



Simultaneous removal of 2,4-dichlorophenol and Cd from soils by electrokinetic remediation combined with activated bamboo charcoal

Jian Wei Ma^a, Fa Yuan Wang^{a,b}, Zheng Hong Huang^c, Hui Wang^{a,*}

^a State Key Joint Laboratory of Environment Simulation and Pollution Control, Department of Environmental Science and Engineering, Tsinghua University, Beijing 100084, China

^b Agricultural College, Henan University of Science and Technology, Luoyang 471003, China

^c Department of Material Science and Engineering, Tsinghua University, Beijing 100084, China

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ABSTRACT

An *in situ* electrokinetic remediation technique was designed by combining the uniform electrokinetic technology with a new-type of bamboo charcoal as adsorbent. A bench-scale experiment was conducted to investigate the application of this technique for simultaneous removal of 2,4-dichlorophenol (2,4-DCP) and Cd from a sandy loam at different periodic polarity-reversals. The contaminated soil was artificially spiked with 100 mg/kg 2,4-DCP and 500 mg/kg Cd. Two modes of polarity-reversal intervals of 12 and 24 h were included. After 10.5 d of operation, about 75.97% of Cd and 54.92% of 2,4-DCP were removed from soil at intervals of 24 h, whilst only 40.13% of Cd and 24.98% of 2,4-DCP were removed at intervals of 12 h. Soil water contents under two operation modes both significantly decreased, but evenly distributed spatially. Soil pH values under two operation modes were all maintained in the range from 7.2 to 7.4, close to the initial value. The electricity consumption per day was 12.24 and 11.61 kWh/m³/d, respectively at polarity-reversal intervals of 12 and 24 h. In conclusion, at polarity-reversal interval of 24 h, electroremediation combined with activated bamboo charcoal was effective in simultaneous removal of 2,4-DCP and Cd from soil. Our results indicate a promising potential in *in situ* electroremediation of soils co-contaminated with organics and heavy metals.

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1. Introduction

Increasing human health and ecological hazards posed by large areas of polluted soils have promoted efforts to develop novel clean-up technologies. As a new, innovative and cost-effective remediation technology, electrokinetic remediation has shown great promise in *in situ* soil remediation and has received increasing attention due to its unique applicability to low-permeable soils. Numerous studies have been demonstrated that the feasibility and effectiveness of electrokinetics for the extraction of heavy metals, such as Cu, Zn, Pb, Cd, Cr, Ni, Hg, As, Co, Sr, radionuclides U [1–4], and organic pollutants, such as phenanthrene [5–8], phenol [9–12], atrazine [13], DDT [14], and BTEX [15].

In electrokinetic remediation process, metals or charged ions are typically removed from soil mainly by electromigration, whereas non-polar contaminants such as most organic contaminants have to be removed by electroosmosis. Removal efficiency generally depends on remediation techniques, contaminants themselves, and soil properties, such as pH, permeability, adsorption capacity, buffering capacity, etc. [16]. Several techniques have been used

with electrokinetics to improve the performance, such as chelating reagents [17], surfactant addition [18–20], design and operation enhancement like using LasagnaTM model, rotational operation mode, or upward process [21–23].

Unlike other integrated electroremediation technologies, LasagnaTM process has several treatment zones (e.g. adsorption, immobilization, degradation) filled with appropriate materials (adsorbents, catalytic agents, microbes, oxidants, buffers, etc.), and transports contaminants from the soil into the treatment zones. Thus, removal efficiency may be drastically increased. LasagnaTM technology showed great potential in remediation of soils polluted with trichloroethylene and *p*-nitrophenol [23–26].

In LasagnaTM process, activated carbon is the most common adsorbent used as the treatment material to trap the contaminants transported from the contaminated soil. However, commercially available activated carbon is expensive, which may limit its use in field-scale remediation. Therefore, there is a need for the development of low-cost, easily available adsorbents. Bamboo charcoal is produced from the rapidly growing moso bamboo plant and its adsorption characteristics have been the subject of many studies [27–28]. Several studies have found that bamboo charcoal has excellent adsorption capacity for a wide variety of substances, such as nitrate-nitrogen [29], heavy metals [30], harmful gases [28,31], and can be used for purification of water or air. Therefore, bamboo

* Corresponding author. Tel.: +86 10 62772137; fax: +86 10 62771472.

E-mail address: wanghui@mail.tsinghua.edu.cn (H. Wang).

charcoal may provide an alternative option of adsorbents. Ma et al. firstly reported the use of bamboo charcoal in electroremediation of Cd from soils [32,33].

Generally, pollutants usually appear not singly but as co-contamination. Soil polluted with organic contaminants often contains also heavy metals. Because of the different nature of the two contaminant groups, between them there may be synergistic or antagonistic effects on electrokinetic removal [34,35]. Thus, suitable remediation techniques should be selected for multiple contaminants. Several research papers have dealt with the simultaneous removal of organics and heavy metals using electrokinetic remediation technology [15,18,22,36–38]. Li et al. [35] found that it is difficult to achieve great removal of hydrophobic organic compounds (HOCs), or HOCs and heavy metal mixed contaminants, by electrokinetic treatment in large scale with the use of hydroxypropyl- β -cyclodextrin. Chlorophenols are regarded toxicologically as priority pollutants. They are generally used as wood preservatives, pesticides and precursors of herbicides. Co-contamination of chlorophenols and heavy metals often occurred due to the conventional use of pesticides [39]. A few studies reported the electrokinetic removal of chlorophenols from contaminated soils [40,41], but there have been no reports on simultaneous removal of chlorophenols and heavy metals.

In the present work, a bench-scale experiment was conducted to investigate the application of electrokinetic remediation combined with bamboo charcoal as an adsorbent for simultaneous removal of 2,4-DCP and Cd from a sandy loam at different polarity-reversals.

2. Materials and methods

2.1. Experimental soil

A natural sandy loam was collected from the topsoil layer (0–30 cm) of a woodland at Tsinghua University. Soil sample was passed through a 2 mm sieve, sterilized three times by alternately using an autoclave (121 °C for 45 min) and a drier (105 °C for 30 min), and stored in a desiccator for later tests. The main characteristics were listed in Table 1.

The contaminated soil was artificially spiked with Cd and 2,4-DCP according to the target concentrations and saturated moisture. Soil samples were prepared carefully and outflow was avoided to achieve the desired target. Cd was added to soil as $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ dissolved in deionized water and the target concentration was 500 mg/kg. The 2,4-DCP required to yield the target concentration of 100 mg/kg was measured and then dissolved in 500 mL of anhydrous ethanol. The solution was mixed homogeneously with the tested soil. The mixture was placed at a thickness of 1 cm beneath a

Table 1
The main characteristics of the tested soil.

Soil sample	
Particle size analysis (%)	
<2 μm	11.21
2–10 μm	5.01
10–50 μm	7.00
50–250 μm	63.31
>250 μm	13.47
BET surface area (m^2/g)	8.10
Organic matter (g/kg)	5.33
CEC (cmol/kg)	11.25
pH	7.74
Air-dried density (g/cm^3)	1.35
Saturated water content (%)	33.0
Point of zero charge	1.5
Effective porosity (%)	0.42
Liquid limit (%)	19.7
Plastic limit (%)	18.2
Stable infiltration rate (mm/min)	3.2

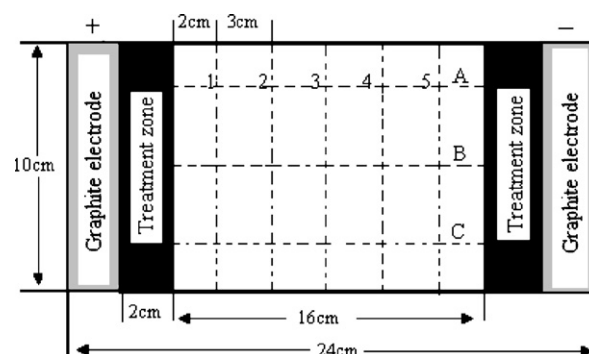


Fig. 1. Schematic diagram of experimental setup and sampling positions.

ventilation hood for about 1.5 d until the ethanol completely evaporated. A measured amount of deionized water was mixed with contaminated soil to obtain the target water content.

2.2. Bamboo charcoal

Bamboo charcoal was provided by Department of Material Science and Engineering, Tsinghua University. Before use, carbonized bamboo was boiled in distilled water for 1 h to remove some ash and impurities, and then oven-dried at 105 °C for 24 h. Then they were ground and passed through 200 mesh (i.e., 0.074 mm) and stored in desiccators. The BET surface area is 1120 m^2/g and the bulk volume is 0.538 cm^3/g .

2.3. Experimental systems

A schematic diagram of the testing system was shown in Fig. 1. It consisted of a soil cell, two graphite electrodes, an electrode control system, an electric current and voltage real-time monitoring system, and a DC power supply [32,33]. The soil cell was made of Perspex with an inner size of 24 cm \times 10 cm \times 10 cm in length \times width \times height. The two electrode compartments (with 240 mL working volume) were placed at each end of the apparatus isolated from the treatment zone by a porous Perspex plate and filter paper. Two plate-shaped electrodes, 10 cm \times 10 cm \times 0.5 cm, were used to generate the uniform electric field. The electrode control apparatus was capable of reversing the polarity of the electric field. Two treatment zones were filled with bamboo charcoal. The data monitoring system was able to monitor electric current and voltage on-line following a preset time step and automatically store them into a personal computer for later analysis. The power supply (WYK2603, Dongfang Com., Yangzhou) provided a constant DC electric voltage in a range from 0 to 60 V for the electrokinetic test.

2.4. Experimental procedures

The uniformly contaminated soil with proper moisture content was placed into the soil cell by layers. Each layer was pressed down using a Perspex pestle and vibrated for 5 min so that the amount of void space was minimized. Then the soil specimen was compacted for 12 h at a pressure of 0.1 kg/cm^2 . The extruded pore fluids were removed from the surface layer using bibulous paper. Before the reactor assembly was conducted, a small fraction of the soil specimen was obtained to determine the initial soil pH, moisture and content of contaminants. The soil beds with the volume of length 16 cm \times width 10 cm \times height 5 cm. Treatment zone was filled with bamboo charcoal and the thickness was 2 cm. After that, the anode and cathode compartments were filled with 0.01 M KNO_3 solution to saturate the bamboo charcoal and soil.

Two electrodes were directly inserted into the electrode chambers with a distance of 20 cm from each other and then connected with two poles of power supply through electrode control apparatus and monitoring equipment. DC power supply, electrode control and monitoring equipment were then connected to the electrodes. Once the reactor assembly was completed, the cell was closed with a Perspex cover to prevent the soil bed from excessive evaporation of water and 2,4-DCP. The test was run at a constant voltage gradient of 1.0 V/cm for 10.5 d. During the test, the electric current and voltage across the soil specimen were automatically measured every 20 min to calculate the electricity consumption. Two bidirectional modes were applied with polarity-reversal intervals of 12 and 24 h, respectively. The electrode control apparatus was capable of reversing the polarity of the electric field periodically in bidirectional operation process. At the same time, a treatment without DC power was conducted as control.

At the end of tests, a fraction of the soil specimen (length 1 cm × width 1 cm × height 5 cm) was taken using a U-shaped sampler and a spatula to determine the soil pH, water content and the residual Cd and 2,4-DCP content. In order to reflect the spatial variation regarding the variables, sampling lines (A and C) were arranged along the middle line (B), and along the sideline with a distance of 4 cm from the middle line. Five spots, with distances of 4, 7, 10, 13 and 16 cm from the anode, were sampled on each line, as shown in Fig. 1.

2.5. Analytical methods

Cd concentrations in soil were determined by inductively coupled plasma atomic emission spectrometry (ICP-AES) [42]. The oven-dried soil samples (0.2 g) were digested with a mixture of concentrated 4 mL HNO₃, 1 mL HCl and 2 mL HF (all guaranteed reagents). ICP (IRIS Intrepid II XSP) was used for Cd analysis.

Acetonitrile was used to extract 2,4-DCP from 1.0 g of soil samples. After ultrasonic extraction for three times (with an extraction efficiency of 94.8%), the extractant was filtered through 0.45 μm Teflon membrane and then analyzed on a high performance liquid chromatograph (HPLC, Hewlett Packard 1050) at 284 nm equipped with a reverse-phase C18 column (Agilent, USA) and a mobile phase containing methanol and 5% acetic acid solution (67/33, v/v). HPLC was calibrated using four external standards prior to performing chemical analysis and using standard 2,4-DCP during analysis to ensure that the system remained good performance and to certify a consistent response. In addition, the syringe for sample injection was rinsed three times between each injection to eliminate cross-contamination.

In addition, soil pH was determined using a soil to water ratio of 1:2.5 and water content was determined using the methods described by Lu [42]. All the analysis was performed in triplicate, and the result was calculated as the average.

The electricity consumption per unit volume of soil was calculated by the following equation [43]:

$$E_u = \frac{1}{V_s} \int_0^t UI dt \quad (1)$$

where E_u is the electricity expenditure per unit volume of soil (kWh/m³), V_s is the soil volume (m³), U is the electric potential difference across the electrodes (V), I is the electric current (A) and t is the treatment time (h).

3. Results and discussion

Removal of Cd and 2,4-DCP from soil by this technique was determined after the tests were terminated. Water content and pH of soils, and the electricity consumption were also determined. These results helped to assess the efficiency of the combined remediation technique to remove Cd and 2,4-DCP from the soil.

3.1. Soil water content

Electroremediation by ionic migration and electroosmosis is restricted to soluble substances. Contaminants that are adsorbed on the soil or are present as precipitates or immiscible liquids cannot be effectively removed unless they can first be absorbed into the aqueous phase. Soil water content is an important factor that alters the electroosmotic flow rate and hence decontamination of the soil by electrokinetic process. Decreased moisture content and/or uneven moisture distributions may lead to low soil conductivity, thus becomes too low for the electrokinetic remediation application.

In the present study, water content of bamboo charcoal in treatment zone was about 75% in both two treatments, significantly higher than that in soils. Compared with the initial soil water content of 33.0%, after rotational operation of 10.5 d, soil water content decreased 12.8–20.0%, and 12.7–18.8%, respectively at intervals of 12 and 24 h, and varied slightly with the distance from anode (Fig. 2). Reversing polarity at intervals of 24 h is a little more suitable for keeping moisture content. Similar water content reduction, 13–20%, was found in clay material after electrokinetic treatment [44,45]. Interestingly, water content did not change remarkably at different lines A, B or C (Fig. 2). An even spatial distribution of soil moisture may be more favorable for electroremediation. In comparison with unidirectional operation, reversing the polarity at appropriate intervals can keep soil moisture at a suitable level [12]. In LasagnaTM process [24], a water layer was designed near the electrodes in the electrokinetics cell, and thus water can be completely recycled without incurring any additional treatment step. In future study, a similar design may be adopted in our technique for recycled use of water.

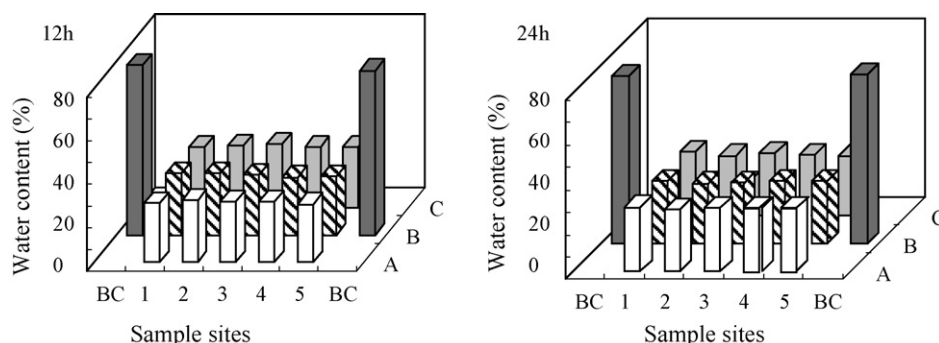


Fig. 2. Variation water content after rotational operation of 10.5 d at intervals of 12 and 24 h.

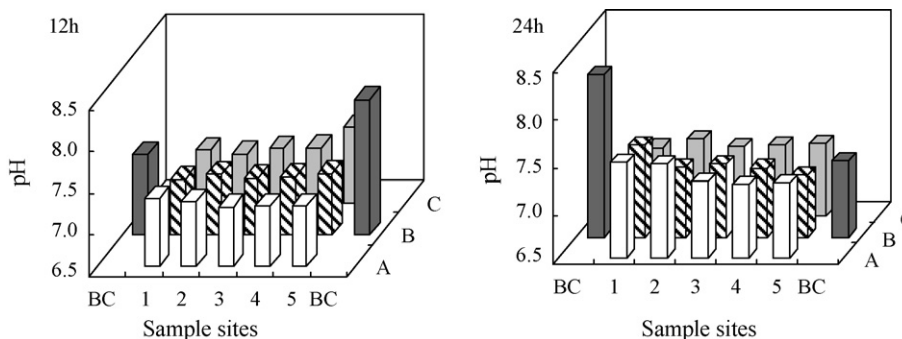


Fig. 3. Variation of soil pH after rotational operation of 10.5 d at intervals of 12 and 24 h.

3.2. Soil pH

Soil pH is another key factor influencing the forms of contaminants, and thus their transport processes. Besides, due to water electrolysis at electrodes, soil pH decreases near the anode and increases near the cathode. The high pH value causes metal hydroxides to precipitate in soil close to cathode, decreasing soil conductivity, while low pH value decreases the soil zeta potential, leading to reduced electroosmotic permeability and water flow rate, and thus limiting electrokinetics of contaminants. To keep pH value suitable and stable in soil is of great importance in removal of heavy metals and/or organics.

In the present experiment, soil pH did not show a dramatic change at the different sample regions and was maintained in the range from 7.2 to 7.4, close to the initial soil pH (7.49) (Fig. 3). It has been demonstrated that polarity reversal could effectively keep soil pH stable when appropriate intervals were chosen [12,24,40]. Our experiment demonstrates that polarity reversal is effective in controlling pH of sandy soil, and favorable for electroremediation of contaminated soil with high permeability.

Additionally, at different intervals, pH showed different variation trends. Soil pH was slightly lower near the anode at intervals of 12 h, but little higher at intervals of 24 h. When the operation was terminated, the polarities at different polarity-reversing intervals were opposite, so pH showed different variation trends.

3.3. Removal of Cd and 2,4-DCP from soil

After 10.5 d of operation, both Cd and 2,4-DCP concentrations in the soil were much lower at intervals of 24 h than those at intervals of 12 h (Figs. 4 and 5). About 75.97% of Cd and 54.92% of 2,4-DCP were removed from soil at intervals of 24 h, while only 40.13% of Cd and 24.98% of 2,4-DCP were removed at intervals of 12 h. The middle region (B) had no significant differences in Cd concentrations with side regions (A and C), 2,4-DCP concentrations in middle region (B) were lower than side regions (A and C) at intervals of 12 h, while at intervals of 24 h, they showed opposite trends.

Electroosmosis and electromigration are two main processes in electroremediation. Electroosmotic flow is able to drive the free-phase dissolved and even sorbed organics toward cathode regions

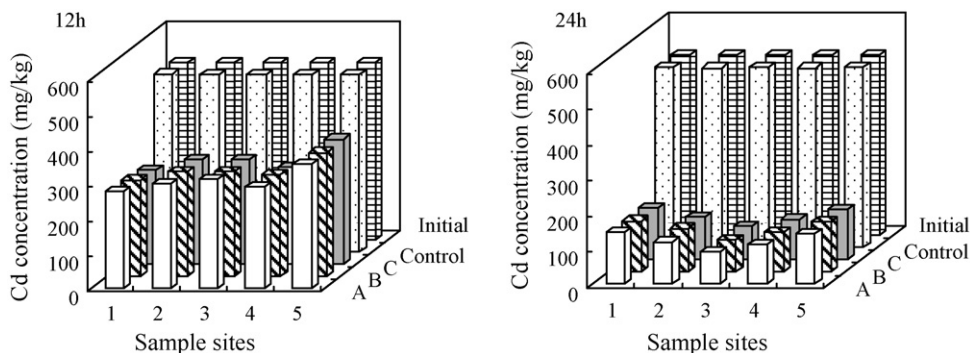


Fig. 4. Spatial distribution of Cd after rotational operation of 10.5 d at intervals of 12 and 24 h.

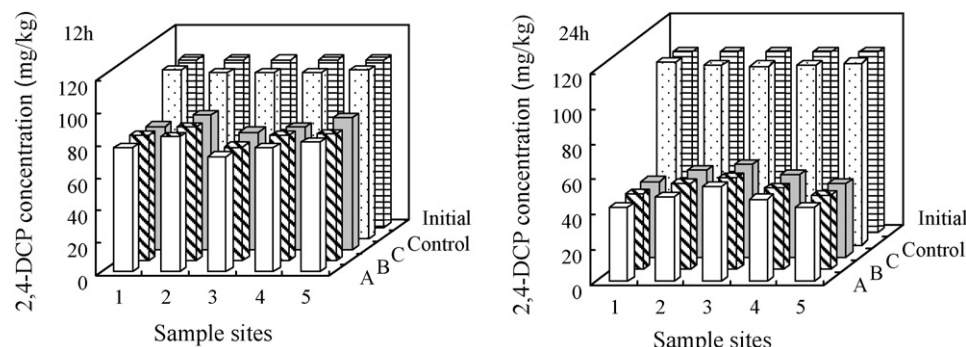


Fig. 5. Spatial distribution of 2,4-DCP after rotational operation of 10.5 d at intervals of 12 and 24 h.

[9]. Charged contaminants can migrate via electromigration in an electric field. Cd^{2+} move towards the cathode while negatively charged 2,4-DCP towards the anode. Thus, although few studies focused have been conducted [12,46], our results confirm that co-contaminants of 2,4-DCP and Cd could be removed simultaneously by electrokinetic remediation.

Sandy soils are characterized by a low total porosity, high permeability and low water-holding capacity. In sandy soil, adsorption of the contaminants to the soil solids is usually low or weak and simple hydraulic flushing will allow the removal of the contaminants. Thus, electrokinetics is generally considered as a useful remediation technique for remediation of clayey soils but not for sandy soils. However, this technique still can be applied in sandy soils. In our previous studies, a series of experiments have been carried out and the results showed that the use of electrokinetics enhances the mobilization and the removal efficiency of phenol and chlorophenol from sandy soil [41]. About 79.6% of Cd was removed from sand loam after 12 d of operation with electrokinetic remediation combined with activated bamboo charcoal [32–33]. The present experiment has proved that co-contaminants of Cd and 2,4-DCP can be removed from sandy soil after application with electroremediation, especially at polarity-reversal intervals of 24 h. The sandy soil used in this experiment contained 5.33% organic matter and 11.21% of soil particles were $<2\ \mu\text{m}$, which may partly explain the success of electroremediation in sandy soils. Vengris et al. [47] investigated electrokinetic remediation of Pb-, Zn- and Cd-contaminated sand and clayey soils under laboratory-scale conditions and found the electrokinetic method is most effective when cleaning polluted sand and sandy loam. Sandy loam and sand have a most coarse grain structure as compared with other types of soil, which ensures a better movement of metal ions and thus better results of cleaning.

In the present experiment, the average removal percentage of Cd is much higher than that of 2,4-DCP, which demonstrates that Cd are more easily removed than 2,4-DCP from sandy soil by use of uniform electrokinetics. In electroremediation process, due to the combined effects of electromigration and electroosmosis, the movement of cationic species towards cathode is enhanced whereas the movement of anionic species towards anode is reduced. In general, the electromigration rate is at least one order of magnitude greater than the electroosmotic flow, and hence electromigration generally dominates mass transport during electrokinetic treatment [2]. In our experiment, soil pH is neutral, thus Cd mainly exists in ionic forms (Cd^{2+} , $\text{Cd}(\text{OH})^+$), which are more readily migrated to cathode by electromigration. The dissolved Cd migrated towards cathode via electroosmotic flow accounted for only a small proportion of total Cd. Therefore, although a sandy soil has a low total porosity and weak electroosmosis because of its high permeability and low water-holding capacity, electroremediation is still effective in removal of Cd from sandy soil. The pK_a value of 2,4-DCP is 7.85 ($\text{C}_6\text{H}_3\text{Cl}_2\text{OH} \rightarrow \text{C}_6\text{H}_3\text{Cl}_2\text{O}^- + \text{H}^+$, $\text{pK}_a = 7.85$). At neutral soil pH, 2,4-DCP mainly exist in the non-dissociated forms, which are easily sorbed on soil particles and difficult to migrate. At the same time, the electroosmosis of neutral 2,4-DCP is reduced due to the decreased soil water content. Moreover, the electromigration of $\text{C}_6\text{H}_3\text{Cl}_2\text{O}^-$ dissociated from 2,4-DCP towards anode, opposite the direction of electroosmotic flow, was reduced by electroosmosis towards the cathode. These may explain the lower removal of 2,4-DCP than Cd from sandy soil.

There may be synergistic or antagonistic interactions on adsorptions and chemical reactions between organics and heavy metals. Organics often affect the chemical fractions and activation of heavy metals, as well as their behaviors. Ricart et al. found that the removal of Cr was improved compared to the experiment where Cr was the only pollutant, and the removal of RB5 (an azo dye) did not decrease [38]. They supposed that the interaction among RB5 and Cr prevented premature precipitation and allowed Cr to migrate

and concentrate in the cathode chamber. Generally, competitive adsorption exists between Cd and 2,4-DCP in soil and bamboo charcoal [48], leading to decreased amounts of ionic Cd and 2,4-DCP molecules adsorbed on soil particles and bamboo charcoal. This may favor the electromigration of Cd and electroosmosis of 2,4-DCP. On the other hand, the negatively charged 2,4-DCP dissociates in aqueous solution may chelate Cd to form a more soluble neutral complex, which is not easily to move by electromigration. Overall, the interactions between 2,4-DCP and Cd may be diverse and need further study.

Bamboo charcoal has excellent adsorption capacity for 2,4-DCP and Cd [48]. This experiment shows that bamboo charcoal is promising as an adsorbent in the electroremediation of soils co-contaminated with 2,4-DCP and Cd. It may be taken as replacement of activated carbon. Certainly, many aspects need to be involved in the future study. First, bamboo charcoal has excellent water holding capacity and high CEC, and may help to keep soil moisture content and pH, which need detailed investigation. Second, the optimal number of treatment zones needs to be determined. Thirdly, other materials for binding the metals and degrading the organics, such as microbes, catalytic agents, oxidants, buffers, should be introduced to treatment zones, and the interaction between these materials and bamboo charcoal also needs further consideration.

3.4. The electricity consumption

The total electricity consumption in 10.5 d was 128.48 and 121.91 kWh/m³, respectively at intervals of 12 and 24 h, and the electricity consumption per day was 12.24 and 11.61 kWh/m³/d, respectively. Clearly, polarity reversal changed migration direction of charged contaminants repeatedly and thus could increase the energy consumption in comparison with unidirectional operation. The shorter the polarity reversal, the higher the electricity consumption. Polarity reversal may induce a frequent change in double layer, and consume a relatively high capacitive part of the current due to the double layer discharging or recharging [49]. Therefore, a proper interval should be adopted for the purpose of saving energy.

Considering both the removal efficiency of contaminants and electricity consumption, polarity-reversal at intervals of 24 h is a more efficient operation mode. When reversing polarity at intervals of 12 h, some of 2,4-DCP and Cd by electromigration and/or electroosmosis maybe did not reach treatment zone before the polarity changed, and then they would move towards the opposite directions, leading to higher power consumption but lower removal efficiency. Just for this reason, the distances between the contaminated soils and treatment zones should be optimized under different polarity-reversal intervals.

Our preliminary results have shown promising potential of electroremediation combined with bamboo charcoal in simultaneous removal of 2,4-DCP and Cd from sandy soil. To our knowledge, this is the first study using electroremediation combined with bamboo charcoal to remove mixed contaminants including organics and heavy metals from sandy soil. In future, additional research will provide both fundamental and practical knowledge that will improve the applications of this technique to actual contaminated sites: (1) the effects of uniform or non-uniform electric fields; (2) the feasibility in other soils such as clay and silt; (3) the application in removal of other co-contaminants; (4) the combination with other remediation techniques such as bioremediation and physical/chemical measures; and (5) the results of pilot-scale and field tests and the feasibility in practical application under field conditions.

4. Conclusions

A bench-scale experiment was conducted to investigate the simultaneous removal of 2,4-DCP and Cd from a sandy loam using

an *in situ* electrokinetic remediation technique combining the uniform electrokinetic technology with a new-type of bamboo charcoal as adsorbent at different periodic polarity-reversals. Our results showed that the electroremediation in this work was effective in simultaneous removal of 2,4-DCP and Cd from sandy soil, but removal percentages were higher when reversing polarity at intervals of 24 h. The electricity consumption was lower at polarity-reversal intervals of 24 h than at polarity-reversal intervals of 12 h. Soil water contents under two operation modes both significantly decreased, but evenly distributed spatially. Soil pH values under two operation modes were all maintained in the range from 7.2 to 7.4, close to the initial value. Our results indicate a promising potential in *in situ* electroremediation of soils co-contaminated with organics and heavy metals.

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