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ORIGINAL PAPER

Pulsed electrokinetic removal of Cd and Zn from fine-grained soil

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Abstract Pulsed electrokinetics studies were carried out to optimize the removal of Zn and Cd from fine-grained soils and to observe the effects of varying the pulse frequency, pulse time ratio (on/off), and DC voltage gradient. Existing forms of heavy metals in the soil matrix were determined using a sequential extraction method. The strongly bound fraction (bound to organic matter and residuals) that is difficult to remove from the soil matrix comprised 74 and 62% of the total Zn and Cd, respectively. In the electrokinetic remediation experiments, $MgSO_4$ was employed to increase the ionic strength of the soil for 2 weeks. Transportation of heavy metals was influenced by the frequency, pulse ratio, and the voltage gradient of the pulsed electric field. Extraction efficiency of Zn and Cd near the anode was correlated positively with the voltage gradient at a given pulse and ratio. A high pulse frequency (1,800 cycles/h) enhanced the removal efficiency of the heavy metals compared to a low pulse frequency (1,200 cycles/h) at a supplied voltage gradient of 1 V/cm. Although pulsed electrokinetics was more effective in extracting and desorbing ions near the anode than conventional electrokinetics, its ability to transport heavy metals from the anode to the cathode was relatively small. Total removals with pulsed electrokinetics were 21–31% for Zn and 18–24% for Cd. In summary, pulsed electrokinetics can enhance removal efficiency of heavy metals and is beneficial with regard to electrical energy consumption.

Keywords Pulse frequency · Pulse ratio · Electrical energy consumption · Heavy metal removal

1 Introduction

Generally, mine tailings contain a variety of toxic metalloids and heavy metals, such as arsenic, cadmium, zinc, lead, and copper, and present significant risks to human health and the surrounding environment. In the Republic of Korea, 1,082 abandoned metal mines exist and approximately 396 of these abandoned metal mines require remediation due to serious contamination with toxic materials. However, most of the stacked mine tailings around the abandoned mines have been left without proper treatment that would prevent environmental pollution, and in addition, large amounts of heavy metals adsorbed onto the mine tailings are available to leach into streams, ground water systems, and surrounding farm lands through oxidation processes [1–4].

At present, many agricultural fields in Korea having a high proportion of silt and clay have become contaminated with various heavy metals from abandoned mine tailings [5]. Electrokinetic remediation is a promising technique for various low permeability matrixes, including silty/clayey soils, mine tailings, saline soils, industrial sites, harbor sediments, sewage sludge, and treated wood contaminated with heavy metals, mixed inorganic species, and organic

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pollutants [3–15]. In electrokinetic remediation systems, charged species migrate toward anodes and cathodes through electro-migration, electro-osmotic flow, electrophoresis, and diffusion [16–18]. However, electro-migration and electro-osmotic flow are the primary mechanisms of heavy metal transport [17].

In order to enhance the mobility and removal of heavy metals in electric fields, several techniques have been applied to electrokinetic systems, such as control of the pH of the electrolyte to prevent metal-hydroxide precipitation [19–22], use of various chelating reagents [7, 9], ion-exchange membranes [13, 20], the polarity exchange technique [23], and pulsed voltage [19, 24, 25]. In this study, a pulsed power supply was used to increase extraction of heavy metals from the soil surface and to decrease polarization and electrical energy consumption by periodically supplying an electric field [19]. Rojo and Hansen have reported on the effectiveness of electrodialytic remediation with a pulsed electric field for removing copper from mine tailings [24]. Reddy and Saichek found that application of periodic electric potential increased phenanthrene solubilization and mass transfer [10]. In addition, Kornilovich et al. [19, 25] reported that pulsed regimes have similar removal efficiency compared to constant regimes but provide noticeable electrical energy savings due to pause time.

The objectives of this pulsed electrokinetics study were to investigate the extraction efficiency of heavy metals compared to conventional electrokinetic approaches with constant DC supply and to characterize transport trends of heavy metals in the soil matrix with variations in pulse frequency, pulse ratio, voltage gradient, and energy expenditure.

2 Materials and methods

2.1 Soil preparation and chemical analysis

The soil used in this experiment was taken from contaminated rice fields near the Sambo abandoned mine in Young-duk, Republic of Korea. The soil sample was allowed to air-dry and was evenly mixed to minimize heterogeneity in the matrix, then sieved using a mesh No. 10 sieve ($\leq$2 mm). Soil properties are shown in Table 1. Particle size and organic content were measured using ASTM methods, and cation exchange capacity (CEC) was measured using EPA methods. The soil composition was approximately 60% of silt and clay. Korea standard test method (KSTM) was employed to measure the initial pH and electric conductivity (EC).

Table 1 Soil properties and initial concentration of heavy metals

Properties	Contents
Particle distribution (ASTM D421)	
Clay (%)	8.0
Silt (%)	50.3
Sand (%)	41.7
CEC (meq/100 g) (EPA 9081)	33.3
Organic contents (%) (ASTM D2974)	2.9
Initial pH	5.0
Electric conductivity (μS/cm)	151
Initial concentration (Aqua regia)	
Zinc (mg/kg)	2,322
Cadmium (mg/kg)	12.7

For pH and EC measurements, 50 mL of distilled water was added to 10 g of dried soil, and the mixture was allowed to settle for 1 h. After filtration, the initial pH and EC of the soil were analyzed. In order to determine the Zn and Cd concentrations, 3:1 HCl:HNO_3 was used to prepare aqua regia for extraction at 70 °C and the metals were analyzed by ICP-OES (730-ES Varian). Also, aqua regia was carried out in triplicate. In order to evaluate the various forms of heavy metals existing in the soils, the sequential extraction was carried out [26]. For sequential extraction procedures, pH 8.2 and 5 of 1 M NaOAc was used as extractants for exchangeable and bound to carbonate fraction, respectively. Also, 0.04 M of $NH_2OH \cdot HCl$ was used to extract the metals bound to Fe/Mn oxide in soil matrix at 85 ± 3 °C. In order to analyze the fraction bound to organic/sulfide matter, 0.02 M HNO_3, 30% H_2O_2 (pH = 2) and 3.2 M NH_4OAc in 20% HNO_3 was sequentially used at 85 ± 3 °C. For the extraction of residual form, digestion procedure using HF–$HClO_4$ mixture was replaced with aqua regia (HCl: HNO_3 = 3:1).

The forms of heavy metals present in the soil significantly affect design and operation of the electrokinetic system for that specific contaminated soil. When the heavy metals predominantly exist as strongly bound fractions (bound to organic/sulfide matter or residual forms), it is very difficult to remove the heavy metals from the contaminated site and a low removal efficiency can be expected using conventional methods. Hence, the selective sequential extraction method provides more detailed information on the forms of the heavy metals for design of the electrokinetic approach, and should be considered for efficient operation of the remediation system.

2.2 Electrokinetic set-up

Figure 1 shows a detailed schematic and dimensions of the electrokinetic apparatus used in this study. The system consisted of four components, namely, the power supply, soil compartment, electrode chamber, and electrolyte reservoir. Conventional (Constant) and pulsed direct current (DC) power sources were used in this experiment. The electrokinetic reactor was made of acrylic and it consisted of one soil compartment (4 × 4 × 20 cm) and two electrode chambers (4 × 4 × 4 cm). A plate-type graphite electrode (4 × 4 cm) was used as the cathode and a mesh of titanium coated with platinum was installed as the anode to prevent corrosion by the strong acid front. Filter paper was inserted to separate the soil from the electrode chamber and to prevent loss of soil particles. A gas vent was installed to release the hydrogen and oxygen gas generated by the electrolysis reaction. One liter of electrolyte was circulated at a flow rate of 4 mL min^{-1} using a peristaltic pump and was refreshed daily.

2.3 Experimental design

In this study, electrokinetic experiments were carried out under various conditions. Tap water was added to the sieved soil to saturation and saturated to 25% water content. The prepared soil was compactly packed into the soil compartment of each electrokinetic reactor. $MgSO_4$ (0.1 M) was employed to enhance the ionic strength of the soil bed. In order to compare the effectiveness of conventional and pulsed regimes, tap water was used as the catholyte without any chemical reagents or pH control to reduce metal-hydroxide precipitation under alkaline conditions. Table 2 summarizes the experimental conditions in detail. All electrokinetic experiments employed a pulsed power supply except Exp. 1 and they lasted for 2 weeks. When the pulsed system was on, an electrical voltage was supplied to the soil bed. However, when it was off, there was no supply of electrical energy. Pulse ratio is time ratio (on/off) with the power source in the position on and off. Together these, on and off pulsed ratio made up one cycle. A depolarization process may have occurred because of the absence of the applied electric field [10]. The pulse ratio (on/off) is directly related to the pulse frequency (cycles/h). From Exps. 2 to 6, different pulse frequencies (cycles/h), pulse ratios (on/off), and voltage gradients (V/cm) were applied.

3 Results and discussion

3.1 Contaminated soil

Table 3 summarizes the results of the sequential extraction. The metals bound to organic matter and residual forms comprise >70 and 60% of the total Zn and Cd amounts,

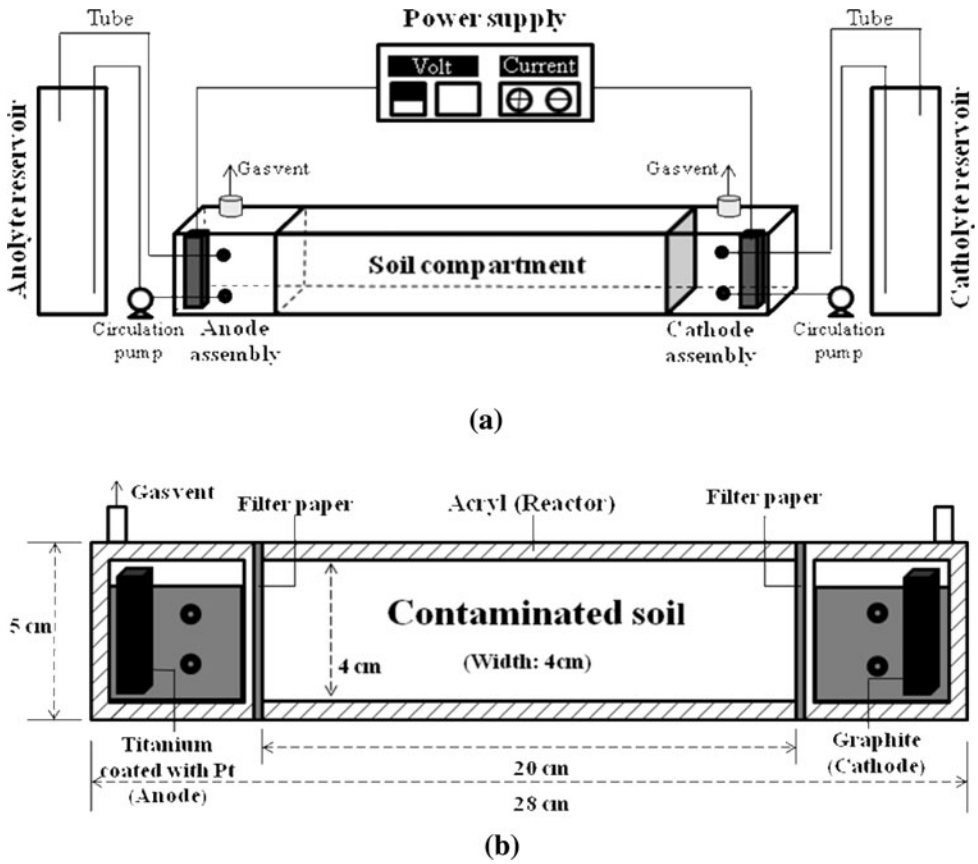

Fig. 1 Schematic and dimension of electrokinetic apparatus. **a** Schematic of electrokinetic set-up and **b** dimension and cell detail of electrokinetic apparatus

Table 2 Detailed experimental conditions

	Power source	Electrolyte		Pulse properties		Voltage gradient (V/cm)
		Anode	Cathode	Frequency (cycle/h)	Ratio (on/off)	
Exp. 1	Conventional	0.1 M $MgSO_4$	Tap water	–	–	–
Exp. 2	Pulse			1,800	1 s/1 s	1
Exp. 3	Pulse			1,800	1 s/1 s	2
Exp. 4	Pulse			1,200	2 s/1 s	1
Exp. 5	Pulse			1,200	1 s/2 s	1
Exp. 6	Pulse			1,200	1 s/2 s	3

respectively. The percentages of weakly bound fractions that are easily desorbed and transported from the soil were significantly lower than the strongly bound forms. In this case, a high removal efficiency for these contaminants would be unlikely.

3.2 pH and electrical current distribution

Figure 2 shows the pH change in the soil compartment after the electrokinetic experiments. In the anode region, the final pH was lower than the initial pH due to formation of hydrogen ions (H^+) generated by the electrolysis reaction. However, hydroxyl ions (OH^-) produced at the cathode contributed to high pH in the soil in the cathode area [14–16]. The acid front extracts cationic metals from the soils and allows the desorbed metals to migrate toward the cathode through electro-migration and electro-osmotic flow. In the anode region, the pH distribution of the soil was affected by the pulse ratio and the voltage gradient. At the same pulse frequency and ratio, greater electric strength resulted in more acidic conditions. Also, applied “on” times of pulse ratio into the soil bed resulted in lower pH regardless of the pulse frequency at a constant voltage gradient (1 V/cm). Metal-hydroxide and sulphate formation may occur under strongly alkaline conditions and could inhibit transport of metal ions in the electrokinetic system. Hence, it is difficult to enhance the mobility of metals or to expect high removal efficiency without any pH control or enhancing reagents.

The time course of the electrical current is shown in Fig. 3. Generally, the electrical current increased slightly with time in Exps. 1, 2, 4, and 5, in which 1 V/cm voltage gradient was applied. For Exps. 3 and 6 (2 and 3 V/cm, respectively), after ~170 h, the electrical current decreased because the ions were desorbed and then removed from the soil bed by electro-migration and electro-osmotic flow under high electric field strength.

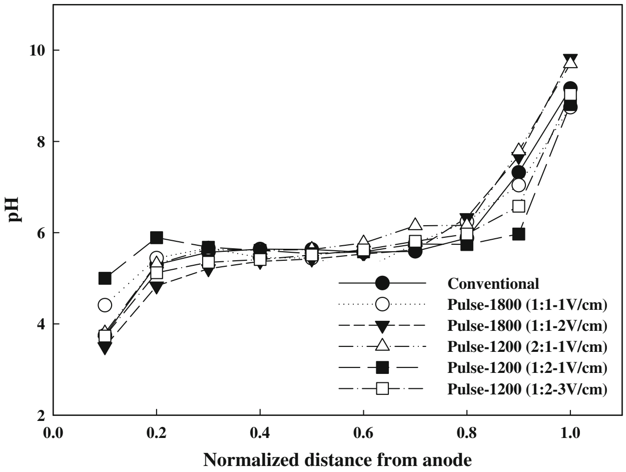

Fig. 2 The variation of pH in soil after electrokinetic treatments

3.3 Removal of Zn

Figure 4 shows the distribution of Zn after electrokinetic treatment. Zn was removed from the soil matrix in the following order of efficiency: Exp. 3 (1,800 cycle—2 V/cm): 30.7% > Exp. 6 (1,200 cycle—3 V/cm): 28.7% > Exps. 2 (1,800 cycle—1 V/cm) and 4 (1,200 cycle—1 V/cm): 23.3% > Exp. 5 (1,200 cycle—1 V/cm): 21.2% > Exp. 1 (conventional): 12.1%.

Overall, Zn migrated toward the cathode from the anode and pulsed electrokinetics was superior to conventional

Table 3 Summarized results of sequential extraction method

Speciation of heavy metals					
Fraction	Exchangeable (%)	Bound to carbonate (%)	Bound to Fe/Mn oxide (%)	Bound to organic matter (%)	Residual
Zn	1.3	5.8	19.4	12.5	61.0
Cd	5.1	7.7	25.6	3.9	57.7

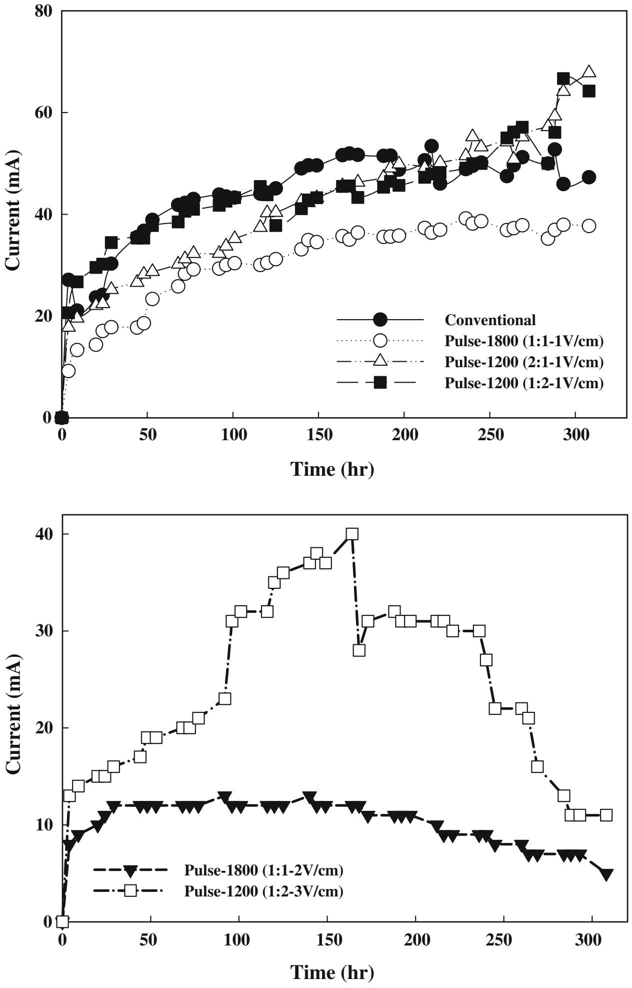

Fig. 3 The variation of electrical current during electrokinetic experiments

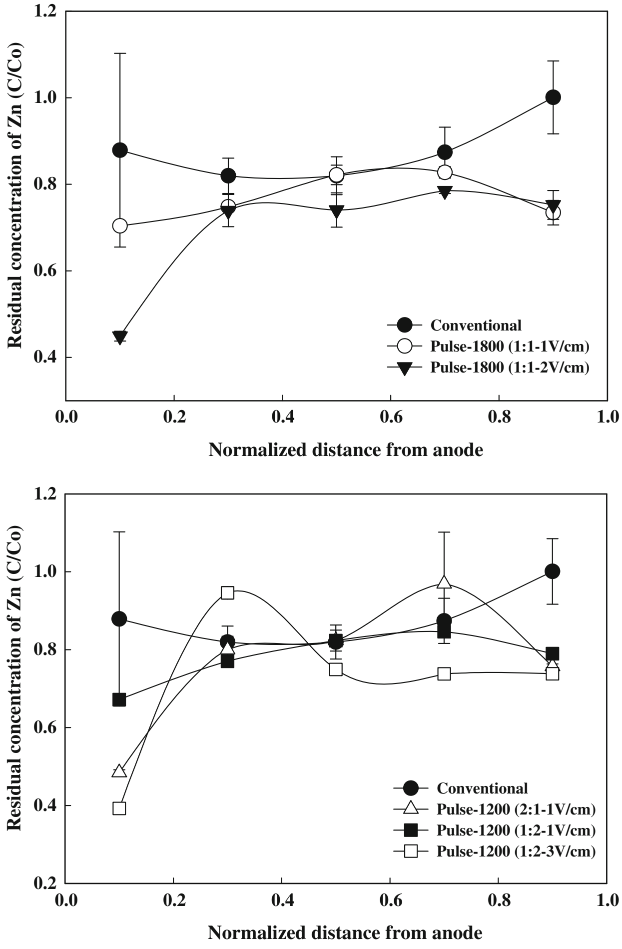

Fig. 4 Residual concentration (C/C_o) of Zn in soil after electrokinetic remediation

electrokinetics for Zn extraction. At a constant voltage gradient (1 V/cm), the overall removal of Zn at a frequency of 1,800 cycles/h was similar to that 1,200 cycles/h. However, the extraction of Zn near the anode was greater with a higher pulse ratio and longer "on" time at the pulse frequency of 1,200 cycles/h. For 1,800 and 1,200 cycles/h, a higher voltage gradient was beneficial for removing Zn from the soil matrix. Although better extraction of Zn occurred in the anode region, greater accumulation of desorbed Zn was observed, especially in Exp. 6. It is believed that the higher pulse ratio and higher voltage gradient primarily contributed to extraction of Zn from the soil surface near the anode; however, the ability of this design to transport heavy metals through the soil compartment is relatively low.

It was observed that lower metal-hydroxide precipitation and formation occurred in the cathode region in the pulsed electrokinetic experiments compared to the conventional electrokinetic approach because fewer hydroxyl ions (OH^-) were generated during the "on" time. Consequently, pulsed electrokinetics has an advantage over conventional electrokinetics for Zn removal, and pulse efficiency was influenced by the pulse ratio and voltage gradient.

3.4 Removal of Cd

Cd distribution is presented in Fig. 5. Cd was removed in the following order of efficiency: Exp. 2 (1,800 cycle—1 V/cm): 24.2% > Exp. 3 (1,800 cycle—2 V/cm): 24.0% > Exps. 4 (1,200 cycle—1 V/cm) and 5 (1,200 cycle—1 V/cm): 18.9% > Exp. 6 (1,200 cycle—3 V/cm):18.4% > Exp. 1 (conventional): 15.7%. The amount of extracted Cd in the anode region was higher than Zn under the same experimental conditions likely because the percentage of weakly bound fractions (exchangeable, bound to carbonate and iron/manganese oxide) was larger than that for Zn, based on the results of sequential extraction. In addition, the self-diffusion coefficient of Cd (7.17×10^{10} m^2/s) is greater than that of Zn (7.02 × 10^{10} m^2/s) in water systems [27]. Although Cd extraction was greater than Zn near the anode, the overall Cd removal efficiency in Exps. 3, 4, 5, and 6 was lower than that of Zn, because Cd significantly accumulated in the third section from the anode. Based on these results, pulsed electrokinetics has a disadvantage in its ability to transport heavy metals in the soil matrix, although its extraction ability is higher than the conventional electrokinetic process.

Similar to Zn, extraction of Cd increased with higher pulse ratio and voltage gradient in the anode region. At 1,800 cycles/h, a higher voltage gradient applied to the soil bed was more effective in extracting Cd near the anode. Although Cd was removed near the anode effectively at 1,200 cycle/h and 3 V/cm, it was difficult to transport the extracted Cd in the pulsed electric field. However, pulsed electrokinetics was a better method than conventional electrokinetics for Cd removal.

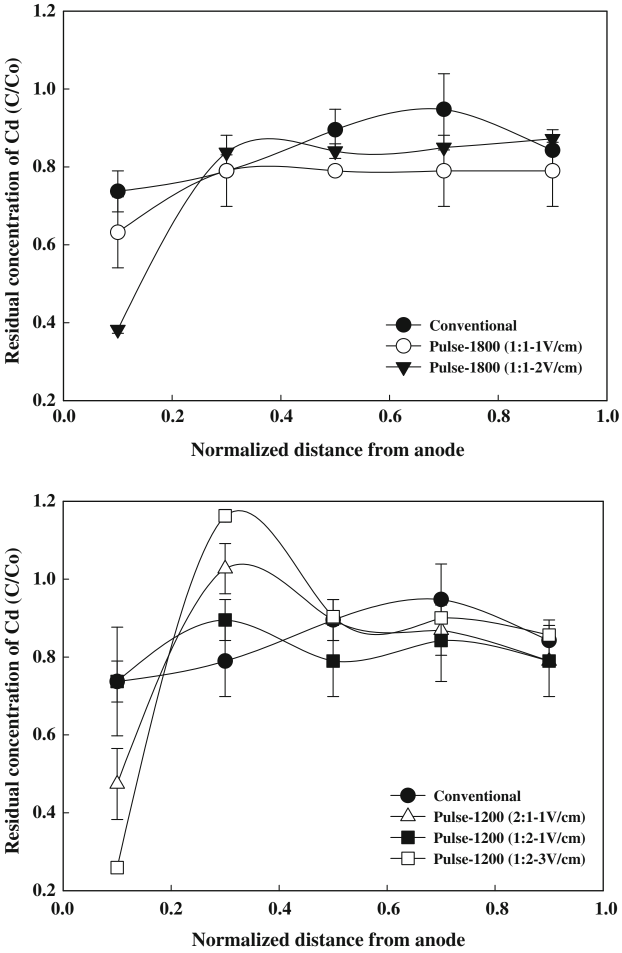

Fig. 5 Residual concentration (C/C_o) of Cd in soil after electrokinetic remediation

3.5 Electro-osmotic flow

In electrokinetic remediation, the principal mechanisms of removal of heavy metals from soil are electro-migration and electro-osmotic flow (EOF) [15–17]. The electro-osmotic flow rate q_{eo} (m^3/s) is defined as follows: [27]:

$$q_{eo} = k_{eo} E_x A$$

where k_{eo} is the electro-osmotic permeability coefficient (m^2/s V), E_x is the average electric field strength or gradient (V/cm), and A is the cross-sectional area of flow (m^2). The electro-osmotic coefficient k_{eo} and the average electric field strength E_x are important factors in determining the electro-osmotic flow rate using this equation.

Cumulative electro-osmotic flow is shown in Fig. 6 and its total amount of electro-osmotic flow was 182, 115, 218, 201, 81, and 401 mL during 2 weeks for Exps. 1, 2, 3, 4, 5, and 6, respectively. The electro-osmotic flow rate was strongly related to the electric field strength regardless of the pulse ratio or frequency. When 1 V/cm voltage gradient was applied to the soil cell, the EOF was related to the amount of applied electrical power and the pulse ratio. However, the removal efficiencies of Zn and Cd did not correlate with EOF, probably because electro-migration was more significant mechanism for mass transport than electro-osmosis. Acar and Alshawabkeh reported that mass transport by electro-osmosis of ionic species including Na^+, Ca^{2+}, Cd^{2+}, Pb^{2+}, and so on was at least 10 times lower than that induced by ionic migration under an electric field [16].

It is also possible to calculate the electro-osmotic permeability coefficient k_{eo}. Figure 7 shows the distribution of k_{eo} (m^2/s V) with elapsed time (h). For pulsed electric fields, the "on" time of pulse ratio has been applied to calculate k_{eo} (m^2/s V). Mitchell reported that k_{eo} for a

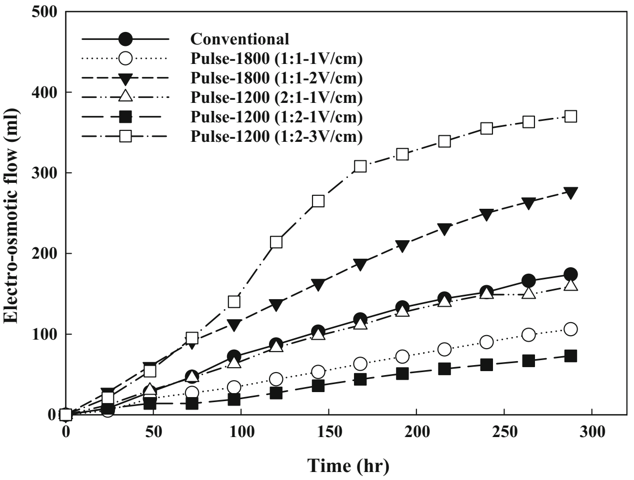

Fig. 6 Cumulative electroosmotic flow with elapsed time

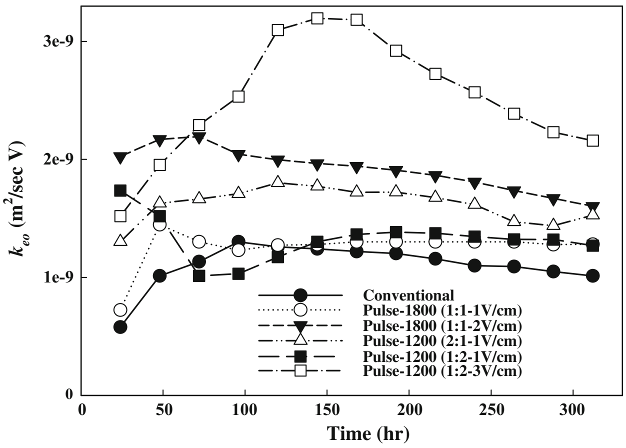

Fig. 7 Variation of electro-osmotic permeability constant (k_{eo}) with elapsed time

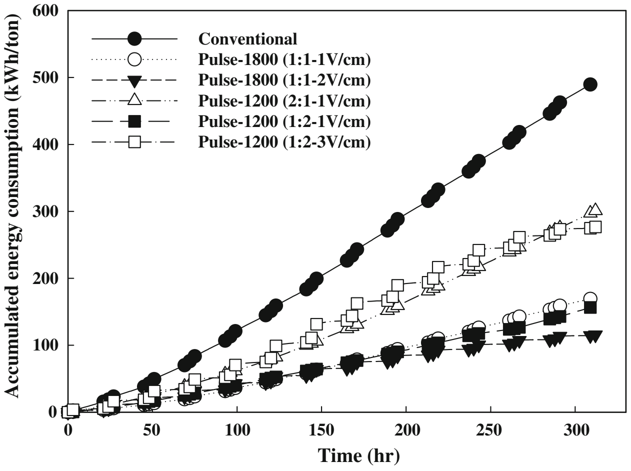

Fig. 8 Cumulative electric energy expenditure with elapsed time

variety of fine sands, powders, silts, and clays ranged from 1.5×10^{-9} to 20.0×10^{-9} m^2/s V [27]. In this experiment, k_{eo} was determined to be 1.01×10^{-9} to 2.16×10^{-9} based on the total "on" time only. It should be noted that pulse electrokinetics can increase electro-osmotic permeability. Generally, Zn removal efficiency was more correlated better with electro-osmotic permeability than with electro-osmotic flow rate. For Cd, it is difficult to compare removal efficiency with electro-osmotic permeability because substantial Cd accumulation occurred in the soil compartment.

3.6 Energy efficiency and the effects of pulsed electrokinetics

Energy consumption is one of the most important considerations for applying electrokinetics techniques to contaminated sites. Cumulative electrical energy consumption in this study is shown in Fig. 8. Because the pulse regime intermittently applied electrical energy to the soil bed in accordance with the pulse ratio, energy efficiency is a strong point of pulsed electrokinetics compared with a conventional electric field [25], as demonstrated by the results of this study. When "on" pulsed ratio increased at a constant voltage gradient, a larger amount of electrical energy was required to remediate contaminants. Although Exps. 3 and 6 supplied a voltage gradient 2–3 times higher than conventional electrokinetics approaches, the amount of electrical energy required was at least 3 times lower. Results for removal efficiency, EOF, k_{eo}, and electrical energy consumption are summarized in Table 4.

The removal of heavy metals versus electrical energy consumption (kWh) is presented in Fig. 9. The amount of Zn and Cd removed by of pulsed electrokinetics was 3.0–10.8 times and 1.9–6.3 times higher than those by conventional electrokinetics based on the same energy consumption. Zn and Cd show similar trends under all conditions; less electrical energy was required to extract and remove these heavy metals at contaminated sites under pulsed electric fields. The removal efficiency of heavy metals was as follows: Exp. 3 (1,800 cycle—2 V/cm) > Exp. 2 (1,800 cycle—1 V/cm) > Exp. 5 (1,200 cycle—1 V/cm) > Exp. 6 (1,200 cycle—3 V/cm) > Exp. 4 (1,200 cycle—1 V/cm) > Exp. 1 (conventional). Considering electrical energy consumption, Exp. 3 (1,800 cycle—2 V/cm) is the most effective method for removing heavy metals, and demonstrates the value of pulsed electrokinetics in reducing energy consumption.

4 Conclusions

In order to enhance heavy metals removal and evaluate the feasibility of pulsed electrokinetics, experiments were carried out under various pulse conditions and voltage gradients. The pH of the soil, removal efficiency of heavy metals, electro-osmotic flow, and electrical energy consumption were influenced by the pulse frequency, pulse ratio, and electrical strength. When a higher "on" time of pulse ratio and voltage gradient were applied at a constant pulse frequency, the pH became more acidic near the anode. A higher pulse frequency (1,800 cycles/h) increased removal efficiency of Zn and Cd compared to a lower frequency (1,200 cycles/h), and greater electrical strength enhanced the extraction and heavy metals removal at a constant pulse frequency. Although a larger fraction of Cd was extracted from the soil matrix compared to Zn in Exp. 6 (1,200 cycle—3 V/cm), Cd removal efficiency was lower than for Zn because significant accumulation of desorbed Cd occurred in the soil. Overall, pulsed electrokinetics was considered superior in extraction and desorption and not as effective in transporting heavy metal ions. In summary, the

Table 4 Summarized the results of electrokinetic experiments

	Removal efficiency (%)[a]		Cumulative EOF[b] (mL)	K_{eo} (m^2/s V)[c]	Electrical energy consumption (kW h/ton)[a]
	Zn	Cd			
Exp.1	12.1 (1.00)	15.7 (1.00)	182	1.01×10^{-9}	489.5(1.00)
Exp.2	23.3 (1.93)	24.2 (1.54)	115	1.28×10^{-9}	169.1(0.35)
Exp.3	30.7 (2.54)	24.0 (1.53)	218	1.60×10^{-9}	115.6 (0.24)
Exp.4	23.3 (1.93)	18.9 (1.20)	201	1.53×10^{-9}	300.8 (0.61)
Exp.5	21.2 (1.75)	18.9 (1.20)	81	1.27×10^{-9}	156.4 (0.32)
Exp.6	28.7 (2.37)	18.4 (1.17)	401	2.16×10^{-9}	276.7 (0.57)

[a] The value in the parenthesis presents the ratio of metal removed and electrical energy consumption in pulsed electrokinetic compared to conventional electrokinetic

[b] Total amount of cumulative electro-osmotic flow during 2 weeks

[c] Electro-osmotic permeability based on total "on" time only

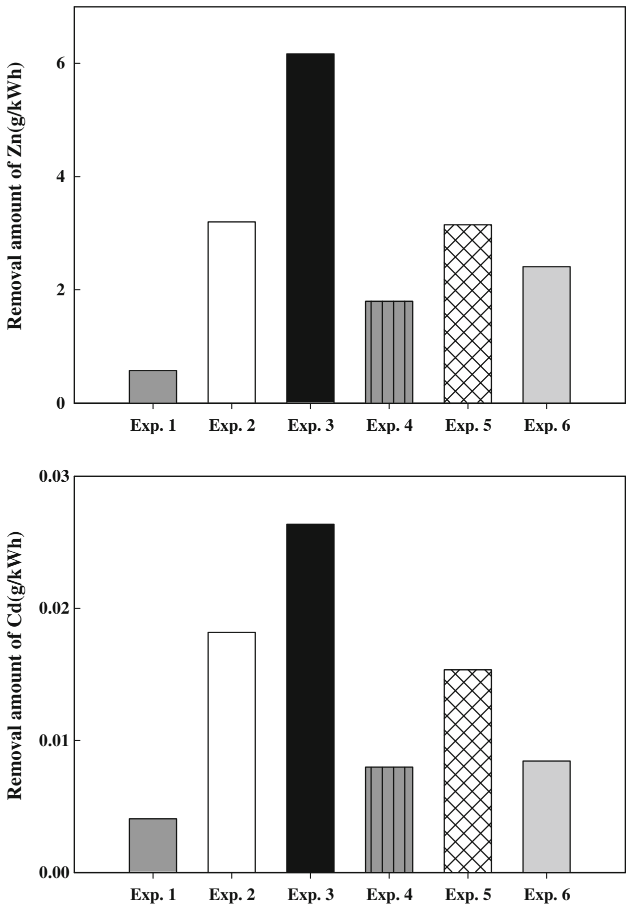

Fig. 9 Removal amount of Zn and Cd based on electrical energy consumption

optimal condition of Zn and Cd removal and energy consumption was under 1,800 cycles/h (1:1)—2 V/cm in this experiment. Also, conventional electrokinetic was not effective in point of removal efficiency of heavy metals and energy consumption compared to pulsed-electrokinetic. Transport of heavy metals was correlated better with the electro-osmotic permeability constant (k_{eo}) than with the electro-osmotic flow rate, and pulse electrokinetics enhanced the electro-osmotic permeability. Consequently, pulsed electrokinetics can significantly reduce electrical energy consumption and can enhance extraction and removal efficiency. However, the removal efficiency of heavy metals was not high because considerable amount of heavy metals exists as strongly bound fraction in soil matrix. Hence, further study is needed to increase its ability to transport and remove heavy metals.

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