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# Monitoring of electrokinetic removal of heavy metals in tailing-soils using sequential extraction analysis

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## Abstract

This research focused on the monitoring of the electrokinetic removal of heavy metals from tailing-soils, and emphasizes the dependency of removal efficiencies upon their physico-chemical states, as demonstrated by the different extraction methods adopted, which included aqua regia and sequential extraction. The tailing-soils examined contained high concentrations of target metal contaminants ( $\text{Cd} = 179 \text{ mg kg}^{-1}$ ,  $\text{Cu} = 207 \text{ mg kg}^{-1}$ ,  $\text{Pb} = 5175 \text{ mg kg}^{-1}$ , and  $\text{Zn} = 7600 \text{ mg kg}^{-1}$ ). The removal efficiencies of the different metals were significantly influenced by their speciations, mobilities and affinities (adsorption capacities) in the soil matrix. The removal efficiencies of mobile and weakly bound fractions, such as the exchangeable fraction were more than 90% by electrokinetic treatment, but strongly bound fractions, such as the organically bound species and residual fraction were not significantly removed (less than 30% removal efficiencies). In accordance with the general sequence of mobilities of heavy metals in soils, the removal efficiencies of more mobile heavy metals (Cd, Cu, and Zn) were higher than that of less mobile heavy metal (Pb). © 2001 Elsevier Science B.V. All rights reserved.

*Keywords:* Heavy metals; Speciations; Sequential extraction; Electrokinetics; Removal efficiency; Mine tailings

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## 1. Introduction

Soils can be contaminated with heavy metals derived from various sources including abandoned mining wastes, industrial wastes, used battery residues, leakage from landfill sites, accidental spills and military activities [1]. Contaminants migrating from these sources threaten human health by contaminating food crops and the ground-water supply. The degree and extent of environmental metal pollution as a direct result of man's activities has been researched by many workers [2–4]. However, technologies for decontaminating these sites

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have not yet been developed. In addition, it has been recently reported that soil contamination is increasing in sensitive areas, such as paddy fields, residential districts near mining areas and reservoirs of drinking water [4].

There are a number of metal mining operations that have been closed in South Korea, but mounds of contaminated tailings remain near mining sites. Many of these contain heavy metals that have the potential to contaminate soil, and surface and groundwater. According to changes in their physico-chemical states and the prevailing chemical conditions, these metals can be transported, dispersed and accumulated in plants and animals, and can be taken in by human beings as the final consumer. Heavy metals in tailings exist in a variety of forms, which determine their environmental mobilities and bioavailabilities [5]. If heavy metals exist as exchangeable or adsorbed forms on the surface of clay, organic matters and oxides with weak bonding strength, they tend to be easily moved and dispersed. However, metals complexed with organic ligands or in crystal lattices are not easily separated or mobilized. To determine the nature of any given system in terms of the chemical species present and their relative mobilities, sequential extraction analysis has been suggested [6–11]. This consists of several steps that allow one to determine the speciation of the contaminating metals, and may help to assess the risk of continued contamination in the long-term.

Decontamination of hazardous waste sites is one of the most important remaining technological challenges. Newly developed techniques for the remediation of soils can be generally classified into two groups. The first is biological remediation, which has been used primarily to detoxify organic contaminants. The other is physico-chemical decontamination, which has usually been applied to remove inorganic contaminants, including heavy metals. The process includes excavation, soil washing and flushing, solidification/stabilization, and electrokinetic soil processing [12]. Although many techniques have been proposed for removing contaminants from waste sites, most suffer from several technical and/or economic limitations [13,14].

Electrokinetic soil processing is also called electrokinetic remediation, electroreclamation, and electrochemical decontamination. It needs a low-level, direct current of the order of  $\text{mA cm}^{-2}$  across the cross-sectional area between electrodes to remove contaminants from soils [15]. The low-level, direct current results in physico-chemical and hydrological changes in the soil mass, and leads to species transport by coupled mechanisms. Electrolysis of water produces hydrogen ions in the anode compartment which causes an acid front to migrate through the soil cell. This, in turn, causes contaminants to be desorbed from the surfaces of soil particles, and results in an initiation of electromigration, i.e. the transport of ions and polar molecules under the influence of the electric field. The applied electrical potential also leads to the process of electroosmosis, i.e. the flow of an ionic liquid under the action of an applied electric field relative to a charged surface [16]. Electromigration and electroosmosis are important mechanisms in electrokinetic soil processing which remove contaminants from soil. Electrokinetic soil processing is an effective technology for the removal of contaminants in low-permeability soils, which range from clay to clayey sands. The advantages of this technology are its relatively low operating cost and its potential applicability to a wide range of contaminant types [17]. The potential of the technique has resulted in a number of studies [18–22]. Electrokinetic remediation technology has recently made significant advances and has been tested for commercial application in the United

States and The Netherlands. Geokinetics (The Netherlands) has successfully completed several field studies [23], and Electrokinetics Inc. (Baton Rouge, LA) has completed several large-scale pilot studies using 2–4 t soil specimens [15]. However, this technique has not been applied to remove heavy metals in tailing-soils and therefore, this investigation was undertaken.

The purposes of this work were to determine the speciations of heavy metals in tailing-soils by sequential extraction analysis and to explore the feasibility of electrokinetic soil processing for the removal of the heavy metals present. The removal efficiencies of different chemical forms of heavy metals in tailing-soils were determined by the different extraction methods.

## 2. Experimental

### 2.1. Preparation of tailing-soil sample and chemical analysis

Soils used in this experiment were tailing-soils taken from an abandoned mining area in Gubong, South Korea. In order to validate the analysis of metal contaminants, tailing-soils were pretreated by sieving (No. 80 sieve, mesh size 180  $\mu\text{m}$ ). For the electrokinetic removal experiment, 4 kg of tailing-soils were mixed with 1 l of distilled water to give a 20 wt.% water content. The slurries of tailing-soils were mixed mechanically for 1 h with an electric stirrer, and these mixtures were then allowed to settle for at least 3 days to stabilize. Triplicate 5 g samples was then taken to determine the initial concentration and chemical forms of the heavy metals. To analyze target metal contaminants, such as Cd, Cu, Pb and Zn in tailing-soils before and after the electrokinetic remediation treatment, metals were extracted from the tailings using 0.1N hydrochloric acid (HCl), aqua regia and by a sequential extraction method. Aqua regia and the sequential extraction method were applied to the analysis of all the target metal contaminants. 0.1N HCl was chosen for the analysis of only two metals, Cd and Pb, in order to monitor the removal trend of the most weakly bound fractions of these two metals, which show different adsorption capacities and mobilities in soils. The sequential extraction method used in this study (Table 1) was originally suggested by Tessier et al. [6] and later revised by the Environmental Geochemistry Research Group at Imperial College, UK [24]. The extracted solutions were analyzed by ICP–AES (Thermo Jarrel Ash, USA) at K-JIST, Korea.

Table 1  
The sequential extraction scheme used in this study

Fractions	Chemical extractants
Exchangeable	0.5 M $\text{MgCl}_2$ + $\text{NH}_4\text{OH}/\text{HOAc}$ (pH = 7)
Bound to carbonate or specially adsorbed	1 M $\text{NaOAc}$ + $\text{NH}_4\text{OH}/\text{HOAc}$ (pH = 5)
Bound to Fe and Mn oxides	0.04 M $\text{NH}_2\text{OH HCl}$ in 25% $\text{HOAc}$
Bound to organics and sulfides	0.02 M $\text{HNO}_3$ + 30% $\text{H}_2\text{O}_2$ + 3.2 M $\text{NH}_2\text{OAc}$ in 20% $\text{HNO}_3$ (pH = 2)
Residual	$\text{HF}/\text{HClO}_4/\text{HNO}_3$ (4:2:15)

## 2.2. Electrokinetic removal experiment

The experimental apparatus used in this study consisted of four principle parts; soil cell, electrode compartments, electrolyte solution reservoirs and power supply as shown in Fig. 1. The acrylic soil cell measured  $9\text{ cm} \times 9\text{ cm} \times 15\text{ cm}$  with a volume of  $1215\text{ cm}^3$ . Each end of the soil cell had 81 holes (diameter,  $0.5\text{ cm}$ ) to enhance uniform electroosmotic flow. At both sides of the soil cell, two sheets of GF/B filter paper were inserted to prevent soil particles from flowing into the electrode compartments. Platinum wires were placed at opposite ends of the soil cell to measure the overall voltage drop over the soil cell and the electrode compartments. Six stainless steel electrodes were then inserted equidistantly into the soil cell to measure the voltage drop between the sampling locations. A platinum mesh anode (overall size  $9\text{ cm} \times 9\text{ cm}$ , mesh size  $1\text{ cm} \times 1\text{ cm}$ ) was used to prevent electrode electrolysis reactions and a titanium plate ( $11\text{ cm} \times 11\text{ cm}$ ), was used as the cathode. The electrode compartments contained  $400\text{ ml}$  of electrolyte solution, which ensured that sufficient volume was present to avoid sudden variation of pH in the electrolyte solution. The compartments also had the function of allowing gas to escape from the electrodes, and to provide water for electroosmosis. Two cylinders (2l) were used as electrolyte solution reservoirs, and the water volume transported was measured. The electrolyte solutions were recirculated in both electrode compartments by peristaltic pumps (Masterflex, 1–100 rpm, three heads), and a BIORAD DC power supply (PowerPac 200, 5–200 V, 0.01–2 A, 200 W) was used.

Anode purging solutions of  $0.005\text{N H}_2\text{SO}_4$  (4l) were used over a 5-day period. Hydroxides were precipitated by hydroxide ions generated by the electrolysis of water in the cathode compartment. These precipitates prevented the removal of contaminants

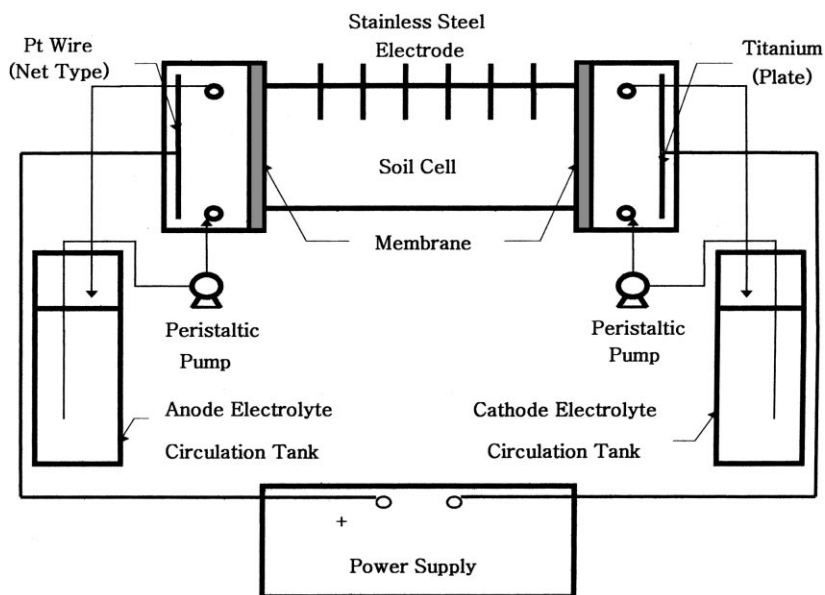


Fig. 1. Schematic diagram of experimental apparatus [25].

Table 2

Summary of experimental program for the electrokinetic removal of heavy metals

Parameters	Values
Soil specimen	Tailing-soils
Contaminants	Cd, Cu, Pb, Zn
Applied current (A)	0.1
Area of soil cell (cm <sup>2</sup> )	81
Length of soil cell (cm)	15
Duration (hr)	120
Anode purging solution	0.005N H <sub>2</sub> SO <sub>4</sub> solution 4 l
Cathode electrolyte solution	0.5N H <sub>2</sub> SO <sub>4</sub> solution 1 l

from the soil cell, and cathode electrolyte solutions may have buffered the concentration of hydroxide ions. Therefore, 1 l of 0.5N H<sub>2</sub>SO<sub>4</sub> solution was used as the cathode electrolyte solution. In order to enhance the effectiveness of the removal process, concentrations and volumes of anode purging and cathode electrolyte solutions were determined by preliminary experiments. A constant current (0.1 A) was used to keep the net rates of the electrolysis reactions constant and to minimize complicated current-boundary conditions. The parameters used in this study are summarized in Table 2.

The prepared tailing-soil samples were packed into the soil cell and the acrylic cover set into position, and sealed tightly with silicone bond to prevent leakage. The anode and cathode electrolyte solutions were pumped into the electrode compartments for 30 min without electric currents to equalize the electrolyte solutions. During the experiment, the overall voltage drops across the soil cell and electrode compartments; the pH and conductivity variations of anode purging and cathode electrolyte solutions; and the soil pH variation and transported porewater volume induced by electroosmotic flow were measured every 6 h. After 5 days of the experiment, the electroosmosis flowed reversely due to the decrease of the soil pH in the anode compartment of the soil cell. For this reason, the experiment was conducted for 5 days only. After the electrokinetic removal treatment, five samples were obtained from the soil bed using a stainless steel sampler (diameter, 1.2 cm) at 3 cm intervals to analyze the concentrations of contaminants and the soil pH. Heavy metals in the samples taken after the removal experiment were extracted with 0.1N HCl (for Cd and Pb), aqua regia and by the sequential extraction method (for all target metals), and all solutions were then analyzed by ICP–AES.

### 3. Results and discussion

#### 3.1. Initial concentrations and chemical speciations of heavy metals in tailing-soils

The composition of tailing-soils used in this study by particle size analysis was: sand (24.8%), silt (62.7%) and clay (12.5%). Table 3 and Fig. 2 show the initial concentrations and chemical forms of metal contaminants in tailing-soils determined by three different extraction methods. The total concentration of Pb and Zn was found to be very high, and Pb

Table 3

Initial concentrations of metal contaminants analyzed by different extraction methods<sup>a</sup>

Extraction methods	Fractions	Concentration (mg kg <sup>-1</sup> )			
		Cd	Cu	Pb	Zn
Sequential extraction	Exchangeable	3.7 ± 0.2	10.0 ± 0.5	262.7 ± 5.3	20.7 ± 0.7
	Carbonates	6.6 ± 0.3	10.3 ± 0.5	977.3 ± 35.7	213.1 ± 8.5
	Fe/Mn oxides	6.0 ± 0.3	7.0 ± 0.4	243.9 ± 5.1	293.8 ± 11.8
	Organic/sulfides	126.0 ± 5.6	112.6 ± 6.8	713.5 ± 23.9	5897.6 ± 294.9
	Residual	36.2 ± 1.5	67.2 ± 2.7	2977.5 ± 160.8	1174.5 ± 82.2
	Sum	178.5 ± 8.0	207.1 ± 7.2	5174.9 ± 326.0	7599.7 ± 456.0
Aqua regia extraction		133.4 ± 7.6	182.2 ± 6.1	3822.8 ± 191.1	5992.0 ± 359.5
0.1N HCl extraction		22.1 ± 0.8	–	1488.4 ± 59.5	–

<sup>a</sup> Values represent the mean of three duplicates ± standard deviation.

predominantly existed in the residual fraction. This may be explained by the facts that the sulfide fraction of the Pb transformed residual species and that the sulfide and organically bound fractions were extracted incompletely by the fourth step of the sequential extraction method. Except for Pb, the three other metals were mostly enriched in the organic and sulfide fractions. Because a complex extractant solution containing hydrofluoric acid (HF), which is a stronger agent than aqua regia, was used for the analysis of residual fractions, the cumulative concentrations analyzed by sequential extraction were higher than those obtained using aqua regia.

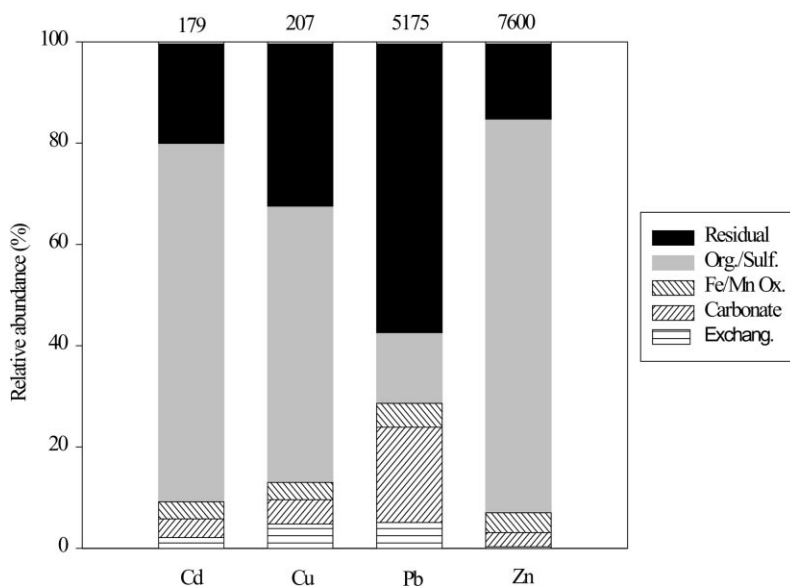


Fig. 2. Initial concentrations of different speciations analyzed by sequential extraction method (total metal concentrations are given in mg kg<sup>-1</sup> on top of each column).

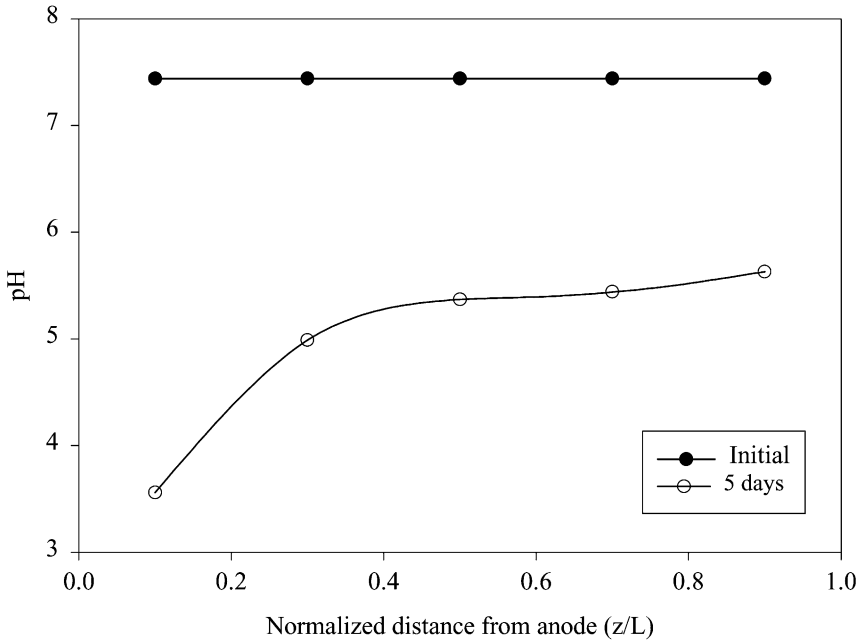


Fig. 3. Variation of pH in the soil cell after the electrokinetic removal experiment ( $z$  = distance from anode,  $L$  = length of soil cell).

### 3.2. Variation of pH and voltage drop in the soil cell during the electrokinetic removal experiment

After the electrokinetic removal experiment, the change of pH in the soil cell was measured at the same sampling points (Fig. 3). The electrolysis of water in the anode compartment generated hydrogen ions and these migrated from anode to cathode. Overall soil pH decreased over the experiment but this decrease was less marked near the cathode compartment. Although the experiment was relatively long (e.g. 5 days), the overall pH in the soil cell did not decrease substantially due to the relatively large pH buffering capacity of the tailing-soils, which decreases the dissolution and desorption rates of adsorbed and/or complexed species in the soil. Therefore, the rate of change of the pH gradient in the soil cell is a significant piece of information in terms of predicting the effectiveness of the electrokinetic remediation of heavy metal contaminated soils. This result is consistent with those of others [16,21,22].

Fig. 4 shows the overall voltage drops between the two electrodes positioned in the soil cell measured at 6 h intervals during the experiment. In the initial stage, the dissolution and desorption mechanism was dominant in the soil system, and this resulted in an increasing resistance and voltage drop within the soil cell. However, in the later stages of the treatment, the migration and transport of hydrogen ions and dissolved and/or desorbed species became more dominant, and the voltage drop decreased gradually. Therefore, the voltage variations

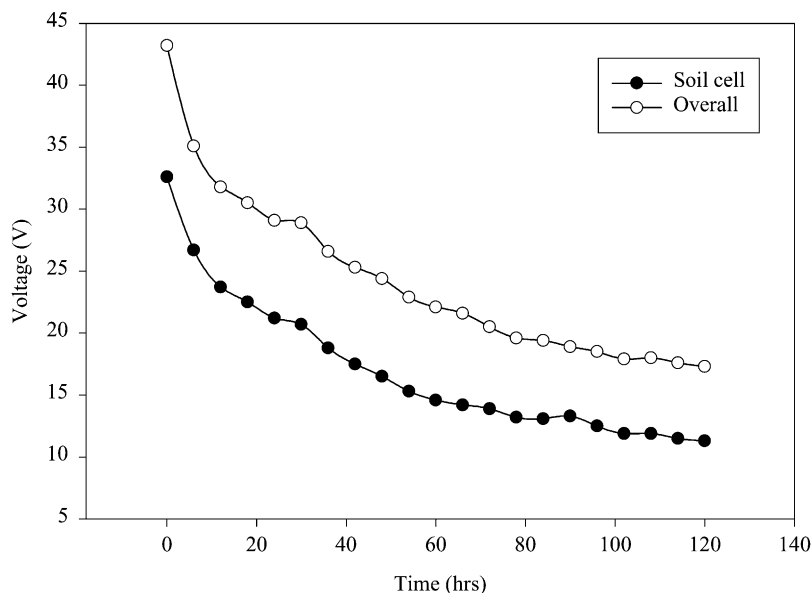


Fig. 4. The voltage profile during the electrokinetic removal experiment.

and trends can explain the resistance, power consumption and transport phenomena of the existing species in the soil system.

### 3.3. Water transport by electroosmosis

During electrokinetic soil processing, pore water is transported by electroosmosis, which is an important mechanism in terms of contaminant removal from soils. Soil pH, applied electric field intensity and soil permeability affect electroosmosis, as illustrated in Fig. 5. Since the pH and conductivity of the anode purging solution determine the pH and conductivity of the soil bed, the anode purging solution may affect the electroosmosis of soils. Electroosmotic velocity on a plane surface,  $U$  (m/s), is expressed as:

$$U = \frac{-\varepsilon\zeta E_x}{\mu} \quad (1)$$

where,  $\varepsilon$  is permittivity of the medium ( $\text{C V}^{-1} \text{ m}^{-1}$ ),  $\zeta$  the zeta potential (V),  $E_x$  the electric field ( $\text{V m}^{-1}$ ) parallel to the direction of electroosmotic flow and  $\mu$  is the viscosity of the medium ( $\text{N s m}^{-2}$ ). This formula for the electroosmotic velocity on a plane charged surface is known as the Helmholtz–Smoluchowski equation [14,17,20,26]. According to this equation (Eq. (1)), the electroosmotic velocity ( $U$ ) is proportional to the zeta potential ( $\zeta$ ). If the cationic concentrations in pore water are increased by increasing the cationic concentrations in the anode purging solution, the cation concentrations adsorbed onto the surfaces of the negative clay particles should also increase, and the increased concentration of adsorbed



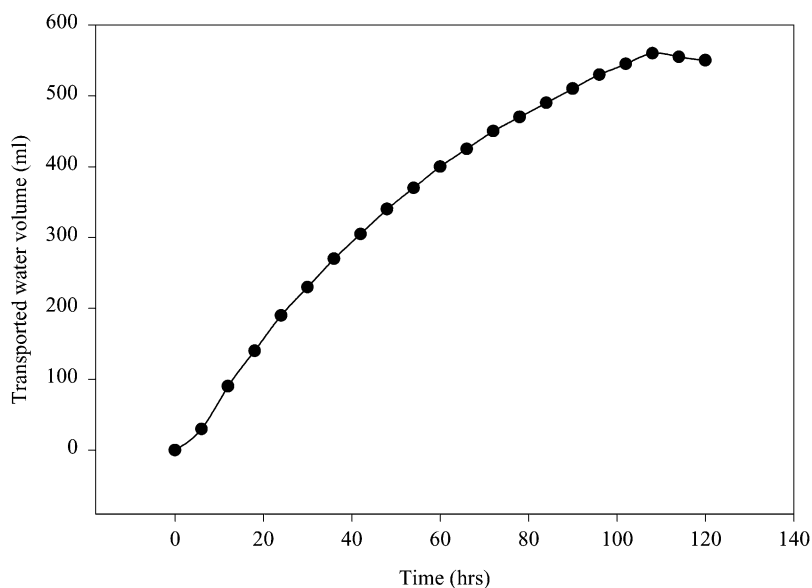


Fig. 5. The water transported by electroosmosis during the electrokinetic removal experiment.

cations would result in a reduction of zeta potential. The electroosmotic velocity then decreases by increasing the cationic concentration in the pore water. Consequently, a decrease of the soil pH and the increase of the conductivity of pore water make the electroosmotic velocity decrease. The volume of transported pore water decreased significantly from the middle stage of experiment (Fig. 5).

#### 3.4. Concentration variations of metal contaminants in the soil cell and removal efficiencies depending on their speciations

The variation of heavy metal concentrations in the soil cell was investigated on the basis of their chemical forms after electrokinetic treatment, as shown in Figs. 6–9. In the electric field, the acid front, produced by electrolysis of water in the anode compartment, migrated toward the cathode by electromigration and electroosmosis with dissolving or desorbing contaminants adsorbed and/or complexed on soil particles. Dissolution and desorption of species in the soil cell occurred concurrently with the acid front migration during treatment, and the contaminant species appeared to be gradually transported toward the cathode under the influence of the electric field. The migration of different contaminants showed similar trends, but these were dependent upon the availability of each species, and naturally, the overall level of soil contamination gradually decreased with time. As shown in Figs. 6–9, the more weakly bound fractions, such as exchangeable and carbonate fractions, the more effectively removed during the treatment. On the other hand, the strongly bound fractions, such as organic/sulfide and residual fractions, were not effectively removed by the electric-field process. The results show the significant dependency of the effectiveness of electrokinetic

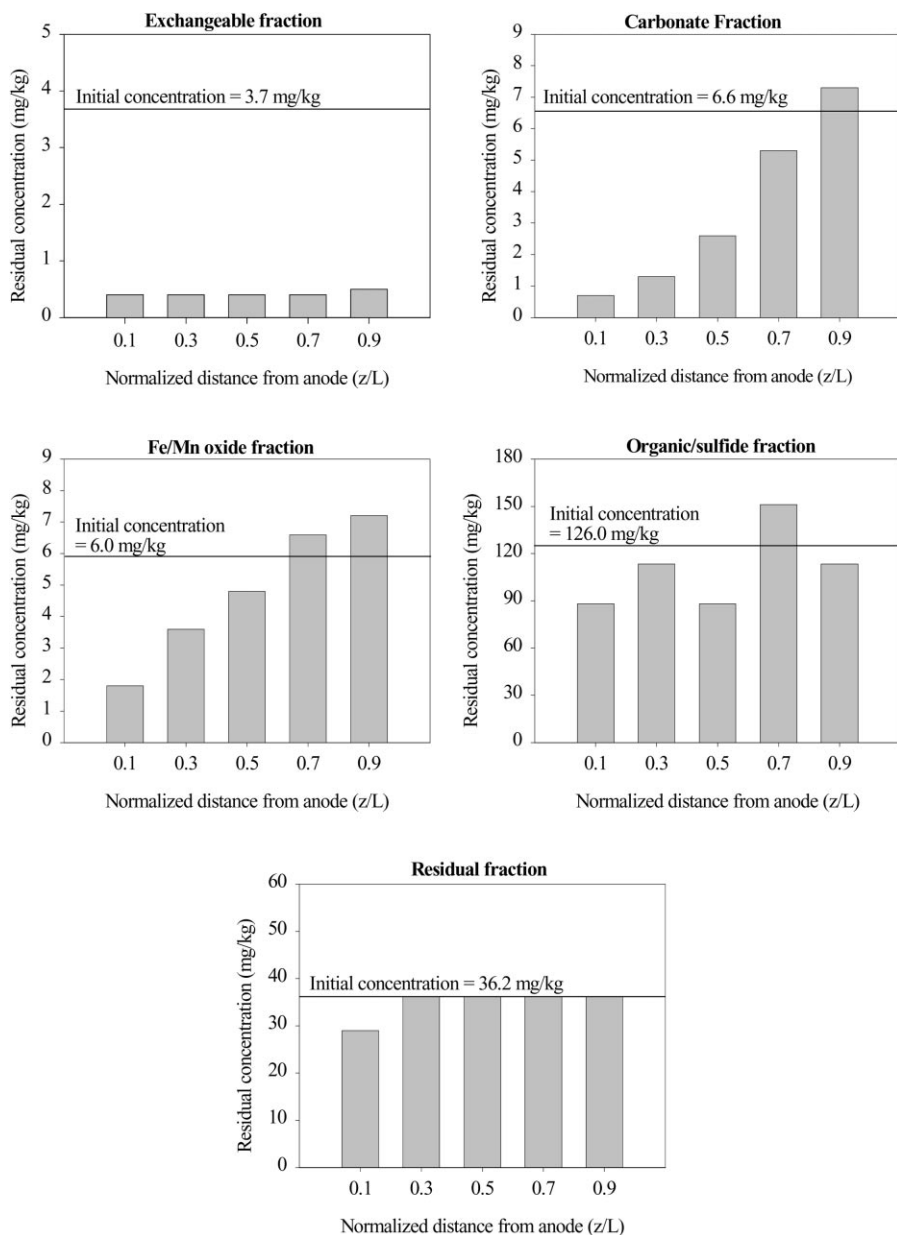


Fig. 6. The variation of Cd concentration in the soil cell depending on speciation after 5-day electrokinetic removal experiment ( $z$  = distance from anode,  $L$  = length of soil cell).

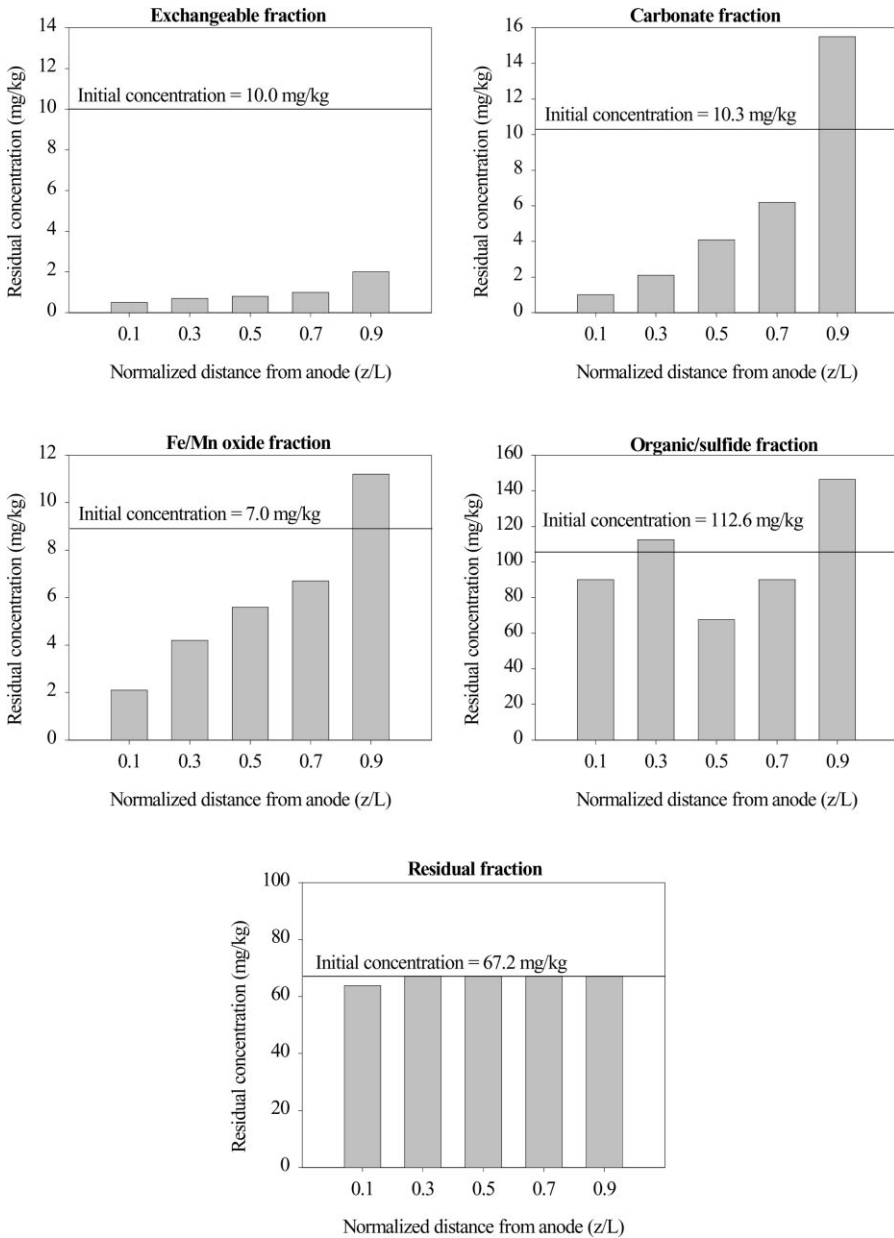


Fig. 7. The variation of Cu concentration in the soil cell depending on speciation after 5-day electrokinetic removal experiment ( $z$  = distance from anode,  $L$  = length of soil cell).

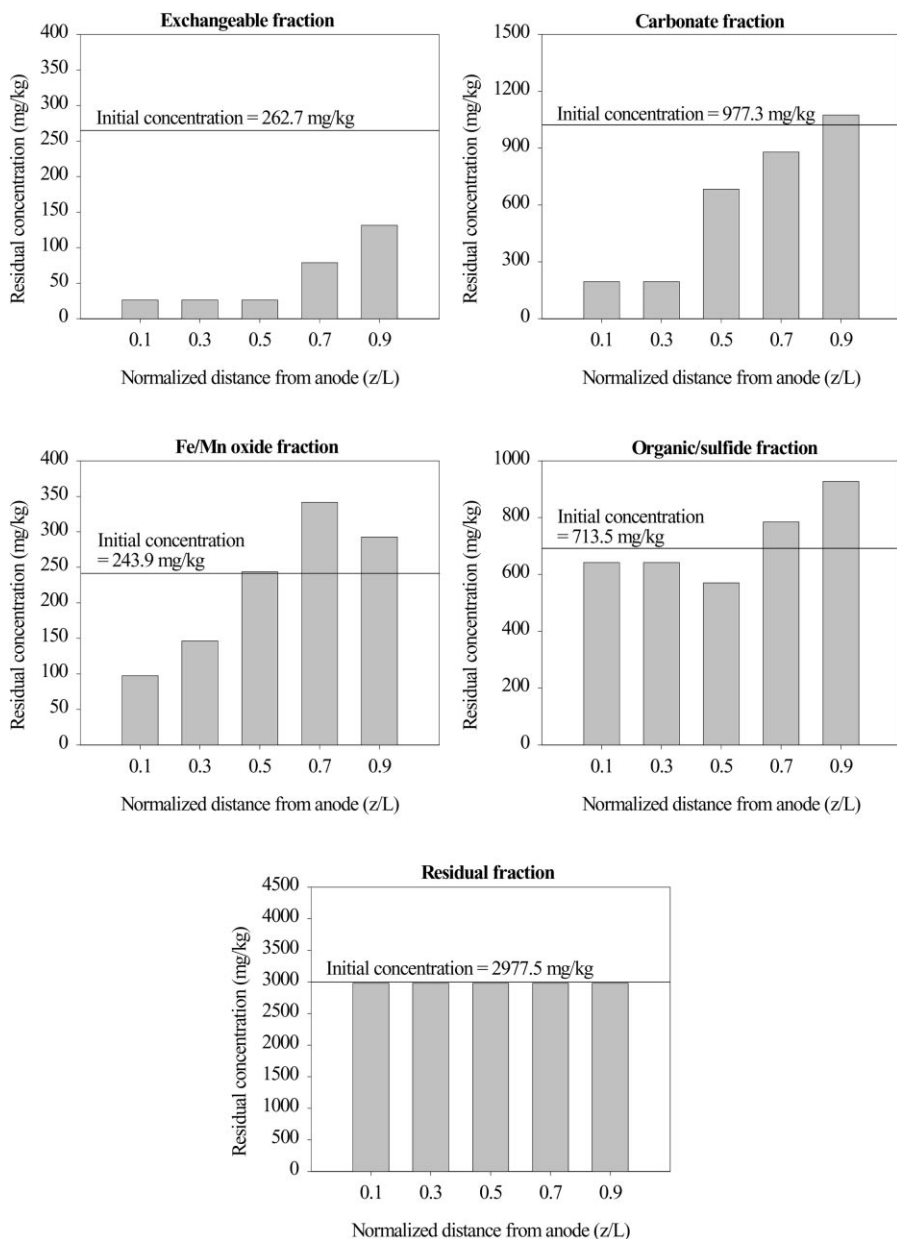


Fig. 8. The variation of Pb concentration in the soil cell depending on speciation after 5-day electrokinetic removal experiment ( $z$  = distance from anode,  $L$  = length of soil cell).

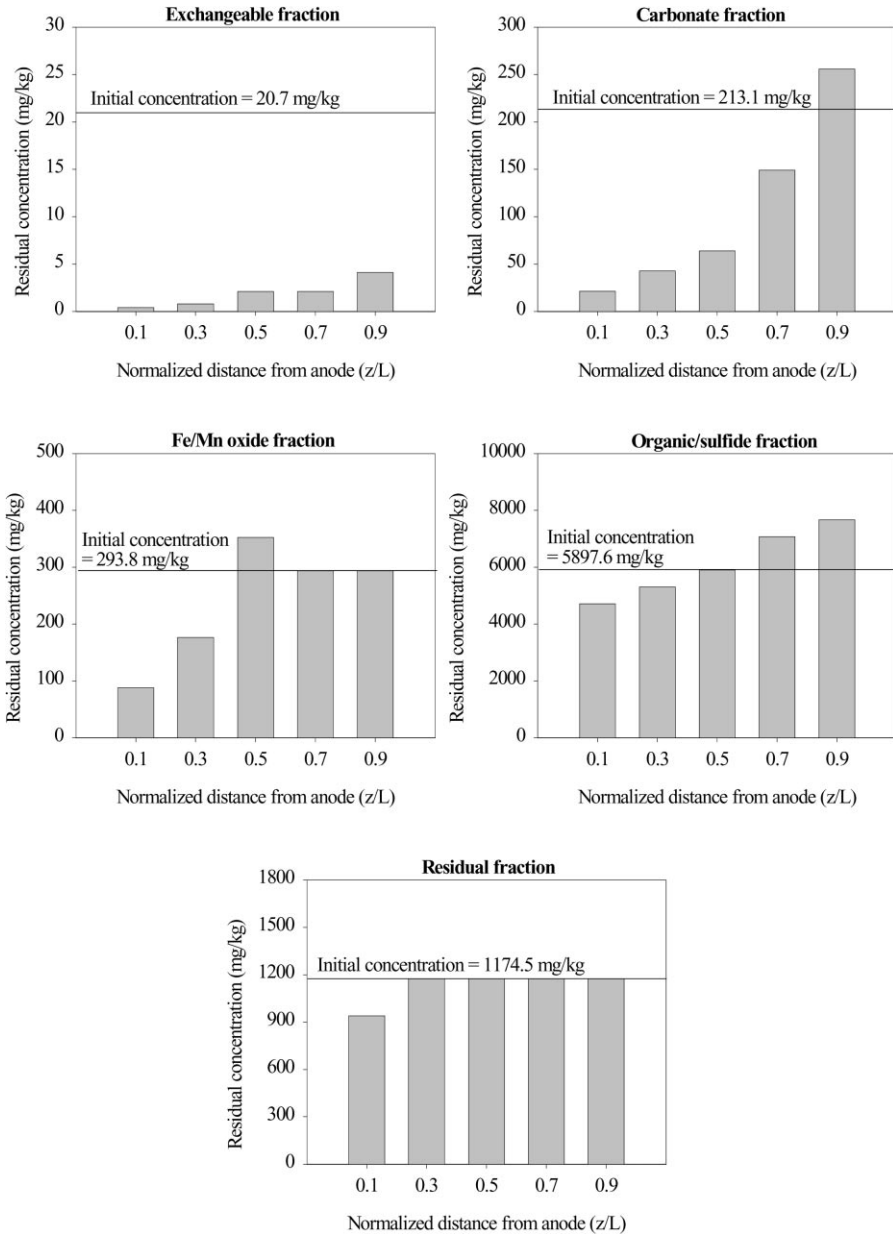


Fig. 9. The variation of Zn concentration in the soil cell depending on speciation after 5-day electrokinetic removal experiment ( $z$  = distance from anode,  $L$  = length of soil cell).

Table 4

The removal efficiencies of metal contaminants analyzed by different extraction methods after electrokinetic treatment

Extraction methods	Fractions	Removal efficiency (%) <sup>a</sup>			
		Cd	Cu	Pb	Zn
Sequential extraction	Exchangeable	94.6	94.8	90.3	95.4
	Carbonates	62.1	66.0	47.8	68.0
	Fe/Mn oxides	21.6	31.8	14.6	27.7
	Organic/sulfides	11.1	20.1	8.4	17.1
	Residual	3.1	2.3	0.3	3.4
	Overall <sup>b</sup>	13.4	20.6	15.6	17.0
Aqua regia extraction		12.5	14.8	10.2	17.4
0.1N HCl extraction		67.6	–	49.8	–

<sup>a</sup> (the initial concentration before treatment/the residual concentration after treatment)  $\times$  100.

<sup>b</sup> Overall efficiency =  $\sum$  (removal efficiency of each fraction  $\times$  percentage of initial concentration of each fraction).

technique on the speciations of metal contaminants. Additionally, the results indicate that the forms of the metals seemed to be altered due to the variation of physico-chemical conditions throughout the cell, such as pH, Eh, and local chemistry of both liquid and solid phase during the electrokinetic treatment. Generally speaking, the metals present in anodic region became partitioned in more mobile forms amenable to migration, whereas the metals near

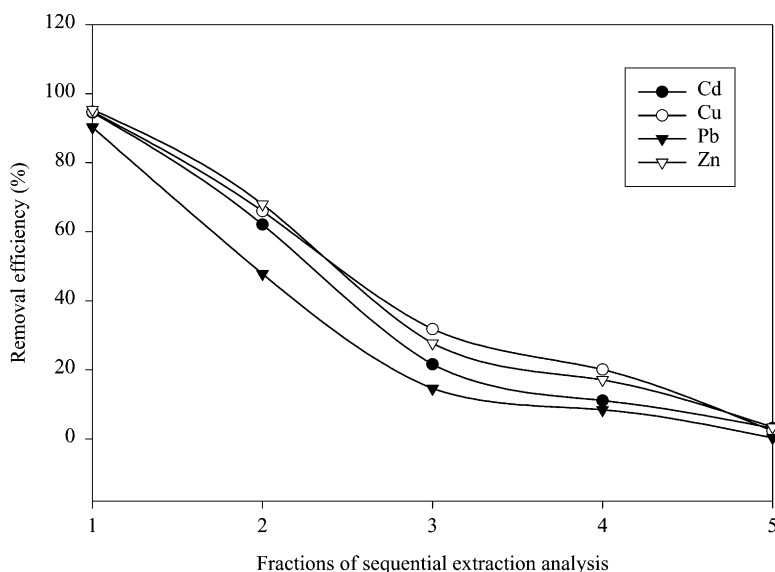


Fig. 10. The removal efficiencies of metal contaminants analyzed by sequential extraction method after the electrokinetic removal experiment (1) exchangeable fraction; (2) bound to carbonate or specially adsorbed fraction; (3) bound to Fe and Mn oxides fraction; (4) bound to organics and sulfides fraction; (5) residual fraction.

the cathode partitioned in less mobile forms, such as precipitated or adsorbed forms, such as the electrokinetic treatment proceeds. Even though metals initially existed in the residual form prior to treatment, part of them could be dissolved and mobilized in the region close to anodic compartment during the treatment, as shown in Figs. 6–9.

Table 4 and Fig. 10 show the removal efficiencies of the different contaminants expressed by the ratio of the amount remaining to that initially present. With the results of sequential extraction analysis, it was suggested that the more weakly bound fractions of metal contaminants were more easily removed by this electrokinetic technique. Differences between the removal efficiencies of the target metal contaminants resulted from their different mobilities and adsorption capacities in soils, e.g. the metals with higher mobilities in soils and weak affinities for the particulate surfaces are more easily removed (Fig. 10). From the results obtained upon the high removal efficiencies of certain weakly bound contaminants (Table 4), it is apparent that the electrokinetic technique can be adopted for the removal of highly mobile and bioavailable metal contaminants from soils, which may cause significant environmental problems. Although the total removal efficiencies calculated by the aqua regia analysis were relatively low, the removal efficiencies of more weakly bound fractions, such as the exchangeable and the carbonate fractions by the electrokinetic technique were reasonably high.

#### 4. Conclusions

From this study on the removal of heavy metals from tailing-soils by the electrokinetic technique, the following conclusions were made:

1. The tailing-soils used in this study contained high concentrations of heavy metals in various chemical forms. In order to precisely estimate the environmental impacts of metal contaminants in tailing-soils, their forms and the actual concentrations of the metal contaminants present were determined by various extraction methods. Pb was the highest contaminant among the target metals. Cadmium, Cu and Zn were mainly enriched in the organic and sulfide fractions, but the residual fraction was dominant in terms of Pb content in tailing-soils.
2. The removal efficiencies of different metals were significantly dependent on their chemical forms, mobilities and affinities (adsorption capacities) in the soil system. The more strongly bound fractions, such as the organic/sulfide and residual fractions were, the less effectively removed by the electrokinetic treatment. More than 90% of the exchangeable heavy metals in tailing-soils were removed by electrokinetic soil remediation, but less than 5% removal efficiency of the residual fraction was achieved. In accordance with the result of various analytical methods, the removal efficiency of Pb was lower than that of any other target metal contaminant, which is not surprising on the basis of its reputation for being an immobile element which has high affinity for soil.
3. As the acid front generated by the electrolysis of water in the anode compartment migrated gradually toward the cathode, the overall soil pH decreased. Complexed and/or adsorbed metal species were dissolved and desorbed by the migrating acid front. Variation in soil pH influenced the efficiency of the electrokinetic removal of heavy metals.

The overall decrease of soil pH was relatively small due to the pH buffering capacity of the tailing-soils. For this reason, the removal efficiency of metal contaminants was relatively low.

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