oren, 2008). Previous research has demonstrated that the efficiency of CDI strongly depends upon the surface properties of the carbon electrodes, such as surface area, pore microstructure, and chemical functional groups (Gabelich et al., 2002; Oh, et al., 2006; Zou et al., 2008a,b; Lee et al., 2009). Many kinds of carbon materials are being investigated as CDI electrodes, such as carbon aerogel, carbon cloth, and carbon nanotubes (Xu et al., 2008; Gao et al., 2009; Oh et al., 2006).

The salt-removal capability of a CDI system may exhibit large differences, even with the same electrode, depending on the adsorption and desorption mechanism in the EDL. During conventional CDI, when an electric potential is applied to the electrodes, ions are adsorbed from the solution into the EDL at the electrode surfaces due to the electrostatic force. Conventional CDI is known to be energy inefficient because of the dissolved salt present in the pore volume of the carbon electrode (Andelman, 2002). When an electric potential is applied, counter-ions in the pore adsorb onto the electrode and co-ions are expelled from the electrodes. This means that ion adsorption and desorption occur simultaneously in the pore volume in the electrode, seriously reducing desalination efficiency. To solve this problem, Andelman (2002) suggested a charge barrier placed adjacent to an electrode of a flow-through capacitor. More recently, a novel membrane-capacitive-deionization (MCDI) device, in which ion-exchange membranes were added to a CDI system, has been developed by Lee et al. (2006). In their work, the salt-removal rate of the MCDI system was 19% higher than that of the CDI system. In a similar work performed by Li et al. (2008), the authors constructed an MCDI device using carbon nanotube and nanofiber electrodes and ion-exchange membranes. They showed that salt removal by the MCDI system was about 50% higher than that by the CDI system. In these works, it was verified that an MCDI system can increase desalination efficiency significantly compared to the CDI system.

Cation-exchange and anion-exchange membranes are selectively permeable to cations and anions, respectively, and are widely used in electro-membrane processes, but the price of commercial ion-exchange membranes remains prohibitively high for CDI applications. Furthermore, when constructing an MCDI cell, ensuring that the membrane and the electrode are in good contact can be problematic.

To overcome these problems, we fabricated a carbon electrode directly coated with an ion-exchange polymer for this study. A mixture of poly(vinyl alcohol) (PVA) and sulfosuccinic acid (SSA) was used as the ion-exchange polymer. The electrochemical properties of the prepared electrodes were analyzed by electrical impedance spectroscopy; we then constructed CDI cells using these electrodes. Several desalination experiments were conducted at various operating conditions, and the conductivities of the effluents and the currents passed through the cells were accurately measured by potentiostat to investigate the performance of the prepared electrodes.

2. Materials and methods

2.1. Fabrication of carbon electrodes

A porous carbon electrode was prepared using activated carbon powder (P-60, Daedong AC Corp., specific surface area = 1260 m^2/g). A carbon slurry was prepared as a suspension of activated carbon powder and poly(vinylidene fluoride) (PVdF, M.W. = 275,000, Aldrich) in di-methylacetamide (DMAc, Aldrich). The PVdF polymer content of the dried carbon electrode was 10 wt%. The mixture was stirred for 12 h to ensure homogeneity. The slurry was then cast onto a graphite sheet (F02511, Dongbang Carbon Corp., Korea) to a thickness of 300 μm. The coated electrode was dried at 50 °C in an oven for 2 h and then in a vacuum oven at 50 °C for 2 h to remove all traces of organic solvent remaining in the micropores of the carbon electrode.

2.2. Coating the carbon electrode with cation-exchange polymer

Following the works of Rhim et al. (2004), we prepared a mixture of poly(vinyl alcohol) (PVA, molecular weight 89,000–98,000, Aldrich) and sulfosuccinic acid (SSA, Aldrich) as a cation-exchange polymer solution. The SSA acted as a crosslinking agent and provided ion-exchange functional groups. To the 10 wt% PVA solution was added 30% SSA by weight and the mixture was vigorously stirred at room temperature for 24 h, after which the polymer solution was cast onto the prepared carbon electrode. The cast electrodes were dried at 60 °C in an oven for 1 h and then heated for 1 h in a thermostatic oven at temperature of 130 °C. The reaction mechanism of PVA and SSA has been described in detail by Rhim et al. (2004).

2.3. Characterization of the carbon electrodes

The surface morphologies of the prepared electrodes were investigated using a scanning electron microscope (SEM, MIRA LMH, TESCAN Ltd.). The sample surfaces were covered with gold using the ion-sputtering method and SEM images were obtained.

To examine the electrochemical properties of the prepared electrodes, electrical impedance spectroscopy (EIS) measurements were made using a three-electrode system (Pröbstle et al., 2003). The carbon electrode specimen, with an exposed surface area of 1.77 cm^2, was inserted into the specimen holder. A platinum rod and a saturated Ag/AgCl electrode were used as the counter- and reference electrodes, respectively. Impedance measurements were performed with an AutoLab PGST30 potentiostat combined with an FRA impedance

analyzer. The impedance spectra were obtained at a potential of 0.0 V in the frequency range of 100 Hz to 20 mHz. An alternating sinusoidal signal of 25 mV peak-to-peak was superimposed on the DC potential. The electrolyte solution used was 0.5 M KCl. All experiments were maintained at 25 ± 0.1 °C in a water bath.

2.4. Desalination experiments using the capacitive deionization unit cells

To compare the performance of the coated-carbon electrode with the uncoated one, two unit cells were constructed. The first was constructed with uncoated-carbon electrodes only (denoted as the CDI cell), and the second with one uncoated and one coated-carbon electrode (denoted as the MCDI cell). In the MCDI cell, the coated-carbon electrode acted as the cathode because the film coating is a cation-exchange layer, which is only permeable to cations.

Each unit cell consisted of two parallel electrode sheets separated by a non-electrically conductive spacer (nylon cloth, 100 μm thick). This prevented an electrical short and allowed liquid to flow. The size of the electrodes was 100 × 100 mm. Graphite sheets were used as inert current collectors on the back sides of the electrodes. A Plexiglas plate was used to assemble the upper and lower parts of the unit cell. A flow channel was created by punching a 1-cm-diameter hole in the center of the electrode so that the solution could be in contact with all sides of the electrode and could run through the spacer to the central hole.

Electrosorption experiments for the two unit cells (CDI and MCDI) were carried out in a flow-through system, depicted in Fig. 1. The system consisted of a reservoir, a peristaltic pump, a unit cell, and a conductivity meter. A NaCl solution (200 mg/L) was supplied to the cell using a peristaltic pump. A given potential was applied to the CDI cell using a potentiostat (WPG100, WonA Tech Corp.). We first conducted an adsorption test while applying a pre-established potential for 180 s, followed immediately afterward by a desorption test for 120 s by changing the cell potential to 0 V. The conductivity of the effluent water was measured by connecting a conductivity meter at the position where the solution exited the cell. The conductivity was automatically measured at 1.0-s intervals by connecting the conductivity meter to a data-acquisition system (midi Logger GL200, GraphTech). The CDI tests were conducted at potentials of 1.2 and 1.5 V to compare the salt-removal efficiency at these values. The effects of changes in the flowrate were examined by supplying 20 or 30 mL/min of solution at 1.5 V.

3. Results and discussion

3.1. Morphologies of the carbon electrodes

Fig. 2 shows the surface morphologies of the carbon electrodes, both uncoated (a), and coated with PVA/SSA polymer solution (b). Although the electrode slurry was cast at 300 μm thick, the thickness of the dried carbon electrode was about 140 μm, reduced by more than half during the drying process. As can be seen in Fig. 2(a), activated carbon powders were covered with PVdF polymer binder, and they bound together well. When the electrode surface was rubbed by hand, carbon powder was not removed from the electrode. Thus, we confirmed that the prepared carbon electrodes had sufficient mechanical strength.

From Fig. 2(b) we can see that the PVA/SSA ion-exchange layer was cast uniformly, and the thickness of the polymer layer was about 10 μm. The ion-exchange polymer was well coated on top of the carbon electrode. In addition, the coated-carbon electrode showed much higher mechanical strength due to the film coating on the electrode surface. From the observation of the SEM images we can conclude that a composite carbon electrode integrating both the advantages of the high capacitance of a carbon electrode and the perm-selectivity of an ion-exchange membrane was successfully produced.

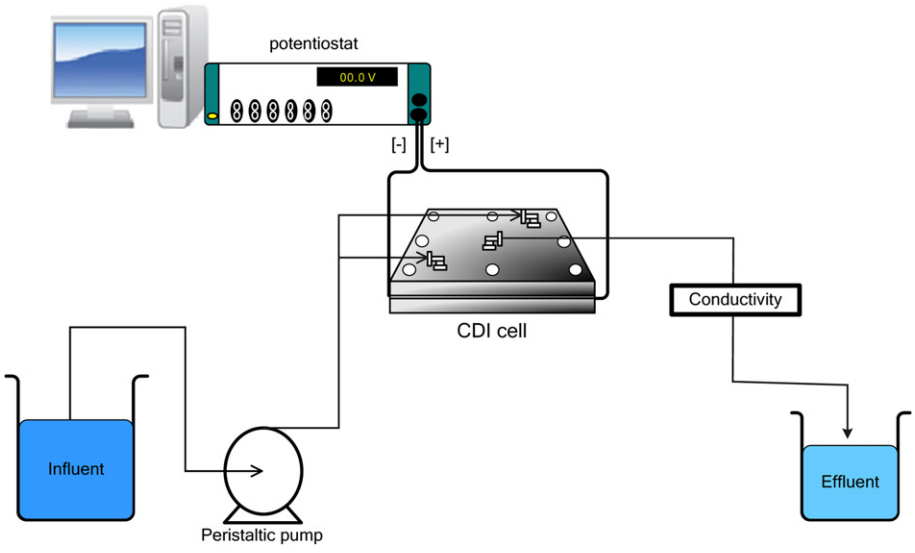

Fig. 1 – Schematic diagram of capacitive deionization experiment setup.

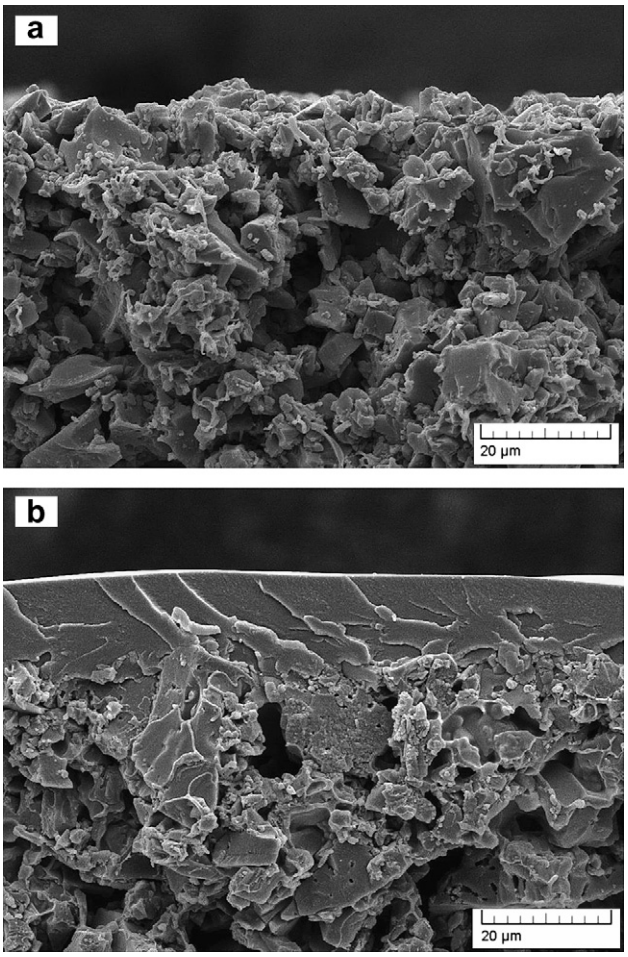

Fig. 2 – The SEM images of carbon electrodes; (a) uncoated and (b) coated with ion-exchange polymer.

3.2. *Electrochemical properties of the prepared electrodes*

In order to analyze the electrochemical properties of the prepared electrodes, electrochemical impedance spectroscopy measurements were made using a frequency-response analyzer (Pröbstle et al., 2003). The impedance value obtained at high frequency (100 Hz) indicates the equivalent series resistance (ESR) composed of the electrode resistance and bulk electrolyte resistance (Yoon et al., 2000). The ESRs for the uncoated and the coated electrodes were 1.30 and 1.66 Ω, respectively. Because the measurements were conducted in the same bulk solution, we could determine the resistance of the coating layer from the difference of the ESR values; the resistance was calculated to be 0.64 Ω cm^2. The resistance of the coating layer is very low compared to commercial ion-exchange membranes. In general, the resistance of commercial ion-exchange membranes are in the range of 1.5–3.0 Ω cm^2 (Strathmann, 2004).

The specific capacitance (C) of a carbon electrode can be derived from the imaginary part (Z″) of the impedance spectrum according to (Pröbstle et al., 2002):

$$C = \left|\frac{1}{\omega Z''}\right| \quad (1)$$

where ω denotes the angular frequency of the applied AC signal. The capacitances of the uncoated and coated-carbon electrodes were determined to be 0.63 and 0.74 F/cm^2, respectively, at 20 mHz, which is a 17.5% increase in capacitance due to the ion-exchange polymer coating. According to the electric double-layer theory, the capacitance of an electrode is proportional to the square root of the electrolyte concentration (Conway, 1999). Activated carbon powders in the coated-carbon electrode were in contact with a cation-exchange polymer in which the fixed ion concentration is higher than that of the bulk solution. This was thought to result in the increase in capacitance of the coated-carbon electrode.

3.3. *Desalination performances of the CDI and MCDI cells*

Desalination experiments for the CDI and MCDI cells were performed by supplying a water solution of 200 mg/L of NaCl at a flowrate of 20 mL/min. After applying a cell potential of 1.2 V for 180 s, the cell potential was immediately changed to 0.0 V for 120 s. Fig. 3 shows the conductivity transients of the effluent for each cell. The conductivity changes show reproducible results for five cycles of sorption and desorption.

As can be seen in Fig. 3, desalination occurred more efficiently with the MCDI cell than with the CDI cell. When the cell potential was applied, ions were quickly adsorbed, so that the conductivities decreased from the initial 420 to the minima of 109 μS/cm and 29 μS/cm for the CDI and MCDI cells, respectively. The average conductivities of the effluent during the sorption period were 193.5 and 73.4 μS/cm for the CDI and MCDI cell, respectively. Notably, a significant difference in the initial conductivity changes was observed for the two cells. The rate of conductivity decrease for the MCDI cell was greater than for the CDI cell. In addition, the conductivities for the MCDI cell increased more slowly after the minimum point than those for the CDI cell during the sorption period. These results demonstrate that the desalination efficiency can be enhanced by using a carbon electrode coated with an ion-exchange polymer.

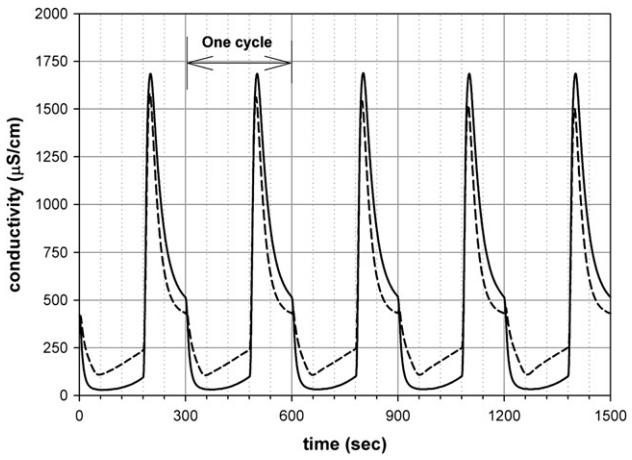

Fig. 3 – The conductivity transient of effluent measured at a cell potential of 1.2 V with a flowrate of 20 mL/min. Solid line: MCDI cell, dashed line: CDI cell.

Regarding the regeneration performance, when the desorption potential (0.0 V) was applied the conductivities increased steeply, showing maximum values of 1580 and 1685 μS/cm for the CDI and MCDI cells, respectively, which is around a four-fold concentration increase of the influent. Most ions were desorbed within 60 s after the application of the desorption potential. Because desorption proceeded rapidly, it was easy to regenerate the electrode during the process operation, meaning that it should be possible to increase the recovery ratio of the feed solution by reducing the desorption time.

The higher desalination efficiency of the MCDI cell compared to the conventional CDI cell could be explained through the adsorption and desorption mechanism of ions at the electrode surface. When the cell potential was applied to the electrode, cations were electrosorbed onto the cathode surface, forming an EDL. When the potential was removed, the cations adsorbed in the EDL moved to the bulk solution to establish electroneutrality, which led to the desorption of ions. However, some anions could move from bulk solution to the anode surface by electrostatic force. Accordingly, some cations adsorbed at the electrode surface could not escape and were held in the carbon electrode. Because these accumulated ions would then be readsorbed at the electrode surface when a potential was again applied, the desalination efficiency would be decreased in the CDI cell. On the contrary, in the MCDI cell only cations in the EDL could penetrate through the cation-exchange layer during the desorption process, but anions in the bulk solution were rejected by the ion-selective layer. As a consequence, desalination efficiency was improved because of the selective transport of ions between the EDL and the bulk solutions.

To confirm the selectivity of the cation-exchange layer in the MCDI cell, desalination experiments were carried out at the cell potential of −1.5 V, and the results are depicted in Fig. 4. When applying a negative potential in the MCDI cell, the coated-carbon electrode acts as the anode and anions in the bulk solution will be adsorbed. As can be seen in Fig. 4, however, few ions were adsorbed and desorbed for the MCDI cell due to anion rejection by the cation-exchange layer of the coated-carbon electrode. From these results, it was verified that the cation-exchange layer coating on the electrode functioned well to transport ions selectively. In contrast, the conductivity changes for the CDI cell showed results similar to those with the cell potential at 1.5 V. In the CDI cell, a positive or negative potential does not affect the desalination efficiency because the electrodes had no ion selectivity.

3.4. Salt-removal efficiencies at various operating conditions

In order to compare the salt-removal efficiencies related to the operating conditions for both cells, electrosorption experiments were carried out at cell potentials of 1.2 and 1.5 V, and flowrates of 20 and 30 mL/min. The average conductivities of the effluents were determined to calculate the amount of salt removed for each set of operating conditions. The NaCl concentration was determined from the linear relationship between NaCl concentration and conductivity. The salt-removal efficiency, η_d, was calculated according to the following equation (Lee et al., 2006):

$$\eta_d(\%) = \left(1 - \frac{C_{eff}}{C_o}\right) \times 100 \quad (2)$$

where C_o and C_{eff} are the influent and average effluent NaCl concentrations, respectively.

Fig. 5 shows the salt-removal efficiencies obtained at various operating conditions. Compared with the CDI cell, the MCDI cell exhibited a much higher removal efficiency. For the case of a 1.5-V cell potential with a flowrate of 20 mL/min, the salt-removal efficiencies of the CDI and MCDI cells were 67 and 85%, respectively, about a 27% improvement in efficiency for the MCDI cell over that of the CDI cell. A maximum salt removal of 75% was recorded for the flowrate of 30 mL/min in the MCDI cell configuration. The salt-removal efficiency of the MCDI cell was therefore enhanced by 27–56% compared to the CDI cell, depending on the operating conditions.

The enhanced salt-removal efficiency in the MCDI cell was attributed to the selectivity of the cation-exchange layer on the carbon electrode. If this is true, significant differences in

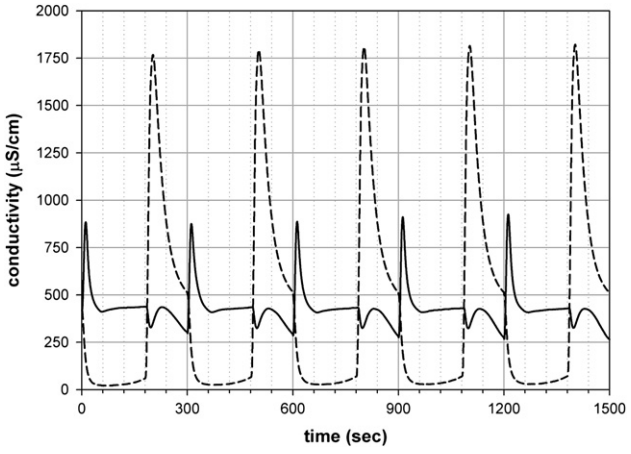

Fig. 4 – The conductivity transient of effluent measured at a cell potential of −1.5 V with a flowrate of 20 mL/min. Solid line: MCDI cell, dashed line: CDI cell.

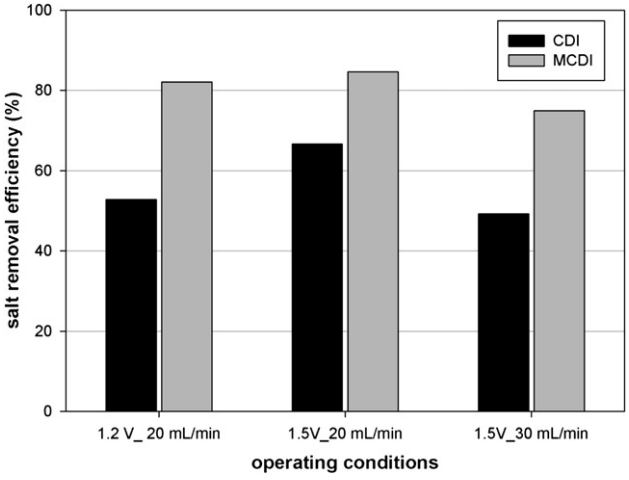

Fig. 5 – Salt-removal efficiencies for the CDI and MCDI cells at various operating conditions.

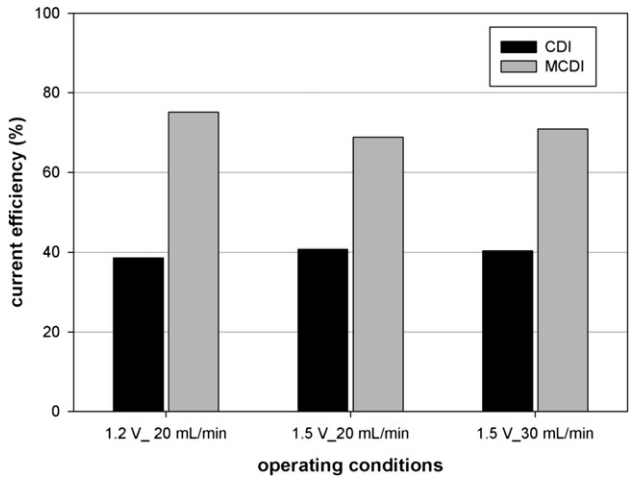

Fig. 6 – Current efficiencies for the CDI and MCDI cells at various operating conditions.

current efficiencies for the CDI and the MCDI cell operation should be observed. Current efficiency ($\eta_{current}$) is defined as the ratio of ions adsorbed to the current passed through the cell, and is determined by the following equation (Choi, 2002):

$$\eta_{current}(\%) = \frac{(C_o - C_{eff}) \cdot V \cdot F}{\int I \, dt} \times 100 \quad (3)$$

where V is the total volume of the effluent flown through the cell in the adsorption period, F is Faraday's constant and I is the current supplied.

When the desalination experiments were carried out at a potential of 1.5 V and a 20 mL/min flowrate, the total charges supplied to the cell during the adsorption period were measured as 32.6 and 24.4 coulombs for the CDI and MCDI cells, respectively. From the average NaCl concentrations of the effluent during the sorption period, we found that 8.1 and 10.2 mg of NaCl were adsorbed, indicating current efficiencies of 41 and 69% for the CDI and MCDI cell operations, respectively.

Fig. 6 shows the current efficiencies obtained at various operating conditions for the CDI and MCDI cells. In the CDI cell the current efficiencies were in the range of 39–41%, while the efficiencies increased to 69–75% for the MCDI cell. The current efficiencies of the MCDI cell were enhanced by about 69–95% compared to the CDI cell, depending on the operating conditions.

4. Conclusions

- In order to improve the desalination efficiency of capacitive deionization, we fabricated a carbon electrode coated with an ion-exchange polymer. The mixture of PVA and SSA was coated onto the carbon electrode and then heated to crosslink PVA with SSA. From the SEM images, we confirmed that a composite carbon electrode, integrating the advantages of both the high capacitance of the carbon electrode and an ion-exchange membrane, was successfully produced.
- Electrochemical properties of the prepared electrodes were studied by impedance spectroscopy. Although the electrical resistance of the coated electrode increased somewhat, the charging characteristics were improved by coating the surface with the ion-exchange polymer.
- Desalination trials were carried out using the CDI cell with carbon electrodes only and the MCDI cell equipped with one coated-carbon electrode (the cathode, in normal operation). The salt-removal efficiencies of the MCDI cell were enhanced by about 27–56% compared to the CDI cell, depending on the operating conditions.
- The current efficiencies of the CDI and the MCDI cell operation showed a significant difference depending on the operating conditions: 39–41% for the CDI cell and 69–75% for the MCDI cell. The enhanced efficiency for the MCDI cell was attributed to the selective transport of ions between the electrode surface and bulk solutions due to the ion-exchange layer.

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