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# Removal of Zinc Ions in Wastewater by Electrodialysis

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**Abstract**−To obtain useful data for treatment of the wastewater discharged from zinc electroplating processes, we investigated the effects of operating parameters, such as the initial concentration of dilute solution, the flow velocity and the applied voltage, on removal rate of $Zn^{2+}$ in the model solutions using an electrodialysis system. Zinc ions in the solutions were effectively removed by the electrodialyzer with CMX cation exchange membranes and AMX anion exchange membranes. The initial concentration of dilute solution, the flow velocity and the applied voltage strongly affected the performance of the electrodialysis system. As the initial concentration of dilute solution, the flow velocity and the applied voltage were increased, the removal ratio was increased. The energy consumption was increased as the initial concentration of dilute solution and the applied voltage were increased, whereas the effect of the flow velocity on the energy consumption was negligible.

Key words: Electrodialysis, Removal Ratio, Zinc Ion, Wastewater Treatment

## INTRODUCTION

Valuable metal ions are dissolved in industrial effluents through rinsing and pickling processes in surface finishing and plating industries. The effluents are both main pollution factors and a considerable loss of raw materials. In particular, if the effluents from electroplating plants are not properly treated, they are a significant source of water pollution. On the other hand, the effluents are the cause of considerable additional cost of raw materials. Therefore, for plating and surface finishing industries, recovery and recycling of metals in the effluents is an important technical and economical problem [Öğütveren et al., 1997].

For wastewater treatment, membrane technology is progressively replacing traditional techniques such as chemical precipitation, coagulation, complexation, activated carbon adsorption, solvent extraction, form flotation, and cementation [Peters et al., 1986]. Membrane separation processes, e.g., electrodialysis, reverse osmosis and ultrafiltration, are very attractive for treatment of industrial effluents because valuable metal ions can be recovered directly for reuse without chemical transformation in these processes [Ramachandraiah et al., 1996].

Electrodialysis was developed mainly for concentration of seawater and water desalination. In recent years, electrodialysis has been widely used for desalination of food or medicines and for recovery of acid or metal from industrial effluents. To concentrate the dilute solution of $ZnSO_4$, Audinos [1983] used an electrodialysis system. As a result of optimization with respect to time, he reported that the concentration factor was high when the product of the exchange area by the inverse of channel width was high.

Wisniewska and Winnicki [1991] removed $Zn^{2+}$ and $Cl^-$ in the solutions by the electrodialyzer with AESD and KESD ion-exchange membranes. They observed that at a current density of 28.6 A/m$^2$, the ion elimination coefficient was high when the energy consumption was equal to or less than 4 kWh/m$^3$.

Sistat et al. [1997] recovered sulfuric acid from the effluents containing metallic salts (Mg, Zn, Mn) by using the electrodialyzer with a modified Nafion cation exchange membrane. They reported that selectivity of the cation exchange membrane towards protons with respect to bivalent metallic cations was enhanced by electrodeposition of the positively charged polyelectrolyte on the surface of the cation exchange membrane through an in situ method.

Aouad et al. [1997] investigated the properties of the anion exchange membranes (Neosepta ACS, Neosepta AMI, Neosepta AFN, Selemion AMV, Morgane ADP) in contact with the aqueous solutions containing zinc chloride complexes. Monovalent cation electrotransport became much easier when zinc chloride was present in the solutions. When electrical current crossed the membranes, large-size ions which equilibrated the fixed sites of the membranes were replaced by chloride ions.

Cho [1989] studied limiting current density and ionic mass transfer rate of zinc ion using an electrodialyzer consisting of an anion exchange membrane (Selemon AMV) and a cation exchange membrane (Selemon CMV). He suggested the two empirical correlation equations for limiting current density and ionic mass transfer rate of zinc ion are as follows:

$$I_{lim} = 66 \cdot V_L^{0.34} \cdot C^{0.34} \tag{1}$$

$$N_u = 2.18 \cdot R_e^{0.34} \cdot S_c^{0.34} \cdot \left(\frac{d_e}{L}\right)^{1/3} \tag{2}$$

In this study, to obtain useful data for treatment of the wastewater discharged from zinc electroplating processes, the effects of operating parameters, such as the initial concentration of dilute solution, the flow velocity and the applied voltage, on removal rate of $Zn^{2+}$ in the model solutions using an electrodialysis system were investigated.

## EXPERIMENTAL

### 1. Preparation of Model Solution

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In this study, the model solutions, which contained 300 mg $L^{-1}$, 600 mg $L^{-1}$ and 1,000 mg $L^{-1}$ of $Zn^{2+}$, respectively, were prepared by means of dissolving a certain amount of $ZnSO_4 \cdot 7H_2O$ in deionized water. 1.5% $Na_2SO_4$ solution was used as the electrode solution containing a common anion. Before making use of the model solutions, $Zn^{2+}$ concentration of the solutions was measured by an atomic absorption spectrometer (Thermo Jarrel Ash, Smith-Hieftje 4000).

## 2. Experimental Apparatus

A schematic diagram of the experimental electrodialysis system (Tokuyama Co, TS-1-10), which was used in this study, is shown in Fig. 1. The membrane stack configuration used in this work is illustrated in Fig. 2. The stack consisted of a collection of alternating cation exchange membranes and anion exchange membranes which were placed between an anode and a cathode. The cation exchange membrane and the anion exchange membrane utilized in the stack were Tokuyama cation exchange membrane CMX and Tokuyama anion exchange membrane AMX, respectively. For the equivalent anion mass transfer between the concentrate solution and the electrode solution, two sheets of the anion exchange membranes were installed near the anode. And the cation exchange membrane and the anion exchange membrane were installed alternatively. The membrane stack configuration permitted the cathode to avoid electroplating. As a result of the arrangement, the stack had two cell pairs and an isolating compartment.

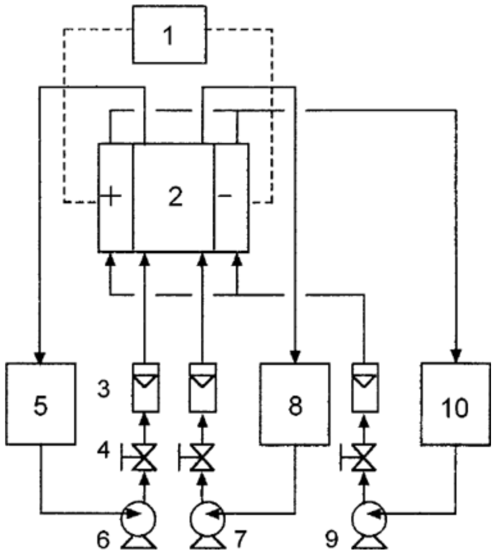

**Fig. 1. A schematic diagram of the experimental apparatus for electrodialysis.**

1. Rectifier
2. Electrodialyzer
3. Flow meter
4. Valve
5. Dilute solution tank
6. Dilute solution pump
7. Concentrate solution pump
8. Concentrate solution tank
9. Electrode solution pump
10. Electrode solution tank

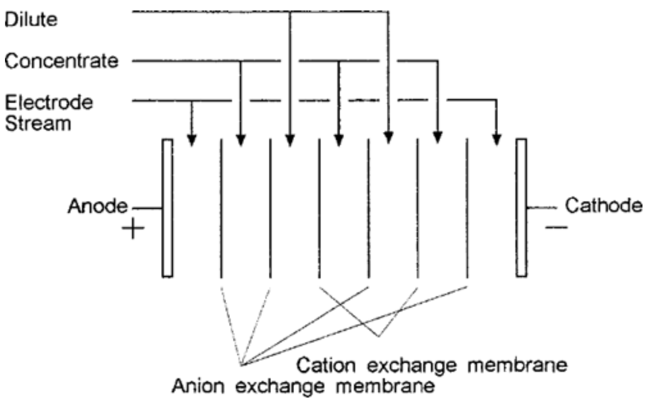

**Fig. 2. Stack configuration used for this experiment.**

As electrical power is applied to the stack while electrolyte streams flow through the stack, cations travel in the direction of electrical field and pass through the cation exchange membranes, but are repulsed upon the anion exchange membranes. Likewise, anions moving in the opposite direction pass through the anion exchange membranes, but are repulsed upon the cation exchange membranes. As a result of this phenomenon, there are two streams except for an electrode stream. One stream (the dilute stream) becomes increasingly depleted of electrolytes and the other stream (the concentrate stream) becomes increasingly enriched with electrolytes. Using such a principle of electrodialysis, zinc ions in the model solutions can be eliminated.

## 3. Experimental Methods

The electrodialysis system was operated in a batch mode. The dilute stream, the concentrate stream, and the electrode stream flowing from their respective holding tanks, were pumped and entered the stack. After that, the streams were recycled to their holding tanks. Flow rates of the dilute stream and the concentrate stream were measured by flow meters. Flow rate of the electrode stream was fixed at 3.252 L/min. The electrodialysis experiments were conducted until zinc concentration of the dilute solution reached 10% of initial value.

The experimental procedure is as follows. First, the experimental apparatus was rinsed by recycling of deionized water for 15 minutes. After the rinsing water was eliminated, the experimental apparatus was rinsed again for 2 minutes using small amount of the solution which would be used for each experiment. At this step, flow velocities of the dilute stream and the concentrate stream were adjusted at the desired values. After the solutions which were used for rinsing were eliminated, the dilute solution tank was filled with 1 L of the model solution. The concentrate solution tank was filled with 1 L of the solution of which $Zn^{2+}$ concentration was 10% of the initial $Zn^{2+}$ concentration of the model solution. After the electrode solution tank was filled with 1 L of the electrode solution, the experiment was started by supply of electrical power to the electrodialysis apparatus.

To obtain experimental data, 5 mL of sample was obtained from the dilute solution tank every 5 minutes. Conductivity of the dilute solution was measured by a conductivity meter (TOA Electrics, CM606). The electric current which flowed through the equipment was measured simultaneously. After the sample was diluted by some deionized water, $Zn^{2+}$ concentration of the sample was measured by an atomic absorption spectrometer (Thermo Jarrel Ash, Smith-Hieftje 4000). The experimental conditions are listed in Table 1.

## 4. Treatment of Experimental Data

Stack resistance is the sum of all electrical resistances present within the stack at any moment. Electrical resistances arise from both membrane and solution contributions. Apparent stack resistance, $R_a$, is calculated from [Gering and Scamehorn, 1988]

$$R_a = \frac{V_a S}{nI} \tag{3}$$

where I is the current over time interval Δt as given by

$$I = \int_{t_1}^{t_2} i(t)\, dt \tag{4}$$

Energy consumption of each experimental run is given by

$$W = V_G \int_{t_1}^{t_2} i(t)\, dt = V_G I \tag{5}$$

On the other hand, removal ratio of $Zn^{2+}$ is calculated as follows [Wisniewska and Winnicki, 1991]:

$$R_r(\%) = \frac{C_0 - C}{C_0} : : 100 \tag{6}$$

## RESULTS AND DISCUSSION

Fig. 3 shows the variation of $Zn^{2+}$ concentration of the dilute solution with time for the different initial concentration of dilute solution. The $Zn^{2+}$ concentration of the dilute solution decreased with increasing time. It can be explained that electrolytes in the dilute solution gradually moved to the concentrate solution by electrodialysis. Roughly, three major periods could be distinguished in the variation of $Zn^{2+}$ concentration of the dilute solution with time. Decreasing rate of the $Zn^{2+}$ concentration for the middle period of electrodialysis was larger than that for the initial and final periods of electrodialysis. The stack resistance for both the initial and final periods was larger than that for the middle period because, for these periods, the $Zn^{2+}$ concentration difference between the dilute solution and the concentrate solution was large. The large stack resistance led to a decrease in the dialytic rate. For the middle period, the stack resistance decreased because the concentration difference was decreased by progress of electrodialysis. Therefore, the dialytic rate was large during the middle period.

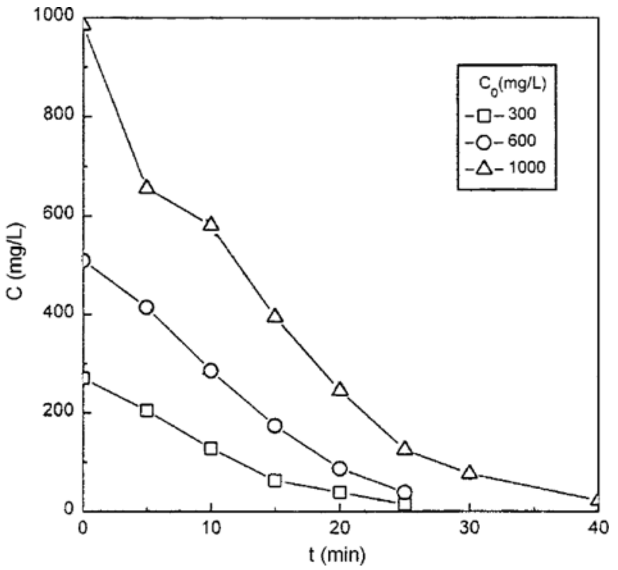

**Fig. 3. Variation of $Zn^{2+}$ concentration of the dilute solution with time for the different initial concentration of dilute solution ($V_L$=6 cm $s^{-1}$, $V_G$=10 V).**

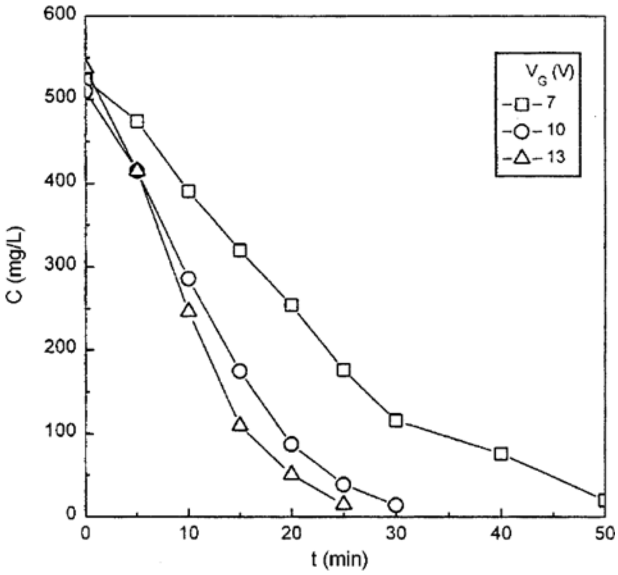

**Fig. 4. Effect of the applied voltage on the variation of $Zn^{2+}$ concentration of the dilute solution with time ($C_0$=600 mg $L^{-1}$, $V_L$=6 cm $s^{-1}$).**

On the other hand, Fig. 3 shows that as the initial concentration of dilute solution increased, the required time for a certain removal ratio was increased. Öğütveren et al. [1997] studied removal of $Cu^{2+}$ in a rinse water using the recirculation electrodialysis system. They reported similar results. In addition, Chung et al. [1996] studied the effects of the initial concentrations of electrolytes ($CuSO_4$ and $H_2SO_4$) on the flux of $Cu^{2+}$ in the electrodialysis system. They reported that as either the initial concentration of $Cu^{2+}$ or the initial concentration of $SO_4^{2-}$ increased, the flux of $Cu^{2+}$ was increased. Choi [1987] observed that the $Cu^{2+}$ flux was proportional to copper ion concentration in the feed.

Fig. 4 shows the effect of the applied voltage on the variation of $Zn^{2+}$ concentration of the dilute solution with time. As the applied voltage increased, $Zn^{2+}$ concentration of the dilute solution suddenly decreased with time. The result can be explained by the electrical power being a driving force for electrodialysis. Ramachandraiah et al. [1996] treated samples of the effluent and the sludge, which were collected from the metal plating industry, using electrodialysis. They reported that percentage reduction of ionic components in the effluent sample was increased gradually as the applied voltage and the current increased. Öğütveren et al. [1977] also reported that percentage reduction of the metal ion was increased as the applied voltage increased. They observed that the required time for a certain value of the percentage reduction of the ion was increased as the applied voltage increased.

Fig. 5 shows the effect of the flow velocity on the variation of $Zn^{2+}$ concentration of the dilute solution with time. In industrial electrodialysis systems, flow velocity of electrolyte solution usually varied from 3 cm $s^{-1}$ to 10 cm $s^{-1}$ [Hattenbach and Kneifel, 1986]. In this study, the flow velocities were 4 cm $s^{-1}$, 6 cm $s^{-1}$ and 8 cm $s^{-1}$. It can be seen in Fig. 5 that an increase in the flow velocity led to an increase in the dialytic rate. It can be explained that as the flow velocity increased, the boundary layer between bulk liquid and the ion exchange membrane thinned and in result diffusion rate of ions

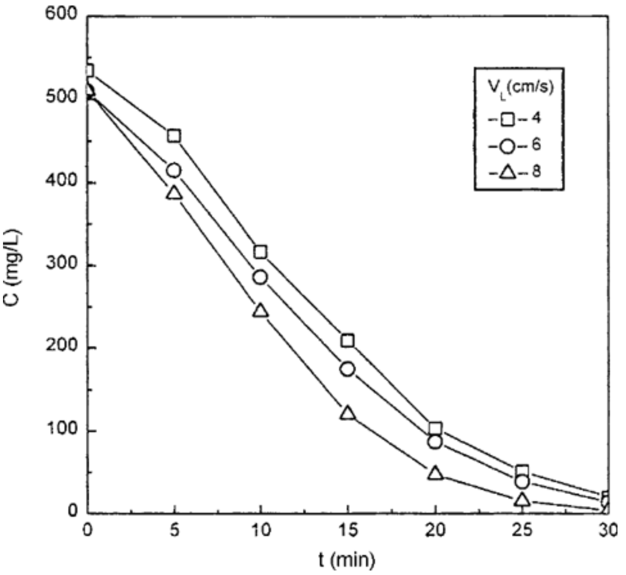

**Fig. 5. Effect of the flow velocity on the variation of $Zn^{2+}$ concentration of the dilute solution with time ($C_0$=600 mg $L^{-1}$, $V_G$=10 V).**

was increased. Huang et al. [1983] studied the effects of the flow velocity, viscosity of electrolyte solution and thickness of electrodialyzer on ionic mass transfer rate of copper ion in an electrodialyzer at limiting current density. They reported that the dimensionless mass transfer rate (Nu) varied directly with $Re^{0.33}$. In addition, Cho [1989] obtained a similar result. On the other hand, in order to obtain data for optimization of electrodialysis membrane stacks in water desalination, Hattenbach and Kneifel [1986] investigated the effects of cell thickness and the flow velocity on water production cost. They reported that for the industrial membrane stacks applied to water desalination, cell thickness of 0.4-0.6 mm and the flow velocity of about 10 cm $s^{-1}$ seem to be the most economical design criteria.

Fig. 6 shows the effect of the initial concentration of dilute solution on the variation of the stack resistance. The stack resistance is the sum of all electrical resistances present within the stack at any given moment. It is well known that solution resistance depends on electrolyte concentration [Gering and Scamehorn, 1988; Chung et al., 1996]. In this study, the initial $Zn^{2+}$ concentration of concentrate solution was 10% of the initial concentration of dilute solution. Therefore, during the initial period of each experiment the stack resistance gradually decreased with time because $Zn^{2+}$ concentration difference between the concentrate solution and the dilute solution was gradually decreased due to ion transfer. The stack resistance showed a minimum value when $Zn^{2+}$ concentration of the concentrate solution almost equaled that of the dilute solution. After showing a minimum value, the stack resistance was increased again because the concentration difference increased. In addition, as the initial concentration of dilute solution increased, the minimum value of the stack resistance was decreased.

Fig. 7 shows the effect of the initial concentration of dilute solution on the variation of the current with time. The current initially increased with time and decreased after showing a maximum value. In the initial period of each experiment, the current gradually increased with time because the stack resistance gradually decreased due to ion transfer. The current showed a maximum value when the stack resistance was the minimum value. After showing the maximum value, the current decreased because of an increase in the stack resistance. Comparied to Fig. 3, the dialytic rate was almost constant when the current remained over initial value of the current, whereas the dialytic rate decreased fast when the current fell to under the initial value. In addition, the maximum value of the current was observed when $Zn^{2+}$ concentration of the dilute solution was about half the initial concentration of dilute solution. Therefore, as the initial concentration of dilute solution was increased, it took a longer time for the current to reach the maximum value. It could be ex-

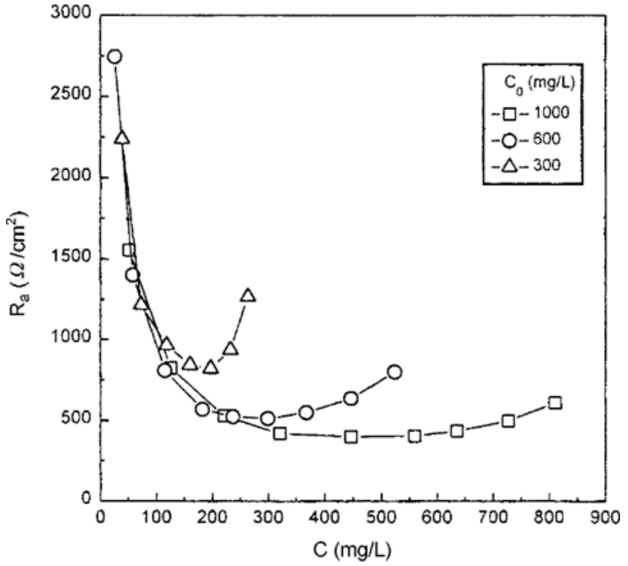

**Fig. 6. Effect of the initial concentration of dilute solution on the variation of the stack resistance with $Zn^{2+}$ concentration of the dilute solution ($V_G$=7 V, $V_L$=4 cm $s^{-1}$).**

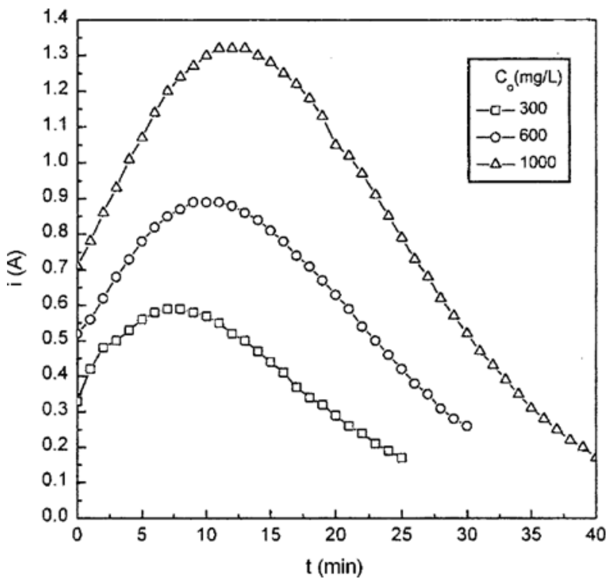

**Fig. 7. Variation of the current with time for the different initial concentration of dilute solution ($V_L$=6 cm $s^{-1}$, $V_G$=10 V).**

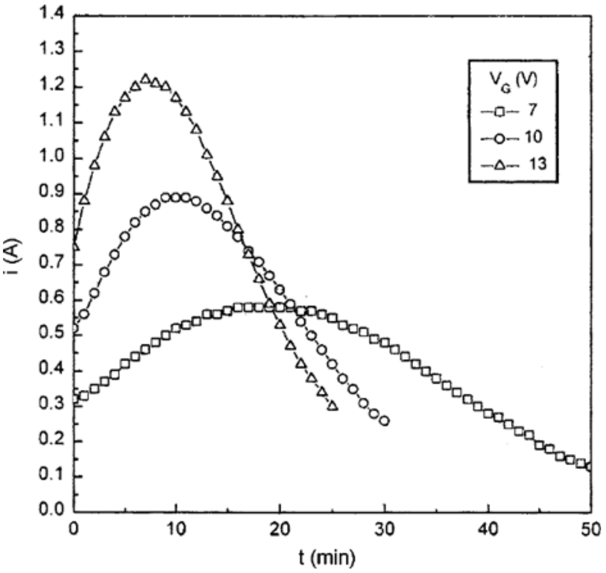

**Fig. 8. Effect of the applied voltage on the variation of the current with time ($C_0$=600 mg $L^{-1}$, $V_L$=6 cm $s^{-1}$).**

plained that as the initial concentration of dilute solution increased, the required time for a certain removal ratio was increased.

The effect of the applied voltage on the variation of the current with time can be seen in Fig. 8. As the applied voltage increased, both the increasing rate of the current before the maximum value of the current and the decreasing rate of the current after the maximum value of the current increased. Regardless of difference of the applied voltages, the maximum value of the current appeared when $Zn^{2+}$ concentration of the dilute solution was about half the initial concentration of dilute solution. When the initial concentration of dilute solution was constant, the maximum value of the current was increased with the applied voltage because the stack resistance was almost same at the time when the maximum value appeared.

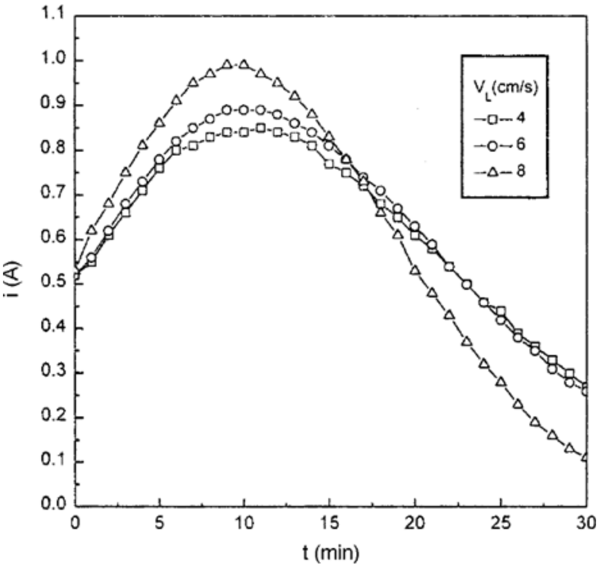

**Fig. 9. Effect of the flow velocity on the variation of the current with time ($C_0$=600 mg $L^{-1}$, $V_G$=10 V).**

**Table 1. Experimental condition**

| | |
|---|---|
| Initial concentration of dilute solution | 300, 600, 1,000 mg $L^{-1}$ |
| Initial concentration of concentrate solution | 30, 60, 100 mg $L^{-1}$ |
| Applied voltage | 7, 10, 13 V |
| Flow velocity | 4, 6, 8 cm $s^{-1}$ |
| Temperature | 23±2 °C |
| Effective membrane area | 100 $cm^2$/sheet |
| Number of cell pairs | 2 |
| Cell thickness | 0.75 mm |
| Volume of dilute solution | 1 L |
| Volume of concentrate solution | 1 L |

Fig. 9 shows the effect of the flow velocity on the variation of the current with time. As the flow velocity increased, the required time for the maximum value of the current was almost same, but both the increasing rate of current before the maximum value of the current and the decreasing rate of the current after the maximum value of the current were increased because the dialytic rate was increased due to the thinned boundary layer. In addition, as the flow velocity increased, the maximum value of the current was increased because the stack resistance was decreased due to the thinned boundary layer.

The effects of the applied voltage, the flow velocity and the initial concentration of dilute solution on the removal ratio for t=25 min are shown in Table 2. On all the operational conditions in this study, the removal ratio for t=25 min was greater than 62%. When $V_G$= 13 V, $C_0$=300 mg $L^{-1}$ and $V_L$=8 cm $s^{-1}$, the maximum value of the removal ratio (99.35%) could be obtained. As the applied voltage and the flow velocity increased, the removal ratio was increased. The removal ratio was increased with a decrease in the initial concentration of dilute solution. However, on the condition ($V_G$=7 V, $C_0$=1,000 mg $L^{-1}$ and $V_L$=4 cm $s^{-1}$), which gave the minimum value of the removal ratio, the removal ratio became 96.95% after 60 mim. The results mean that zinc ions in the model solutions were effectively removed by the electrodialysis system with CMX cation exchange membranes and AMX anion exchange membranes.

Table 3 shows the effects of the applied voltage, the flow veloc-

**Table 2. Effects of applied voltage, flow velocity and initial concentration of dilute solution on removal ratio for t=25 min**

(unit: %)

| Applied voltage (V) | Flow velocity (cm $s^{-1}$) | Initial concentration of dilute solution (mg $L^{-1}$) | | |
|---|---|---|---|---|
| | | 300 | 600 | 1000 |
| 7 | 4 | 69.67 | 64.97 | 62.24 |
| | 6 | 72.8 | 70.67 | 66.26 |
| | 8 | 75.85 | 74.73 | 66.46 |
| 10 | 4 | 93.17 | 91.53 | 84.39 |
| | 6 | 95.17 | 93.6 | 87.51 |
| | 8 | 98.29 | 97.48 | 89.44 |
| 13 | 4 | 95.04 | 93.03 | 84.56 |
| | 6 | 98.14 | 97.58 | 89.99 |
| | 8 | 99.35 | 97.92 | 94.5 |

**Table 3. Effects of applied voltage, flow velocity and initial concentration of dilute solution on energy consumption for $R_r$= 90%** (unit: Wh)

| Applied voltage (V) | Flow velocity (cm $s^{-1}$) | Initial concentration of dilute solution (mg $L^{-1}$) | | |
|---|---|---|---|---|
| | | 300 | 600 | 1000 |
| 7 | 4 | 1.135 | 2.246 | 3.174 |
| | 6 | 1.134 | 2.281 | 3.223 |
| | 8 | 1.140 | 2.304 | 3.241 |
| 10 | 4 | 1.655 | 2.841 | 4.747 |
| | 6 | 1.673 | 2.820 | 4.885 |
| | 8 | 1.657 | 2.629 | 4.831 |
| 13 | 4 | 2.300 | 4.309 | 7.263 |
| | 6 | 2.242 | 4.148 | 7.371 |
| | 8 | 2.278 | 4.175 | 6.932 |

ity and the initial concentration of dilute solution on the energy consumption for $R_r$=90%. As both the applied voltage and the initial concentration of dilute solution increased, the energy consumption was increased. However, the effect of the flow velocity on the energy consumption was negligible. A useful correlation for the prediction of the energy consumption was obtained by exponential multiple regression on the experimental results. In this study, the applied voltage, the flow velocity and the initial concentration of dilute solution were considered as the main parameters which influence the energy consumption. The energy consumption data were well correlated by Eq. (5) with correlation coefficient of 0.987.

$$E_C=4.796 \cdot 10^{-4}\, C_0^{1.00} V_G^{1.03} V_L^{0.01} \tag{5}$$
(R.C.=0.987, S.D.=0.348)

## CONCLUSIONS

In this study, to obtain useful data for treatment of the wastewater discharged from zinc electroplating processes, the effects of operating parameters, such as the initial concentration of dilute solution, the flow velocity of electrolyte solution and the applied voltage, on removal rate of $Zn^{2+}$ in the model solutions using an electrodialysis system were investigated.

Zinc ions in the model solutions were effectively removed by the electrodialysis system with CMX cation exchange membranes and AMX anion exchange membranes.

As the initial concentration of dilute solution, the flow velocity and the applied voltage increased, both the dialytic rate and the maximum value of the current were increased. The required time for the maximum value of the current was increased with an increase in the initial concentration of dilute solution. However, as the flow velocity and the applied voltage increased, the required time for a certain removal ratio was decreased.

The energy consumption for $R_r$=90% was increased as the initial concentration of dilute solution and the applied voltage were increased, whereas the effect of the flow velocity on the energy consumption was negligible. As the applied voltage and the flow velocity increased, the removal ratio for t=25 min was increased. The removal ratio was increased with a decrease in the initial concentration of dilute solution.

## NOMENCLATURE

C : concentration of dilute solution [mg $L^{-1}$]
$C_0$ : initial concentration of dilute solution [mg $L^{-1}$]
$d_e$ : equivalent diameter [cm]
$E_C$ : energy consumption [Wh]
I : current during time interval [A·h]
i : current [A]
$I_{lim}$ : limit current density [A/$cm^2$]
L : length of effective area for current [cm]
n : number of cell pairs
$N_u$ : Nusselt number
$R_a$ : apparent stack resistance [Ω·$cm^2$]
R.C. : regression coefficient
$R_e$ : Reynolds number
$R_r$ : removal ratio [%]
S : effective area of cell pairs [$cm^2$]
$S_c$ : Schmidt number
S.D. : standard deviation
t : time [s]
$V_G$ : applied voltage [V]
$V_a$ : apparent or observed stack voltage [V]
$V_L$ : flow velocity [cm $s^{-1}$]

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