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Electrodialysis of groundwater with heavy metal and nitrate ions under low conductivity and effects of superficial velocities

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ABSTRACT

The efficiency of separation of lead, manganese, arsenic, and nitrate from groundwater with relatively low conductivity by electrodialysis (ED) was investigated. The effects of the superficial velocities in either the diluate or concentrate cell were also evaluated to understand the relative separation rate of the four ionic compounds by ED and to examine the possibility of reducing the energy for pumping. Significant removal (i.e. 92.8–99.2%) of manganese, arsenic, and nitrate from Ilgam (Seoul, Korea) groundwater was achieved, along with removal (61.3%) of lead comparable to that of Sungbuk (Seoul, Korea) water. The superficial velocity in the concentrate cell exerted no effects on either the conductivity or current reduction, or on the separation ratios. The superficial velocity in the diluate cell increased the operation time required to obtain the requisite conductivity reduction, but exerted little effect on the separation ratios. ED could be performed at reduced water velocity to treat groundwater with low conductivity.

Keywords: Electrodialysis; Heavy metal; Nitrate; Separation; Superficial velocity

1. Introduction

The presence of species such as lead, arsenic, and nitrate in groundwater has become a worldwide problem, especially in areas where groundwater is the only source of drinking water. For instance, more than 100 million people in India have been exposed to serious risk due to the consumption of arsenic from groundwater for drinking purposes [1,2]. High concentrations of arsenic, lead, manganese, and nitrate in groundwater are also frequently observed in Korea. A monitoring system operated by the Korean Rural Community Corporation measures thirty-three parameters of water quality at intervals of every other month [3]. For instance, between 2001 and 2006, the reported concentrations of arsenic in groundwater in Korea were 5.3–188 mg/L [4]. The average maximum annual

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concentration detected by the monitoring system was approximately 100 μg/L of arsenic, 27 mg/L of nitrate, and 4 mg/L of manganese, with a conductivity of 400 μs/cm [5].

Various methods to treat ionic compounds in groundwater include adsorption, membrane separation, and chemical, photochemical, or photocatalytic oxidation [6–8]. Among the membrane separation technologies, nanofiltration (NF), reverse osmosis (RO), and electrodialysis (ED) could be considered. Membrane-based desalination processes have great removal of ionic compounds and the automated operation offers the advantage for application in small villages in rural areas. ED is an electrochemical separation process in which ions pass across charged membranes from one solution to another under the influence of an electrical potential difference, as a driving force. ED has been applied to heavy metal removal, wastewater minimization, nitrate reduction, treatment of concentrates from RO systems, in addition to desalination of salt water. The ion removal efficiency of ED is comparable to that of RO membrane processes, whereas the former has lower energy consumption and less fouling tendency.

Several recent studies have examined ED desalination on sensitivity to the superficial velocities, chemical composition, hydraulic recovery, and temperature and competitive separation of ions with different valences during operation. The superficial velocity (i.e. water flow rate divided by cross-sectional area of flow path without spacers) on the membrane surface was reported to be inversely proportional to the boundary layer thickness. The thickness of the boundary layer, which is considered as the extent of the concentration polarization (CP) from the ion exchange membrane surface, is controlled by the hydrodynamic condition [9–11]. When CP occurs in the concentrate compartment, the higher concentration of rejected co-ions on the membrane surface induces back diffusion of ions to the bulk solution and cancels the movement of ions by the applied electrical potential [12,13]. The ion concentration on the membrane surfaces in the diluate cell is reduced by CP, which increases the electrical resistance, thereby increasing the required electrical potential for ion transport. Kim et al. investigated the competitive separation of divalent and monovalent cations during ED. They found that the thin boundary layer due to the increased flow rate in the cell preferentially accelerated the separation rate of calcium compared to potassium due to the greater ionic charge of calcium that facilitated the transfer of calcium through the cation exchange membrane [14].

Most studies on ED have been conducted with relatively high salt concentrations, i.e. approximately 8,000 mg/L of total dissolved solids, although ED is known to be effective at moderate salt concentrations between 1,000 and 10,000 mg/L [15]. However, 42.6% of groundwater is reported to be less than 2,000 μS/cm and 18.5% of groundwater is reported to be greater than 15,000 μS/cm in Korea [3]. In this study, ED is applied to the removal of common problematic species such as lead, manganese, arsenic, and nitrate from groundwater under the usual conditions in Korea, i.e. at relatively low conductivity. In addition, the superficial velocities in the cell are varied to understand the relative rate of separation of the four compounds during ED and to examine the possibility of reducing the energy for pumping. The groundwater was collected from two locations, Ilgam and Sungbuk, and was then concentrated and spiked with lead, manganese, arsenic, and nitrate to simulate the properties of the groundwater under investigation. The concentrations of Pb, Mn, As, and NO_3^- were designated as 0.05, 0.5, 0.1, and 30 mg/L, respectively.

2. Methods and materials

2.1. Preparation of groundwater

The Ilgam groundwater located at the third basement of a building in the Konkuk university was selected because of the convenient accessibility. Since a pilot study using ED is planned, a location close to this laboratory is important. The Sungbuk groundwater was sampled due to its high concentrations of arsenic. The water quality, including the concentrations of Pb, Mn, As, and NO_3^-, was analyzed as summarized in Table 1. The conductivity of the raw water samples ranged from 100 to 200 μs/cm, which is very low and is expected to cause high resistance during ED operation. A nanofilter (NE1812–70, Toray Chemical, Korea) with a nominal molecular cutoff of

Table 1
Water quality of groundwater from Sungbuk and Ilgam

Location	Ilgam		Sungbuk	
Parameters	Raw	Spiked	Raw	Spiked
Mn^{2+} (μg/L)	1.65	453.1	0.16	450.3
Arsenic (μg As/L)	ND	92.9	118.1	1,013.3
Pb^{2+} (μg/L)	ND	40.5	1.02	43.4
NO_3^- (mg N /L)	3.64	34.8	2.17	31.4
pH	6.82	7.67	8.00	7.75
Conductivity (μs/cm)	258.3	2,288	317.0	1,406
Turbidity (NTU)	0.201	3.15	0.226	0.109
UV absorbance (1/cm)	0.0037	0.055	0.007	0.056
TOC (mg/L)	1.03	2.44	1.02	2.56

Table 2
Characteristics of nanofilter

Model name	Monovalent ion rejection (NaCl)	Divalent ion rejection ($MgSO_4$)	Pore size
NE1812-70	40–70%	95%	0.001–0.01 μm

100 Da was used to concentrate the groundwater. The characteristics of the nanofilter are summarized in Table 2. The water recovery was maintained at 95% so that the concentrate flow was 5% of the feed flow. After NF, the water was spiked with Pb, Mn, As, and NO_3^- to reach the designated concentration. The feed water parameters after spiking with the ionic compounds are also presented in Table 1.

2.2. Preparation of synthetic model water

To elucidate effects of the superficial velocity on the relative separation rate of Pb, Mn, As, and NO_3^-, a model water sample was prepared in the laboratory. Stock solutions of As, Pb, Mn, and NO_3^- were produced by dissolving appropriate quantities of sodium arsenate ($Na_2HAsO_4{\cdot}7H_2O$, 99.5%, Sigma-Aldrich, Germany), lead nitrate ($Pb(NO_3)_2$, 99.0%, Sigma-Aldrich, Germany), manganese sulfate ($MnSO_4$, 99.0%, Sigma-Aldrich, Germany), and sodium nitrate ($NaNO_3$, 99.0%, Showa, Japan) in 1 L of deionized (18.2 MΩ) water before use. The model water sample was prepared by adding stock solutions to give final concentrations of 0.1 mg/L of arsenic, 0.05 mg/L of lead, 0.5 mg/L of manganese, and 30 mg/L of nitrate nitrogen and by dissolving sodium chloride (NaCl, 99.5%, Showa, Japan) to give a concentration of 400 mg/L in the tap water at the laboratory.

2.3. Electrodialysis (ED) system

The bench-scale ED system is shown in Fig. 1. The ED system (CJ-S3, Changjo Techno, Korea) consisted of reservoirs, pumps, an ion-exchange membrane stack, power supply, electrodes, and a data acquisition system. The system was operated at a constant voltage of 12 V and the separation efficiency was measured as changes in the current. A pump (NH-3PX, Pan World, Ibarakiken, Japan) with a speed of 2,700–3,200 rpm was used. The ED cell was packed with five pairs of ion exchange membranes (cation and anion) and an electrode pair. The cathode and anode were made of stainless steel coated with platinum. The area of each electrode was $4.7 \times 10.0\ cm^2$. The ion-exchange membranes were purchased from ASTOM Company (Neosepta, Tokyo, Japan). Neosepta CMX-SB and AMX-SB membranes were used as the cation and anion exchange membranes, respectively. Each membrane had an effective surface area of $275\ cm^2$. The thickness of the CMX and AMX membranes was 170 and 140 μm, respectively. The specifications of the ED stack are shown in Table 3. The properties of the ion exchange membranes are summarized in Table 4. The ED operation was terminated at a conductivity of 20 μs/cm.

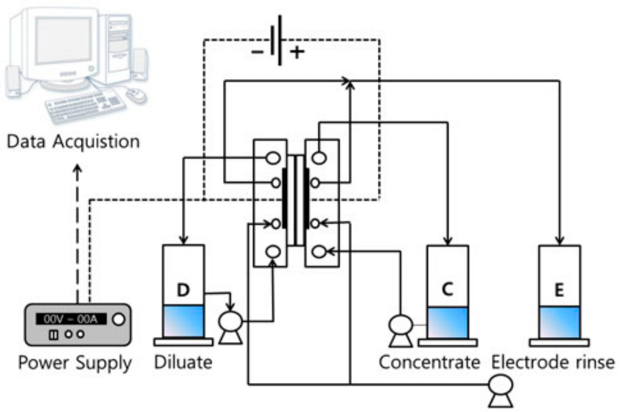

Fig. 1. Schematic of electrodialysis system used in this study.

2.4. Water quality analysis

In all experiments, the pH and conductivity of the solution were monitored using a Thermo pH-Conductivity Meter Orion 5 Star setup (Orion 5 STAR, Thermo Fisher Scientific Inc. Beverly, USA).

In addition to the pH and the conductivity, current in each reservoir was measured and recorded every second using a customized supervisory data acquisition system. The diluate from the ED system was sampled and analyzed by inductively coupled plasma-optical emission spectrometry (Thermo scientific, ICAP 6000 series) to determine the arsenic, lead, and manganese concentrations and using a spectrophotometer (HACH DR/2500, USA) for the nitrate nitrogen. The removal ratio of each ion is defined in Eq. (1) as:

$$\text{Ion removal } (\%) = \frac{C_0 - C}{C_0} \times 100 \qquad (1)$$

Table 3
Specifications of electrodialysis system

Parameter	Values/Material
Electrode	Stainless steel coated with platinum
Current	0–3 A
Voltage	0–30 V
Number of cell pair	5
Spacer material	Polypropylene
Spacer thickness (mm)	0.72

Table 4
Properties of ion exchange membranes (obtained from manufacturer)

Type	CMX	AMX
Characteristics	Strong acid (Na^+ type)	Strong base (Cl^- type)
Mechanical strength	High	High
Electric resistance[a] (Ω cm^2)	3.0	2.4
Destructive strength (MPa)	$\geqq$0.40	$\geqq$0.25
Thickness (μm)	170	140
Operating pH	0–10	0–8

[a] Equilibrated with a 0.5 N-NaCl solution at 25˚C.

where C_0 and C correspond to the concentration of an ion in the diluate at the beginning of the experiment and at time t, respectively [16].

Dissolved organic carbon concentrations of groundwater samples were analyzed using a Total Organic Carbon Analyzer (Sievers 5130C Laboratory TOC Analyzer, USA).

3. Results and discussion

3.1. Efficiency of separation of ions in groundwater with low conductivity

ED of Ilgam and Sungbuk groundwater was conducted for approximately 20 min until the conductivity reached 20 μs/cm. The initial conductivity was 2,280 μs/cm for Ilgam water and 1,450 μs/cm for Sungbuk groundwater. The reduction in the conductivity occurred rapidly after initiating the operation, as shown in Fig. 2. The reduction rate slowed down after 8 min. The variation in the conductivity in the concentrate compartment was characterized by a small valley right after the start of operation for both water samples. The removal rate for the ions in Ilgam water was in general greater than that of Sungbuk water, which indicated that the high conductivity, i.e. great amount of electric potential carriers, enhanced the ED performance.

The ED system produced high quality permeate. Removal of the specified ions was mostly achieved to an extent exceeding 90%, except in the case of Pb and As. The separation ratio for lead in Ilgam water was 61.3%, compared to 64.7% for Sungbuk water, whereas the separation ratio for arsenic in Sungbuk water was 76.5%. In general, the arsenic removal efficiency of NF is dependent on the arsenic concentrations and is reportedly approximately 80% for As(V) [17,18]. The arsenic removal efficiency achieved with RO is generally reported to be in the range of 80–86% [19,20]. The results of this study indicate that a comparable separation ratio of arsenic can be achieved with ED. The quality standard for drinking water in Korea states that the arsenic, lead, manganese, and nitrate nitrogen ion concentrations should be less than 0.01, 0.01, 0.05, and 30 mg/L, respectively. The ionic compounds such as Mn and nitrate in the diluate satisfy the standard requirements; however, species such as As and Pb did not satisfy the standard requirements, as summarized in Table 5.

The removal ratio of manganese was greater than that of Pb and the nitrate removal ratio was greater than that of arsenic for both groundwater samples. The competitive ion transport was governed mainly by the membrane selectivity and boundary layer thickness [14]. Accelerated transport of Mn and nitrate compared to Pb and arsenic was observed in the experiments.

3.2. Ion separation efficiency of synthetic model water

The ion separation ratios were investigated using synthetic model water, which was manufactured to understand the effects of the water flow velocity on the removal of the four ions. The removal of each ionic compound was analyzed at different operation times, and the conductivity of the diluate was also varied. As the ED operation time was increased to 9 min, the conductivity decreased to 100 μs/cm. The conductivity reached 50 μs/cm at 12 min and 20 μs/cm at 15 min of ED operation. Where the total dissolved solids concentration is concerned, only 4 min of operation would be sufficient to meet the water quality standards. The removal of the ions of interest is summarized in Fig. 3. The concentration of lead, manganese, and nitrate ions satisfied the drinking water quality standards for all times after 9 min of operation, where the conductivity reached 100 μs/cm. However, arsenic required at least 15 min of operation to satisfy the standard. Further experiments were therefore terminated when the conductivity reached 20 μs/cm. At the terminated conductivity, the removal

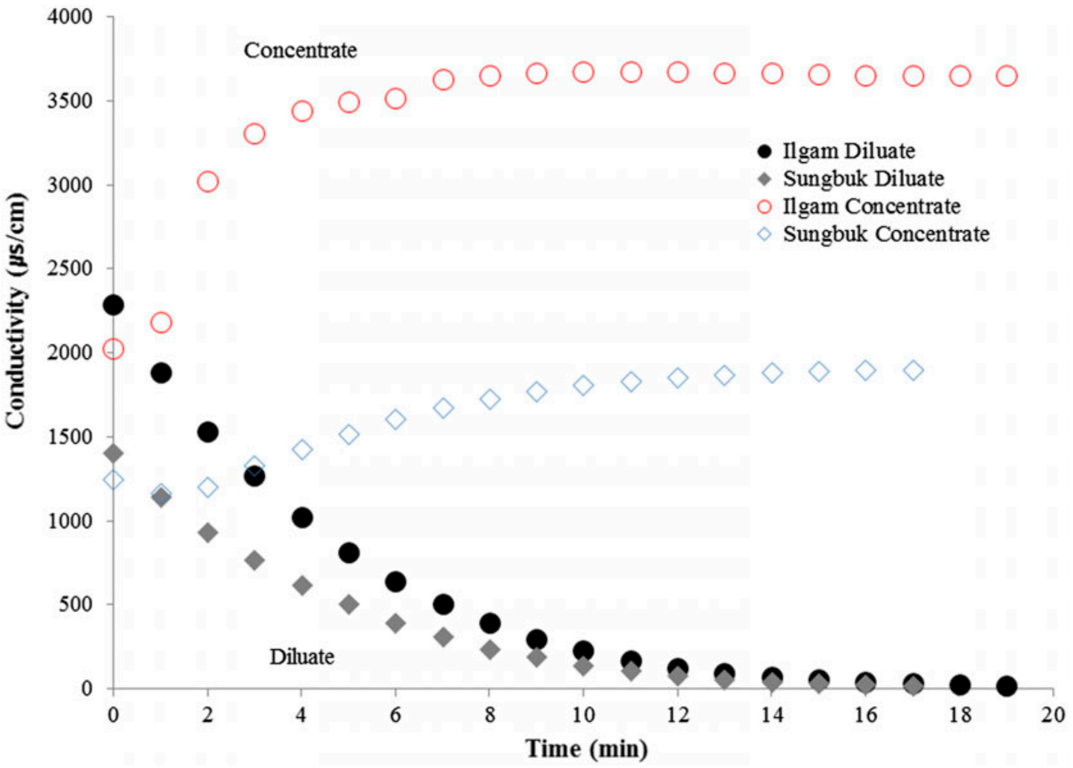

Fig. 2. Separation efficiency of ions in groundwater collected from Ilgam and Sungbuk.

Table 5
Removal ratio of ionic compounds and conductivity by ED

	Ilgam			Sungbuk		
Parameters	Feed	Diluate	Removal (%)	Feed	Diluate	Removal (%)
Mn^{2+} (µg/L)	453.084	3.68	99.2	450.33	8.71	98.1
Pb^{2+} (µg/L)	40.46	15.66	61.3	43.44	19.15	64.7
NO_3^- (mg as N/L)	34.80	0.7	99.2	31.40	0.93	97.0
Arsenic (µg as As/L)	92.85	6.64	92.8	1,013.27	237.97	76.5
pH	7.67	4.92		7.75	5.19	
Conductivity (µs/cm)	2,288	19.99	99.1	1,400	19.75	98.6

efficiencies of Mn, Pb, and nitrate exceeded 98% and the removal of arsenic was 90.4%. Compared to the results of the groundwater sampled in Ilgam and Sungbuk, the removal of lead from the synthetic model water increased remarkably. The increased removal of lead for the synthetic model water might indicate that numerous ions or organics in the real groundwater deteriorated the transport mechanism of lead due to interactions with suspended particles, sediments, and organics that are strongly affected by the chemical forms of the metals [21]. In addition, Robert et al. [22] reported that lead proceeded reduction reaction to form 1:2 aqueous lead-organic complexes. In addition, increases in voltages, flow rate, and temperature resulted in enhancing lead removal during ED operation [23].

3.3. *Effect of superficial flow velocities on diluate compartment*

CP can be avoided by increasing the water flow velocity in the diluate cell. During CP, the concentrations of the ions at the membrane surface become lower than the bulk concentration, thereby reducing the desalination rate. Increases in the flow velocity have been applied to achieve higher concentrations of ions on the ion exchange membrane surface and to improve the ED performance by decreasing the thickness of the diffusion boundary layer.

The CP may be minimized in this study due to the low salt concentrations in the diluate cell. Therefore, the superficial velocity in the diluate cell compartment was reduced from 13.5 to 1.9 cm/s, while the superficial velocity in the concentrate cell compartment was fixed at 15.4 cm/s. Taking into account the variation in the cell velocity from 1 to 10 cm/s in previous studies, the velocities in this study spanned from the far lowest to the far highest [24]. The lower velocity in the diluate cell could decrease the energy cost for pumping. The ED performance should be evaluated under the conditions where the ion separation ratios and electrical currents are maintained.

The variations in the conductivity and electrical current are presented in Fig. 4. The conductivity declined more rapidly at the higher velocity of 13.5 cm/s compared to the lower velocity of 1.9 cm/s, as expected. The reduction of the electrical current followed different trends under the two flow conditions, although the same water sample (i.e. equal ion concentrations) was injected as feed water. The maximum current was 350 mA and the current was declined rapidly to 50 mA within 8 min of operation at the flow velocity of 13.5 cm/s. In contrast, the maximum current was only 200 mA and the current declined gradually to reach 50 mA within 17 min of operation at the flow velocity of 1.9 cm/s. Furthermore, abrupt changes in the pH of the diluate cell were observed at the flow velocity of 13.5 cm/s, as shown in Fig. 5, which corresponded to the rapid decline in the conductivity and electrical current. Due to the limited ion concentrations of the feed water, the extremely low ion concentrations at the membrane surfaces induced decomposition of water to hydrogen and hydroxide ions under the voltage condition used in this study. Although the lower velocity in the diluate cell resulted in a longer operating time to achieve desalination, the low velocity lessened the abrupt depletion of the ions and the significant pH variation in the water with low salt concentration.

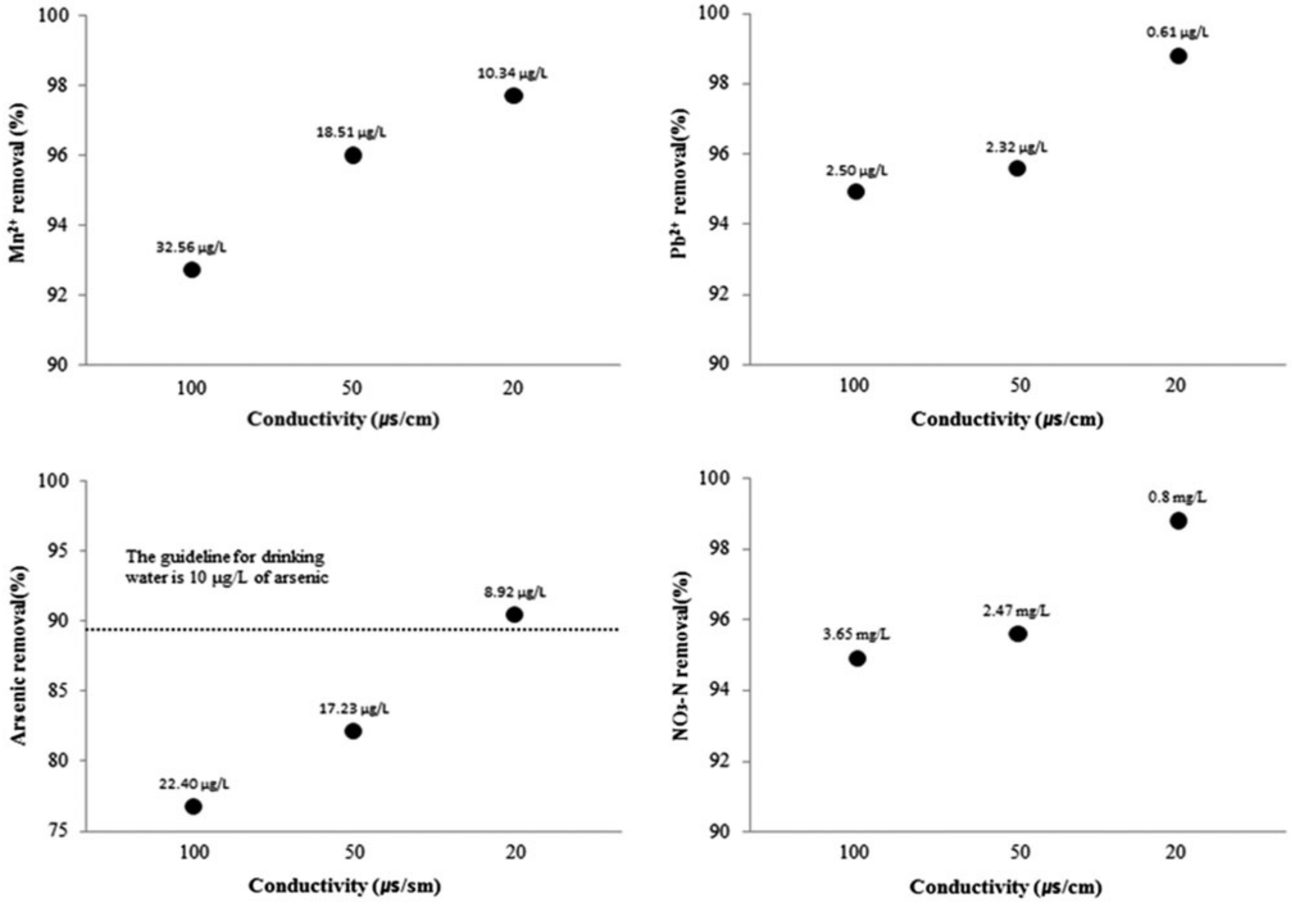

Fig. 3. Separation ratios of each ion in the synthetic model water.

The relative separation ratios of the ions were evaluated as shown in Table 6. The ion removals for manganese and nitrate were high, ranging from 89.8 to 99.0%. The removal of arsenic was 89.8% at a velocity of 13.5 cm/s and 92.4% at a velocity of 1.9 cm/s. The results showed slight increases in the removal at a velocity of 1.9 cm/s. The effects of superficial velocity on the separation efficiencies are controversial. A previous study by Midaoui et al. [25] revealed that the nitrate removal increased from 78 to 83% with an increase in the water flow rate from 100 to 180 L/h after 10 min of operation. However, Kabay et al. [26] concluded that the effect of the flow rate on the separation performance was not so apparent when mono- and di-valent ions were treated by ED. The flow rate was varied as 0.6, 1.2, and 1.8 L/min in their study, and the separation efficiency of calcium ions was greatest at the flow rate of 0.6 L/min, which is consistent with the results of this study. The applied voltage is the main driving force affecting the rate of separation [27,28]. The solution velocity, as one of the process control parameters, in general determines the thickness of the diffusion boundary layer. Decreasing the inter-membrane water flow velocity decreases the amount of mixing, which decreases the mass transfer to the membrane surfaces. Seemingly, the influence of the applied voltage dominated the decreases in the mass transfer limitation due to the low velocity used in this study.

3.4. Effect of superficial velocities on concentrate compartment

The water flow rates in the concentrate cell were varied as 1.6, 0.5, and 0.2 L/min, which corresponded to superficial velocities of 15.4, 4.8, and 1.9 cm/s, respectively, while the water flow rate in the diluate cell was fixed at 14.5 L/min, corresponding to a superficial velocity of 13.5 cm/s. The reduction in the conductivity and the removal of the four ions were measured at three superficial velocities as shown in Fig. 6. The conductivity decreased at a similar rate for the three velocity conditions, such that approximately 15 min was required to reduce the conductivity to 20 μs/cm, which is different from the result of the conductivity reductions at the two superficial velocity in diluate cell. CP in the concentrate cell is generally problematic due to the fact that the high concentration of ions developed at the membrane surface could induce solid precipitation and thus restrict ion transport. However, the low salt concentrations of the feed water used in this study precluded the detrimental effects exerted by high salt concentrations.

The removals of the individual ion species at the three different velocities were also identical. As explained earlier, the limited effect of the water velocity on reducing the conductivity was due to the fact that the electrical potential, as the main driving force, governed the desalination performance for the model feed water with low conductivity.

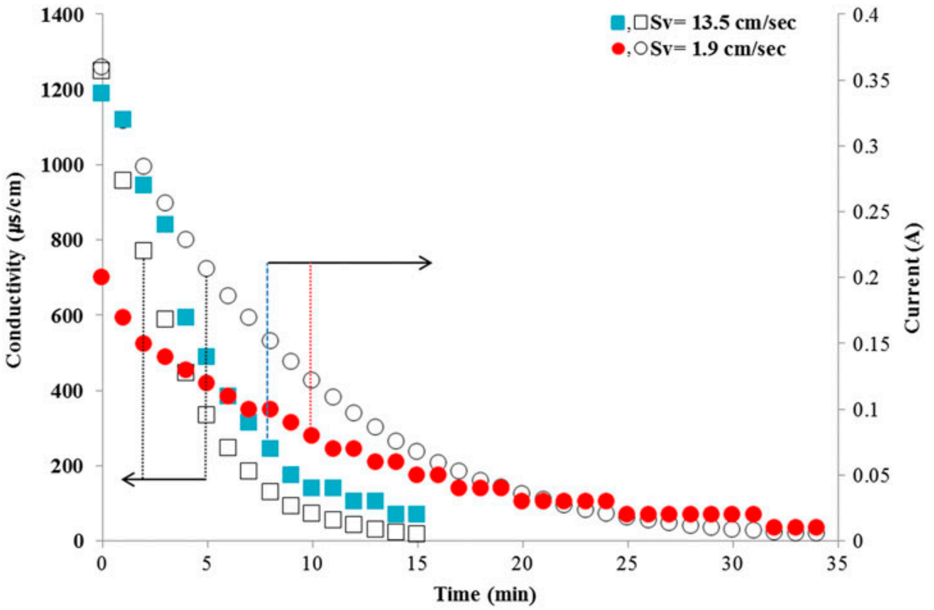

Fig. 4. Changes in conductivity and electrical current at two water flow velocities in diluate cell.

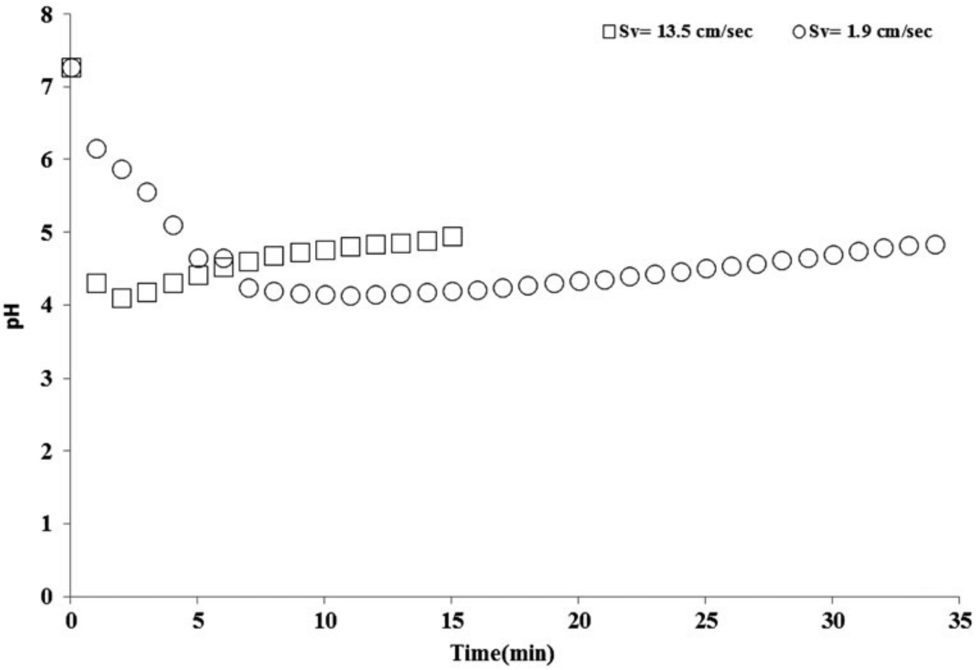

Fig. 5. pH variation at superficial velocities of 1.9 and 13.5 cm/s during ED.

Table 6
Characteristic of water quality based on superficial velocities on diluate cell

	Sv = 13.5 cm/s			*Sv* = 1.9 cm/s		
Ions	Feed	Diluate	Removal (%)	Feed	Diluate	Removal (%)
Mn^{2+} (μg/L)	460.63	7.26	98.4	468.08	4.61	99.0
Pb^{2+} (μg/L)	41.46	2.11	94.9	30.26	ND	NA
NO_3^- (mg N/L)	34.67	0.60	98.3	31.07	0.97	99.0
As (μg/L)	96.53	9.89	89.8	99.57	7.57	92.4
pH	7.27	4.94	–	7.27	4.82	–
Conductivity (μs/cm)	1,248	19.75	98.4	1,258	19.98	98.4

The relative removal ratio of each ion species showed interesting results. Two anions (i.e. nitrate and arsenic) and two cations (i.e. manganese and lead) were evaluated in this study. The separation of nitrate and manganese was superior to the separation of arsenic and lead, although the relative separation ratio became substantial during 5 to 10 minutes of operation and the ratios became similar as the operation neared the end. Transport through the ion exchange membrane has been explained in terms of the selectivity of the membrane and the diffusivity [22]. The two cations have the same charge, leading to similar preference by the cation exchange membrane. The diffusivity values of manganese and lead are 0.712×10^{-5} and 0.945×10^{-5} cm^2/s, respectively. However, the high concentration of manganese would overcome the preferable effect of the slightly high diffusivity of lead in this study. For two anions, the higher separation of nitrate relative to arsenic was apparent. The higher diffusivity of nitrate, 1.902×10^{-5} cm^2/s resulted in preferential separation compared to arsenic, where the diffusivity of H_2AsO4^- is 0.905×10^{-5} cm^2/s. In addition, arsenic occurs as various species in water depending on the pH and oxidation states. H_3AsO_3, $H_2AsO_3^-$, $H_2AsO_3^{2-}$, and AsO_3^{3-} species occur for arsenite, (As(III)), while H_3AsO_4, $H_2AsO_4^-$, $HAsO_4^{2-}$, and AsO_4^{3-} occur for arsenate, (As(V)). Thus, changes in the pH and oxidation–reduction potential during ED may be possible causes of the low separation ratio of arsenic because arsenite may exist in a neutral molecular form in the pH range of 0–10, while arsenate exists in a negative monovalent form [20].

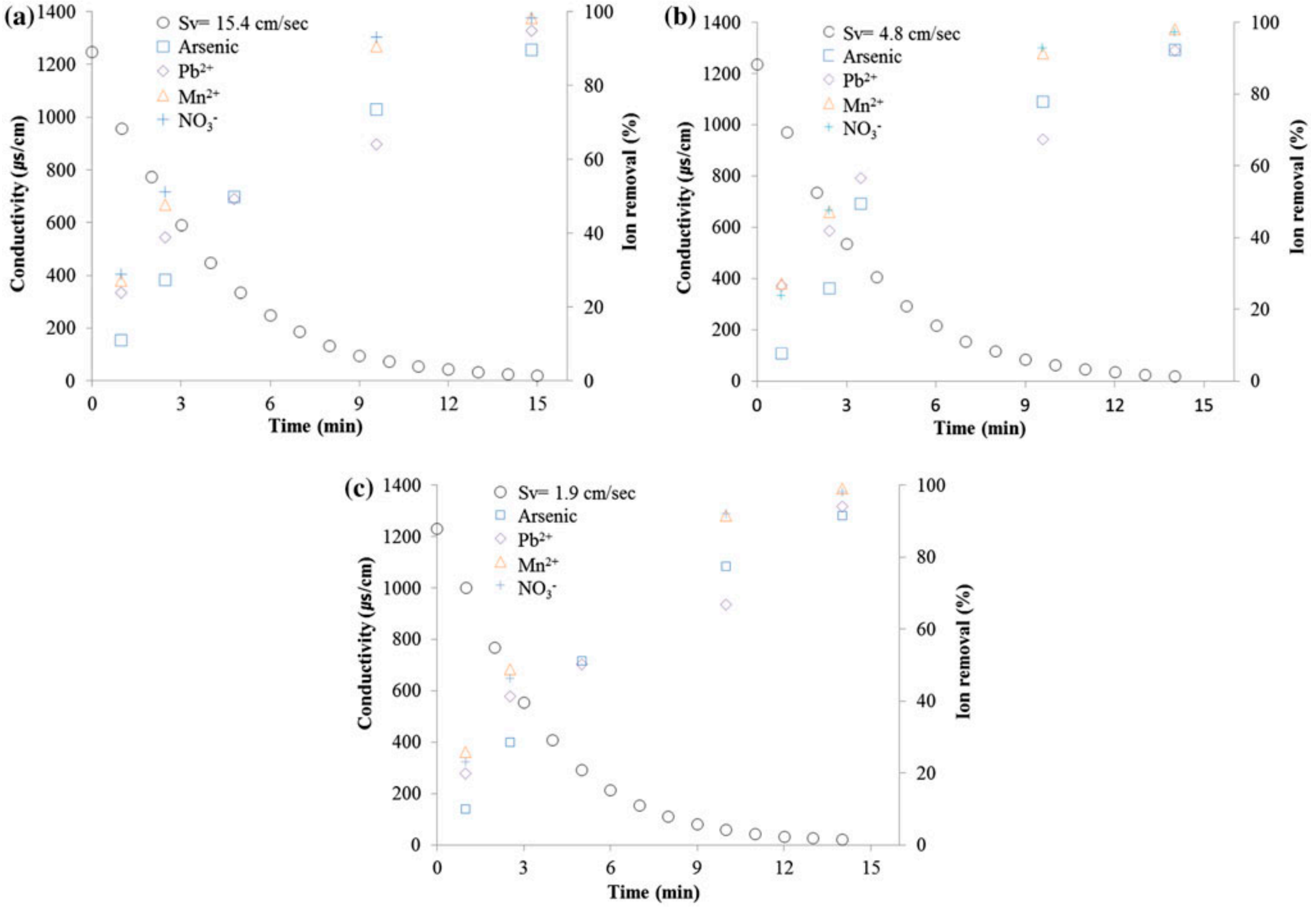

Fig. 6. Effects of the superficial velocities of the concentrate cell on ion removal at (a) 15.4 cm/s, (b) 4.8 cm/s, and (c) 1.9 cm/s.

The initial pH was 7.26 and the pH dropped to approximately 4.93 at the diluate and 2.38 for the concentrate. The observed decreases in the pH of the concentrate due to water dissociation (co-creating H^+ and OH^-), CP in the boundary layer, and/or transfer of weak acids (e.g. carbonate species) are consistent with other studies [29,30]. The low conductivity of the model synthetic water might be the cause of the low pH condition during ED.

4. Conclusions

The efficiency of lead, manganese, arsenic, and nitrate separation from groundwater with relatively low conductivity by ED was evaluated. Significant removal (i.e. 97.0–99.2%) of manganese and nitrate were achieved for both Ilgam and Sungbuk ground water samples. The lead removal (61.3%) comparable to that of Sungbuk water (64.7%) were achieved for Ilgam water whereas the arsenic removal from the Sungbuk water only 76.5% due to the high concentration of the feed water, i.e. 1,013 µg/L. The ion separation was substantial within approximately 10 min of operation corresponding to diluate conductivity of 100 µs/cm.

The effects of the superficial velocities in either the diluate or concentrate cell were also evaluated to understand the relative separation rate of the four ions during ED and to examine the possibility of reducing the energy for pumping. The decrease in superficial velocity in the diluate cell increased the operation time required to obtain the requisite conductivity reduction, but had little effect on the separation ratios. The superficial velocity in the concentrate cell showed no effects on either the conductivity or the current reduction, or on the separation ratios. The low water velocity in the cell has been proposed to limit transport of ions through the boundary layer, but resulting in infinitesimal effects on the separation ratios in this study, which was in part due to the low conductivity of the feed. ED could be performed at reduced water velocity to treat groundwater with low conductivity.

Acknowledgment

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