

## 한계전류밀도 이상에서 전기투석공정의 운전

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### Operation of Electrodialysis at Over Limiting Current Density

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**요약** : 이온교환막의 전압-전류곡선의 plateau length를 결정하는 변수를 다양한 NaCl 농도와 유속 하에서 연구하였다. 또한, 한계전류밀도 이상의 전류에서 전기투석공정 운전의 타당성을 검토하기 위해 다양한 전류밀도의 전원을 공급하면서 0.1 M NaCl 용액의 탈염실험을 실시하여 이온의 제거효율, 전류효율, 에너지소비량, 물분해 현상을 측정하였다. NaCl 용액의 농도와 유속이 감소하면서 확산경계층의 두께도 함께 감소하였으며, 본 확산경계층의 두께는 plateau length와도 밀접한 관련이 있는 것으로 나타났다. 탈염실험에서 측정된 이온 제거 효율 및 전류효율은 한계전류밀도 이상에서도 한계전류밀도 이하에서의 탈염실험과 크게 차이 나지 않은 것으로 보아 한계전류밀도 이상에서도 대부분의 전류는 이온교환막 표면의 물분해에 의한 것이 아니라 막을 통한 이온의 이동에 의한 것으로 사료된다. 한계전류밀도 이상에서의 탈염운전에 대한 에너지소비량은 plateau length의 영향으로 한계전류밀도 이하에서의 탈염운전 보다 다소 높지만, 한계전류밀도 이상에서는 전류밀도의 증가에도 에너지소비량이 증가하지 않았다. 이러한 결과들은 물분해 현상이 심각하게 일어나지 않는 한 한계전류밀도 이상에서도 매우 경제적으로 전기투석 공정을 운전할 수 있다는 것을 제시해 주는 것이다.

**Abstract** : The parameter which determines the plateau length of current-voltage curve for ion-exchange membranes was studied at various concentrations of NaCl and different flow rates. Moreover, the feasibility of the electrodiolytic removal of 0.1 M NaCl solution at various current densities was tested by assessing the electrodiolysis performance parameters such as salt removal efficiency, current efficiency, energy consumption, and water dissociation. The diffusion boundary layer (DBL) thickness decreased with the NaCl concentration and flow rate of feed solution, and it was observed that the plateau length of current-voltage curves was related with the DBL thickness. The removal efficiency and current efficiency were not affected significantly by the current densities even at the overlimiting current region, indicating that most current were passed by electrolyte, and water dissociations

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are not responsible for the overlimiting current. Energy consumption increased when the current density supplied exceeded the limiting current density (LCD) values, because additional energy was necessary to overcome the plateau potential. Beyond the LCD values the energy consumption required to get a certain removal efficiency was not affected by the current density applied. The result suggests that it is allowed to operate electro dialysis processes at as high as possible current density unless water-splitting does not occur.

*Keywords* : *electrodialysis, ion-exchange membrane, current-voltage curve, plateau length, over-limiting current density*

## 1. Introduction

For years the most important application of electro dialysis (ED) is water desalination and production of potable water from brackish water [1]. Ion-exchange membranes, a key element of ED, have been developed to fit the purpose ideally: high permselectivity, even at high concentration, low electric resistance, and discrimination between mono-valent and divalent ions[2]. The availability of these membranes enables the use of ED in a number of industrial applications such as demineralization of whey[3], recovery of metals from metal plating rinse waters[4], recovery of inorganic and organic acids[5-11], separation of amino acids from a fermentation broth[12,13].

The important factor hindering the wider spread of ED is the cost of the stack including ion exchange membranes. Since the ED process cost is depending on the membrane area, it is desired to operate at the highest practicable current density to get the maximum ion flux per unit membrane area. Operating current levels are, however, restricted by the concentration polarization phenomenon. It is traditionally believed that the limiting current density is the maximum current allowed to operate an ED process [14]. If the current exceeds limiting one, the process efficiency diminishes with increasing in the electrical resistance and the occurrence of water dissociation.

Although there were potential detrimental effects, such as the increase in electrical resistance and occurrence of water dissociation at extremely high

current density, it was observed from the previous studies that the over-limiting current is still carried dominantly by the electrolyte, and the loss of permselectivity or water dissociate are not responsible for the over-limiting current [15-18]. It was also found that the plateau length of current-voltage curve determining the voltage increase at the over-limiting current might be reduced by controlling the membrane heterogeneity[19-21]. In order to obtain a cost effective operation of ED at higher current densities, the plateau length of the current-voltage curve for an ion-exchange membrane is required to be as short as possible. In addition it was expected that water dissociation could be suppressed with more stable anion-exchange groups in an anion-exchange membrane in a high alkaline condition.

Even though much work has been done on the concentration polarization phenomena and on the occurrence of water dissociation at the ion-exchange membrane, there has been relatively little experimental study of the behavior of ED systems at high current densities, under conditions of severe concentration polarization. If the economic effectiveness of the process is to be improved, it is important to understand the behavior of the system under such operating conditions. This may open the way to the more efficient use of the ED system at higher current densities.

In this study the current-voltage curves were analyzed under different experimental conditions to investigate parameters determining the plateau length of an ion-exchange membrane. In addition, the

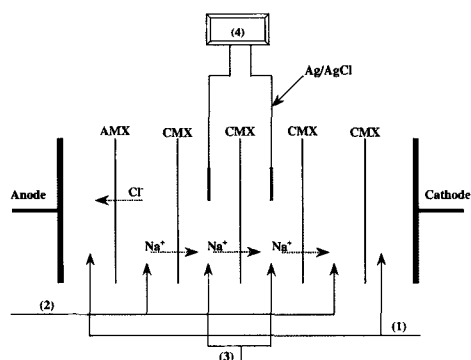


Fig. 1. Schematic diagram of current-voltage experimental set-up for the CMX membrane: (1) electrode solution (0.5 M  $\text{Na}_2\text{SO}_4$ ), (2) 0.2 M NaCl, (3) feed solution, (4) Multimeter. The three CMX membranes between the outer AMX and CMX membrane were replaced by the AMX membrane to obtain the current-voltage curve for the AMX membrane.

feasibility of ED operation at over-limiting current densities was discussed in terms of current efficiency, salt removal efficiency, energy consumption, and water dissociation.

## 2. Experimental

### 2.1. Current-voltage Curve of Ion-exchange Membrane

Current-voltage curves were obtained with a plexiglas membrane cell shown in Fig. 1. The cell consists of six separate compartments. The two outer compartments contain the working electrodes, i.e. a platinized titanium anode and a stainless steel cathode. The membranes used in the experiments were the Neosepta CMX and the Neosepta AMX that were purchased from Tokuyama Soda Co., Japan. The effective membrane area was  $25 \text{ cm}^2$ . The anion membrane between compartments 1 and 2 was used for blocking proton transport from the anolyte solution while the cation membrane between compartment 5 and 6 blocked the flow of  $\text{OH}^-$  ions from catholyte solution.

Current-voltage curves were determined for four dif-

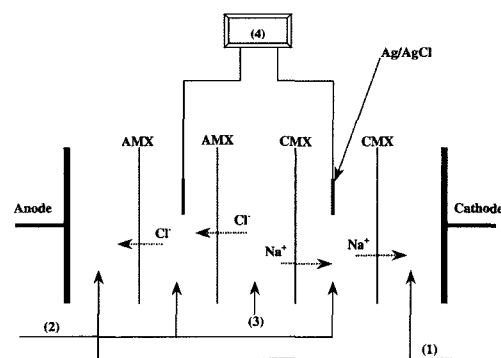


Fig. 2. Schematic diagram of the membrane arrangement for the desalting experiments: (1) electrode solution (0.5 M  $\text{Na}_2\text{SO}_4$ ), (2) concentrate compartment solution (0.1 M NaCl), (3) feed solution (0.1 M NaCl), (4) Multimeter.

ferent concentrations of NaCl solution (0.025, 0.050, 0.075, and 0.10 M). The solution was circulated through compartments 3 and 4 at a flow rate of 150 mL/min in each compartment. Also the effect of flow rate on the current-voltage curve was examined in 0.1 M NaCl solution. In the electrode compartments (compartments 1 and 6) 0.5 M of  $\text{Na}_2\text{SO}_4$  was used. The solutions in compartments 2 and 5 were 1.0 L of 0.5 M NaCl so that any interference due to concentration polarization did not occur on any membrane other than the test membrane (positioned between compartments 3 and 4). Current-voltage curves were obtained by a step-wise increase of current. After an increase in the current, the electrical potential difference across the membrane was measured every 3 seconds by multimeter (HP34401) for 5 minutes, followed by the next current increase. Average values of the potential were plotted against current densities to get the current-voltage curves.

### 2.2. ED Experiment of NaCl Solution

A plexiglas cell was used in desalting experiment. Fig. 2 shows the cell configuration with the transport direction of the different ionic species.

The feed solution (200 mL of 0.1 M NaCl) circulated through the central compartment at a flow rate of 150 ml/min. The concentrate compartments on

both sides of the feed compartment were adjacent to the electrode rinse compartments in which 1.0 L of a 0.5 N of  $\text{Na}_2\text{SO}_4$  solution was circulated, and 1.0 L of a 0.1 M of NaCl solution in each of the concentrate compartments. Using an Orion pH meter (Model 250 A) and conductivity meter (Cole-Parmer, Model 124) the pH and conductivity of the feed solution were measured as a function of time. The concentration of sodium ions in the feed solution was analyzed using ion chromatography (Dionex DX-500, ED 40 conductivity detector, CS 12 A column).

The ED experiments were performed at a constant current operation mode. The current densities supplied (HP 66332, 0-5 A, 0-20 V) were 6.0, 12.0, 18.0, and 24.0  $\text{mA}/\text{cm}^2$ . The voltage drop across the unit cell was measured by Ag/AgCl electrodes connected to a multimeter (HP 34401 A). The potential data were automatically collected by a computer. New membranes were used for each experiment. All experiments were carried out at room temperature ( $23 \pm 1^\circ\text{C}$ ).

### 3. Results and Discussion

#### 3.1. Relationship between Plateau Length and Diffusion Boundary Layer Thickness

Current-voltage curves obtained at the CMX membrane in 0.025 M sodium chloride solution were shown in Fig. 3. The potential data is the average value at a given current density. The curve shows the three characteristic regions as expected: the first region of approximately ohmic behavior, the second region showing a plateau, followed by the third region of a rapid current increase. In order to operate ED at higher (over-limiting) current density, it is required to decrease the plateau length that causes the high power consumption. Therefore, it is necessary to know the factors that determine the plateau length of a current-voltage curve. The plateau length is defined as the projection of two points; one is the intersection of the ohmic and plateau region, and another is the

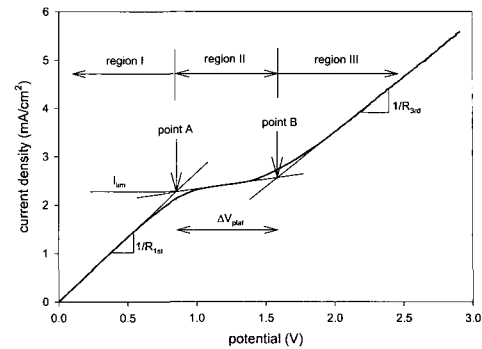


Fig. 3. Current-voltage curves obtained at the CMX membrane in 0.025 M of sodium chloride solution. The solid line corresponds to Eq. (1).

intersection of plateau and the third region of current-voltage curve as can be seen in Fig. 3.

The limiting current density (LCD) has been determined by indirect methods, such as Cowan plots or the tangent method[15,22]. If the concentration of electrolyte or flow rate increase, then the plateau value becomes less pronounced [23]. Thus those methods are not accurate to determine the LCD value. Barragan et al[24]. developed a new method for determination of the LCD from the adjustment of the data corresponding to the first region of the current-voltage curves. Taking into account the classical theory of concentration polarization, they derived the following equation which relates the membrane potential to the current density.

$$\Phi = \left( R_o - \frac{2RT}{F i_{lim}} \right) i + \frac{RT}{F} \left[ (2 \Delta t_i) + (2 \Delta t_i)^{-1} \right] \ln \left( \frac{i_{lim} + i}{i_{lim} - i} \right) \quad (1)$$

where  $\Phi$  and  $i$  are the electric potential difference and current density,  $R_o$  is the ohmic resistance before the polarization layers are formed,  $i_{lim}$  is the limiting current density,  $\Delta t_i$  is the difference between the counterion transport numbers in the membrane and in the solution,  $F$  is the Faraday constant, and  $R$  and  $T$  are the gas constant and absolute temperature, respectively. The equation is only applicable to current

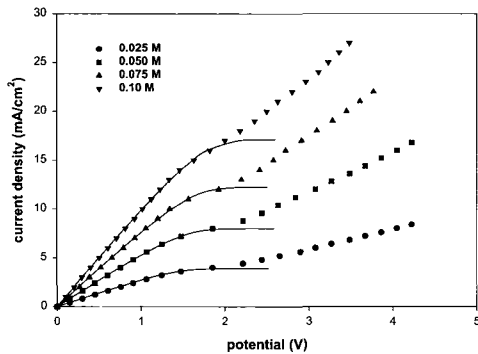


Fig. 4. Current-voltage curves for the AMX membrane at different concentration of sodium chloride solution. The solid lines correspond to Eq.(1).

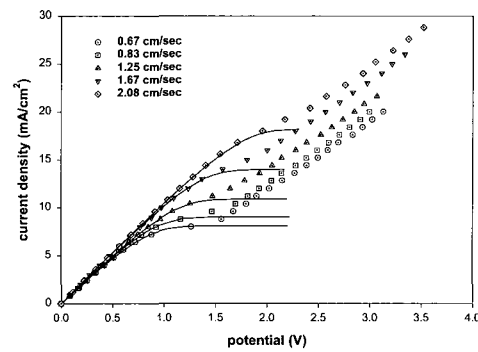


Fig. 6. Current-voltage curves obtained for the CMX membrane at various linear velocity of feed solution in 0.1 M NaCl. The solid lines correspond to Eq. (1).

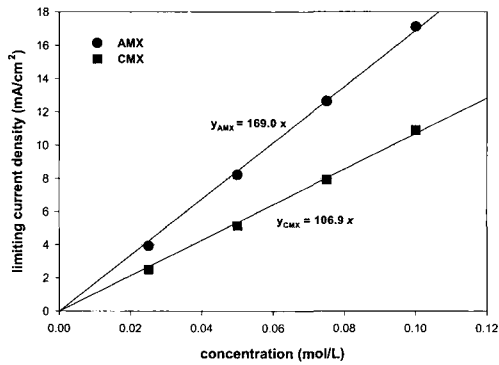


Fig. 5. Limiting current densities (LCD) obtained for the CMX and AMX membranes as a function of NaCl concentration.

densities such as  $i < i_{lim}$ . From the data obtained in the first region of the current-voltage curve, the value the LCD and  $R_o$  were obtained by fitting the data to Eq. (1).

Fig. 4 shows the current-voltage curves for the AMX membrane at different concentration of sodium chloride. It can be observed that the plateau region of the curves becomes less pronounced and the length becomes short when the concentration increases. The similar feature of current-voltage curves was observed for the CMX membrane. The parameters for each concentration were determined by data regression using Eq. (1).

Fig. 5 shows the results of LCD values obtained as a function of concentration. The LCD for the AMX

membrane is higher than that for the CMX membrane at a given concentration of sodium chloride. This is due to the difference in transport number of the counter ions in solution. The limiting current density is expressed by

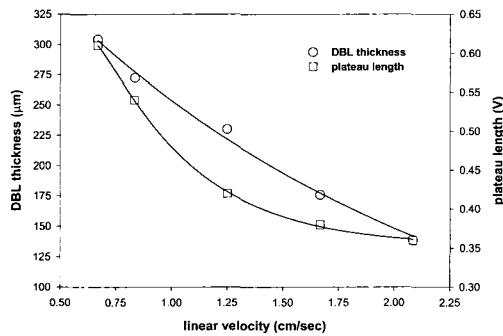
$$i_{lim} = \frac{FDC_o}{\delta \Delta t_i} \quad (2)$$

where,  $D$  is the diffusion coefficient,  $C_o$  is the concentration of electrolyte, and  $\delta$  is the diffusion boundary layer (DBL) thickness. The transport number of chloride ion is 0.61 and the sodium ion is 0.39 in sodium chloride solution[25]. The transport numbers of chloride and sodium ion in the AMX and CMX membrane were measured by the emf method, and the values were measured as 0.97 and 0.96, respectively. If it is assumed that  $D$ ,  $\delta$  and  $\Delta t_i$  remained constant, Eq. (2) represents a proportional relationship between the limiting current density and the concentration of the salt in the bulk phase. This feature was well illustrated in Fig. 5.

Fig. 6 shows the current-voltage curves obtained for the CMX membrane at various linear velocity of solution in 0.1 M NaCl solution. All the curves show three characteristic regions. However, it is observed that the plateau region of the curves becomes unclear and the length decreases as the linear velocity increases. The  $R_o$  and  $i_{lim}$  values are determined by

**Table 1.** LCD ( $i_{lim}$ ) and  $R_o$  Values Obtained for the CMX Membrane at Various Linear Velocity of Solution in 0.1 M NaCl Solution

Linear velocity (cm/sec)	LCD (mA/cm <sup>2</sup> )	$R_o$ (Ohm-cm <sup>2</sup> )
0.67	8.09	88.10
0.83	9.04	86.51
1.25	10.70	88.70
1.67	14.01	85.29
2.08	17.73	87.34



**Fig. 7.** Diffusion boundary layer thickness and plateau length as a function of linear velocity of feed solution for the CMX membrane in 0.1 M NaCl solution.

fitting the current-voltage data obtained in the first region to Eq. (1), and the results are listed in Table 1. The limiting current density increases as the flow rate increases. It is well known that the DBL thickness is affected by the flow rate of the solution[22]. The increase in flow rate caused a decrease of the DBL thickness and enhanced the transport process. On the contrary, the value of  $R_o$  for the given concentration, as could be expected, practically independent of the flow rate.

Using the LCD values determined by data regression (Table 1), the DBL thickness were calculated from Eq. (2) and their values are plotted in Fig. 7 along with the plateau length as a function of the lineary velocity of feed compartment solution. As expected the DBL thickness is strongly affected by the linear velocity. The plateau length also decreased with the linear velocity. However, it was noticeable

that the plateau length decreased exponentially, approaching to about 0.35 V. It was reported that the plateau length depended on the nature of an electrolyte solution and a membrane[26]. It is thought that an ion-exchange membrane has an intrinsic value of plateau length that is not affected by the DBL thickness but by electrolyte and the membrane inherently.

Fig. 7 show that the plateau length of the current-voltage curve is related to the DBL thickness. The relationship could be explained by the transport phenomena in an ion-exchange membrane system at over-limiting current region. The plateau region of the current-voltage curve is distinguished by the development of a weak space charge within a local electroneutral diffusion layer. Recalling the electroconvective transport mechanism at the over-limiting region[27], the plateau length can be regarded as a minimum potential that can cause the electroconvective mixing in the diffusion boundary layer. Therefore, a short plateau length is favorable to create an electroconvective mixing in a thin DBL. Concentration polarization can never be completely avoided. Its magnitude, however, can be minimized by the proper equipment and process design. The boundary layer thickness is a function of the hydrodynamics of the feed solution, i.e., the flow velocities, the cell geometry, the spacer design, etc. It is preferred to decrease the DBL thickness because the LCD can be increased by decreasing the DBL thickness. In addition, it was considered that the DBL thickness is an another factor determining the plateau length of an ion-exchange membrane.

### 3.2. Desalting of NaCl Solution at Higher Current Densities

As shown in Fig. 5 the LCD for the AMX membrane is about 1.6 times higher than that for the CMX membrane at a given concentration of sodium chloride solution. Therefore, the LCD value for desalting of NaCl solution is controlled by the CMX

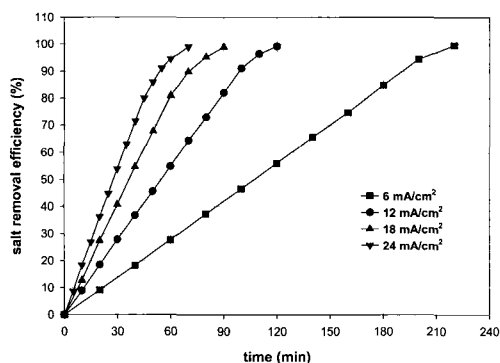


Fig. 8. Time course of the electrodyalytic removal efficiencies of NaCl at different current densities.

membrane and is  $10.7 \text{ mA/cm}^2$  in  $0.1 \text{ M NaCl}$  solution. The feasibility of the batch ED of  $0.1 \text{ M NaCl}$  solution at various current densities ( $6, 12, 18$  and  $24 \text{ mA/cm}^2$ ) was carried out to assess the ED performance parameters, i.e., salt removal efficiency, current efficiency, energy consumption, and water dissociation.

The time course of the electrodyalytic removal percentage of NaCl at different current densities are shown in Fig. 8. Removal of sodium chloride at the current density of  $6, 12, 18,$  and  $24 \text{ mA/cm}^2$  were completed within about 220, 120, 90, and 70 minutes, respectively. The transport rate exhibited a linear relation with the current density applied, and the removal efficiency was not affected by the current density even for the over-limiting region. The result indicates that the membrane area required to remove salts for a given feed solution could be reduced with the increasing current density.

Using the current and potential data energy consumptions were calculated and plotted as a function of removal efficiency of NaCl in Fig. 9. As expected, the energy consumptions increased exponentially with the removal efficiency because the electrical resistance in the dilute compartment solution increased with the removal efficiency. Further it was found that energy consumption increased with the current density supplied to get the same removal efficiency. The specific energy consumptions at the

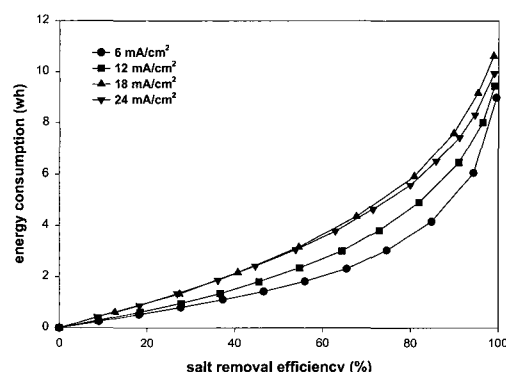


Fig. 9. Energy consumptions as a function of removal efficiency of NaCl at different current densities.

current density of  $6, 12, 18,$  and  $24 \text{ mA/cm}^2$  were calculated as  $5.18, 6.71, 7.82, 7.10 \text{ kWh/kg NaCl}$ , respectively. The high specific energy consumption was due to the dimension of the electrodyalytic cell used in this study. In general ED stack, the thickness of flow channel is less than  $1 \text{ mm}$ , while the channel thickness of the cell used in the study was  $8 \text{ mm}$ .

The increase in energy consumption with the current density was attributed to the plateau length of current-voltage curves. For  $0.1 \text{ M NaCl}$  solution  $6 \text{ mA/cm}^2$  of current density is in the under-limiting current region for both CMX and AMX membrane as shown in Fig. 5. The current density of  $12 \text{ mA/cm}^2$ , however, is in the over-limiting current region for the CMX membrane, but still under-limiting current for the AMX membrane. This indicates that additional potential corresponding to the plateau length is necessary to supply the over-limiting current through the CMX membrane, resulting in higher energy consumption. Since the current densities exceeding  $18 \text{ mA/cm}^2$  are over-limiting currents for both AMX and CMX membranes, additional potential is required to transport over-limiting current for the AMX membrane as well as for the CMX membrane. It was noteworthy that there were no significant differences in energy consumptions between the current density of  $18$  and  $24 \text{ mA/cm}^2$ . In the over-limiting current region the energy consumption per unit mass transported was not

affected by the current density supplied, implying that the ED operation at a high current density is feasible without significant energy consumption. This indicates that it is preferred to operate ED at as high current densities as possible if there is no deleterious effects on the membranes, such as, membrane damage and scale formation.

Process design and economics are closely related in ED operation. The total costs are the sum of fixed charges associated with amortization of the plant investment costs and of operating costs[28]. The total investment cost of an ED process is a function of the required membrane area that is inversely proportional to the current density. The operating costs are mainly determined by the energy required. The energy in ED is determined by the electrical energy required for transporting ions and for pumping the solution through the stack. The pumping energy depends on the ion removal rate which is related to the operating current density. Eventually, although the energy consumption required to transport ions increased in the over-limiting current region, operation of ED at higher current density has advantages of decreasing the pumping cost and the reduction of membrane area required.

One of the problems in operating ED at an over-limiting current is water dissociation which gives rise to scale formation and decrease in current efficiency[15]. Fig. 10 shows the pH change of dilute compartment solution during the ED experiment at different current densities. As expected there was little pH change at the current density of 6 mA/cm<sup>2</sup> until 100 min because the current density supplied was under limiting current density. After 100 min, however, the pH increased steeply up to 9.16 at 160 min and then decreased to 3.74 at 200 min. The pH change at the current density of 12 mA/cm<sup>2</sup> shows a similar pattern with that of 6 mA/cm<sup>2</sup> but the pH change occurred at the beginning of the experiment. On the contrary pH increase was not observed at the current density of 24 mA/cm<sup>2</sup>. The pH changes with

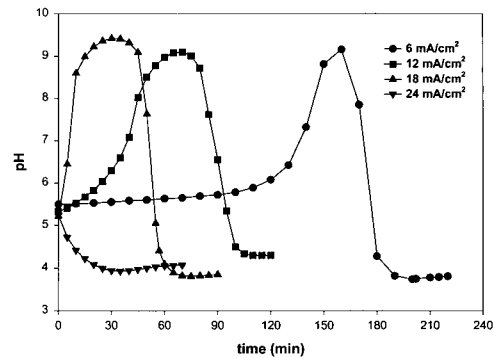


Fig. 10. pH changes of dilute compartment solution during the electro dialysis experiment at different current densities.

the current density are related to the water dissociation reaction at the AMX and CMX membrane. Recalling the results of water dissociation obtained in the previous study that water dissociation was more pronounced with the anion-exchange membrane, the pH changes occurred in ED experiments seemed to be natural.

The current efficiency relates the performance of a process to a theoretical maximum and is therefore useful for process evaluation. The current efficiency is the ratio of stack current to the number of ions passed through the membrane. The current efficiency ( $\eta_i$ ) was calculated by the previously defined mathematical equation [29].

$$\eta_i = \frac{(C^o - C^t) \cdot V \cdot F}{\int_0^t i A dt} \times 100 \quad (3)$$

where,  $C^o$  are  $C^t$  salt concentration in the feed solution initially and at time  $t$ .  $V$  is the volume of feed solution,  $i$  the current density supplied, and  $A$  the effective membrane area.

Fig. 11 shows the current efficiencies at different operating current densities as a function of salt removal efficiency. The current efficiencies did not change significantly until most salts were removed, and decreased slightly from 0.96 to 0.93 with the increasing current densities. Usually, the current



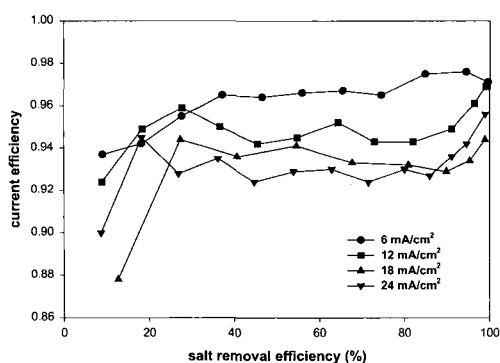


Fig. 11. Current efficiencies as a function of salt removal efficiency at various current densities.

efficiency is related to the membrane permselectivity. Assuming that the membrane permselectivities are not affected by the current density, the decrease in current efficiency was resulted from the water dissociation. The permselectivity of commercial ion-exchange membrane is known to be greater than 0.95[30]. Even if the permselectivity of AMX and CMX membrane is assumed to be 0.96, the amount of water dissociation is estimated to be less than 3% of the total current supplied. This indicates that most current were passed by the electrolyte, and water dissociation was not responsible for the over-limiting current at the over-limiting current density investigated in this study.

#### 4. Conclusions

The parameter which determines the plateau length of an ion-exchange membrane was studied using the characteristic of current-voltage curves obtained at various flow rates. The DBL thickness decreased with the linear velocity of feed solution. It was noticeable that the plateau length decreased exponentially with the linear velocity approaching to a certain value that is considered to be an intrinsic value of the membrane.

In batch ED experiments of 0.1 M NaCl solution at various current densities, the removal efficiency and current efficiency were not affected significantly by the current densities even at the over-limiting current

region. Energy consumption increased when the current density supplied exceeded the LCD values, because additional energy was necessary to overcome the plateau potential. In the over-limiting current region, however, the energy consumption per unit mass transported was not affected by the current density supplied, implying that the ED operation at a higher current density is feasible without significant energy consumption. From the current efficiency data, we observed that most currents were carried by the electrolyte, and water dissociation was not responsible for the elevated current at the over-limiting current density.

Through this study it can be concluded that operating of electro dialysis at overlimiting current region could be possible economically if the plateau length of the current-voltage curve are shorten by proper spacer design for the feed solutions which does not contain the scale forming ions such as metal ions.

#### 5. Nomenclature

$A$	Effective membrane area
$C^0, C^t$	Salt concentration in the feed solution at initial and time $t$
$F$	Faraday constant
$i$	Current density
$i_{lim}$	Limiting current density
$R$	Gas constant
$R_o$	Ohmic resistance before the polarization layers are formed
$\Delta t_i$	Difference between the transport numbers in the membrane and in the solution
$t$	Time
$T$	Temperature
$V$	Solution volume
$\delta$	Diffusion boundary layer thickness
$\Phi$	Electric potential difference
$\eta_i$	Current efficiency

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