



Influence of linear flow velocity and ion concentration on limiting current density during electro dialysis

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ABSTRACT

The effects of linear flow velocity and ion concentration on limiting current density (LCD), an important parameter in the design and operation of electro dialysis (ED) devices, was examined. As the applied voltage increased, reduction rate of the conductivity increased. However, it did not increase beyond a certain limit because of the effect of concentration polarization. The maximum applied potential of synthetic wastewater, prepared with NaCl (2,000 mg/L), was 15.4 V and the LCD was estimated to be 77.9 mA/cm². When the linear flow velocity of the diluate was increased by 1, 2, and 6 cm/s, LCD increased in direct proportion to the increase in electrical conductivity. The relationships between LCD, linear velocity, and ion concentration were established and parameters *a* and *b* were experimentally estimated. In contrast, as a result of changing the linear flow velocity of the concentrate by the same levels, there was no significant difference in the rate of reduction in conductivity. LCD measurements using synthetic wastewater containing various ions, including copper and nickel, and plating wastewater showed that the LCD was slightly higher because the ionic charge was higher than NaCl under the same conductivity conditions. Overall, LCD showed a tendency to be proportional to electrical conductivity.

Keywords: Electro dialysis; Linear flow velocity; Limiting current density; Metal; Potential

1. Introduction

Increasing use of water from rivers, lakes, and reservoirs due to population growth and industrial development in recent decades has resulted in corresponding amount of wastewater being generated; the treatment and reuse of wastewater is still a challenge worldwide [1,2]. To prevent over exploitation of water resources, water treatment systems for reclamation of water must be increasingly practiced [3,4]. Recently, several wastewater treatment processes have been proposed that minimize the impacts of secondary environmental pollutants and enable water reuse following its effective treatment for hazardous materials such as heavy metals [5,6].

One of these proposed processes for the reclamation of wastewater is electro dialysis [7]. The electro dialysis system is a membrane separation process in which ions in a solution move through an ion exchange membrane with an electric field as the driving force. Electro dialysis results in a solution with a high concentration of ions and treated (reclaimed) water with few a reduced ion concentration [5,8,9]. Membrane-based wastewater treatment technology reduces the consumption of chemicals, energy, and water as well as land space through the compact facility. In addition, minimization of wastewater and solid waste, and automation of operation can reduce operating costs [5,6,8–10]. Electro dialysis can concentrate heavy metals to a high

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concentration. Therefore, when combined with electrolytic extraction technology, heavy metals can be recovered at a minimum cost and recycled as raw materials for industrial processes [11,12].

In general, factors that influence the efficiency of separation of ions in electro dialysis include influent characteristics, composition ratio of ions, intrinsic properties of ion exchange membranes, electrical characteristics due to electropotential differences, hydrodynamic characteristics, physicochemical properties of metal ions, and operating conditions [13,14]. In particular, ion separation efficiency is determined by the hydrodynamic characteristics, including ion composition ratio, concentration, current density, and linear flow velocity of the influent [15,16].

When the anion or cation charged species is transported to the concentrate cell through the cation/anion exchange membrane, counter-ions are concentrated in the laminar boundary layer on the membrane surface adjacent to the diluate cell and are increased on the surface facing the concentrate cell [13,17]. In general, when the ion concentration at the surface of the cation and/or anion exchange membrane in the cell with the diluate is zero, the current density approaches the maximum value in the process, which is called LCD [17]. In the electro dialysis process, when the LCD is exceeded, the electrical resistance of the diluate increases sharply due to depletion of ion in the laminar boundary layer of the film surface. Therefore, when the electro dialysis process is operated beyond the LCD, the electrical resistance increases further and utilization efficiency of the current decreases [5,14]. The LCD is considered to be one of the most important design parameters that determine the efficiency of the process in the operation of the plant. Thus, it is important to provide basic information on these operating factors of the LCD in order to efficiently treat wastewater using electro dialysis [5,14,17].

The aims of this study were to (a) investigate the balance of counter-ion and non-ionic fluxes affecting the change of LCD in the separation of heavy metals in wastewater; (b) determine the effects of conductivity on the dilution–water flow rate and ion concentration on the LCD and their correlations; and (c) determine the effect of LCD on conductivity using various concentrations of ions and wastewater containing copper and nickel.

2. Experimental

2.1. Materials

The most important factors determining the performance of electro dialysis are the structure and electrochemical

characteristics of the ion exchange membrane. Separation performance indices, including electrical resistance, and selective and ion exchange capacities of ion exchange membranes depends on the composition of the backbone polymer, functional groups, and reinforcement. In this study, Neosepta CMX-SB (a cation exchange membrane) and AMX-SB (an anion exchange membrane) of ASTOM (Tokyo, Japan) among a series of commercial membranes were used. The physical properties of the ion exchange membrane are summarized in Table 1 [18]. The ion exchange membranes were treated with distilled water at room temperature for 1 h before use, followed by treatment with 0.36% HCl for 1 h to remove impurities. Finally, the treated ion exchange membranes were immersed in the concentrated electrolyte solution at $25^{\circ}\text{C} \pm 1^{\circ}\text{C}$ for 24 h, prior to being used for the experiments.

2.2. Experimental set-up

A schematic diagram of the electro dialysis system (CJ-S3, CHANG JO TECHNO Co. Ltd., Korea) used in this experiment is shown in Fig. 1. The device consists of a dilution/concentration/electrolyte solution tank, an ion exchange stack, a diluate/concentrate/electrolyte feed pump, and a power supply. The solution tanks were made of polyvinyl chloride with a volume of 500 mL. The ion exchange stack was 115 mm (W) \times 225 mm (H) and consisted of an anion/cation permeable membrane, dilution stream spacer, and concentrate spacer. A cathode/anode electrode was installed at each end of the stack to conduct direct current. A silicone rubber seal was used to prevent leakage from each compartment, and a steel block for compressing the ion exchange membrane and spacer was installed at the upper and lower ends, and then screwed into place. The effective surface area of the anion and cation permeable membrane was 55 cm², and the effective surface area of the stack of five pairs was 275 cm². The polypropylene spacer was fabricated to have a tortuous path flow. Channel flows in the dilution and concentration compartments were independent of each other, and the path of each channel flow was maximized. The dimensions of each electrode were 45 mm (W) \times 105 mm (H) \times 2 mm (D). To prevent corrosion, the electrode was plated with a mixture of platinum and ruthenium. The power supply (P3030, ADV @ NTEK Co. Ltd., Korea) was capable of supplying 3 A (DC) at 30 V.

2.3. Wastewater

In this study, wastewater from electroplating facilities was investigated to determine the copper and nickel

Table 1
Physical properties of the Astom ion exchange membranes used [18]

Membrane	Type	Thickness (mm)	Electrical resistance ^a ($\Omega \text{ cm}^2$)	Burst strength (kPa)	Counter-ion transport number ^b
CMX-SB	Strongly basic	0.14–0.20	1.8–3.8	40	>0.98
AMX-SB	Strongly acidic	0.12–0.18	2.0–3.5	30	>0.98

^aFor membranes equilibrated in 0.5 equiv/L NaCl solution at 25°C.

^bFor a current density of 200 A/m² in 0.5 equiv/L NaCl solution at 25°C.

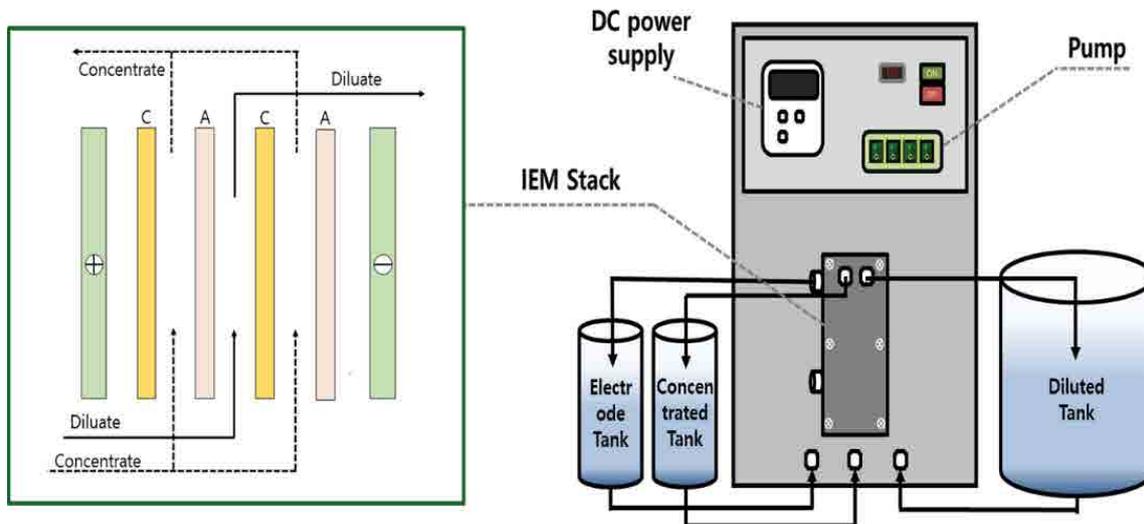


Fig. 1. Schematic representation of the lab-scale electrodiagnosis system.

concentrations. Concentrations of copper and nickel were 22–300 and 24–120 mg/L, respectively. The electrical conductivity was $6,000 \pm 300 \mu\text{S/cm}$ and pH was 2.2 ± 0.2 . Synthetic wastewater was prepared by using nickel (II) chloride hexahydrate ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, Sigma-Aldrich, Saint Louis, MO, USA) and copper (II) chloride dihydrate ($\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$, Sigma-Aldrich, Saint Louis, MO, USA), and sodium chloride (NaCl , Sigma-Aldrich, Saint Louis, MO, USA) was used to adjust the total electrical conductivity. In the experiment using divalent anions, synthetic wastewater was prepared by using nickel (II) sulfate hexahydrate ($\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, Sigma-Aldrich, Saint Louis, MO, USA) and copper (II) sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, Sigma-Aldrich, Saint Louis, MO, USA). In experiments evaluating the effect of LCD and linear flow velocity, synthetic wastewater was prepared by adjusting the conductivity of the solution using only NaCl . Table 2 summarizes the properties of synthetic wastewater.

2.4. Operation conditions

The lab-scale electrodiagnosis system used Astom's CMX-SB and AMX-SB membranes. In the LCD experiment, synthetic

wastewater with a conductivity of 2,000–12,000 $\mu\text{S/cm}$ prepared using NaCl was used. The current and resistance changes were measured at each potential; the potential was increased by 1 V up to 10 V, followed by an increase of 2 V up to 30 V with a stabilization interval of 5 min.

We also measured changes in current and resistance as described previously, using calcium chloride (2,000 mg/L), iron chloride (2,000 mg/L), and copper and nickel (at increasing concentrations of 20, 100 and 200 mg/L) [19]. The linear flow velocity of the diluate and the concentrate was adjusted to 1, 2, and 6 cm/s for the experimental examination of the influence of changing the linear flow velocity of the membrane on LCD. The electrode solution used was (w/w) Na_2SO_4 [20,21].

2.5. Analytical methods

Ion separation efficiencies were investigated using the changes in conductivities of the diluate and the concentrate during electrodiagnosis. To remove the suspended solids in all samples, a 0.45- μm filter was used. The pH and conductivity of all samples were measured at regular intervals using an

Table 2
Characteristics of the synthetic wastewater

	Main cation	Main anion	Linear flow velocity (cm/s)
NaCl	Na 1,000, 2,000, 4,000, 5,000, 6,000 mg/L	Cl^-	1, 2, 4, 6
CaCl_2	Ca^{2+} 2,000 mg/L	Cl^-	4
FeCl_2	Fe^{3+} 2,000 mg/L	Cl^-	4
$\text{CuCl}_2(20) + \text{NiCl}_2(20)$	Cu^{2+} 20 mg/L, Ni^{2+} 20 mg/L	Cl^-	4
$\text{CuSO}_4(20) + \text{NiSO}_4(20)$	Cu^{2+} 20 mg/L, Ni^{2+} 20 mg/L	SO_4^{2-}	4
Wastewater ($\text{Cu}20 + \text{Ni}20$)	Cu^{2+} 22 mg/L, Ni^{2+} 24 mg/L	–	4
$\text{CuSO}_4(100) + \text{NiSO}_4(100)$	Cu^{2+} 100 mg/L, Ni^{2+} 100 mg/L	SO_4^{2-}	4
$\text{CuSO}_4(200) + \text{NiSO}_4(200)$	Cu^{2+} 100 mg/L, Ni^{2+} 100 mg/L	SO_4^{2-}	4

Orion 5 Star instrument (Thermo Fisher Scientific Inc., USA). The decrease in conductivity with time in the dilution tank was determined using Eq. (1):

$$\frac{C_t}{C_0} = e^{-k_1 t} \tag{1}$$

where C_0 and C_t correspond to the conductivity ($\mu\text{s}/\text{cm}$) of the diluate at the beginning of the experiment and at time t (min), respectively, and k_1 is a first-order constant [22].

3. Results and discussion

3.1. Effects of LCD

In electro dialysis, upon the application of the electrical potential gradient, anions migrate toward the anode, cations migrate to the cathode, and cross the anion and cation exchange membranes, thereby leading to the recovery of the metals. Therefore, the applied voltage affects the process, and by increasing the voltage, the efficiency of separation increases.

Fig. 2 shows the change in conductivity as a function of the applied potential. The first-order rate constant, which represents the separation rate of the electro dialysis, was derived by regression analysis of the experimental data. As the applied potential increased, the rate of ion transfer through the membrane also increased, thereby increasing the reaction rate constant (Fig. 2). However, there was no significant difference in rate constants between 18 and 24 V (Fig. 2). The possible reason for this observation was the concentration polarization that occurred at the surface of the film when the voltage exceeded 18 V [5,23]. When the current density exceeded the LCD during electro dialysis, the electrical resistance of the solution increased, and the efficiency of the process rapidly deteriorated. In addition, H^+ and OH^- produced by the dissociation of water can cause the pH of the solution on the surface of the membrane to change; scaling can also occur as a result of the deposition of metal hydroxide on the surface of the ion exchange membrane [17].

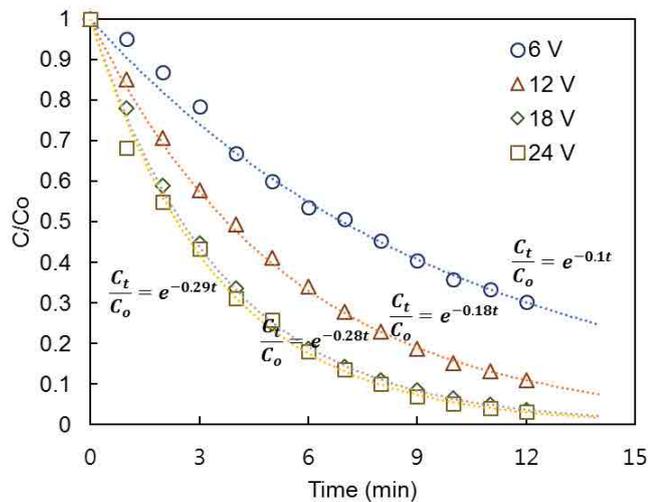


Fig. 2. Change of treatment rate as a function of the applied voltage.

If the ion exchange membrane is not sufficiently resistant to an acidic or alkaline environment, it may degrade and its performance reduced [5]. Therefore, LCD is a very important parameter in the electro dialysis process.

The LCD can be determined by measuring the electric resistance in the dilution compartment as a function of current, potential, or current density [24]. Fig. 3 shows the calculation of LCD, using the relationship between resistance and current in the diluate cell. The current at the point where the slope changes due to concentration polarization on the membrane surface is the critical current [14,24]. The maximum value of the potential that can be applied and the LCD at this point were estimated to be 15.4 V and

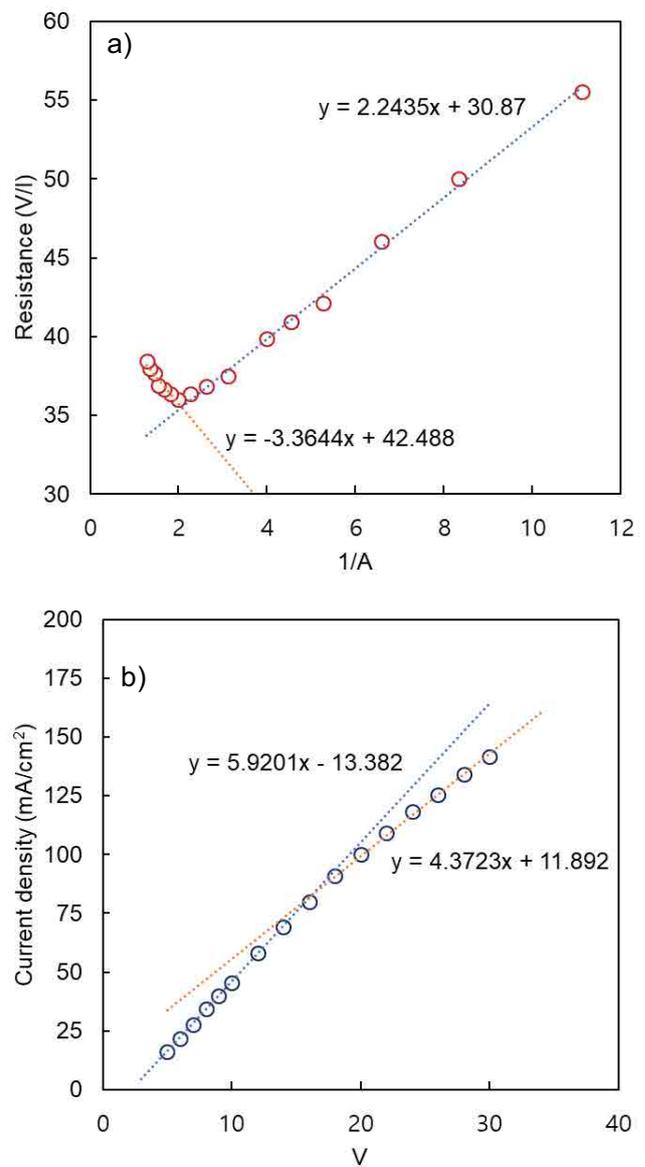


Fig. 3. Determination of the limiting current density: (a) the change in resistance according calculated as the reciprocal of the current and (b) the change in current as a function of the applied potential (diluate solution: conductivity 2,000 $\mu\text{s}/\text{cm}$ with NaCl, cell pair: CMX and AMX, linear velocity 1 cm/s).

77.9 mA/cm², respectively. Therefore, since the electro-dialysis system must operate at currents below the LCD, the appropriate potential was determined to be 15 V.

3.2. Effects of flow velocity

In physical-based models, LCDs have empirical correlations with fluid velocity and bulk concentrations of diluate [25]. Some researchers have reported that these can be expressed as a function of velocity with constant coefficients, which is influenced by fluid dynamics, including linear flow velocity [24,26]. However, calibration is necessary because most experiments were performed on a specific electrochemical cell using only one ion exchange membrane and one solution. Therefore, in practice, it is necessary to measure LCD under the conditions that are applicable for various wastewaters [25,27].

The solution linear flow velocity in the dilution tank can promote turbulence, reducing concentration polarization [28,29]. In this study, the LCDs were estimated at 1, 2, and 6 cm/s linear flow velocity of the diluate with varying conductivity of 2,000, 4,000, 8,000, 10,000, and 12,000 μs/cm using NaCl. The LCD also increased proportionally as conductivity increased for the same linear flow velocity (Fig. 4). The LCD also showed a tendency to increase with increasing the linear flow velocity even at the same conductivity.

Mass transfer coefficients are generally unknown, but can be calculated from the experimentally determined limiting current [30]. The mass transfer coefficient *k* can be written as a non-linear function of the linear velocity as follows [31]:

$$k = a(v_d)^b \tag{2}$$

where *v_d* is the linear flow velocity(cm/s) of diluate, and *a* and *b* are empirical parameters. Using Eq. (2), the basic empirical equation that provides a reasonable result within practical limits as a function of concentration and linear flow velocity is widely accepted, and can be expressed as follows [17]:

$$LCD = kC = a(v_d)^b C \tag{3}$$

$$a(v_d)^b = ae^{b \ln(v_d)} \tag{4}$$

where *v_d* is the linear flow velocity (cm/s) in the diluate and *C* is the electrical conductivity (μs/cm) of the influent.

In this study, coefficients *a* and *b* were estimated to be 0.0085 and 0.84, respectively, in the linear velocity range between 1 and 6 cm/s. Aider et al. [29] reported that if the solution was well mixed in electro-dialysis, combined with an ultrafiltration membrane, the solution linear flow velocity did not significantly affect electrolyte transport through the membrane. However, other researchers have found that the linear flow velocity of the solution varies the Reynolds number and pressure; as a result, that there is a significant difference in the solute permeation of the membrane depending on the electric field [32,33].

In contrast, when the effect of the linear flow velocity of the concentrate on ion separation efficiency of the electro-dialysis process was examined, and the flow rate of the dilution tank was fixed at 6 cm/s, separation efficiency was not significantly affected when the flow rate of the dilution tank was increased to 1, 2, or 6 cm/s (Fig. 5).

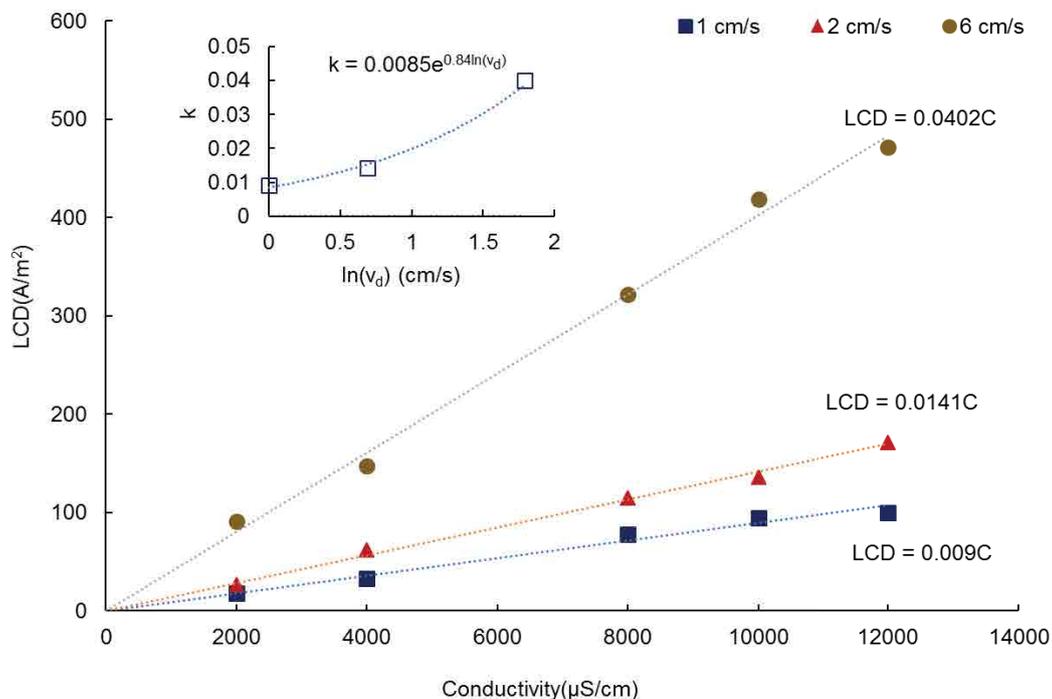


Fig. 4. LCD as a function of conductivity concentration determined at different linear flow velocities of the diluate and determination of coefficients *a* and *b*.

3.3. Impact of various ions on LCD

The current was mostly transferred through movement of counter-ions except co-ions. Since ion exchange membranes contain more counter-ions than are in solution, ion exchange membrane migration is faster in solution. Therefore, the concentrations of ions in the diluate compartment are reduced and the concentrations in the concentrate compartment are increased. Consequently, concentration gradients are formed (called the diffusion boundary layer) in the region adjacent to the ion exchange membrane. At this point, the concentration at the surface of the ion exchange membrane of the diluate compartment reaches a value close to zero, resulting in polarization [5,23]. Therefore, the LCD can differ depending on the counter-ions present in the solution [13,20].

Actual plating wastewater had various ions and a different composition ratio compared with the artificial wastewater. Therefore, there may be differences in LCD for these two types of wastewater. To explore these differences, LCDs were investigated using calcium chloride and iron chloride, and various concentrations of copper and nickel, using chloride ions and sulfate ions in synthetic wastewater as counter-ions (Fig. 6).

In this experiment, it was confirmed that there was a difference in the LCD depending on the concentration of the metal ions in the solution and composition of the counter-ions. Based on the conductivity of the synthetic wastewater using NaCl, the calcium chloride and the ferric chloride at the same concentration resulted in a higher LCD, according to the increase in conductivity, but they did not deviate largely from the baseline. In the solution containing copper chloride and nickel chloride, the LCD was 61.5 mA/cm², a value slightly lower than the LCD of NaCl (65.6 mA/cm²). Despite having a conductivity of 2,500 μ S/cm², the LCD was almost equal to that for NaCl, at 68.2 mA/cm². Furthermore,

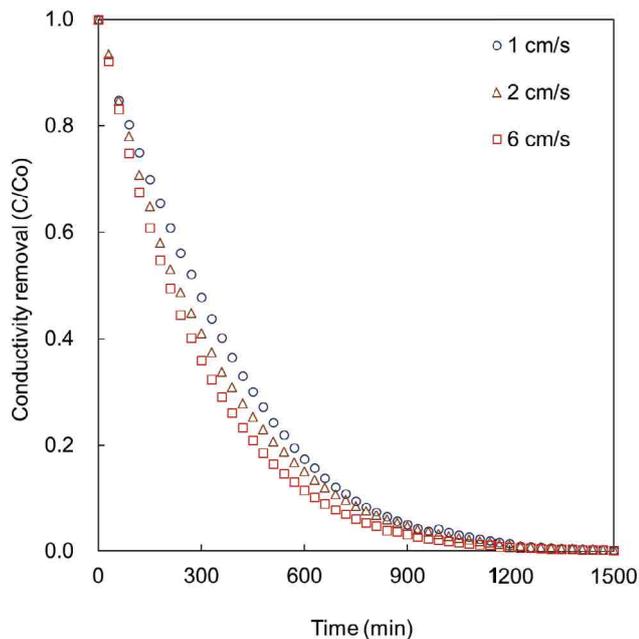


Fig. 5. Concentration change with linear flow velocity in the concentrate cell.

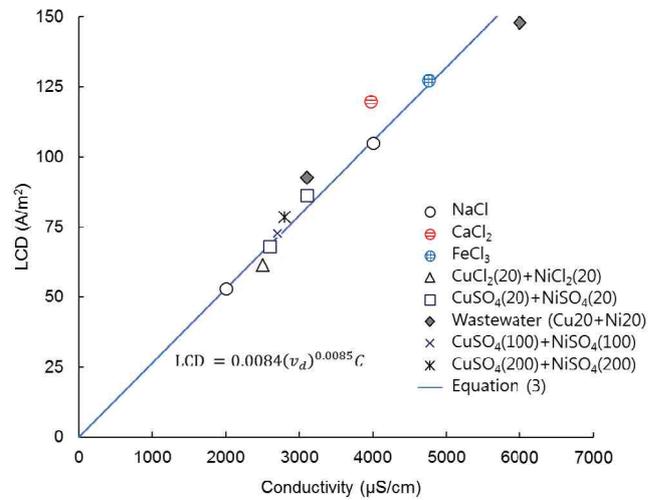


Fig. 6. Relationship between conductivity and LCD of a solution containing various ions and plating wastewater.

solutions of higher concentrations of copper chloride and nickel chloride exhibited a generally higher LCD compared with that of sodium chloride of the same conductivity.

These differences could be attributed to the slower transport of sulfate ions in the membrane due to the greater affinity of these ions for chloride ions than for AMX-SB [13,20]. Charged membranes contain many ionizable groups within their structure. Since the charge of the membrane plays a very important role in the transport of the electrolyte, ion mobility is greatly influenced by the fixed charge on the ion exchange membrane. Monovalent ion binds with one fixed ion on the ion exchange membrane, and replaces one adjacent fixed ion. However, the divalent ion binds to two fixed ions on the membrane, and replaces two adjacent fixed ions; the relative migration of the divalent ions is slow [34,35]. As a result, the flux of cations is decreased due to the slow migration of the divalent counter-ion (sulfate) [13,20,35]. Nevertheless, the overall tendency was increased LCD as the conductivity increased.

4. Conclusions

The applied voltage used in electrodialysis is a very important driving factor for determining energy consumption as well as separation efficiency of metal ions. The rate constant increased proportionally with the increase of the electric potential at 2,000 μ S/cm, but the rate constant and LCD showed little difference above 18 V. The LCD had a proportional correlation with the dilution water flow rate and influent concentration. The linear flow velocity and concentration dependencies of the critical current density were evaluated and the parameters a and b were estimated to be 0.0085 and 0.84, respectively. LCD increased with the increase in flow rate, and application of increased applied voltage led to a reduction in the treatment time. In addition, it is possible to effectively isolate the highly concentrated metal solution. Therefore, in cases of high concentration wastewater flows, increasing the linear flow velocity and voltage of the diluate are effective methods to shorten

processing times. When the flow rate of the concentrated water was increased, ion separation efficiency of the process did not differ substantially. LCD measurements using various cations, anions, and copper and nickel containing synthetic and actual plating wastewater, showed slight differences under the same conductivity values, depending on the concentration of metal ions and counter-ions; LCD tended to be proportional to conductivity.

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