Removal of lead from a silt loam soil by electrokinetic remediation

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Abstract

In this study, a silt loam soil spiked with lead (ca. 1000 mg/kg) was treated by electrokinetics using an electric gradient of 1 V/cm. In all tests, 0.1 M acetic acid was used as the cathode reservoir fluid. However, anode reservoir fluids used were 0.1 M of acetic acid, citric acid, ethylenediaminetetraacetic acid (EDTA), respectively. Experimental results have shown that the electroosmotic (EO) flow in a direction towards the cathode would be reversed if the system pH is less than the point of zero charge (PZC) of the soil particles. Besides, the pH of both electrode reservoirs were found to be important to the removal of lead ions from soil. Experimental results have also shown that using acetic acid, citric acid, and EDTA as anode reservoir fluids resulted in lead removal efficiencies of 27.6%, 53.3%, and 11.2%, respectively. © 1998 Elsevier Science B.V.

Keywords: Contaminated soil; Electrokinetic remediation; Point of zero charge; Electroosmotic flow; Electrode reservoir fluid

1. Introduction

In recent years, a number of laboratory-scale and field-scale studies have shown the technical feasibility of electrokinetic processing in removing various contaminants from soils [1–7]. Removal efficiencies of lead ranging from 75 to 95% [8–13] and 90–95% for cadmium [14] have been reported. In addition, removal of copper [15–17] and many other elements such as As, Cr, Cs, Ni, and Sr [18] from contaminated soils can also be found in the literature. Aside from heavy metals, electrokinetic soil processing has also been employed for treating organic contaminants such as acetic acid, phenols, and BTEX [5,19–22] and even for controlled radionuclide migration [23].

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Four approaches have been identified for metals to be mobilized in soils: (1) changing the acidity, (2) changing the system ionic strength, (3) changing the redox potential, and (4) forming complexes [24]. In the last technique, the addition of a chelating agent (e.g. ethylenediaminetetraacetic acid, EDTA) can convert soil-bound heavy metal ions into soluble metal complexes. Thus, chelating agents are promising extractants for treatment of heavy metal-contaminated soils. The results of many previous studies have also shown that lead and cadmium can be easily removed by EDTA [25–30]. Therefore, EDTA among others, can be a potential conditioning agent used in the electrode reservoirs for removing heavy metals from soil by electrokinetic processing.

It is well known that upon application of low-level direct current to the saturated porous medium, the water in the immediate vicinity of electrodes is electrolyzed. Hydrogen ions will be generated by the anode, thereby resulting in an acid front. Likewise, a base front will be created at the cathode. For example, in a soil column, the acid front will advance across the soil specimen towards the cathode by advection of the pore fluid, migration of ions, and chemical diffusion. While advancing across the soil specimen, the acid front exchanges with adsorbed cations in the electric double layer of contaminated soil particles [23]. This can be viewed as an acid flushing process for treating contaminated soils. In the light of this principle, the anode reservoir fluid would play an important role as a flushing fluid in electrokinetic soil processing. Two selected approaches identified by Pickering [24], namely changing the acidity and forming complexes, were adopted in this work to verify the role of the anode reservoir fluid during electrokinetic remediation of heavy metal-contaminated soil. Acetic acid, citric acid, and EDTA solution are acidic in nature and they are widely used in soil washing/flushing. Thus, they were selected as the anode reservoir fluids in this study. By using acetic acid as the cathode reservoir fluid in all tests, the removal efficiencies of lead from an artificially contaminated soil by electrokinetic processing were determined and compared.

During the electrokinetic soil processing, electroosmotic (EO) flow, ionic migration, chemical diffusion, etc., are considered to be important mass transfer mechanisms. The transport of pollutants is believed to be influenced by the distribution of H^+ and OH^- in the soil system. Therefore, the in situ pH in the soil column is of concern in this study. The EO flow is widely considered to be responsible for the removal of pollutants. As a result, the quantity and direction of the EO flow were also included in this investigation.

The objective of this study was to determine the roles of electrode reservoir fluid, soil system pH, and EO flow in removing lead from an artificially contaminated soil.

2. Experimental

2.1. Materials

In this work, the original soil sample was obtained from a college campus in southern Taiwan. This soil is considered to be an 'uncontaminated' one. After collection, the soil

sample was air dried first. Pebbles, stones, and plant debris were hand-picked and discarded. The soil sample was further subjected to screening to obtain the minus 10-mesh fraction (i.e. < 2.00 mm) for later uses.

Acetic acid (100% in purity; Merck), citric acid (99.5% in purity; Showa), ethylene-dinitrilo tetraacetic acid disodium salt dihydrate (designated EDTA in this study; 99% in purity; Merck), lead nitrate (99.5% in purity; Showa), and other chemical compounds used in this work are all reagent grade. Standard ASTM Type I deionized water was used throughout this study.

2.2. Scheme

In this work, three electrode reservoir fluids were used to investigate the removal efficiency of lead from an artificially contaminated soil by electrokinetics. After the sample pretreatment, the soil specimen was first characterized and then spiked with lead. The contaminated soil specimen, thus, obtained was electrokinetically treated by a constant voltage of 20 V. 0.1 M acetic acid was used as the cathode reservoir fluid throughout this study; whereas 0.1 M of acetic acid, citric acid, and EDTA aqueous solution, respectively were used as the anode reservoir fluid. Using graphite electrodes, an electric field of 1 V/cm was applied to the soil column for 20 days in all tests. Each of test soil specimens was first put in a cylindrical cell having a diameter of 9.5 cm, then it was compacted longitudinally by a weight of 10 kg for 24 h. The soil specimen was then removed to the soil chamber of the experimental apparatus. After the whole apparatus was connected, the desired electrode reservoir fluids were injected. Before the application of the external electric field, the soil specimen was conditioned with the electrode reservoir fluids for 24 h. The values of pH and electric conductivity in both electrode reservoirs were determined as the initial pH and electric conductivities. Table 1 summarizes the test program employed in this work. In the course of testing, pH values of both electrode reservoir fluids and EO flow were measured every 24 h; whereas the pH distribution in the soil column were determined every five days. At the end of each test, the soil column was longitudinally divided into five equal-length fractions. For each treated soil fraction, the contents of residual moisture and lead were analyzed.

Table 1
The summary of test program for electrokinetic soil processing

| Experimental parameter	Test 1	Test 2	Test 3
Electric field applied (V/cm)	1	1	1
Cathode reservoir fluid	0.1 M acetic acid	0.1 M acetic acid	0.1 M acetic acid
Anode reservoir fluid	0.1 M acetic acid	0.1 M citric acid	0.1 M EDTA
Initial pH in cathode reservoir	2.9	2.8	2.8
Initial pH in anode reservoir	2.9	1.9	4.3
Initial electric conductivity in cathode reservoir (mS/cm)	4.7	5.2	4.5
Initial electric conductivity in anode reservoir (mS/cm)	4.6	27.6	91.6
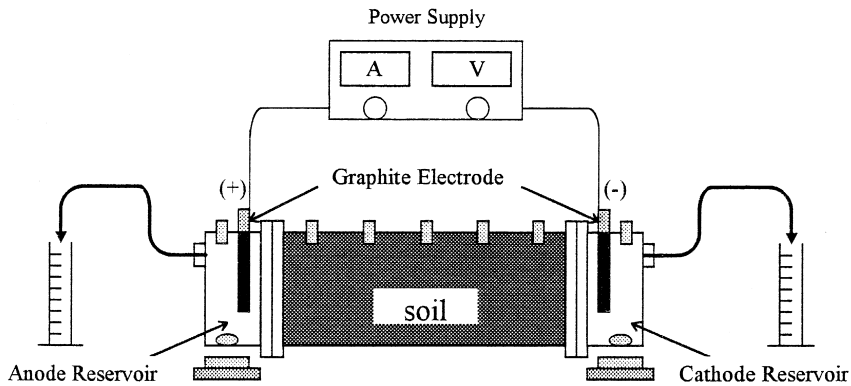
Fig. 1. A schematic diagram showing the electrokinetic soil processing system.

2.3. Apparatus

Fig. 1 shows the schematic diagram of the electrokinetic soil processing system used in this work. The soil specimen was placed in an acrylic, cylindrical cell (i.e. soil cell) of 20 cm in length and 9.5 cm in diameter. Along the longitudinal axis of the cylindrical cell, five holes of equal distance were drilled for the purpose of in situ soil pH and electric potential measurements. At each end of the soil cell, a set of filter media (a Whatman No. 42 filter paper was sandwiched in two pieces of nylon clothes) were used to separate the soil particles and the respective electrode reservoir fluid. Each of the electrode reservoir compartments that is connected to the soil cell has a holding capacity of 850 ml. Graphite anode and cathode, partly immersed in the respective electrode reservoir fluids, were connected with a direct-current power supply. The graphite electrodes (coded EGF 264) were manufactured by Tokai Carbon (Japan). Each electrode has a specific gravity of 1.85 and an electric resistance of 1200 $\mu\Omega$/cm.

2.4. Characterization of the original soil

The minus 10-mesh fraction of the original soil was further subjected to the following analyses: (1) particle size analysis, by dry sieving and Coulter LS100 Particle Size Analyzer; (2) pH, by Method 9045, SW-846, U.S. EPA; (3) density, by ASTM D584-83; (4) cation exchange capacity, by Method 9081 (sodium acetate), SW-846, U.S. EPA; (5) loss on ignition: according to Ref. [31]; (6) point of zero charge (PZC), by electrophoresis; (7) surface area, by Micrometritics ASAP 2010 Surface Area Analyzer; (8) carbon analysis: according to Ref. [32]; (9) major mineral constituents: by X-ray diffraction [33]; (10) total contents of heavy metals, by NIEA S321.60 T, R.O.C. EPA [34]; and (11) 0.1 M HCl extractable concentrations of heavy metals, by a R.O.C. EPA standard method to be presented in more details in Section 2.6.

2.5. Preparation of the test soil specimen

In this investigation, the artificially contaminated soil was prepared by mixing the pretreated, original soil with a lead nitrate stock solution of 1000 mg/l. First, 3 l of lead

nitrate solution was added to 2.5 kg of soil. The mixture was then agitated for 1 h using a horizontally rotary shaker at 120 rpm. After mixing, the soil slurry was kept still for 24 h before filtering out the extra solution. To have a similar soil compaction in all test soil specimens, the wet soil mass then was placed in a vertical column (9.5 cm in diameter) for compacting, assisted with a 10 kg weight from the top for 24 h. After this, the compacted, wet soil mass was placed in the soil cell of electrokinetic processing system. At this time, the moisture content and total Pb content were determined.

2.6. Analysis of lead concentration of the test soil specimen

To compare with the tentative management standards of soil quality in Taiwan, the lead concentration in soil specimen was determined according to NIEA S320.60 T, an R.O.C. EPA standard method [35]. This was carried out by mixing 10 g of soil with 100 ml of 0.1 M HCl for 1 h at 120–180 rpm. The soil slurry was filtered with a Whatman No. 42 filter paper. The filtrate was analyzed by flame atomic absorption spectroscopy for determining the lead concentration.

2.7. Measurements during the experiments

As indicated in Section 2.2, the measurements of pH values of both electrode reservoir fluids, the quantity of the EO flow, and the pH distribution in the soil column were made in this study. Every 24 h about 30 ml of each electrode reservoir fluid was taken out for the determination of its pH value. The fluid was then put back to the reservoir after each determination. As shown in Fig. 1, due to the phenomenon of the EO flow, the reservoir overflows were collected separately and determined for their quantities every 24 h. For the determination of in situ soil pH, a pH electrode was put into the soil through each measuring hole on the top of the soil chamber. The pH distribution in the soil column was determined in this manner every 5 days.

3. Results and discussion

3.1. Characteristics of the original soil

Table 2 shows the characterization results of the pretreated, minus 10-mesh fraction of the original soil. Based on the result of particle size analysis, this soil is categorized as a silt loam according to the U.S. Department of Agriculture classification system. The soil pH and PZC were determined to be 5.12 and 5.10, respectively. Thus, this soil is acidic in nature. Besides, it has a rather high CEC value of 27.6 cmol/kg (or 27.6 meq/100 g). But this value is close to the CEC value (i.e. 25.6 cmol/kg) reported for a silt clay loam by Thompson et al. [13]. From the background values of heavy metal contents, this original soil can be considered as a clean, uncontaminated soil according to the soil quality standards of R.O.C. EPA. Thus, it is a suitable one for preparing an artificially contaminated soil.

Table 2
The characteristics of the original soil

Item determined	Value
Particle size analysis	$< 2\ \mu$m: 19.17%
	2–50 μm: 75.61%
	50–2000 μm: 5.22%
Texture	Silt loam
pH	5.12
Density	2.54 g/cm^3
Cation exchange capacity	27.6 cmol/kg
Point of zero charge	5.10
BET surface area	36.70 m^2/g
Loss on ignition	6.64%
Carbon content	Inorganic: 0.01%
	Organic: 0.67%
Major mineral constituent	Quartz, kaolinite, and illite/vermiculite
Total content of heavy metal	Cd: 0.50 mg/kg
	Pb: 36.28 mg/kg
	Cr: 51.52 mg/kg
	Cu: 20.22 mg/kg
	Zn: 59.90 mg/kg
0.1 M HCl extractable heavy metal	Cd: Not detected
	Pb: 7.95 mg/kg
	Cr: 5.51 mg/kg
	Cu: 1.23 mg/kg
	Zn: 1.41 mg/kg

3.2. Changes of in situ pH in the soil column

Figs. 2–4 show the variations of the in situ pH profiles with time for the soil columns of Tests 1–3. For all the tests, the initial, in situ pH was about 4.6–4.9 for the tested soil columns. As the test proceeded, roughly speaking, the in situ soil pH on the 5th day increased. Then the pH throughout the soil systems of Tests 1 and 2 began to decrease, while the in situ pH of Test 3 remaining above the original soil pH profile. On the 10th day, the in situ pH profiles of Tests 1 and 2 dropped to the neighborhood of four; whereas the in situ pH of Test 3 ranged from 5.2 to 5.6. Apparently, the acidification of soil in Tests 1 and 2 has begun by this time. Experimental data of the Days 15 and 20 did not show further decreasing of in situ pH throughout the soil systems for Tests 1 and 2. For Test 3, a slight decrease of in situ pH after the 10th day till the end of the test has been observed. But the in situ pH near the cathode end increased slightly. Perhaps, this was influenced by a very high pH (e.g. 12.93) of the cathode reservoir fluid at the end of Test 3 (see Fig. 5). For Test 3, it is important to bear in mind that the in situ soil pH profile remained above the initial one almost all the time throughout the test period (see Fig. 4).

Experimental results shown in Figs. 2 and 3 are in good agreement with the results reported by Reed and Berg [12] and Thompson et al. [13]. In their systems of flushing lead from silt loam by electrokinetics, acetic acid was used as the cathode reservoir fluid
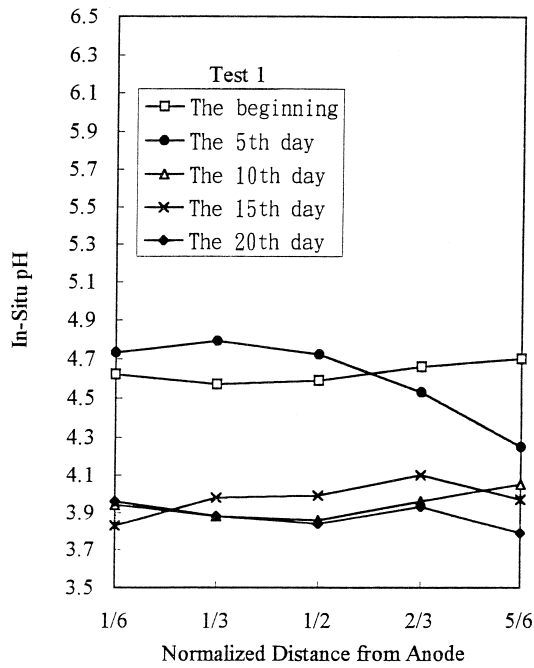
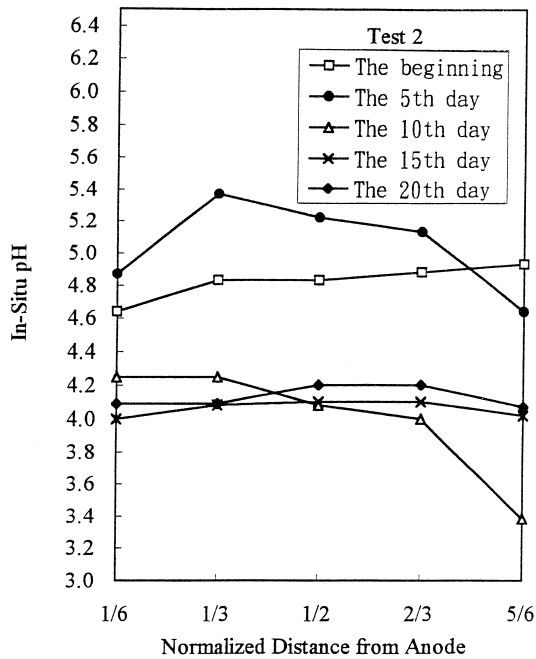Fig. 2. The in situ pH distribution in the soil column for Test 1.

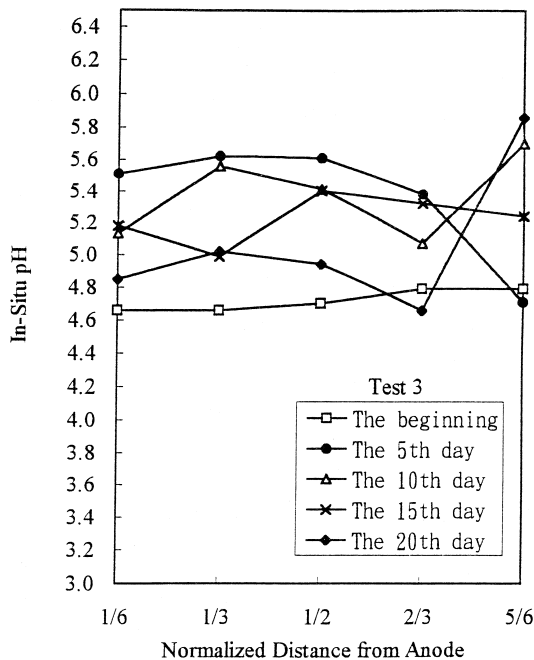Fig. 3. The in situ pH distribution in the soil column for the Test 2.

Fig. 4. The in situ pH distribution in the soil column for Test 3.

while using 0.1 N HCl as a conditioning agent in the anode reservoir. HCl was used in the anode reservoir to increase the amount of H^+ available for transfer through the soil column. Their final soil pH ranged from 3 to 3.5. In our study, much weaker acids (i.e. 0.1 M acetic acid and citric acid, respectively) were used as conditioning agents in the anode reservoir in Tests 1 and 2. Patterns of soil pH profiles similar to that of reported by Reed and his co-workers were obtained, but the final soil pH was less than one unit higher. As for Test 3, EDTA was used as in the anode reservoir instead. As shown in Fig. 5, the pH profile of the anode reservoir of Test 3 is about one pH unit higher than that of Tests 1 and 2. In other words, EDTA had a weaker capability of acidifying this soil as compared with acetic acid and citric acid. Hence, it is understandable that the final pH profile for EDTA is also about one pH unit higher than the other two acids.

3.3. The relationship between electroosmostic (EO) flow and point of zero charge (PZC)

Although a normal EO flow towards the cathode is commonplace in the system of electrokinetic soil processing, different directions of the EO flow were observed in this work (see Fig. 6). Here, a positive value of the flow quantity is designated to a normal EO flow towards the cathode; whereas a negative value for a reverse EO flow towards the anode. In Test 1, 101.4 ml of the reverse EO flow was collected for the very first day of the test. A reverse EO flow continued for the following two days with a smaller quantity each day. During Day 4 to Day 9, almost no EO flow was collected. Starting from Day 10, it began to have a reverse EO flow again and it continued till the end of the test. For Test 1, the total cumulative flow quantity was determined to be 660 ml. No
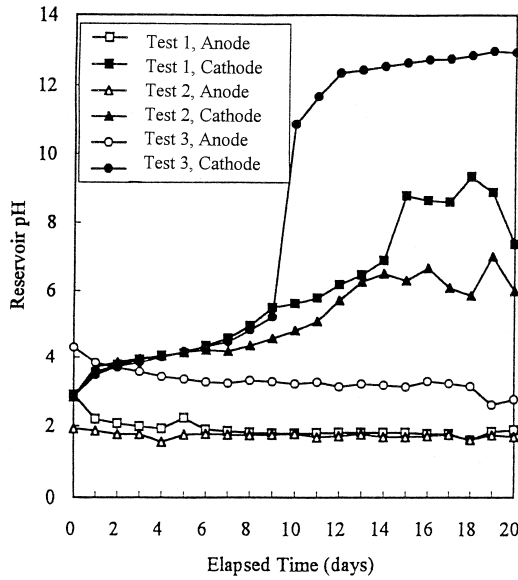
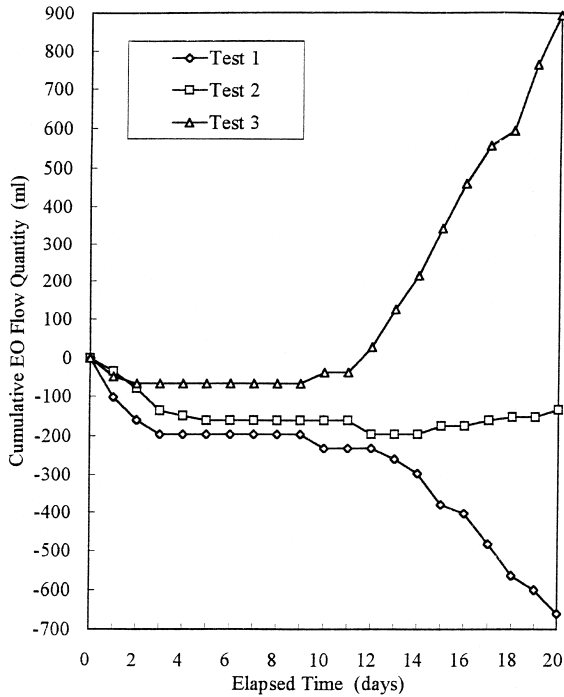Fig. 5. Anode and cathode reservoir pH vs. time for Tests 1–3.

Fig. 6. Cumulative EO flow quantities vs. time for Tests 1–3.

normal EO flow was observed and collected at the cathode reservoir for Test 1 where acetic acid was used in both electrode reservoirs. A similar finding was also reported by Eykholt and Daniel [17]. In their test, an obvious reverse EO flow was generated using sodium citrate as the electrode reservoir fluid for both anode and cathode. Like that of Test 1, a certain quantity of the reverse EO flow was determined for each of the first 5 days in Test 2. During the period of Day 6 to Day 11, no obvious EO flow was observed. Then a small quantity of the reverse EO flow was determined during the next few days. On Day 15, it began to have a normal EO flow and it continued until Day 20. The cumulative reverse EO flow amounted to 196 ml, whereas the cumulative normal EO flow totalled 62.5 ml. In other words, a net value of 133.5 ml of the fluid was collected from the anode reservoir. For Test 3, a total of 66.3 ml of the reverse EO flow was collected for the first 2 days of the test. No EO flow was observed from Day 3 to Day 9. On the 10th day, a normal EO flow began to observe. Starting from Day 12 through Day 20, a large amount of the normal EO flow was collected each day. The cumulative normal EO flow totalled 960.5 ml, resulting in a net 894.2 ml of the normal EO flow for Test 3. From the test results presented above, Test 1 (acetic acid–soil–acetic acid system) yielded the largest net quantity of the reverse EO flow and Test 3 (EDTA–soil–acetic acid system) yielded the largest net quantity of the normal EO flow. Test 2 (citric acid–soil–acetic acid system) yielded only a nominal amount of the reverse EO flow. No normal EO flow was observed in this test.

The experimental results of this work have clearly indicated that the normal EO flow occurred when the in situ soil pH profile remained above the PZC of the soil particles in the system. Namely, $pH_{pzc} = 5.1$ in this study. On the other hand, when the in situ soil pH profile was lower than the PZC of the soil particles, the reverse EO flow prevailed. Experimental results also suggested that a greater cumulative quantity of the reverse EO flow in Test 1 was due to a greater difference between the system pH and the PZC of the soil particles as compared with Test 2 (see Figs. 2 and 3). This observation is in good agreement with that proposed as a result of an electroosmosis modeling study [17]. In addition, our finding is supported by the experimental results of other researchers. In a work by Weng et al. [36], the soil pH and PZC of soil particles were 8.1 and 6.8, respectively. In all their tests, the in situ soil pH in the soil columns remained at a pH between 8 and 9. A normal EO flow was generated in each of their tests. It is clear that both system pH and the PZC of the soil particles play important roles in affecting the direction of the EO flow.

3.4. Residual lead concentration distribution in the soil column

Figs. 7–9 show the residual lead concentration distributions in the soil columns for Tests 1–3, respectively. As pointed out above, acids were used to provide additional H^+ for flushing lead from the contaminated soil in Tests 1 and 2. This of course would assist the positively charged lead ions to migrate towards the cathode during the electrokinetic soil processing.

In Test 1, the residual lead concentration near the anode end in the soil column decreased from the initial value of 1026.6 mg/kg to 92.7 mg/kg resulting in a removal efficiency of 90.97%. However, the removal efficiencies of lead for the rest soil
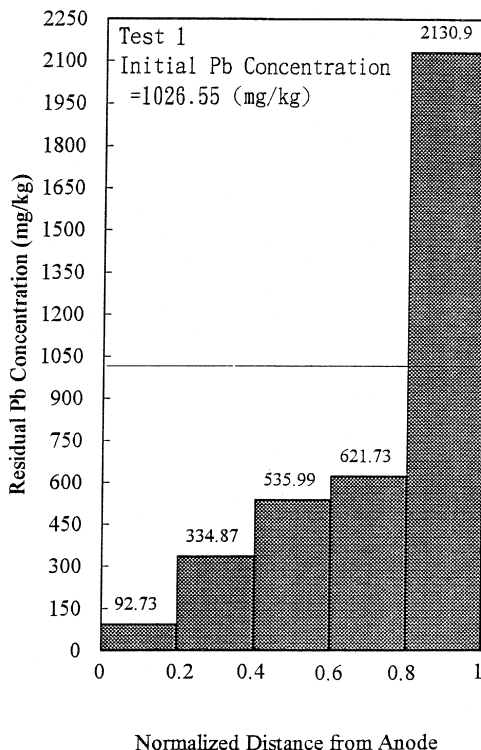
Fig. 7. The residual lead concentration distribution in the soil column for Test 1.

fractions were found to be decreasing towards the cathode end. It was also determined that the residual lead concentration for the soil fraction nearest the cathode end was 2130.9 mg/kg (see Fig. 7). This value is 2.08 times of the initial lead concentration of the soil. Obviously, it had a very serious deposition of lead or its compounds there. Perhaps this was due to the reaction of lead ions and electrons forming lead metal at the cathode end of the soil column. Owing to the electrolysis of water at the cathode, the cathode reservoir pH of Test 1 has increased to the neighborhood of nine starting from Day 15 (see Fig. 5). The base front thus formed would enter the soil cell. However, an immediate contact of lead ions in the soil column and the base front at this pH would yield the precipitate of $Pb(OH)_2$ on soil near the cathode end. Besides, the precipitate of lead carbonate might also exist in this case. The overall efficiency of lead removal for Test 1 was only 27.60%. In a test conducted by other researchers [12] using 0.1 N hydrochloric acid–soil–1 M acetic acid system for flushing lead (1000 mg/kg) from contaminated soil, the reported removal efficiency was 97%. Apparently, this is due to the fact that 0.1 N hydrochloric acid is a much stronger acid than 0.1 M acetic acid used in Test 1. Therefore, a proper selection of electrode reservoir fluids is very important in removing heavy metals from soil.
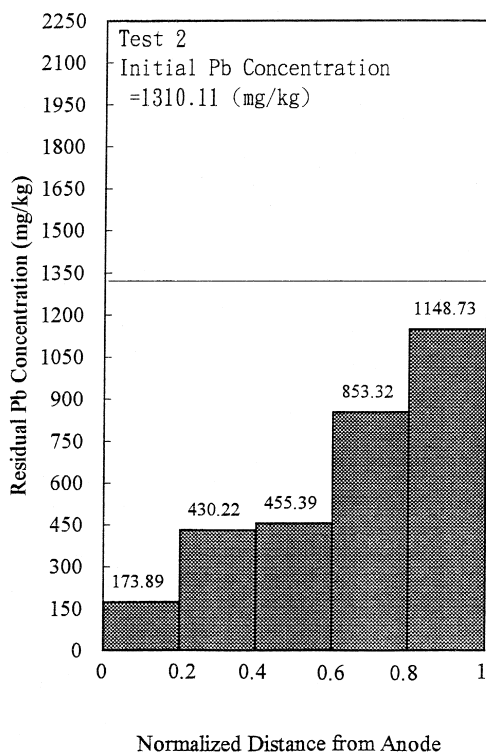
Fig. 8. The residual lead concentration distribution in the soil column for Test 2.

For Test 2 of the present study, as compared with Test 1, a somewhat similar pattern of the residual lead concentration in the soil column was obtained (see Figs. 7 and 8). The removal efficiencies of lead for the soil fractions near the anode end and the cathode end were determined to be 86.73% and 12.32%, respectively. The overall removal efficiency of lead was 53.26% for Test 2. Namely, citric acid outperformed acetic acid as a conditioning agent in the anode reservoir in this work. From Fig. 5, it is clear that the cathode reservoir pH of Test 2 remained lower than that of Test 1 throughout the test period. Perhaps it would explain why there was no serious deposition of lead and/or its precipitates near the cathode end of the soil column.

In the case of EDTA (i.e. Test 3), a very different pattern of the residual lead concentration in the soil column was obtained (see Fig. 9). The removal efficiencies of lead for the first three soil fractions from the anode were determined to be 42.91%, 108%, and 117%, respectively. A high accumulation of lead in the middle part of the soil column was observed. For the soil fraction near the cathode end, its lead removal efficiency was 23.04%. Thus, the overall removal efficiency of lead was only 11.16%. The experimental results showed that the normal EO flow towards the cathode was prevailing in Test 3 (see Fig. 6). Therefore, it was favorable for the anode reservoir fluid (i.e. EDTA in this case) to continuously flow into the soil column. According to Fig. 5,
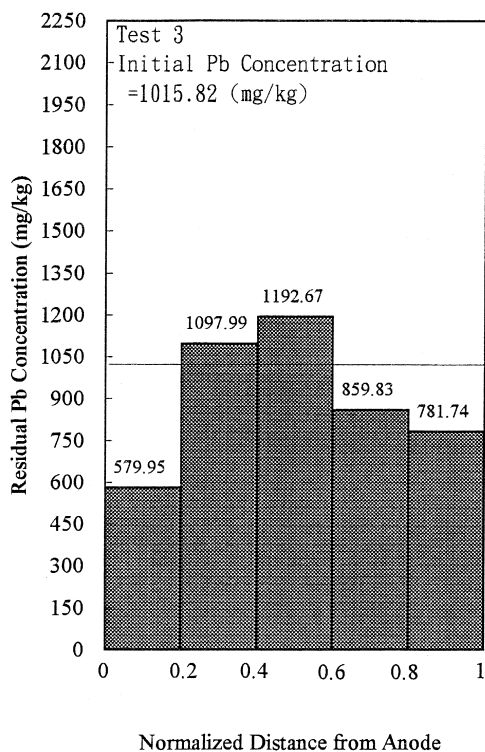
Fig. 9. The residual lead concentration distribution in the soil column for Test 3.

the pH of the anode reservoir fluid remained in the neighborhood of 3.0 throughout the test period. At this pH, EDTA species are in the forms of H_3Y^- and H_2Y^{2-} [37]. After complexing with lead ions in the soil column, they should migrate towards the cathode. The in situ soil pH profile ($< pH\ 6$) shown in Fig. 4 also suggested so. In other words, these conditions are favorable for the migration of EDTA–lead complexes towards the cathode. Perhaps when EDTA–lead complexes flowed through the soil column and entered the cathode reservoir after Day 10, a pH of greater than 10 further dissociated EDTA to a species of Y^{4-} [37]. Consequently, the EDTA–lead complexes became negatively charged and began to migrate back towards the anode. As a result of this conflict, lead was accumulated in the middle part of the soil column. If so, the proposed hypothesis satisfactorily explains the experimental finding of Test 3 in this work.

It is worth pointing out that some film-like materials formed on the cathode and in the cathode reservoir compartment during the experiments of all tests. However, they were not analyzed in this study. In this study, no sample of electrode reservoir fluids was taken out for the determination of lead concentration so that the pH and electric conductivities of electrode reservoir fluids would not be altered. Due to the difficulties indicated above, a material balance of lead was not conducted in this study. But it is believed that a portion of Pb has been removed from the soil, then passed through the filter media and entered the cathode reservoir compartment.

4. Conclusions

Acetic acid, citric acid, and EDTA (a chelant) were used as the conditioning agents in the anode reservoir, respectively, with acetic acid as the cathode reservoir fluid in an electrokinetic soil processing system. A silt loam soil spiked with lead (ca. 1000 mg/kg) was thus treated by applying an electric gradient of 1 V/cm to the soil system. Based on the experimental results obtained, several conclusions can be drawn.

(1) The soil system pH and the PZC of the soil particles are controlling the direction of the EO flow. When the former is greater than the later, the EO flow direction is towards the cathode. Otherwise, it is towards the anode.

(2) Properties of both electrode reservoir fluids and reservoir pH are important to the removal efficiency of lead. A poor selection of EDTA and acetic acid as the anode and cathode reservoir fluids resulted in a serious accumulation of lead and/or its compounds in the middle part of the soil column.

(3) In terms of the removal efficiency of lead, citric acid outperformed acetic acid and EDTA. EDTA yielded the lowest removal efficiency. For citric acid, an overall removal efficiency of 53.26% is not satisfactory. Thus, a better experimental condition has to be sought to enhance its performance in removing lead from soil.

Acknowledgements

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