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Pulsed Electrokinetic Decontamination of Agricultural Lands around Abandoned Mines Contaminated with Heavy Metals

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Abstract: The feasibility of pulsed electrokinetic remediation under laboratory conditions was investigated using soil samples from contaminated agricultural land around an abandoned area. The major contaminants of the soil were zinc and cadmium. Sequential extraction showed that most heavy metals existed as strongly bound residual fractions, meaning that they were very difficult to remove. Based upon the overall removal of the cadmium and zinc, pulsed electrokinetic has an advantage over normal electrokinetics, and the electromigration was superior to the electroosmotic flow in this study. Catholyte circulation of 0.1 N HNO_3 in the cathode chamber enhanced the overall removal of heavy metals. The electric energy expenditure for pulsed electrokinetics was half of that for normal electrokinetics under identical conditions yet still had similar removal efficiency. Therefore, pulsed electrokinetics significantly affects energy consumption in a beneficial way. These results suggest that enhanced pulse eletrokinetic remediation is a cost-effective and economical technique.

Keywords: Catholyte, electric energy expenditure, pulsed electrokinetic, removal efficiency, sequential extraction analysis

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INTRODUCTION

Of the roughly 2,000 mines in Korea, more than 80% are inadequately abandoned with a large amount of mine tailings remaining near the mine sites. Many of these abandoned mine tailings contain high concentrations of various heavy metals including Zn, Cd, and As. These contaminants leach from the mine tailings; migrate into the surrounding streams, groundwater, and farm lands, and threaten human health (1). Currently, many agricultural lands near the abandoned mines are seriously contaminated with heavy metals and toxic inorganic materials. So far, several trial methods have been applied to remove inorganic materials from the contaminated soil (2,3).Generally, soil washing is used to remove heavy metals, but this is not suitable for agricultural lands in Korea, which often have a high proportion of clay or silt in the soil. Electrokinetic technology is a promising process for the remediation of low-permeability soils contaminated with heavy metals, radionuclides, certain organic compounds, or mixed inorganic species (4).

In electrokinetic remediation, electrodes are inserted into the contaminated zone and a low level direct current is applied to the electrodes to create an electric field (1,4–7). In an electric field, ionic species in the soil are transported through electromigration, electroosmotic flow, electrophoresis, and diffusion (4–9). The major removal mechanisms of heavy metals from contaminated soil during electrokinetic treatment are electromigration and electroosmotic flow (3–10). Electromigration is the transport of ions due to the electric potential gradient towards an electrode. Contaminant transport due to electromigration is greater than electroosmotic flow. Therefore, electromigration plays an important role in decontamination during electrokinetic treatment.

Heavy metals exist in various forms in the soil (11–15). Existing forms of heavy metals on the soil surface is the key parameter for measuring the desorption and transport of the contaminants. When these contaminants exist as weakly bound fractions such as the exchangeable and carbonate bound forms, these fractions of heavy metals are easily desorbed from the soil surface, and migrate and diffuse towards an electrode. On the other hand, heavy metals that are bound strongly to the soil matrix are very difficult to extract from the soil. Consequently, the fractional ratio of metal speciation needs to be considered to enhance the efficiency of the electrokinetic process. For many researchers, selective sequential extraction has been the method of choice to evaluate the fraction of metal speciation in soil (9,11–14).

In electrokinetic remediation, a few researchers have focused on changing the applied field from a normal direct current to a cyclic or pulsed field (16,17). This was tested to minimize or eliminate concentration gradients that could form during applications of a direct current field. Hansen and Rojo reported that pulsed electric fields seemed to increase the efficiency for copper removal from mine tailings (16). Reddy and Saicheck reported that a cyclic electric field improved the removal of phenanthrene from spiked clay (8).

In this study, the feasibility of pulsed electrokinetic remediation to remove zinc and cadmium from contaminated soil was investigated in the laboratory with soil samples using the analytical procedure of sequential chemical extraction. The enhanced electrokinetic remediation using a pulsed electric field was expected to diminish the polarization process and to increase the desorption of heavy metals from the soil surface as well as reduce the electrical power consumption.

MATERIALS AND METHOD

Soil Sampling and Preparation

The soil used in this study was taken from the surface soil (depth $\leq$30 cm) of a rice field around the Sambo abandoned mine, which used to extract manganese steel in Youngduk, Republic of Korea. The sampled soil was air-dried and passed through a No.10 mesh sieve ($\leq$2.0 μm). The properties of the soil are shown in Table 1. The initial pH of the soil was 5.0, and the initial concentrations of zinc and cadmium were 2,120 and 6.27 mg/kg respectively. A mass of 4.0 kg of soil with grain sizes of less than 2.0 μm was mixed with 1 liter of tap water to form a sample with

Table 1. Properties of soil and initial concentration of heavy metals

Properties	Contents
Initial pH (KSTM)	5.0
Electric conductivity (μS/cm) (KSTM)	150.5
CEC (meq/100 g) (ASTM)	124.14
Organic contents (%) (ASTM)	2.92
Particle distribution (ASTM)	
Clay (%)	8
Silt (%)	50.27
Sand (%)	41.73
Initial concentration (KSTM)	
Zinc (mg/kg) (Aqua regia)	2120
Cadmium (mg/kg) (0.1 N HCl)	6.27

20 wt% water content. The mixtures were saturated with water for 24 h and then inserted into the electrokinetic cell.

Experimental Setup

Figure 1 shows the schematic diagram and dimensions of the electrokinetic setup used in this test. The apparatus basically consists of a soil compartment (4:4:20 cm), two electrode chambers, two electro-reservoirs, and a power supply. A mesh-type electrode (4:4 cm) of titanium coated with platinum was used as the anode to avoid corrosion of electrode; a plate electrode of graphite (4:4 cm) was used for the cathode. Filter paper was inserted between the soil compartment and electrode chamber to prevent soil particles from moving away from the soil compartment. Gas vents were installed to allow the gases caused by the electrolysis reaction in the electrode chamber to escape, and a peristaltic pump was used to circulate electrolytes into the chambers at a flow rate of 4 mL min^{-1}.

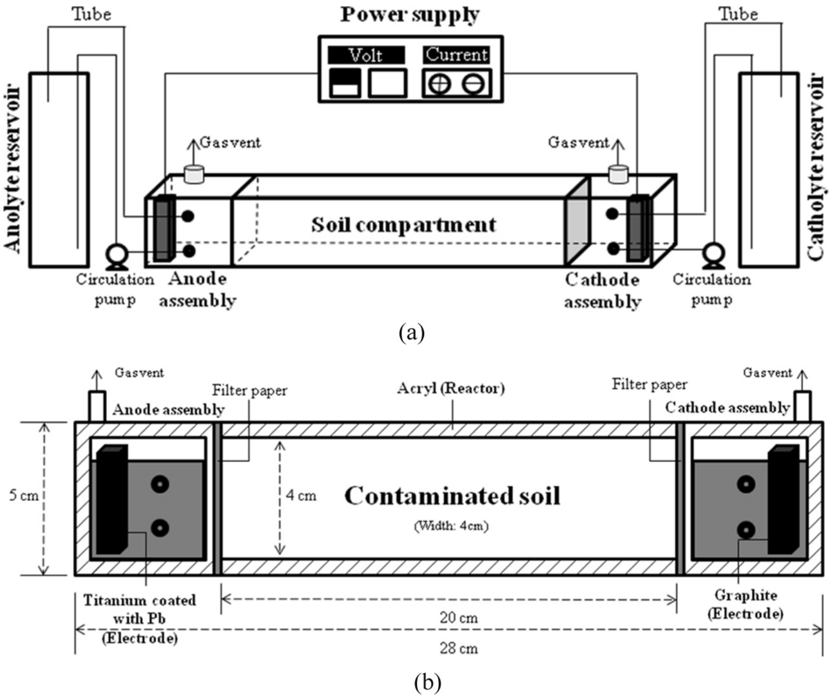

Figure 1. Schematic and dimension of electrokinetic apparatus. (a) Schematic of electrokinetic set-up and (b) dimension and cell detail of electrokinetic apparatus.

Experimental Program

The experimental conditions for four different tests are summarized in Table 2. In general, a constant voltage gradient of 1 V/cm was applied to the electrodes over 14 days. The power supply providing a direct current was used for Exps. 1 and 2, while the pulsed power supply was applied to Exps. 3 and 4. A solution of 0.1 M $MgSO_4$ was circulated in the anode chamber to supply a sufficient amount of ions into the soil cell. At alkaline conditions, the mobility of zinc or cadmium seriously diminished because cationic metals form metal hydroxide near the cathode area. Because of this, 0.1 N HNO_3 was supplied to the cathode chamber to prevent the formulation of immobile precipitates and to increase the mobility of cationic heavy metals. Nitric acid was used as a catholyte for Exps. 2 and 4. When the pulsed power supply equipment is "on," an electric current is supplied. However, when it is "off," there is no electric current and the polarization generated when it was "on" is reduced (15). For Exps. 3 and 4, the pulse cycle was adjusted to 2 s intervals consisting of 1 s each of "on" and "off" time respectively.

Chemical Analysis

The Korean standard test method (KSTM) for soil was used to analyze the concentration of Zn and Cd in soil; the sequential extraction method suggested by Tessier et al. (15) was used to investigate the speciation of metals in the soil. The residual fraction was calculated by subtracting the other fractions from aqua regia extraction. For the Cd analysis, 0.1 N HCl was used to extract Cd from the soil at a solid/liquid ratio of 1:5. After extraction, the mixture was filtered to separate the extractant from the soil; the extractant was then analyzed using an atomic absorption spectrophotometer (AAS, AA 6300, Shimadzu, Japan). Aqua regia extraction was used to extract Zn from the soil. The details for the experimental method are summarized in Table 3.

Table 2. Experimental conditions

	The type of power supply	Electrolyte		Period of pulse (ON/OFF)	Duration (days)	Voltage gradient (V/cm)
		Anolyte	Catholyte			
Exp. 1	Normal (DC)	0.1 M $MgSO_4$	Tab water	–	14	1
Exp. 2	Normal (DC)	0.1 M $MgSO_4$	0.1 N HNO_3	–	14	1
Exp. 3	Pulse	0.1 M $MgSO_4$	Tab water	1sec:1sec	14	1
Exp. 4	Pulse	0.1 M $MgSO_4$	0.1 N HNO_3	1sec:1sec	14	1

Table 3. The method of extraction heavy metals and analysis equipment

Extraction method	Solution used for extraction	Analysis
Soluble formation	0.1 N HCl	AAS (AA-6300, Shimadzu)
Aqua regia	$HCl + HNO_3$ (3:1)	
Sequential extraction		
Exchangeable	1 M NaOAc (Ph = 8.2)	ICP-OES (730ES, Varian)
Bound to Carbonate	1 M NaOAc (pH = 5)	
Bound to Fe/Mn oxide	0.04 M $NH_2OH \cdot HCl$ in 25% HOAc	
Bound to organics and sulfides	3.2 M NH_2OAc in 20% HNO_3+0.02 M HNO_3 +30% H_2O_2	
Residual (Aqua regia)[a]	$HCl + HNO_3$ (3:1)	

[a] The residual fraction was calculated by subtracting the other fractions from the aqua regia extraction.

RESULTS AND DISCUSSION

pH Distribution and Electric Conductivity

Figure 2 shows the distribution of soil pH after electrokinetic treatment. The change in pH was directly related to the electrolysis reaction and catholyte purging using nitric acid. Hydrogen ions generated by electrolysis at the anode migrated toward the cathode and contributed to the low pH around the anode. The soil pH in the anode region (5.02) was lower than the initial pH value; the acidic pH enhanced the ion exchange reaction between the hydrogen ions and the cationic metals adsorbed onto the soil surface, which allowed the desorbed cationic metals to move toward the cathode through the electromigration and electroosmotic flow mechanisms more easily.

In Exps. 1 and 3, hydroxyl ions produced at the cathode migrated toward the anode and maintained the high pH near the cathode. In Exps. 2 and 4, however, the circulation of 0.1 N HNO_3 as a catholyte purging solution compensated for the hydroxyl ions, so the pH of the soil around cathode is similar to the pH value of initial soil. The circulation of the low pH solution created acidic conditions in the cathode chamber and prevented the precipitation of the cationic ions in alkaline conditions.

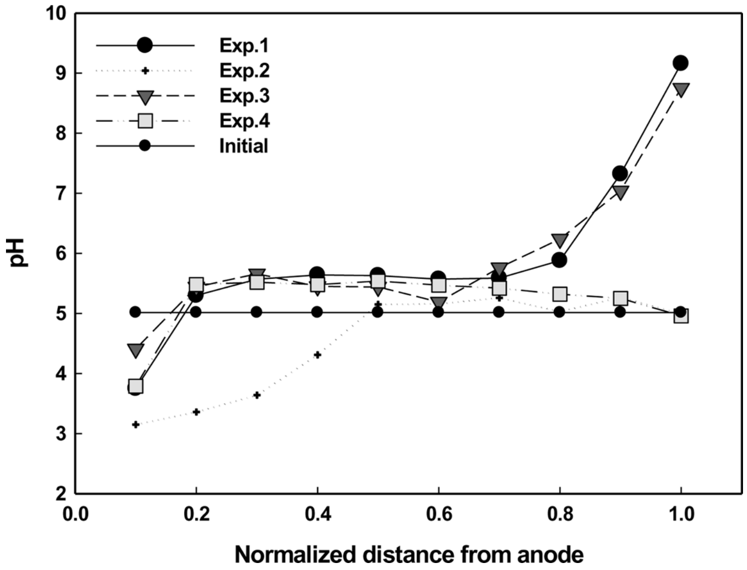

Figure 2. The variation of pH in soil compartment after electrokinetic treatments.

Figure 3 shows the variation in electric conductivity (EC) for the soil compartment after electrokinetic treatment. EC is highly correlated to the concentration of exchangeable ions, such as salts, existing in the soil or pore water. The overall EC value increased compared to the initial value after electrokinetic treatment because the electromigration and electroosmotic flow caused by the electric field provided ions to the soil. In particular, nitrates from the catholyte purging solution were easily transported toward the anode by electromigration because nitrate adsorption onto the soil surface was negligible.

Electrical Current and EOF

The variation of electrical current during electrokinetic experiments is shown in Fig. 4. To interpret the variation in current, two points need to be considered: (1) the catholyte purging solution and (2) the type of power (pulsed vs. normal). The catholyte purging solution HNO_3 supplied a huge amount of ions to soil compared to scenarios with no purging; the ions increased the current and decreased electrical resistance. A higher current in Exps. 2 and 4 across the soil section was observed in comparison with Exps. 1 and 3. For Exps. 1 and 3—which had no catholyte purging—the current increased gradually during the test period.

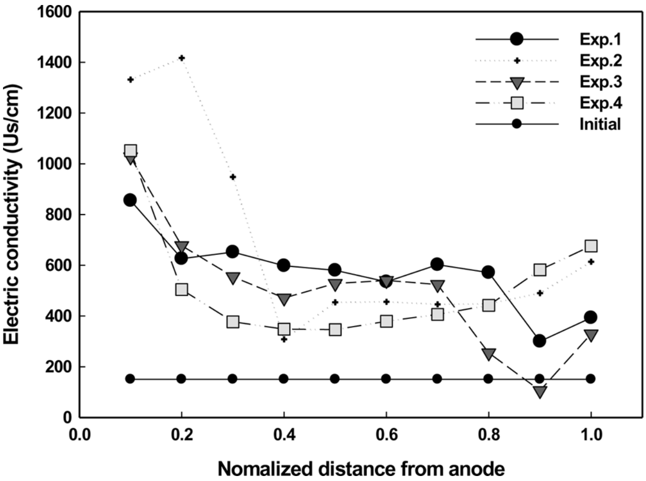

Figure 3. The distribution of electric conductivity in soil cell after electrokinetic experiments.

The electrical resistance in soil decreased gradually due to the continuous supply of ions generated by electrolysis. Also, the current for experiments using normal power (Exps. 1 and 2) was higher than that for pulsed

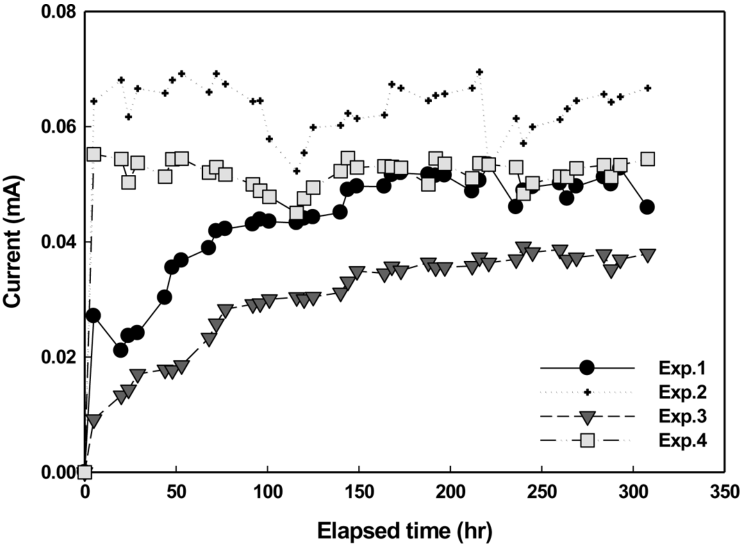

Figure 4. The variation of electrical current during electrokinetic tests.

power (Exps. 3 and 4) because the pulsed power provided the soil compartment with electrical energy intermittently, decreasing the transport of ions toward an electrode. However, the higher electrical current also caused higher energy consumption.

Figure 5 shows the accumulated electroosmotic flow during electrokinetic remediation. The direction of electroosmotic flow was from anode to cathode. Mitchell defined the electroosmotic flow rate q_{eo} through the following conduction equation [6]:

$$Q_{eo} = k_{eo} E_x A,$$

where k_{eo} is the electroosmotic permeability (cm/s)/(V/cm), E_x denotes the average electric field strength or gradient (V/cm), and A is the cross-sectional area of flow. According to this equation, the electroosmotic flow rate is highly related and proportional to the value of k_{eo}, E_x, and A. In this test, the critical point for determining the electroosmotic flow rate was the voltage gradient (V/cm). Even though the exact same constant voltage gradient of 1 V/cm was applied to all experiments, the period of applied voltage was different between the normal and pulsed power supplies. Consequently, the flow rate for normal power (Exp. 1) was approximately twice as high as for pulsed power (Exp. 3). For Exps. 3 and 4, the electroosmotic flow rate could not be measured accurately due to the catholyte purging.

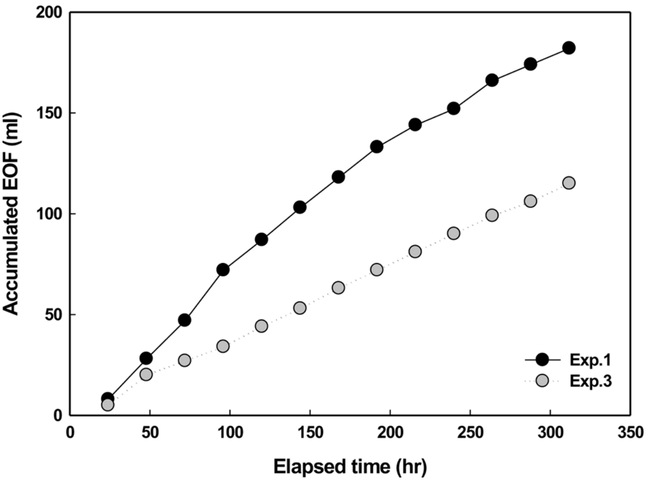

Figure 5. Accumulated electroosmotic flow with elapsed time.

Removal of Cd and Zn and Energy Consumption

Figure 6 shows the distribution of Zn in the soil cell after electrokinetic treatment. The removal of Zn was 2.1%, 23.4%, 14.0%, and 22.9% for Exps. 1, 2, 3, and 4 respectively. In particular, the residual fraction of Zn in the soil near the anode was significantly reduced when catholyte purging using 0.1 N HNO_3 was employed. In acidic conditions, the Zn adsorbed on the soil surface was exchanged with hydrogen ions generated by electrolysis, and then migrated toward the cathode by electromigration, contributing to the positive electroosmotic flow. In Exps. 1 and 3, the removal efficiency was relatively lower than for Exps. 2 and 4 because Zn was transported toward the cathode by electromigration and electroosmosis, formed a hydroxide complex with hydroxyl ions, and was precipitated at neutral or alkaline conditions. The catholyte purging with an acidic solution maintained an acidic pH near the cathode, preventing the precipitation of Zn with hydroxide. When considering the electroosmotic flow of Exps. 1 and 3 as shown in Fig. 5, it should be noted that the major mechanism for Zn removal was electromigration. The removal efficiency for Exp. 3 was higher than for Exp. 1, meaning that pulsed electrokinetic remediation is an effective technique for removing Zn from soil. On the other hand, Zn removal efficiency of Exp. 2 was slightly higher than Exp. 4 because desorption of Zn in acidic condition by the circulation of nitric acid is superior to the effectiveness of pulse power.

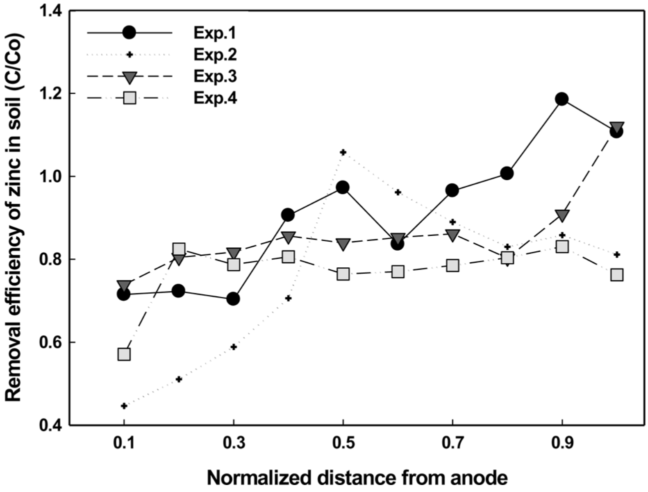

Figure 6. The removal efficiency (C/C_0) of zinc in soil compartment after electrokinetic remediation.

However, pulsed-electrokinetic is more economically reasonable method than normal electrokinetic in same experiment conditions in considering energy expenditure.

Figure 7 shows the distribution of Cd in the soil cell after electrokinetic treatment. The removal efficiency of Cd was 32.1%, 21.5%, −24.0%, and −15.7% for Exps. 1, 2, 3, and 4, respectively. In this study, only the soluble form of Cd was extracted from the soil by 0.1 N HCl. However, the other forms strongly bound to the soil matrix were changed into more weakly bound forms through exterior physicochemical reactions when pulsed electrical energy was applied to the soil cell. One probable reason was that the pulsed power enhanced desorption of the metals from the soil surface. Because of this, the concentration of Cd in Exp. 3 and 4 after treatment seemed to be higher than the initial concentration. However, catholyte purging was not effective in removing Cd from the soil.

The electrical energy expenditure is shown in Table 4 and Fig. 8. The energy consumption calculated based upon the amount of electrical current was 269.2, 387.5, 92.9, and 158.7 Wh for Exps. 1 (normal), 2 (normal and catholyte purging), 3 (pulsed), and 4 (pulsed and catholyte purging) respectively. Although 2.1% and 14.0% of Zn were removed in Exp. 1 and 3 respectively, the energy consumption in Exp. 3 was one-third that of Exp. 1. The Zn removal rate was similar for Exps. 2 and 4, but the energy expenditure of Exp. 2 was 2.5 times higher than for Exp. 4. In general, catholyte purging spent more electrical energy

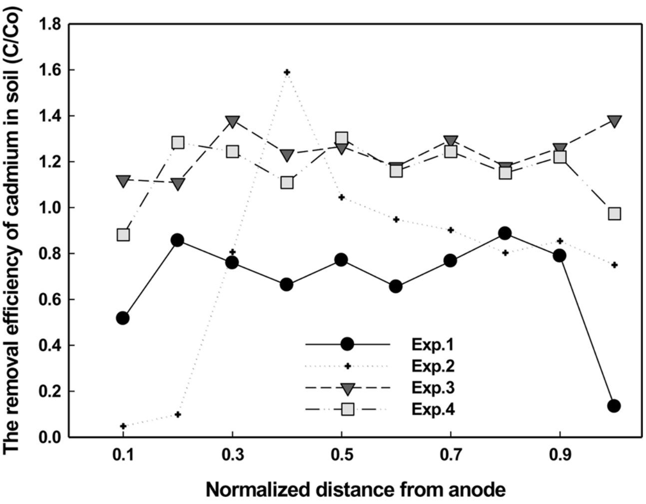

Figure 7. The removal efficiency (C/C_0) of cadmium in soil compartment after electrokinetic remediation.

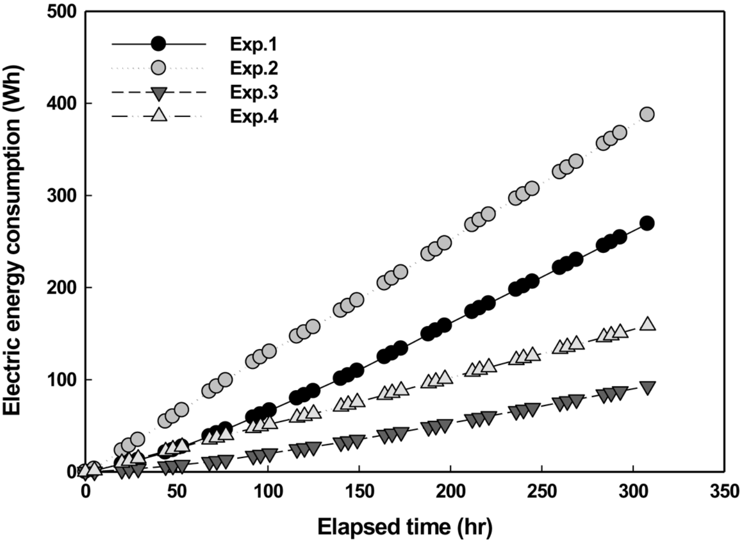

Figure 8. The accumulated electric energy consumption with elapsed time.

because the abundance of electrolytes increased the electric current, causing higher energy consumption. In light of this, pulsed electrokinetic remediation is a very cost-effective technique.

Distribution of Zn and Cd in Soil

The variation in fractionation of Cd and Zn in soil was investigated by using the sequential extraction method. Table 5 shows the fraction of heavy metals in original soil before the application of electrokinetic treatment. The fractions weakly bound (exchangeable or carbonate bound) to the soil are very sensitive and susceptible to pH, and can be desorbed

Table 4. The removal efficiency of zinc and cadmium and electric energy consumption

Experiment	Zn removal (%)	Cd removal (%)	Energy consumption (Wh)
Exp. 1	2.1	32.1	269.2
Exp. 2	23.4	21.5	387.5
Exp. 3	14.0	−24.0	92.9
Exp. 4	22.9	−15.7	158.7

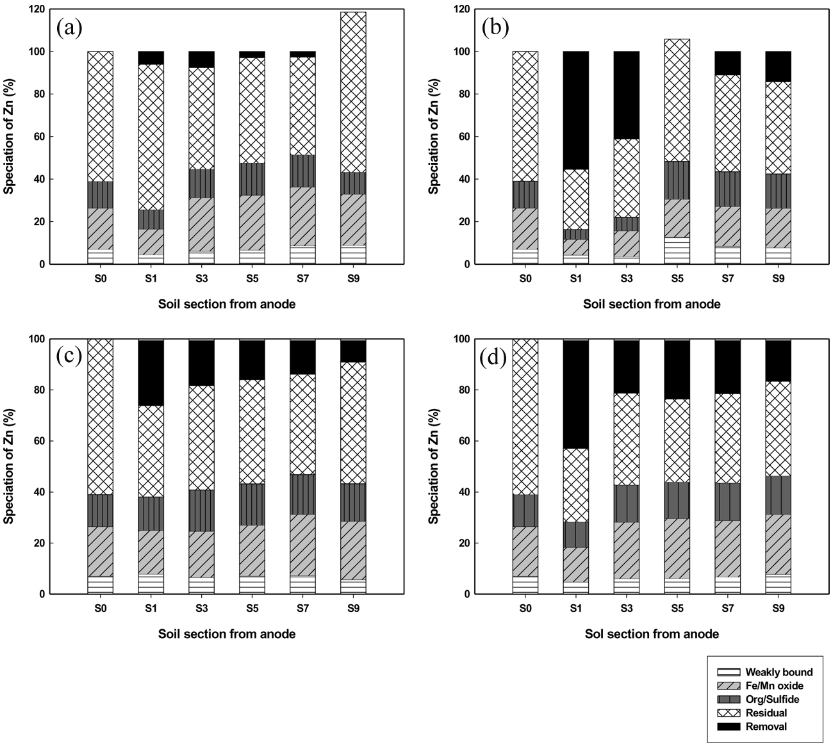

Figure 9. The distribution of zinc in soil depended on the change of fraction after electrokinetic remediation. (Weakly bound = exchangeable + bound to carbonate). (a) Exp. 1, (b) Exp. 2, (c) Exp. 3, and (d) Exp. 4.

from soil quite easily. On the other hand, the species strongly bound to the soil are considerably immobile. The speciation and relative portion of each fraction influence the removal of metals in electrokinetic remediation. In this test, 61% of Zn and 57.67% of Cd existed as residual fractions. The exchangeable and carbonate-bound fractions were 1.27% and 5.77% for Zn and 5.09% and 7.75% for Cd, respectively. This indicated that the heavy metals remaining in the soil were difficult to remove and low removal rates for Zn and Cd were to be expected.

Figures 9 and 10 present the distribution of Zn and Cd speciation in the soil after electrokinetic treatment. Weakly bound fractions of Zn were expected to move actively and to be mostly eliminated from the soil. However, in actuality the residual fraction near the cathode region clearly increased (Fig. 9(a)). The Cd extracted by 0.1 N HCl in Exps. 3 and 4 was shown to be present in higher amounts than the initial concentration, as

Table 5. The concentration and composition percentage of heavy metals depend on each fraction prior to electrokinetic experiment

	Zn		Cd	
Fraction	Conc. (mg/kg)	Composition (%)	Conc. (mg/kg)	Composition (%)
Exchangeable	27.02	1.27	0.65	5.09
Bound to Carbonate	122.39	5.77	0.98	7.75
Bound to Fe-Mn oxides	410.83	19.38	3.24	25.59
Bound to Organic matter	265.70	12.53	0.49	3.89
Residual	1294.06	61.04	7.30	57.67
Total	2120.00	100	12.66	100

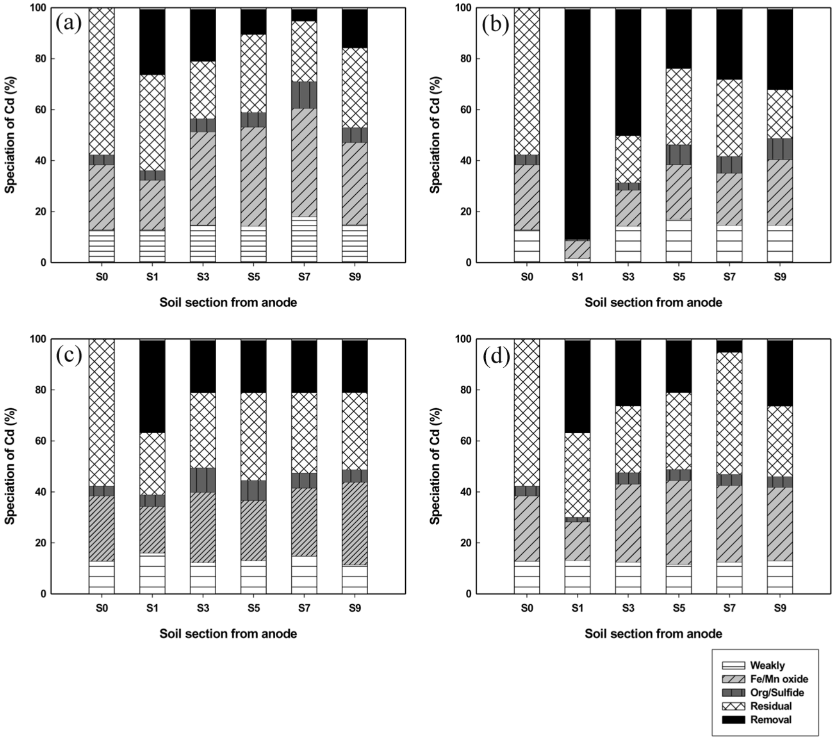

Figure 10. The distribution of cadmium in soil depended on the change of fraction after electrokinetic remediation. (a) Exp. 1, (b) Exp. 2, (c) Exp. 3, and (d) Exp. 4.

shown in Fig. 7. The transport of the weakly bound fraction in Exps. 3 (Fig. 10(c)) and 4 (Fig. 10(d)) showed that the residual fraction decreased after electrokinetic treatment but the weakly bound fraction increased. This means that heavy metals strongly adsorbed onto the soil surface can change into other fractions, such as exchangeable or carbonate bound. In terms of the total mass of Cd, approximately 20% of Cd was removed from the soil through the pulsed electrokinetic technique (Figs. 10(c) and 10(d)).

CONCLUSION

The feasibility of pulsed electrokinetic process was evaluated using Zn- and Cd-contaminated agricultural field soil in the laboratory. Even though normal electrokinetic remediation showed a higher electroosmotic flow, the overall removal efficiency of the pulsed electrokinetic process was higher. This result demonstrated that electromigration was the major removal mechanism for this study. The overall removal efficiency was not high enough because more than 50% of the Zn and Cd existed as a residual fraction strongly bound to the soil surface. Pulsed electrokinetics changed the residual fraction into weakly bound fractions. The electric energy expenditure for pulsed electrokinetic was less than a half of the energy consumption for normal electrokinetic under the same conditions, and removal efficiency was similar. Therefore, pulsed power has a great energy consumption advantage for applications of electrokinetic remediation.

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