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Ion-selective composite carbon electrode coated with TiO_2 nanoparticles for the application of electrosorption process

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ABSTRACT

Composite carbon electrodes with ion selectivity were fabricated using mixtures of sulfonated polystyrene and Titanium dioxide (TiO_2) nanoparticles that were coated onto carbon electrodes. After composite carbon electrodes with various TiO_2 contents were coated in the coating solution in the range of 0–20 wt%, the scanning electron microscopy, cyclic voltammetry (CV), and electrical impedance spectroscopy (EIS) were performed. In addition, the desalination performance was evaluated through the use of a capacitive deionization (CDI) unit cell. The CV and EIS analyses of the composite carbon electrodes showed that the electrical resistances of the coating layers decreased significantly as the TiO_2 content increased. In contrast, the ion selectivity decreased as the TiO_2 content increased because of the pores formed among the particles. In this study, the optimal content of TiO_2 in the composite carbon electrodes in terms of electrical resistance and ion selectivity was found to be approximately 10 wt%. In addition, the desalination experiments confirmed that the desalination efficiency of the composite carbon electrodes was improved by approximately 30% over that of unmodified carbon electrodes. The composite carbon electrodes fabricated in this study can be used effectively in the CDI process.

Keywords: Composite carbon electrode; TiO_2 nanoparticle; Capacitive deionization; Ion selectivity; Electrical resistance

1. Introduction

The desalination technologies used to separate ionic species include the evaporation, ion-exchange, reverse osmosis, and electrodialysis methods. The efficacy of these processes has already been technically proven and they are currently being used in various commercial processes. However, current desalination technologies have several disadvantages, including high operational and maintenance costs and the release of secondary pollutants during the operation [1–4]. Hence, a strong demand exists for new desalination technologies that address these issues.

Recently, studies of capacitive deionization (CDI) technology as an approach to desalinization that overcomes the problems of conventional desalination technologies have gained attention as interest in energy costs and environmental protection has grown [4–8]. CDI technology removes ions through adsorption by electrostatic force at the electric double layer formed on the electrode surface. One characteristic of CDI

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technology is its ability to drastically reduce energy consumption because it operates at a low electric potential and can reutilize the charges acquired during adsorption [2,4]. CDI also has the advantage of easy operation because adsorption and desorption are possible with the adjustment of the electrode potential. As a result, CDI is known as an environmentally friendly desalination process because it does not release secondary pollutants [9–12].

CDI technology uses carbon electrodes with high specific surface area and a high electric conductivity because it involves adsorption at the electrode surface. However, this technology has the drawback that the charge efficiency is greatly reduced because of the porous structure of carbon electrode [13,14].

Andelman reported that the desalination efficiency of the CDI process can be enhanced by the coupling of a "charge barrier" to carbon electrodes [15]. Lee et al. [16] reported a 50% increase in the desalination efficiency compared to the current CDI process when the membrane capacitive deionization (MCDI) technology, which involves the combination of carbon electrodes and an ion-exchange membrane, was applied.

The MCDI process can improve desalination efficiency; however, the cost of the device is prohibitively high because it requires the use of an expensive ion-exchange membrane. In addition, contact resistance can occur at the interface of the ion-exchange membrane and the carbon electrodes. To resolve these issues, ion-selective composite carbon electrodes (ISCCEs) were fabricated with an ion-exchange polymer coated onto the surface of the carbon electrodes [14]. In ISCCEs, the ion-exchange membrane and the carbon electrodes are combined into one structure and are considered to deliver economical desalination performance.

The CDI process operates at a low electric potential (approximately 1.2 V) at which electrode reactions do not occur at the electrode surface [11,17–19]. Therefore, reducing the electrical resistance of the electrodes is important to improve the electrosorption capacity and the electrosorption rate. In ISCCEs coated with an ion-exchange polymer, in particular, most of the electrode resistance is caused by the coating layer [14]. As a result, the reduction of the electrical resistance of the coating layer while maintaining the ion selectivity of the electrodes is important for improving the performance of the ISCCEs.

In this study, ISCCEs were fabricated by coating a mixture of metal-oxide nanoparticles and ion-exchange polymers onto a carbon electrode surface. The metal oxides can be effective at maintaining the ion selectivity of the electrodes because their surfaces are charged. In addition, the electrical resistance of the coating layer is thought to be reduced by the formation of miscellaneous pores among the nanoparticles when the nanoparticles are added to the coating layer. The ISCCEs were fabricated by mixing Titanium dioxide (TiO_2) nanoparticles and sulfonated polystyrene (SPS) and coating the carbon electrodes with the mixture. The ISCCEs were fabricated with various amounts of TiO_2, and the changes in the properties of the electrodes were analyzed as a function of the metal-oxide content. The surface structure, capacitance, ion selectivity, and the change in the electrical resistance of the fabricated electrodes were measured using scanning electron microscopy (SEM), cyclic voltammetry (CV), and electrical impedance spectroscopy (EIS). The desalination performance was confirmed by the fabrication of a CDI unit cell using the fabricated ISCCE.

2. Experimental

2.1. Preparation of SPS

SPS, a cation-exchange polymer, was prepared for the fabrication of composite carbon electrodes that can exhibit cation selectivity. Polystyrene (PS, Aldrich, M.W. 350,000) was dissolved in 1,2-dichloroethane (DCE, Samchun) in a four-necked flask and then, sulfuric acid was added to induce sulfonation. The sulfonation reaction was conducted for 90 min during which the reaction temperature was maintained at 60°C. After the reaction was completed, the reaction mixture was gradually added to methanol to recover the solidified SPS. The SPS was washed with distilled water several times to remove the remaining sulfuric acid and was then thoroughly dried for 12 h at 50°C in a vacuum oven. The ion-exchange capacity of the prepared SPS was measured to be 1.62 meq/g.

2.2. Fabrication of ISCCEs

2.2.1. Fabrication of carbon electrodes

An electrode slurry was prepared by mixing activated carbon powder (P-60, Daedong AC) and polyvinylidene fluoride (PVdF, Aldrich, M.W. = 530,000) as a polymer binder in an organic solvent, *N*,*N*-dimethylacetamide (DMAc, Aldrich). The electrode slurry was made uniform by blending the mixtures vigorously in a stirrer for 12 h. Carbon electrodes were fabricated by drying at 50°C in a convection oven for 6 h after the electrode slurry was cast onto graphite foil (F02511, Dongbang Carbon) using a doctor blade. The polymer binder (PVdF) content in the fabricated carbon electrode was 10 wt%.

2.2.2. Ion-exchange polymer coating

The coating solution was prepared by mixing TiO_2 and SPS. The coating solution was prepared with various contents of TiO_2 (nano powder, Aldrich) to examine the change in the properties of the ISCCEs as a function of the metal-oxide content. The coating solution was made uniform by stirring the solution vigorously for 24 h after the SPS and TiO_2 were mixed, so that the content ratio of TiO_2 was 0, 5, 10, 15, or 20 wt%. The ISCCEs were prepared by coating the solution onto the fabricated carbon electrode.

2.3. Analysis of the physical and electrochemical properties of ISCCE

The physical structure of the fabricated composite carbon electrodes was examined by SEM (MIRA LMH, TESCAN) analysis. On the basis of the cross-sectional analysis of the carbon electrodes, the thickness of the coating layer of the carbon electrode and the coupling between the polymer and carbon electrode were confirmed. In addition, the change in the miscellaneous pores on the surface of the coating layer as a function of the TiO_2 content was examined.

The electrochemical properties of the fabricated electrodes were analyzed with EIS and CV using a three-electrode system. The fabricated ISCCE (diameter 1.5 cm) was fixed as the working electrode; a Ag/AgCl electrode was used as the reference electrode; and a platinum electrode was used as the counter electrode. CV and EIS analyses were performed using a potentiostat (PGSTAT30) from AutoLab. A 0.5 M KCl solution was used as the electrolyte.

CV was measured in a potential range of –0.5 to +0.5 V at a potential scan rate of 5.0 mV/s. In addition, EIS was measured using a frequency response analyzer coupled to the potentiostat. The impedance was measured at each frequency in the range from 100 Hz to 20 mHz. The AC amplitude used in the EIS measurement was 25 mV and the potential was 0.0 V.

2.4. The CDI experiment

The CDI unit cell was made using the fabricated electrodes, and the desalination performance was evaluated. Desalination experiments were performed with fabricated electrodes cut into $10 \times 10\,cm^2$ sections. The desalination efficiency of the cell composed of uncoated carbon electrodes was compared to that of the cell composed of an ISCCE (TiO_2 content 10 wt%) as the cathode and a carbon electrode as the anode; all desalination experiments were performed under the same conditions.

Desalination was performed by charging the CDI unit cell at a potential of 1.2 V for 3 min using a potentiostat (WPG100, WonA Tech) and the adsorbed ions were subsequently desorbed by immediately changing the cell potential to 0.0 V. The desalination experiment was performed by uniformly supplying a 200 mg/L NaCl solution at a rate of 20 mL/min. To measure the change in the conductivity of the effluent in real time, a conductivity meter was connected to the data collector (Vernier Labquest, Vernier). The composition of the CDI unit cell and the experimental procedure are described in detail elsewhere [14,19,20].

3. Results and discussion

3.1. SEM analysis of the ISCCEs

The structure of the miscellaneous pores of the coating layer was examined via SEM micrographs of the fabricated composite carbon electrode. The SEM images of the cross-section and the surface of the composite carbon electrodes with TiO_2 ratios of 0, 10, and 20 wt% are shown in Fig. 1. As shown in the figure, the thickness of the coating layer appears to be approximately 10 μm and the coating is uniformly deposited onto the surface of the carbon electrode. In particular, the coating solution appears to have coupled well with the carbon electrode as it solidified after penetrating into the pores on the surface of the carbon electrode. In the case of an MCDI, where the carbon electrode and the ion-exchange membrane are used separately, stacking of the carbon electrodes can be difficult. In addition, contact resistance between the carbon electrode and the ion-exchange membrane can occur, which can, in turn, lead to reduced adsorption performance of the electrode. However, the SEM images confirmed that the structure of the composite carbon electrode fabricated in this study can overcome these problems.

When TiO_2 was added to the coating solution, an important difference was observed in the surface structure of the composite carbon electrode. The coating surface did not appear smooth but rather was partially nonuniform for a composite carbon electrode (Fig. 1(a)) that is coated only with SPS without TiO_2. This lack of uniformity is due to the bubbles produced during the drying process of the coating solution. As bubbles inside the carbon electrode escape during drying after the application of the SPS coating solution, the bubbles form on the electrode surface due to the high viscosity of the SPS. As the drying progresses, these bubbles either burst or are hardened, which makes the surface nonuniform, as shown in the figure. In contrast, when TiO_2 nanoparticles were

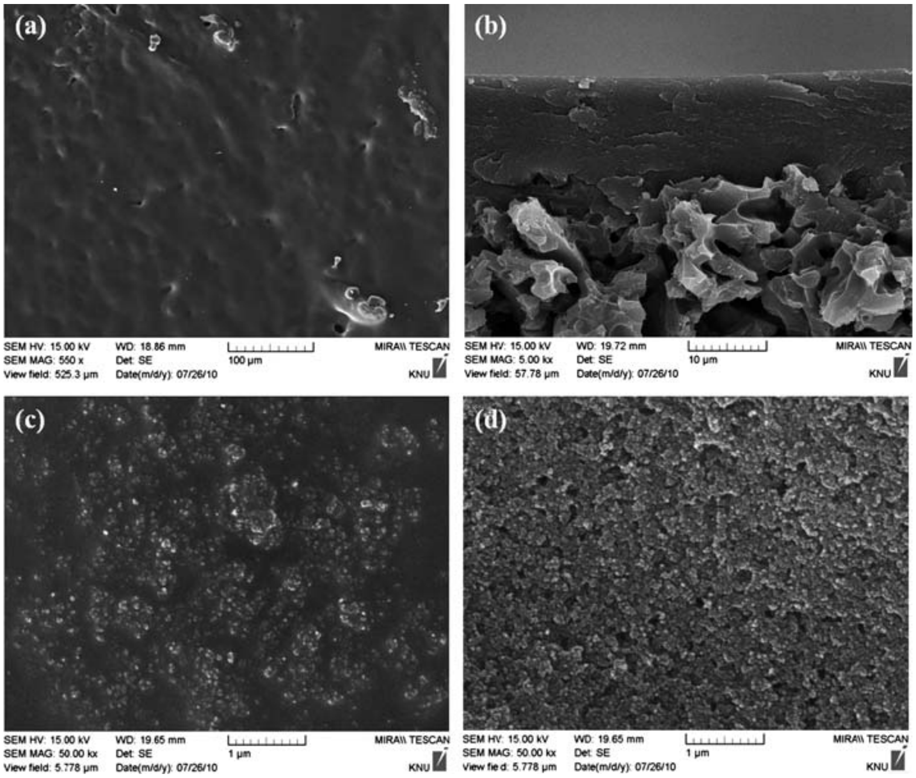

Fig. 1. SEM images of composite carbon electrodes coated with a mixture of TiO_2 nanoparticles and SPS. (a) TiO_2: 0 wt% (top), (b) TiO_2: 0 wt% (cross-section), (c) TiO_2: 10 wt%, and (d) TiO_2: 20 wt%.

added to the coating solution, no bubbles formed in the fabricated carbon electrode because the bubbles escaped through miscellaneous metal-oxide particles. As a result, a uniform coating layer was formed.

The size of the pores on the surface of the composite carbon electrode was observed to increase as the amount of metal oxide was increased. When the TiO_2 content was 10 wt% (Fig. 1(c)), SPS and TiO_2 formed a coating layer that was mixed uniformly without the formation of bubbles on the surface of the electrode. In contrast, when the metal-oxide content was 20 wt% (Fig. 1(d)), the size of the pores on the coating layer increased. The composite carbon electrode in this study was fabricated to show cation selectivity by coating SPS with a cation-exchange group. However, when the size of the pores increases as metal oxide is added, the ion selectivity of the electrode may decrease. Therefore, it is important to determine the optimal content of metal oxide that reduces the electrical resistance while maintaining the ion selectivity.

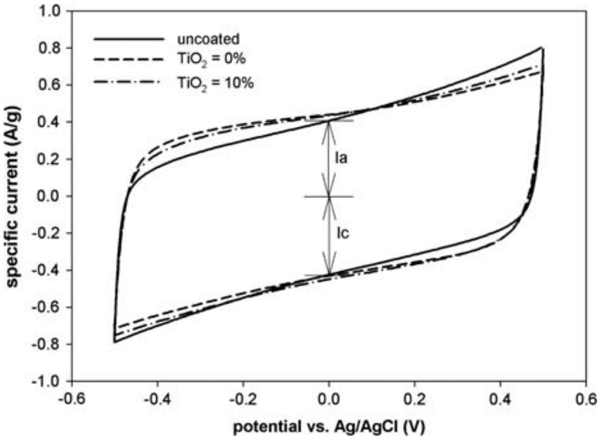

Fig. 2. Cyclic voltammograms for the carbon electrode and the composite carbon electrodes at a potential scan rate of 5.0 mV/s.

3.2. *The capacitance and ion selectivity of the composite carbon electrodes*

CV was used to analyze the capacitance and ion selectivity of the composite carbon electrodes. The current at each electric potential was measured by varying the potential in the range of –0.5 to +0.5 V (vs. Ag/AgCl) at a scan rate of 5 mV/s. Fig. 2 shows the cyclic voltammograms for an uncoated carbon electrode, a carbon electrode coated with only SPS

($TiO_2 = 0\%$), and a composite carbon electrode with a TiO_2 content of 10 wt% ($TiO_2 = 10\%$). For an ideal capacitor, the CV profile assumes a rectangular shape; however, as shown in Fig. 2, the CV profile of the carbon electrode fabricated in this study exhibited a slightly inclined rectangular shape. The nonideal shape of the voltammogram is thought to be either due to resistance of the carbon electrode or to an oxidation or reduction reaction at the electrode surface [21–23].

The specific capacitance of the carbon electrode can be calculated by dividing the current by the potential scan rate [24]:

$$C_{dl} = \frac{I_a - I_c}{2\left(\frac{dE}{dt}\right)} = \frac{\Delta I}{2\left(\frac{dE}{dt}\right)} \tag{1}$$

where I_a is the oxidation current (A/g), I_c is the reduction current (A/g), C_{dl} is the capacitance (F/g), and dE/dt is the potential scan rate (V/s). The capacitance according to the electrode potential was calculated by substituting the CV results for all of the carbon electrodes into Eq. (1); the results are shown in Fig. 3.

As shown in Fig. 3, a large difference in the capacitance was observed depending on the coating of the carbon electrode. The capacitance of the carbon electrode increased after the surface of the carbon electrode was coated. In the case of the uncoated carbon electrode, the capacitance was 86 F/g at –0.3 V but was 93 F/g when coated, which represents an 8% increase in the capacitance. In the case of the uncoated carbon electrode, the capacitance was the lowest in the range of –0.1 to 0.0 V and exhibited a symmetrical shape. The potential with the lowest capacitance is known as the potential of zero charge (PZC) of the corresponding electrode, i.e. the cations are adsorbed at potentials lower than the PZC and the anions are adsorbed at potentials higher than the PZC.

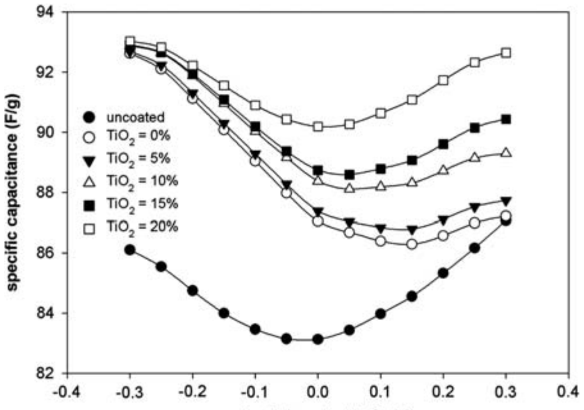

Fig. 3. The specific capacitances of various carbon electrodes in the potential range of –0.3 to +0.3 V vs. Ag/AgCl.

In contrast, in the case of the coated composite carbon electrode, the capacitance did not exhibit symmetry as a function of negative potential or positive potential. This electrode exhibited a tendency similar to that of the uncoated carbon electrode at negative potentials (below 0 V) because, when a negative potential is applied, cations can easily pass through the coating layer and be adsorbed onto the carbon electrode. However, in the positive potential range, the capacitance exhibited a constant value with no increase as a function of the electrode potential. This behavior can be explained by the inability of anions to pass through the coating layer when a positive potential is applied.

The ion selectivity of the electrodes can be interpreted from the change in the capacitance of the composite carbon electrode as a function of the electrode potential [24]. As was previously described, if the carbon electrode does not exhibit ion selectivity, then capacitance tends to increase according to electrode potential based on the PZC. However, if the carbon electrode exhibits cation selectivity, it will interfere with the anions being adsorbed in the positive potential range by the influence of cation-exchange groups in the coating layer. As such, the ion selectivity of the carbon electrodes can be analyzed from the change in capacitance as a function of the electrode potential.

As shown in Fig. 3, in the case of electrode coated with SPS on carbon electrode surface, excellent cation selectivity was observed. However, the ion selectivity decreased as the content of TiO_2 in the coating layer was increased. When the content of TiO_2 was 5 wt%, the change in the ion selectivity was similar to that of the electrode coated only with SPS. When the content of TiO_2 was greater than 10 wt%, the ion selectivity slightly decreased and when it was 20 wt%, the ion selectivity decreased significantly. The reason for the decreased ion selectivity with increased TiO_2 content is thought to be the pores formed on the coating layer.

3.3. The electrical resistance of the ISCCEs according to metal-oxide content

CDI technology operates at low electric potentials, at which no oxidation or reduction reaction occurs at the electrode. Therefore, the electrical resistance of the electrode is an important factor in determining the adsorption capacity and the adsorption speed of an electrode. The efficiency of the CDI process can be improved while maintaining the ion selectivity through the use of a composite carbon electrode with a low electrical resistance.

EIS analyses were performed to measure the electrical resistance of the fabricated composite carbon electrodes. Fig. 4 shows impedance graphs for carbon electrodes in the frequency range of 100 Hz–20 mHz. In the frequency range of 1 Hz–100 Hz, the real part (Z') and the imaginary part (Z'') increased by approximately 45°; however, at frequencies of less than 1 Hz, the imaginary part drastically increased. The fact that the imaginary part of the change in impedance changed with the frequency indicates that the phase angle of the AC wave increased as the charges were stored in the electrode [23].

The electrical resistance of the coating layer of the composite carbon electrodes can be calculated from the impedance data. The impedance at a frequency of 100 Hz can be considered as resistance because the phase angle is close to 0. Therefore, the Z' value of the impedance measured at 100 Hz indicates the following equivalent series resistance (ESR) [25]:

$$\mathrm{ESR} = R_{\mathrm{ele}} + R_{\mathrm{sol}} \tag{2}$$

where R_{ele} is the resistance of the carbon electrode and R_{sol} is the electrical resistance of the electrolyte between the carbon electrode and the reference electrode. In this study, the same electrolyte was used in the impedance measurements; therefore, the difference in impedance is considered to be due to the resistance of the electrode. The electrical resistance of the coating layer can be calculated from the difference between the ESR of the coated composite carbon electrode and that of the uncoated carbon electrode. The calculated electrical resistances of the coating layer for each composite carbon electrode are shown in Fig. 5.

The resistance of the coating layer was 1.6 $\Omega\,\mathrm{cm}^2$ when the carbon electrode was coated only with SPS. However, when TiO_2 was added, the electrical resistance significantly decreased to a minimum of 0.56 $\Omega\,\mathrm{cm}^2$ in the electrode containing 20 wt% TiO_2. As previously mentioned, the decrease in electrical resistance can be explained by the pores formed in the coating layer. In the case of the previously discussed analysis of ion selectivity, a TiO_2 content of 10 wt% was thought to show enough ion selectivity for the MCDI process. Therefore, the results suggested that the electrical resistance could be reduced by approximately 40% while maintaining the ion selectivity if the composite carbon electrode with a TiO_2 content of 10 wt% was used compared to when only SPS was used. Therefore, these results also suggest that the optimal content of TiO_2 for minimizing electrical resistance while maintaining the ion selectivity for the coating layer is 10 wt%.

3.4. CDI performance using the ISCCEs

Desalination experiments were performed to evaluate the desalination performance of the fabricated ISCCEs. A CDI unit cell was fabricated with an electrode that contained 10 wt% TiO_2. The desalination performance of the ISCCE was compared to that of the uncoated carbon electrode by performing the desalination experiments under the same conditions. Fig. 6 shows the NaCl concentration for the effluent when a cell potential of 1.2 V was applied.

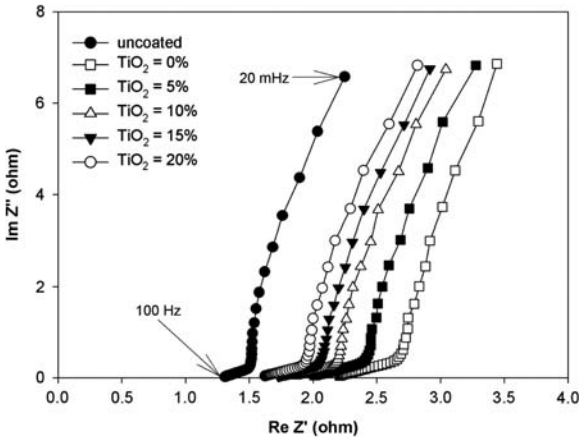

Fig. 4. A Nyquist plot for various carbon electrodes coated with TiO_2 and SPS mixtures with different ratios.

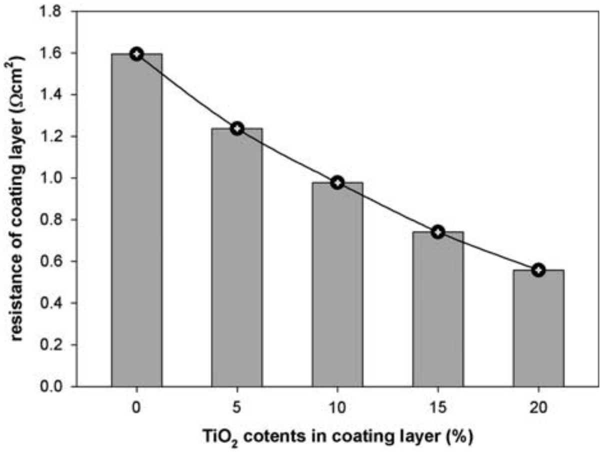

Fig. 5. The electrical resistances of the coating layer of the composite carbon electrodes according to the TiO_2 content.

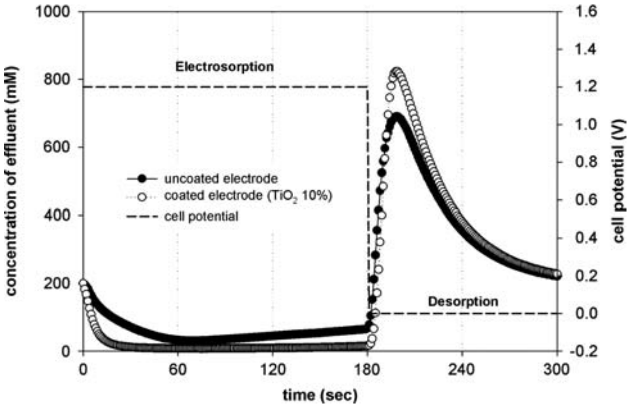

Fig. 6. The changes in effluent concentration during the adsorption and desorption period for the CDI unit cell constructed with uncoated carbon electrodes.

The desalination experiment with the uncoated carbon electrode showed that the concentration gradually decreased to 30.1 mg/L after approximately 70 s. The concentration of the effluent began to increase to 67.3 mg/L at 180 s. In contrast, when the ISCCE was used, the concentration decreased to 8 mg/L at approximately 45 s and to 15.1 mg/L at 180 s. The calculated average removal efficiency of NaCl during 3 min of adsorption time was 70.8% when the carbon electrode was used, but the efficiency improved to 91.8% when the ISCCE was used. In addition, desorption progressed rapidly when the charging potential was changed to 0.0 V after adsorption. The CDI desalination experiment using the ISCCE with a TiO_2 content of 10 wt% showed that the ISCCE can improve the desalination efficiency by 30% while effectively maintaining the ion selectivity.

4. Conclusion

An ISCCE was fabricated and applied to the CDI process in this study. The TiO_2 nanoparticles were mixed with the ion-exchange polymer and the mixture was coated onto the surface of the carbon electrode to reduce the electrical resistance of the coating layer while maintaining the ion selectivity of the electrode. The electrode was fabricated with various TiO_2 content ratios and the performance was analyzed using SEM and electrochemical methods.

The SEM images showed that the coating solution permeated into the surface of the carbon electrode, coupled with the carbon electrode, and formed a stable coating layer. Miscellaneous pores were formed among the metal oxide particles as the TiO_2 content was increased. As a result, the resistance of the composite carbon electrode was significantly reduced as the TiO_2 content was increased; however, the ion selectivity simultaneously decreased. The fabrication of the composite carbon electrode with the addition of TiO_2 showed that the optimal TiO_2 content to reduce the electrical resistance while maintaining the ion selectivity was approximately 10 wt%. In addition, the CDI desalination experiment using the ISCCE showed that the desalination efficiency was improved by approximately 30% compared to that of the uncoated carbon electrode. The ISCCE fabricated in this study is expected to apply to improve the existing CDI technology, which is considered to be the next generation of desalination technology.

Acknowledgments

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