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CONSIDERING THE ION TYPES WHILE EVALUATING THE PERFORMANCE CRITERIA IN ELECTRODIALYSIS SYSTEMS

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Abstract

The removal/recovery of heavy metals has been conducted through a number of processes. Electrodialysis is one of the most important ones among others in diluting and concentrating the ionic pollutants. In this study, the performance criteria of the electrodialysis system such as removal efficiency, current efficiency, amount of ion removal and specific electrical energy consumption (SEEC) have been investigated in the presence of more than one pollutant species in wastewater. With constant operating parameters of: 200 mL/min flow rate, natural pH of water which is $\text{pH} \approx 6$ and 0.05 M electrolyte concentration prepared by NaCl, 30, 45 and 60 V of potentials has been applied to the wastewater containing 6 mg/L lead, nickel and copper ions. Experimental results indicated that removal efficiencies of different ion types with an equal concentration in a solution could be different depending on hydration shells, hydration bonds, hydration energies and ionic mobilities of the ions. The percent of current used by each ion type from total current transferred in the system has been calculated by transference number. Different ions had different current efficiencies and specific electrical energy consumptions depending on the transference number of the ion.

Keywords: current efficiency, electrodialysis, hydration shells, ionic mobilities, specific electrical energy consumptions

Received: February, 2020; *Revised final:* July, 2020; *Accepted:* July, 2020; *Published in final edited form:* January, 2021

1. Introduction

Heavy metals are extremely toxic to living organisms (Chaturvedi et al., 2018). For decades heavy metal pollution caused numerous diseases and became a huge problem for the environment (Ma et al., 2018). The key origins of metals in water are battery, mining and metal plating industries (Gopi et al., 2018). Many treatment methods had been applied to the heavy metal-containing wastewaters such as biosorption (Demey et al., 2018), adsorption (Huang et al., 2018; Son et al., 2018), membrane filtration (Huang et al., 2017), chemical precipitation (Ku and Jung, 2001), electrochemical processes (Martins et al., 2012; Nanseu-Njiki et al., 2009) or hybrid systems (Gonzalez-Munoz et al., 2006; Kaykioglu 2016; Mahdavi et al., 2018).

Electrodialysis (ED) have the ability to separate ionic species from wastewater with an

applied current or voltage (Luiz et al., 2018). With applied current/voltage, a potential gradient is formed. This potential migrate ions from solution to pass through cation exchange membranes (CEMs) or anion exchange membranes (AEMs) depending on their charge (Ward et al., 2018). Because of the nature of the ED process, ED cell is consecutively divided by multiple CEMs and AEMs. These membranes create individual cells and two solutions types are formed, one is diluted cell (wastewater after treatment) and the other one is concentrated cell (recovered ions) (Scarazzato et al., 2017). Then the recovered ions in the concentrated cell are subjected to a second treatment to remove pollutants. Chemical precipitation (Li et al., 2019), flotation (Hoseinian et al, 2020) and electrocoagulation (Doggaz et al., 2019) processes can be used for the final treatment of ions collected in the concentrated cell of the ED stack. Recent trends in electrodialysis are production of new

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types of ion exchange membranes such as homogeneous, inorganic-organic (hybrid), bipolar, amphoteric and mosaic membranes (Xu, 2005).

Electrodialysis is a low energy consuming technique because it is a no-phase transition process (Nie et al., 2017). Some advantages of ED are: operates at low pressures, the process is very quiet and simple and operational maintenance costs are low. ED also has a big disadvantage as a treatment process that is only applicable to ionic contaminants (Scott, 1996). A simple ED stack consists of electrodes (anode & cathode), ion exchange membranes (AEMs & CEMs), power supply, ED cell - which contains frames, separators and plates, and pumps - for recycling diluted and concentrated cell.

Removal or recovery of different types of heavy metal pollutants in an ED process has been examined by researchers intensively. Lead (Gherasim et al., 2014), cadmium (Marder et al., 2004), zinc (Babilas and Dydo, 2018), copper (Caprarescu et al., 2015; Peng et al., 2011), silver (Guvenc and Karabacakoglu, 2005), chromium (dos Santos et al., 2019; Lambert et al., 2006) and nickel (Benvenuti et al., 2014) are among these heavy metal pollutants. Although the amount of pollutants eliminated in the ED systems and the removal efficiencies (RE %) have been widely studied, the individual effects of the ionic species to the performance criteria of the treatment have not been mentioned enough. In particular, the current efficiency (CE%) and the SEEC values, which are the performance criteria of an ED system have not been investigated in a parallel manner.

Within this study we investigated which mechanisms and models are effective in the removal of individual ion types and how effective they are in the performance of the whole ED system. The removal efficiency, the amount of ion removal, the current efficiency (CE %) and the SEEC value of the lead, copper, and nickel were examined and presented in a comparative manner.

2. Material and methods

2.1. Experimental setup

The experimental cell (ElectroCell A/S), provided from Microflowcell has platinum covered titanium material as electrodes with 0.01 m² active area. The system had three compartments. The wastewater circulation is generated with peristaltic pumps. The DC power supply was used to satisfy electrical demand. The specifications of ion exchange membranes used in the study can be seen in Table 1.

Synthetic wastewater with a concentration of 6 mg/L including 2 mg/L Pb²⁺ from lead (II) nitrate (Pb(NO₃)₂), 2 mg/L Ni²⁺ from (II) nitrate hexahydrate (Ni(NO₃)₂·6H₂O) and 2 mg/L Cu²⁺ from copper (II) sulphate pentahydrate (CuSO₄·5H₂O). The wastewater had pH ≈ 6 and, 0.05 M sodium chloride (NaCl) containing solution was used as electrolyte. The wastewater and electrolyte solutions were recirculated to the system as batch flow with a flow rate of 200 mL/min. The setup for the experiments can be seen in Fig. 1.

Table 1. Specifications of the membranes used in the study

Membrane type	Thickness (µm)	Ion exchange capacity (meq/g)	Water content (%)	Conductivity (S/cm)	Electrical capacitance (ohm-cm ² , 25 °C)
Nafion 117	183	0.94	5	0.083	n/a
Neosepta ACM	120	1.4 – 1.7	13 – 18	n/a	2.5 – 5.5

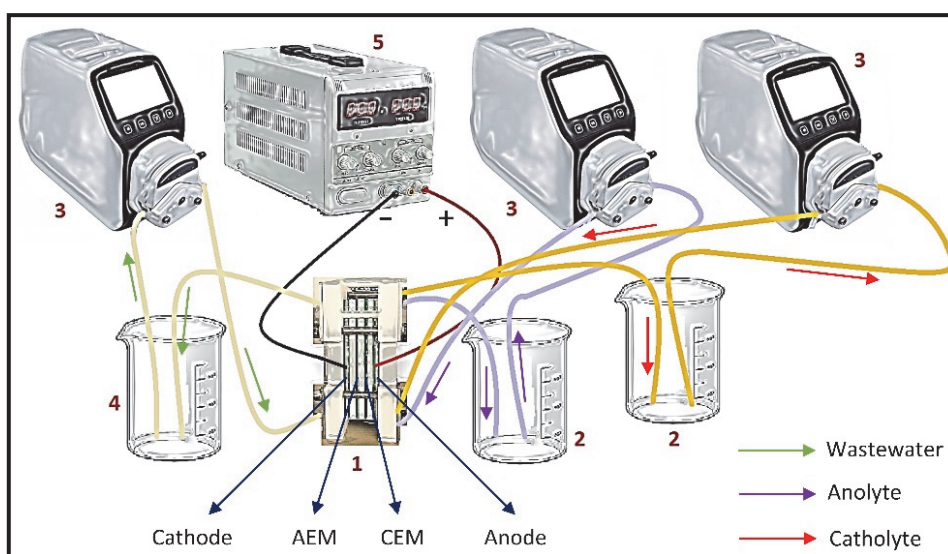


Fig. 1. Setup for the experiments (1 – Electrodialysis cell, 2 – electrolytes, 3 – pumps for circulation, 4 – wastewater, 5 – power generator)

2.2. Analysis

Each experiment was performed for six hours. Sampling was done at the tenth, thirtieth and sixtieth minutes within the first 60 minutes. The frequency of sampling after the first hour has been increased to 1 hour. Atomic Absorption Spectrophotometer (Thermo Scientific AAS ICE 3300) was used for heavy metal analysis.

The removal efficiency calculated with the Eq. (1) (Marder et al., 2004):

$$RE \% = ((C_0 - C_i) / C_0) 100 \quad (1)$$

where: C_0 is the initial concentration of metal ion; C_i is the concentration of metal ion at time t .

With Eq. (2) (Yu et al., 2000) current efficiency was determined.

$$CE \% = (z F \Delta n / N I \Delta t) 100 \quad (2)$$

where: z is the valence of the ion; F is Faraday constant which is $96485 \text{ A s mol}^{-1}$; Δn is the number of moles removed in time t (mole); N is the number of compartments in ED stack; I is the electrical current applied (A); Δt is the time interval (s).

Transference number of a cation (t_+) in a solution was calculated by Eq. (3) (Zugmann and Gores, 2014):

$$t_+ = \lambda_+ / A_0 \quad (3)$$

where: λ_+ is the limiting molar ionic conductivity of cation; A_0 is the limiting molar ionic conductivity of the total solution.

Limiting molar conductivity of a solution can be expressed by Eq. (4) using the Nernst – Einstein equation (Daintith, 2008):

$$A_0 = (F^2 / RT) (v_+ z_+^2 D_+ + v_- z_-^2 D_-) \quad (4)$$

where: R is the gas constant; T is the thermodynamic temperature; v_+ and v_- are the number of cations and anions per formula unit of electrolyte; z_+ and z_- are the valences of the ions; D_+ and D_- are the diffusion coefficients of the ions.

The diffusion coefficient of charged particles (D) can be calculated by the Eq. (5) (Waigh, 2014):

$$D = k T u / z \quad (5)$$

where: k is the Boltzmann's constant; u is the ionic mobility.

Associating Eqs. (3-5) will result in the following equation (Eq. 6):

$$t_+ = (v_+ z_+ u_+) / (v_+ z_+ u_+ + v_- z_- u_-) \quad (6)$$

where: u_+ and u_- are the ionic mobilities of cation and anion respectively.

Since all ions are in the same solution that has a molar concentration of c the transference number of an ion can be calculated with Eq. (7):

$$t_+ = (z_+ u_+ d_+ c) / (z_+ u_+ d_+ c + z_- u_- d_- c) \quad (7)$$

where d_+ and d_- are the molar fractions of cation and anion respectively.

Specific electrical energy consumptions ($SEEC$) was calculated with Eq. (8) (Abou-Shady et al., 2012).

$$SEEC (W h mg^{-1}) = E \int_0^t I dt / \Delta m \quad (8)$$

where: Δm is the amount of ions removed during time t (mg).

3. Results and discussion

An electro dialysis cell can be operated potentiostatically or galvanostatically. In an ED process voltage or current determines the amount of ions passing through the ion-selective membranes (Guvenc and Karabacakoglu, 2005). Since it is difficult to operate an ED system at high voltage when the pollutant concentration in the diluted cell is low, our ED system was operated potentiostatically to keep the system at low voltage values (Gherasim et al., 2014). One issue to consider before applying a voltage to the system is the limiting current density (LCD). The largest amount of current that can move through a membrane region is the LCD. Exceeding this phenomenon means wasting current, leading to energy losses (Ravikumar et al., 2013). Fig. 2 shows voltage – current curves determined for 200 mL/min flow rate, 6 mg/L pollutant concentration, 0.05 M electrolyte concentration and with a pH of 6. As can be seen in Fig. 2, applying a voltage of 60 V to the ED cell would be adequate for not exceeding LCD value. Depending on LCD values 30, 45 and 60 V were studied to certify and compare the intended results.

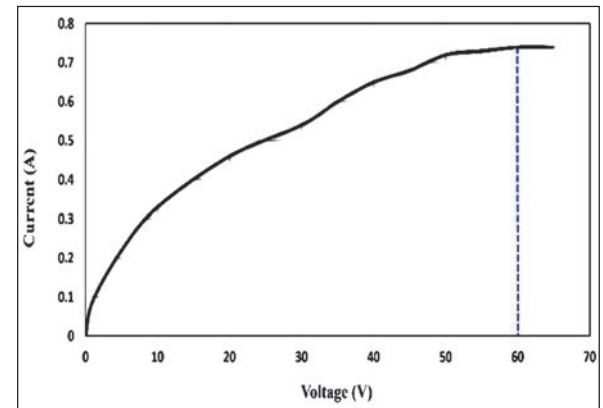


Fig. 2. Voltage – Current curve for LCD determination (pH \approx 6, electrolyte concentration = 0.05 M NaCl, Flow rate = 200 mL/min)

3.1. Removal and current efficiencies

The removal efficiency (RE%) which has to be taken into account when analyzing the performance of the electro dialysis system indicates how much percentage of the pollutant has been eliminated at the end of the operating period. The current efficiency (CE%) is the proportion of the quantity of ions extracted from the system, measured experimentally, to the potential quantity of ions that should be removed. It depends on the Faraday's law (Lux et al., 1998).

Removal and current efficiencies of the total system and the ions types depending on altering voltages under mentioned constant operational conditions have been examined and can be seen in Fig. 3. It was observed that the removal efficiencies of all three types of metal ions and the total ion removal increased with increasing voltage. This phenomenon can be explained as the enhanced electrical driving

force improving the ion removal (Guvenc and Karabacakoglu, 2005). But the most remarkable part is the differences in the removal efficiencies of all three types of ions. For all applied potentials, the removal efficiencies are arranged as $Pb^{2+} > Cu^{2+} > Ni^{2+}$. When regarding removal efficiencies in 60 V, the removal efficiencies of lead, copper, and nickel ions were determined as 77.4233%, 72.5542%, and 68.1579%, respectively. There are a wide variety of reasons for this distinction in the removal efficiencies.

One of these reasons was the hydration bonds of the ions with water molecules. In a solution, water molecules surround ions. A water molecule is a structure in which the hydrogen and oxygen atoms are joined at an angle of 104.5° . This configuration causes a very weak polarization in water molecules. Depending on the polarity, water molecules form layers called hydration shells around cations and anions. This formation is illustrated in Fig. 4.

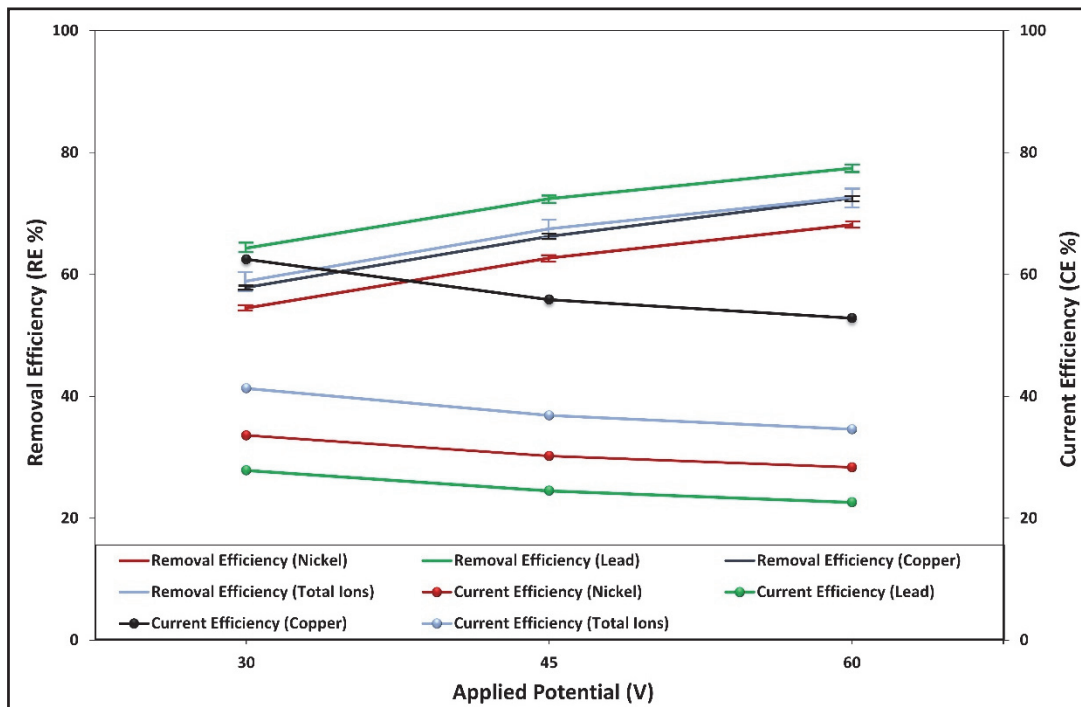


Fig. 3. Removal efficiencies and current efficiencies of ions and total solution (Wastewater conc. = 6 mg/L, Flow rate = 200 mL/min, pH ≈ 6, electrolyte conc. = 0.05 M NaCl, t = 6 h)

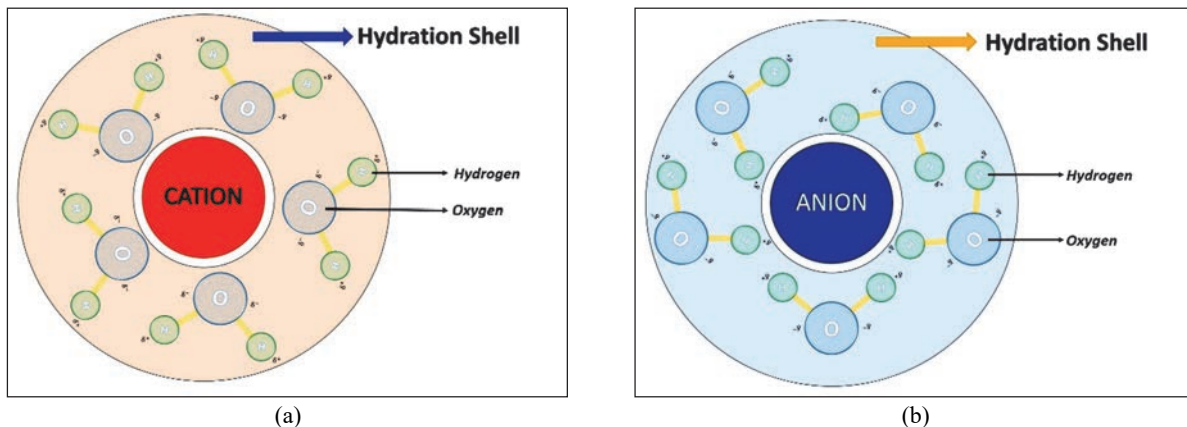


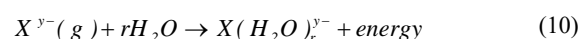
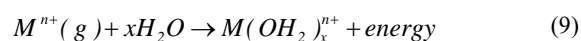
Fig. 4. Hydration shells around (a) cation and (b) anion

Table 2. Parameters affecting the removal efficiencies of lead, copper and nickel ions

Parameter	Ion		
	Pb ²⁺	Cu ²⁺	Ni ²⁺
Removal Efficiency (%)	77.423	72.554	68.158
Ionic Radii** (Å)	1.19	0.73	0.68
Hydration Energy*** (kJ mol ⁻¹)	1481	2100	2105
Ionic Mobility Value*	0.480	0.385	0.337

Note: * Larger is better for RE % performance (Relative to K⁺ (Ionic Mobility of K⁺ = 7.62x10⁻⁸ m² s⁻¹ V⁻¹)); ** Larger is better for RE % performance; *** Smaller is better for RE % performance.

Positively charged ions are attracted to the negative side of the water molecule (oxygen) and negatively charged ions affect the positive side (hydrogen) of the water molecule (Tansel et al., 2006). The number of water molecules that covers the ion and moves with it is called hydration number (*n*) (Impey et al., 1983). A hydrated ion can be expressed as in Eqs. (9-10) for cations (*Mⁿ⁺*) and anions (*X^{y-}*) respectively (Whitten et al., 2009):



Ions with small ionic radii set up stronger hydration bonds and it is more difficult to split them from their hydration shells. Ions with a large ionic radius can be more easily separated from the hydration shells because they have weaker hydration bonds (Tansel et al., 2006). When different ion types pass through membrane pores, those with large ionic radii have easier transition and have higher removal efficiencies because they can split from their hydration shells well. The ionic radii's of heavy metal types used in the study was 1.19 Å for Pb²⁺, 0.73 Å for Cu²⁺ and 0.69 Å for Ni²⁺ (Ruben, 1998). Regarding the ionic radii, Pb²⁺ showed the highest removal efficiency and Ni²⁺ showed the lowest removal efficiency.

The other reason for the differences in removal efficiencies was hydration energy. Hydration energy is described as the energy change that occurs as a result of the hydration of one-mole ion in gas form (Whitten et al., 2009). Hydration energy can also be described as the strength of the hydration bond of an ion with water molecules. Ions with low hydration energy can easily be separated from its hydration shell. The hydration energies of the ions in the feed solution are 1481 kJ/mole, 2100 kJ/mole, 2105 kJ/mole for Pb²⁺, Cu²⁺ and Ni²⁺ ions respectively (Smith, 1977). It can be concluded that with the lowest hydration energy Pb²⁺ has ability to easily split from its hydration shell to pass the other side of the CEM. It should be noted that hydration energy and the ionic radius of ions are directly related.

The last reason for the removal efficiency distinctions was ionic mobility. Ionic mobility is described as the measure of how rapidly an ion can move under the influence of an electric field depending on its size, shape, and charge (Jiang and Robinson, 2013). Ions with greater ionic mobility have

the ability to move faster with the applied current. The ionic mobilities of ions used in the study were 0.480 for Pb²⁺, 0.385 for Cu²⁺ (Dean, 1999) and 0.337 for Ni²⁺ (Vanysek, 2002) relative to K⁺ ion which has a mobility value of 7.62x10⁻⁸ m² s⁻¹ V⁻¹. With respect to all three aforementioned grounds, removal efficiencies of the ions are arranged as Pb²⁺ > Cu²⁺ > Ni²⁺ with the values of 77.423%, 72.554%, 68.158% respectively. Table 2 summarizes the parameters affecting the removal efficiencies of all these three metal ions.

As can be seen in Fig. 3 current efficiency has a downward trend with growing voltage for each ion type and for the total ion removal. This phenomenon can be explained as the transition of ions is not increasing enough to cover the increment in applied current (Kanavova et al., 2014).

In order to understand at which ratio of an individual ion carry of the total current in a solution, it is necessary to know the transference number of this specific ion. Transference number (transport number) (*t_i*) refers to the part of the current that moves through the solution carried by *i*-ion.

With the help of Eq. (7), the transference number of each ion has been calculated. Since current efficiency examinations have a dependence on pollutant species (Benneker et al., 2018; La Cerva et al., 2018; Sadrzadeh and Mohammadi, 2009) to be treated in ED systems, anions originated from the referred salts did not included in transference number computations to proceed in parallel with the literature. The transference number for Pb²⁺, Cu²⁺ and Ni²⁺ were calculated as *t_{Pb²⁺}* = 0.2089, *t_{Cu²⁺}* = 0.2732 and *t_{Ni²⁺}* = 0.5178 respectively. These transference numbers can also be explained in fractional numbers as, Pb²⁺ used 20.89 % of the total current, Cu²⁺ used 27.32 % of the total current and Ni²⁺ used 51.78 % of the total current applied to the system. Average of the overall current that moves through the ED stack for 30, 45 and 60 V in 6 hours of treatment time were 0.48, 0.61 and 0.71 A respectively.

With Δn values and specific current used by each ion type, the current efficiencies for Pb²⁺, Cu²⁺ and Ni²⁺ have been utilized. In each applied voltage, current efficiency of each ion type has been arranged as Cu²⁺ > Ni²⁺ > Pb²⁺. According to these results, we can conclude that each ion type uses the applied current in different yields. Even Pb²⁺ has the best removal efficiency, when dealing with current efficiency Cu²⁺ showed the worst performance.

3.2. Amount of ion removal and specific electrical energy consumptions

In an ED process applied voltage or current determines the electrical energy/power consumption of the system (Banasiak et al., 2007). The SEEC value specifies the amount of ions removed per electrical energy consumed and this data is an important parameter in calculating the cost of the electro dialysis process. Fig. 5 illustrates the variations of SEEC values and the amount of ions removed from the wastewater depending on the applied voltage. Analysis of the sum of ions extracted indicated that, there was also a positive correlation between applied potential and ion removal. This data is also connected with the removal efficiency of the ions mentioned in section 3.1. As the applied voltage increased the amount of ion removed from the system increased. For voltages of 30, 45 and 60 the total removal of ions in mg were 3.5337, 4.0073 and 4.3627 respectively. When each type of ion is handled one by one, the removal amount of lead had the biggest value in every applied voltage. The second-best ion removal was copper and the least removed was nickel. The removal

of each ion type had also dependence on hydration shells, hydration bonds, hydration energies and ionic mobilities of the ions. Table 3 gives the quantity and percent changes with voltage transitions for each ion type and the total ions. Table 3 indicates that quantity and percent changes in all ion types have very close values. But when comparing transition from 30 V → 45 V and 45 V → 60 V, quantity, and percent changes indicate a downward rate. This phenomenon can be explained as system was approaching the limiting current density value. Around LCD value, depletion of electron carriers in the feed solution has occurred and concentration polarization on the membrane boundary layer had formed (Banasiak et al., 2007) which led the system to a saturation point.

One of the main aims of an ED process is to operate the system with minimum electrical energy consumption and with maximum ion removal. Lower SEEC values exhibit that the system is operating more efficiently. With the help of Eq. (8), SEEC values of the total ion removal from the solution have been calculated. The specific electrical energy consumption values were computed as 0.048, 0.082 and 0.118 W h/mg for 30, 45 and 60 volts respectively

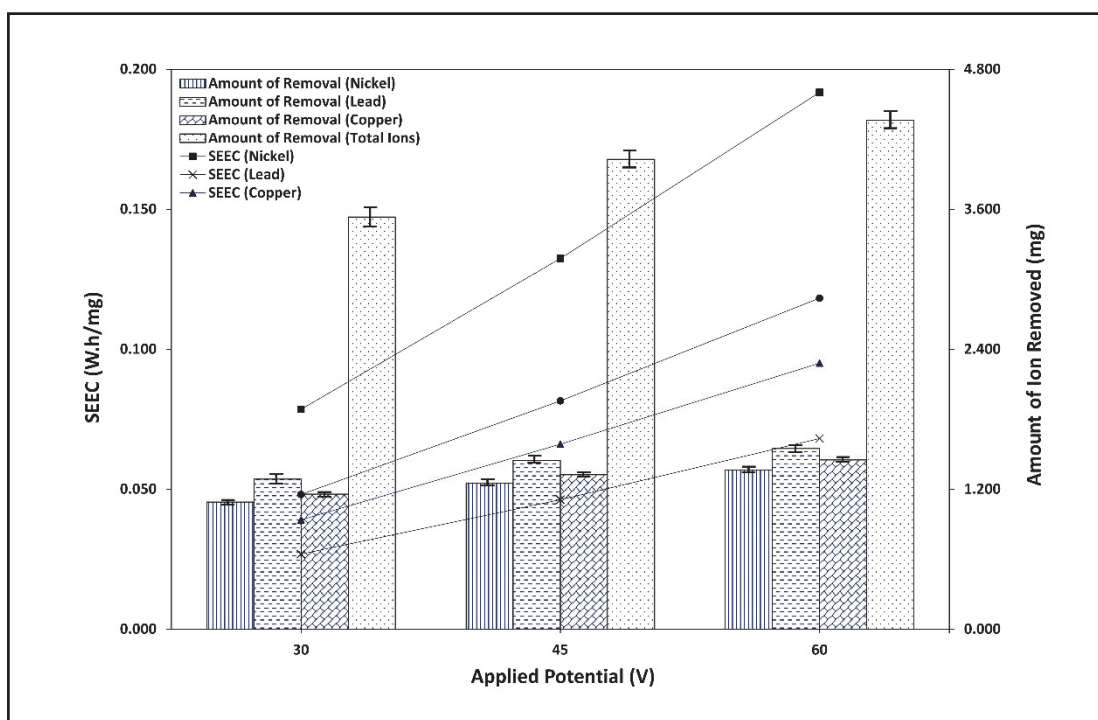


Fig. 5. Specific electrical energy consumptions and removed amount of ions (Wastewater conc. = 6 mg/L, Flow rate = 200 mL/min, pH ≈ 6, electrolyte conc. = 0.05 M NaCl, t = 6 h)

Table 3. Quantity and percent change in voltage transitions

Metal Type	Voltage Transitions			
	30 V → 45 V		45 V → 60 V	
	Quantity change (mg)	Percent Change (%)	Quantity change (mg)	Percent Change (%)
Pb ²⁺	0.1619	8.09	0.1002	5.01
Cu ²⁺	0.1672	8.36	0.1263	6.32
Ni ²⁺	0.1645	8.23	0.1089	5.45
Total	0.4936	8.23	0.3354	5.59

The current that moves across the ED system was low when the applied voltage was low and that led to nominal electrical energy consumption. As can be seen in Fig. 5, SEEC values had shown a dramatic increment. Ascending voltages led to increasing currents and depending on the quantity of the removed ions, SEEC values also raised. The quantity of removed ions was not linearly correlated to the energy consumed in the system and this event led to inefficient operations.

To evaluate the SEEC values of different metal types, transference numbers of the ion types should be taken into account. With the help of specific current used by each ion type depending on their transference numbers, SEEC values have been utilized. For 60 V of applied potential, the specific current used by each ion types were 0.148 A for Pb^{2+} , 0.194 for Cu^{2+} and 0.368 A for Ni^{2+} . Quantities of ions treated for the same voltage value were 1.5485 mg for Pb^{2+} , 1.4511 mg for Cu^{2+} and 1.3631 mg for Ni^{2+} . With the obtained data SEEC values were determined as 0.068, 0.095 and 0.192 W h/mg for Pb^{2+} , Cu^{2+} and Ni^{2+} respectively. Lead ion had the lowest energy consumption and nickel ion had a mass removal with much more energy. SEEC results indicated that lead ion needed less electrical energy per mg to be purified from the system.

4. Conclusions

The removal efficiency of pollutants in an ED system was directly proportional to the voltage. Removal efficiencies of ions with an equal concentration were detected at different values. Hydration shells, hydration bonds, hydration energies and ionic mobility's of the ions were the most important factors in such differences. When the voltage applied to the system increased, the current efficiency decreased.

The increment in voltage value triggered the current value, however, because the amount of metal ions eliminated in the system did not react to this rise, the applied current was used more inefficiently. According to the transference numbers, the specific currents of ions determined and the current efficiencies of each ion type calculated separately. The eliminated ions in mg were positively affected by the voltage increment but this phenomenon was at a degressive rate when the system reached to LCD value. SEEC criterion was found to be directly proportional to the voltage and also this situation reduced the efficiency of the method.

The ion types used in the study showed performances as follows: for removal efficiency, amount of removal and SEEC $Pb^{2+} > Cu^{2+} > Ni^{2+}$ and for current efficiency $Cu^{2+} > Ni^{2+} > Pb^{2+}$.

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