Enhanced Electrokinetic Remediation of Cadmium (Cd)-Contaminated Soil with Interval Power Breaking

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RESEARCH PAPER

Enhanced Electrokinetic Remediation of Cadmium (Cd)-Contaminated Soil with Interval Power Breaking

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Abstract

This study compared electrokinetic (EK) remediation with and without interval power breaking in the removal of total and plant available cadmium (Cd) in the soil. Two laboratory experiments, i.e. EK remediation with interval power breaking (24–12 h power-on-off cycles) and conventional EK remediation (continuous power supply), with the same accumulated time (192 h) of power supply, were conducted to remove soil Cd. After the EK remediation with interval power breaking, the total Cd removal efficiency in the soil rose to 38%, in comparison to 28% after the conventional EK remediation. As for the plant available Cd, the removal efficiency was enhanced from 52 to 63%. Additionally, the electric current during the EK remediation and electric conductivity after the EK remediation were higher in the soil treated by interval power breaking, which indicated an enhanced desorption and/or migration of charged species. It further meant that the higher removal efficiency of soil Cd by interval power breaking could be related to the enhanced desorption and/or migration of Cd species. This study indicated that both conventional EK remediation and EK remediation with interval power breaking were effective methods to remove soil Cd but EK remediation with interval power breaking was more efficient.

Article Highlights

- **Plant available Cd has higher removal efficiency than total Cd after EK remediation.**
- **EK remediation with interval power breaking enhanced the removal of both Cd forms.**
- **The enhanced removal was due to enhanced ion desorption and migration.**

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Introduction

Heavy metal contamination in the soil has been a long-time issue across the world (Su et al. 2014; Huang et al. 2019; Kumar et al. 2019). Not only does heavy metal contamination inhibit the growth of plants, but also affects other living creatures, including human beings through the food chain (Singh et al. 2011, 2017; Emamverdian et al. 2015). Cadmium (Cd) is one of the most toxic heavy metals that are studied vastly on the patho-physiological effects on the reproductive system, nervous system, bones, gastrointestinal tract, etc. (Singh et al. 2011, 2017).

Over the past few decades, electrokinetic (EK) remediation has become one of the most effective methods for in situ or ex-situ soil decontamination. Numerous EK remediation investigations have shown success in removing soil pollutants including heavy metals from agricultural soils and industrial sites (US Army Environmental Center 2000; Kim et al. 2011, 2012, 2013; Lu et al. 2012; Ottosen, 2014; He et al. 2015; Ribeiro et al. 2016; Shin et al. 2017; Han et al. 2021). EK remediation of heavy metals is a result of many driving forces, including electro-migration, electro-osmosis, electrophoresis, etc.; the heavy metal species are expected to accumulate at either the cathode or anode and are removed afterwards (Acar et al. 1995; Hansen et al. 2016; Han et al. 2021). However, this technology also has some bottlenecks. For example, the generation of OH^- at the cathode can lead to the precipitation of heavy metals in the soil, which is referred to as "focusing" effect and can inhibit the removal of heavy metals from the soil (Probstein and Hicks 1993; Acar et al. 1995; Han et al. 2021). In this case, acidic electrolytes are usually added to enhance the removal efficiency. Ion exchange membrane (IEM) and electrode polarity exchange have also been adopted to prevent the spreading of OH^- from the cathode compartment into the main soil matrix (Kim et al. 2005; Ji et al. 2019; Mao et al. 2019).

Soil heavy metals are in different chemical forms. Some are soluble in the soil solution and some are exchangeable by other ions. The others are bound to carbonates, Fe/Mn oxides, organic matter and sulfides, and the mineral matrix (residual). They can be extracted by the famous BCR method (Pueyo et al. 2001; Ahmadipour et al. 2014; Ning et al. 2019). Among these fractions, the water and acid soluble and the exchangeable fractions (plant available) have the highest mobility and are the easiest to be removed by the EK remediation especially with the addition of acids, while the last fraction is tightly integrated in the soil matrix and is very hard to be removed (Kim and Kim 2001; Ryu et al. 2009; Han et al. 2021). The addition of acidic electrolytes such as lactic acid and acetic acid, and complexing agents such as EDTA and citric acid (Kim et al. 2011; Song et al. 2016;

Cai et al. 2021; Han et al. 2021) has proven to increase the desorption, solubility, mobility and thereafter the removal efficiency of heavy metals. However, the desorption and mobility enhancement reagents need to be chosen carefully according to the soil characteristics and heavy metals (Ottosen et al. 2005; Yang et al. 2009; Cai et al. 2021). Other modifications, such as the use of approaching electrodes, permeable reactive barrier, bipolar electrodes and/or interval power breaking have also been made to improve the removal efficiency by EK remediation (Hansen et al. 2007; Shen et al. 2007; Sun and Ottosen, 2012; Cai et al. 2016; Yao et al. 2020).

Previous researchers have found that EK remediation with interval power breaking could enhance the removal efficiency as well as save energy consumption due to the boosted power strength upon power connection (Hansen and Rojo, 2007; Ryu et al. 2009, 2010; Sun and Ottosen, 2012). However, Ryu et al. (2010) also reported that although interval power breaking could enhance the desorption of Cd close to the anode, it did not necessarily ensure an efficient migration (accumulation of heavy metals near the cathode) and the relevant removal efficiency can be very close to that of the conventional EK remediation (without power breaking). The removal efficiency and accumulation near the cathode can be affected by the power breaking frequency (Hansen and Rojo, 2007; Ryu et al. 2010). It is not surprising that despite the pulsed energy upon power connection, the accumulated time of power supply also accounts.

Despite the superiority of EK remediation with interval power breaking in the removal of soil heavy metals, the removal effect on different fractions of soil heavy metals by interval power breaking has not been clearly illustrated. It is important because accumulation of heavy metals near the cathode after the EK remediation with interval power breaking has been observed, so has the fraction transfer from the strongly to the weakly bound after the EK remediation (Hansen and Rojo 2007; Ryu et al. 2009, 2010). It is especially important to study the removal effect on the plant available fraction (weakly bound) since it is the most toxicity relevant. In this study, EK remediation of Cd-contaminated soil with and without interval power breaking but with the same accumulated time of power supply were compared on the removal of soil total Cd and plant available Cd. Additionally, electric current throughout the experiment and soil electric conductivity before and after the EK remediation were compared between the two EK remediation methods to support that EK remediation with interval power breaking could increase the removal efficiency of soil Cd by enhancing the Cd desorption from soil particles and/or migration across the soil matrix.

Materials and Methods

Soil Sampling and Processing

The soil used in this study was sampled near lead and zinc mine tailings, in Shaoguan city, Guangdong province, China. The sampled soil was processed before the EK remediation. The sampled soil was first air-dried. Stones, soil fauna, plant residues, etc. were removed. Then the soil was crushed and passed through a 2 mm nylon sieve. The soil was well mixed and further ground in an agate mortar until all of them passed through a 0.149 mm nylon sieve. The soil consisted of 61.4% silt, 19.3% sand, and 18.3% clay. The total organic carbon (TOC) content was 5.7%. The soil pH was 5.3 and the soil electric conductivity (EC) was 43 μS cm^{-1}.

Experimental Setup

The experimental setup (Fig. 1) included mainly a rectangular plexiglass as the EK remediation tank (length = 26 cm, width = 10 cm, and height = 10 cm) with two electrode chambers on the sides and a soil cell in between. The soil cell was divided into three measuring parts, named S1, S2, and S3 in the direction from the anode to cathode. The EK remediation tank was equipped with an external power supply of 20.0 V (1 V cm^{-1}) during the experiment. To prevent the soil in the soil cell from entering the electrode chambers, multiple layers of filter paper were placed between the electrode chambers and the soil cell. The electrodes were made of graphite and the effective surface area of the electrodes was 54 cm^2, all covered by electrolyte. The electrode chambers were filled with distilled water and the electrolyte (0.5 mol L^{-1} lactic acid) was replenished by a pump in circulation to keep the liquid level along the soil surface. During the experiment, a current recorder (model l99-dl-4, Hangzhou

luge technology co., LTD.) was used for an automatic collection and recording of current data (once per hour).

Two experiments were carried out with the same experimental setup. One experiment was conventional EK remediation with a continuous power supply for 192 h. The other, i.e. EK remediation with interval power breaking, had an alternate 24-h power-on and 12-h power-off cycle where the accumulated power-on time was 192 h. Both experiments were repeated three times. In each setup, about 1000 g of air-dried soil was loaded into the soil cell.

Chemical Analysis

Total Cd Measurement

An air-dried sample of 0.2–0.5 g was weighed in a 50 mL teflon crucible. A couple of drops of distilled water were added to wet the soil and 10 mL of hydrochloric acid (1.19 g mL^{-1}) was added. The crucibles were then heated at 120 °C on an electric heating plate in the fume hood so that the soil was decomposed preliminarily. After the liquid in the crucibles was evaporated to about 3 mL, the samples were cooled down. Then 5 mL of nitric acid (1.19 g mL^{-1}), 5 mL of hydrofluoric acid (1.49 g mL^{-1}), and 3 mL of perchloric acid (1.68 g mL^{-1}) were added to the crucibles. The crucibles were then sealed and heated at 230 °C for 1 h. After that, the seal was removed and the heating continued to let the silicon tetrafluoride gas that was produced by the reaction volatilize. The crucibles were shaken frequently during the heating process for a complete digestion of the soil samples. When the samples started to emit thick perchlorate white smoke, the crucibles were covered by lids so that the black organic matter was fully decomposed. When the black organic matter on the wall of the crucible disappeared, the lids were removed and the heating continued until the samples turned viscous and thick. Depending on the digestion degree, the whole digestion process could be repeated. After the samples cooled down for a while, 1 mL of nitric acid (0.24 g mL^{-1}) was added to dissolve the samples. The dissolved samples were then transferred to volumetric flasks (25 mL) and 3 mL of diammonium phosphate (5% w/w) was added to the flasks. After the flasks turned room temperature, nitric acid (0.2% v/v) was added to fill the flasks so that the final volume was 25 mL. The solution was run in ICP-MS (Agilent).

Plant Available Cd Measurement

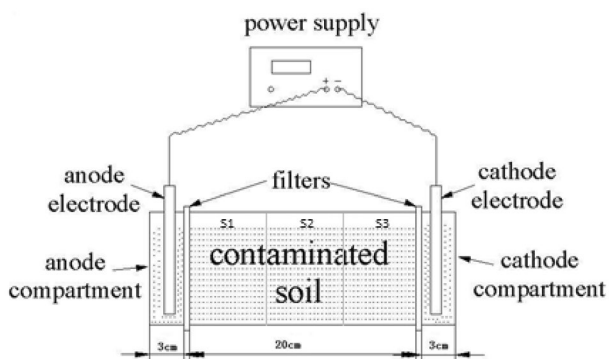About 10 g of air-dried soil was weighted, soaked in the 20 mL extraction solution, and shaken at 200 rpm for 2 h

Fig. 1 Schematic diagram of the EK remediation. Laboratory apparatus

at room temperature. The extraction solution contained 0.1 mol L^{-1} TEA (triethanolamine), 0.01 mol L^{-1}, and 0.005 mol L^{-1} DTPA (diethylenetriaminepentaacetic acid), and the pH of the extraction was around 7.3 adjusted by hydrofluoric acid. After the shaking, the samples were centrifuged at 5000 rpm for 10 min. The liquid phase was then filtered through a filter paper (30 μm) and run in ICP-MS (Agilent).

Statistical Analysis

IBM SPSS 28 was used for statistical analysis. Total Cd and plant available Cd in the soil were compared before and after the EK remediation using Repeated Measures ANOVA with simple main effects test, having the EK remediation method as the between-subject factor. The removal efficiency was compared between the two EK remediation methods using One-way ANOVA.

Results and Discussion

Soil pH

Low pH in soil is often crucial for the removal of heavy metals including Cd in EK remediation, although an alkaline condition can be favored for some other heavy metals such as As (Acar et al. 1995; Zhou et al. 2005; Yang et al. 2009). High pH can result in the metal hydroxide precipitation in the soil, or worse the formation of a negatively charged complex of metal elements and consequently disturb the migrating direction of metal elements (Acar et al. 1995; Zhou et al. 2005; Han et al. 2021). After the EK remediation with the addition of lactic acid, the soil pH was lowered from 5.3 to below 3.6 in all the soil sections and in either EK remediation experiment (Fig. 2). The pH was higher in soil section S3 than S1 in either remediation experiment. It was due to the OH$^-$ and H$^+$ production at the cathode (S3) and anode (S1), respectively by electrolysis, which is a normal phenomenon in EK remediation.

Electric Current

Electric current is an indicator of the number of ions that migrated in the electric field, i.e. electro-migration. As shown in Fig. 3, the electric current rose in the first 12 h in both experiments: EK remediation with interval power breaking and conventional EK remediation, as the electrolyte entered the soil matrix, which increased the concentration of soluble ions in the soil fluid. After the electric current reached its peak, it decreased gradually in about 24 h and became stable in the conventional EK remediation experiment. The electric current in the EK remediation with
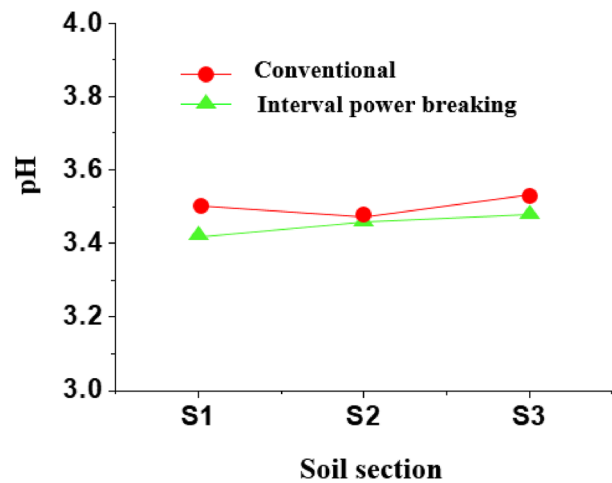

Fig. 2 Soil pH after the EK remediation in the soil sections S1, S2 and S3
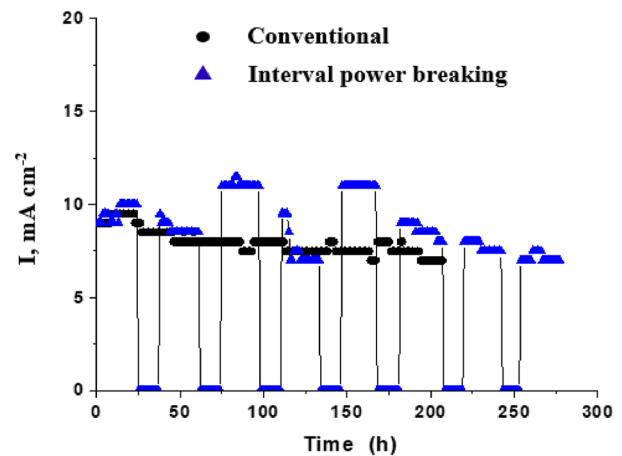

Fig. 3 Electric record throughout the experiment

interval power breaking also displayed a decrease with the power being intermittently on and stayed around the same level as in the conventional EK remediation after 216 h.

With the increase in remediation time, the current tended to decrease, which indicated that the number of migrating ions in the soil was reduced. This could be caused by several factors. It could be due to the precipitation of metal ions with OH$^-$ at the cathode where OH$^-$ was electrolyzed or in the main soil matrix when OH$^-$ migrated to the anode. Metal cations could also electrodeposit onto the cathode, which could increase the electric resistance. It is a very common phenomenon in EK remediation (Acar et al. 1995; Han et al. 2021). Another reason could be the ion neutralization in the soil fluid where H$^+$ and OH$^-$ came across each other while migrating across the soil to the opposite electrodes. Additionally, when ions migrated to the oppositely charged

electrodes, the electric field created by the external power supply would be compensated, resulting in a gradually weakened electric field and lower current (Acar et al. 1995).

When the power was on, the electric current in the soil treated by interval power breaking was generally higher than that in the soil treated by conventional EK remediation, especially after 72 h and before 144 h. The higher current in the soil treated by interval power breaking indicated that interval power breaking enhanced the desorption and/or migration of charged species (Ryu et al. 2009, 2010; Sun and Ottosen 2012).

Electric Conductivity

After the EK remediation, the electric conductivity increased dramatically in either EK remediation experiment (Fig. 4). The highest electric conductivity reached around 260 μS cm^{-1}, which was observed in the soil section S3 in the EK remediation with interval power breaking. The lowest that was about 185 μS cm^{-1} was observed in the soil section S1 in the conventional EK remediation. In general, the soil electric conductivity increased from section S1 to S3 and was higher in all the soil sections after the EK remediation with interval power breaking compared to the conventional EK remediation. Similar to electric current, since electric conductivity depends on the free mobile ions in the system. The higher electric conductivity in the soil treated by EK remediation with interval power breaking compared to the conventional EK remediation was related to the enhanced desorption of charged species (Ryu et al. 2009, 2010). The reason why the electric conductivity increased from the soil section S1 to S3 could be the migration of desorbed metal cations from the anode to the cathode and the sufficient supply of lactic acid prevented the formation of metal precipitates near the cathode (Cai et al. 2021).

Cd Removal

The total Cd concentration decreased from 2.45 mg (kg dry wt soil)$^{-1}$ to 1.52 mg (kg dry wt soil)$^{-1}$ and 1.76 mg (kg dry wt soil)$^{-1}$ on average after the EK remediation with interval power breaking ($F = 2029.8$, $df = 1$, $p < 0.001$) and conventional EK remediation ($F = 1098.7$, $df = 1$, $p < 0.001$), respectively (Fig. 5). The concentration of the plant available Cd decreased from 0.19 mg (kg dry wt soil)$^{-1}$ to 0.07 mg (kg dry wt soil)$^{-1}$ and 0.09 mg (kg dry wt soil)$^{-1}$ after the EK remediation with interval power breaking ($F = 881.2$, $df = 1$, $p < 0.001$) and conventional EK remediation ($F = 604.1$, $df = 1$, $p < 0.001$), respectively (Fig. 6).

The removal efficiency of total Cd and plant available Cd was about 38% and 63%, respectively after the EK remediation with interval power breaking, which were higher ($F = 98.4$, $df = 1$, $p < 0.001$) than 28% and 52%, respectively, after the conventional EK remediation. This was in accordance with the higher electric current and electric conductivity in the soil treated by EK remediation with interval power breaking. Higher electric current and electric conductivity in the soil treated by interval power breaking indicated an enhanced desorption and/or migration of charged species. Therefore, the higher removal efficiency of Cd by interval power breaking could imply an enhanced desorption, migration, and removal of Cd elements. There could be several mechanisms involved, e.g. cation exchange, electro-migration, and electro-osmosis (Acar et al. 1995; Hansen et al. 2016; Han et al. 2021).

Soil Cd is adsorbed to soil particles through various mechanisms that are affected by the soil type, surface loading, and pH, although the mechanisms are not fully understood yet (Ning et al. 2019; Mo et al. 2021). Competitive adsorption with other heavy metals, e.g. Pb, Cr, Zn, and Cu also affects the fractionation of Cd species (Campillo-Cora et al. 2020).
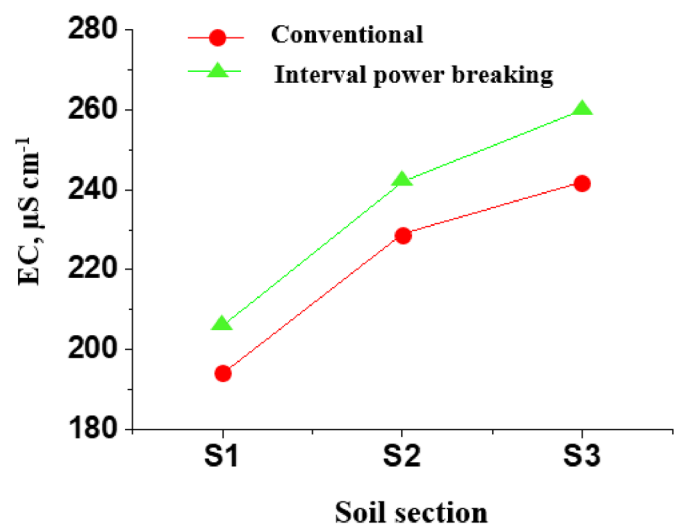

Fig. 4 Soil electric conductivity after the EK remediation in the soil sections S1, S2 and S3

Fig. 5 Concentration of total Cd before and after the EK remediation in the soil sections S1, S2 and S3
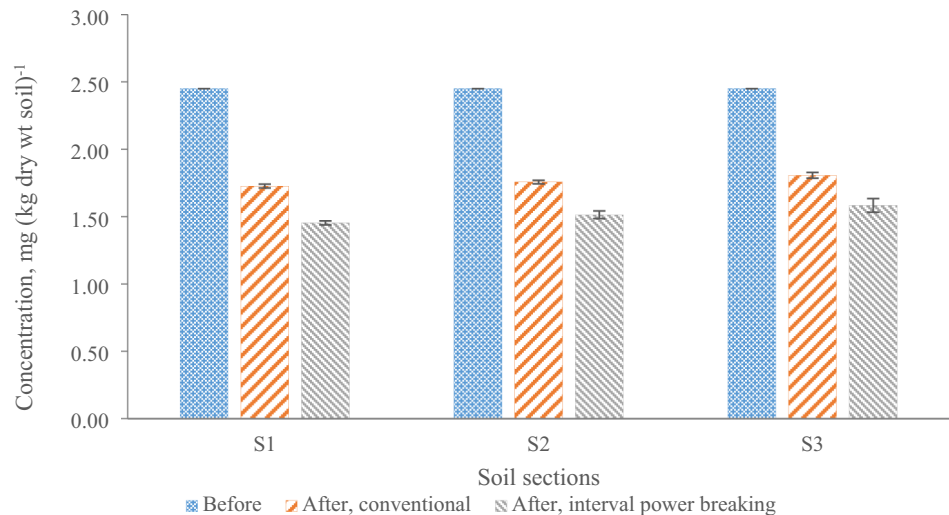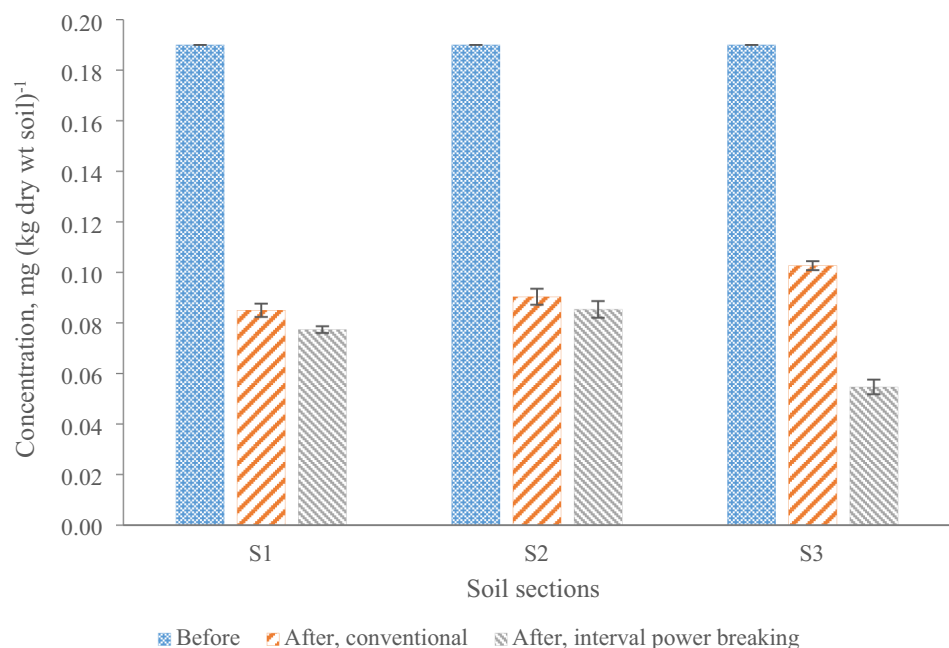

Fig. 6 Concentration of plant available Cd before and after the EK remediation in the soil sections S1, S2 and S3

The soil studied here was mineral (TOC 5.7%), the adsorption mechanisms between which and Cd could involve electrostatic attraction (outer-sphere complexes), covalent bond formation (inner-sphere complexes), and surface precipitation. The outer-sphere complexes are weakly bound to the soil from which Cd^{2+} can be exchanged out easily; other readily exchangeable cations in soil include K, Na, Mg, Ca, and Al (Campillo-Cora et al. 2020). H^+ plays an important role in soil EK remediation in the desorption of metals through cation exchange and dissolution of heavy metal precipitates, e.g. carbonates and hydroxides (Acar et al. 1995; Han et al. 2021). In this study, the soil pH was adjusted by lactic acid so that it stayed below pH 3.6. Therefore, the lower soil pH resulted from interval power breaking could lead to higher H^+ exchange of Cd^{2+} from the exchangeable and acid-soluble species in the soil and consequently the higher removal of Cd through electro-migration and electro-osmosis (Acar et al. 1995; Han et al. 2021). Additionally, interval power breaking could enhance the desorption and migration of charged Cd species by instant higher electric attraction (pulsed energy). Furthermore, higher electro-osmosis by interval power breaking could convey the Cd species that were dissolved or suspended in the soil water out of the soil matrix more efficiently.

The plant available Cd is the weakly bound fraction, i.e. the soluble and exchangeable fraction that can be easily dissolved, desorbed by H^+ exchange, and migrated out of the soil matrix by the EK remediation. Therefore, the removal efficiency of the plant available Cd was higher than the total Cd by either EK remediation method.

Additionally, no accumulation of total Cd or plant available Cd occurred near the cathode in comparison to conventional EK remediation, unlike the findings in earlier studies (Hansen and Rojo 2007; Ryu et al. 2009, 2010). In these studies, accumulation of Cd near the cathode was observed in the soil treated by interval power breaking, which was owed to the inefficient migration of Cd compared to the conventional EK remediation. In this study, the same accumulated time of power supply was provided, which allowed sufficient migration of soil Cd.

Conclusions

EK remediation successfully treated Cd-contaminated soil in 192 h in this laboratory study. The removal efficiency of the plant available Cd was higher than that of the total Cd. EK remediation with interval power breaking enhanced the removal of total and plant available Cd in the soil compared to the conventional EK remediation with the same accumulated time of power supply. The higher removal efficiency of Cd by the EK remediation with interval power breaking was in accordance with the higher electric current and soil electric conductivity, which implied that the higher removal efficiency by interval power breaking was due to the enhanced desorption and/or migration of soil Cd.

Availability of data and material The datasets used and/or analysed during the current study are available from the corresponding author on reasonable request.

Code availability Not applicable.

Declarations

Conflicts of interest/Competing interests The authors have no relevant financial or non-financial interests to disclose.

Ethics approval Unethical actions were not made.

Consent to participate All authors gave consent to participate in this publication.

Consent for publication All authors gave consent for publication in the International Journal of Environmental Research.

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