

Effects of sodium hypochlorite and high pH buffer solution in electrokinetic soil treatment on soil chromium removal and the functional diversity of soil microbial community

Long Cang^a, Dong-Mei Zhou^{a,*}, Akram N. Alshwabkeh^b, Hai-Feng Chen^a

^a State Key Laboratory of Soil and Sustainable Agriculture, Institute of Soil Science, Chinese Academy of Sciences, Nanjing 210008, China

^b Department of Civil and Environmental Engineering, Northeastern University, Boston, MA, USA

Received 27 September 2005; received in revised form 25 July 2006; accepted 28 July 2006

Available online 3 August 2006

Abstract

Effects of sodium hypochlorite (NaClO), applied as an oxidant in catholyte, and high pH buffer solution on soil Cr removal and the functional diversity of soil microbial community during enhanced electrokinetic treatments of a chromium (Cr) contaminated red soil are evaluated. Using pH control system to maintain high alkalinity of soil together with the use of NaClO increased the electrical conductivities of soil pore liquid and electroosmotic flux compared with the control (Exp-01). The pH control and NaClO improved the removal of Cr(VI) and total Cr from the soil. The highest removal percentages of soil Cr(VI) and total Cr were 96 and 72%, respectively, in Exp-04 when the pH value of the anolyte was controlled at 10 and NaClO was added in the catholyte. The alkaline soil environment and introduction of NaClO in the soil enhanced the desorption of Cr(VI) from the soil and promoted Cr(III) oxidation to mobile Cr(VI), respectively. However, the elevated pH and introduction of NaClO in the soil, which are necessary for improving the removal efficiency of soil Cr, resulted in a significantly adverse impact on the functional diversity of soil microbial community. It suggests that to assess the negative impact of extreme conditions for enhancing the extraction efficiencies of Cr on the soil properties and function is necessary.

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Keywords: Electrokinetics; Chromium; Sodium hypochlorite; Red soil; Microbial functional diversity

1. Introduction

In recent years, electrokinetic soil remediation has shown fast development and its application has gained an increasing interest around the world [1–3], in which a direct current is applied in polluted soil to drive the transport of pollutants from soil towards electrodes by electromigration, electroosmosis and electrophoresis. However, its application on Cr contaminated soils is complex because Cr usually exists in two different valences; Cr(III) and Cr(VI), depending on the soil environment. Cr(III) exists as cations, while Cr(VI) usually existed as toxic oxyanions hydrochromate (HCrO_4^-), dichromate ($\text{Cr}_2\text{O}_7^{2-}$) and chromate (CrO_4^{2-}). As a result of electric field application, Cr(VI) oxyanions migrate towards the anode [4]. On the other hand, cationic Cr(III) migrates towards the cathode. Although

Cr(III) is less toxic than Cr(VI) and less practically removed from soil, it can be oxidized into mobile and toxic Cr(VI) in certain conditions such as by MnO_2 [5].

Several studies exist on the electrokinetic treatment of soil Cr(III). Using lactic acid [6], citric acid, ethylene diamine tetraacetic acid (EDTA) and sodium dodecyl sulfate (SDS) [7,8] as complexing reagents, improved solubility and mobility of Cr(III) in clays and correspondingly enhanced its removal. But, it is difficult to meet similar removal efficiencies in real soil because Cr(III) strongly adsorbed in soil or existed as chromite. When $\text{pH} > 11$, the remediation efficiency of soil Cr(III) improves, because it can form mobile anionic hydroxyl complexes $\text{Cr}(\text{OH})_4^-$ and $\text{Cr}(\text{OH})_5^{2-}$ [9].

Adding oxidant in soil is an alternative for increasing the treatment and extraction effectiveness of Cr(III) by oxidizing Cr(III) into Cr(VI). The extent and effect of Cr(III) oxidation on the electrokinetic remedial efficiency in manganese-enriched clay has been assessed. In the presence of manganese, oxidation of Cr(III) to Cr(VI) ranged from 67% in kaolin to 28% in

* Corresponding author. Tel.: +86 25 86881180; fax: +86 25 86881180.
E-mail address: dmzhou@issas.ac.cn (D.-M. Zhou).

Table 1
Selected soil physicochemical characteristics

pH _{1:2.5}	EC ($\mu\text{S cm}^{-1}$)	OM (g kg^{-1})	CEC (cmol kg^{-1})	Soil texture (μm , %)					Heavy metal (mg kg^{-1})				
				<2	2–10	10–50	50–250	>250	Cr	Cu	Zn	Pb	Cd
4.81	28	22.5	12.2	24.6	24.6	22.5	24.3	4.0	78.1	19.3	57.0	31.3	0.07

glacial till, before the application of electric potential [5]. However, Cr(VI) adsorbed to soil surfaces near anode region due to low soil pH environment which was resulted from the H⁺ production at anode during electrokinetic [10–13], and correspondingly limited Cr(VI) removal from soil. For a glacial till with high pH, Cr(VI) adsorption was low which resulted in a high Cr(VI) removal during the electrokinetics [9]. NaClO can oxidize soil Cr(III) into mobile Cr(VI). Therefore, to obtain a quantitative removal of chromium with electrokinetic treatment, the success of the process is governed by the control of alkaline pH and by the injection of oxidant in the soil sample [14]. The use of alkaline and oxidizing environments resulted in removal of almost all chromium from kaolinite after 18 day and removal of 74% of chromium from natural soil after 27 day.

However, the effect of increasing the alkalinity and adding an oxidant in electrokinetic treatment on soil properties is unclear. The objective of this paper is to optimize the treatment conditions and to evaluate the effect of injecting NaClO together with electrolyte control at high pH in a chromium-contaminated red soil on the electrokinetic processes and the functional diversity of soil microbial community performed by BIOLOG analysis.

2. Materials and methods

2.1. Soils

The red soil (Hapludults) was sampled from the top 20 to 40 cm of an uncultivated land in Yingtan, Jiangxi province. The soil was air-dried, ground, and sieved through a 0.84 mm diameter sieve. Physicochemical characteristics of the soil are listed in Table 1. Soil pH and EC were measured by preparing a slurry of 1:2.5 soil to water ratio and using a pH meter and an EC meter, respectively. Soil Cu, Pb, Zn, Cr, Cd were analyzed by a Hitachi 180–80 atomic absorbance spectrometer (AAS) after digesting 0.5 g of soil with HF–HNO₃–HClO₄. Two certified reference soil samples were used for quality control of the analytical results. Soil Cr(VI) was analyzed by USEPA method 3060A. According to the method, 2.5 g of soil was extracted by 50 mL of 20 g L⁻¹ NaOH and 30 g L⁻¹ Na₂CO₃ and 0.5 mL of 1.0 mol L⁻¹ phosphate buffer (pH 7.0) at 90–95 °C for 1 h, and then adjusted with 5.0 mol L⁻¹ HNO₃ to pH 7.5 ± 0.5 and

filtered through a 0.45 μm membrane. The Cr(VI) concentration in the supernatant was determined by AAS.

The Cr polluted soil was prepared by spiking 1 L of 1000 mg Cr(VI) L⁻¹ solution with 1000 g soil to produce a final Cr concentration of 1000 mg kg⁻¹. The soil was thoroughly mixed, maintained in a pot, and was covered with 2 cm water at room temperature. After curing the mix for 1 month, the soil was air-dried, crushed, sieved through a 20-mesh sieve again, and was preserved for experimental testing. The pH, EC, total Cr and Cr(VI) of the contaminated red soil after the cultivation were 4.56, 2.65 × 10² $\mu\text{S cm}^{-1}$, 1009 and 488 mg kg⁻¹, respectively.

2.2. Methods

The set-up used for the electrokinetic experiments consists of a soil column, two electrode reservoirs, an electric power supply, and a four-channel peristaltic pump, as shown in Fig. 1. The peristaltic pump was used for circulating the electrolyte in the electrode reservoirs at a flow rate of 18 mL min⁻¹. Square titanium alloy sheets (3 cm × 3 cm) were used as the working electrodes. The soil column was a 12 cm long PVC cylinder with an inner diameter of 6.6 cm. Two porous plates of fritted glass were used for preventing soil dispersion or leakage from soil column to electrode reservoirs.

About 580 g of soil was compacted in the soil column. The set-up was assembled and deionized water was cycled for 24 h prior to the application of electric current. A DC voltage was applied to the cell and water or pH buffer solutions (pH 10), depending on the test conditions, were cycled through the anode and cathode reservoirs. A pH control system was used to control the rate of addition of the buffer solution and to keep its pH at 10. Four experiments were performed, and shown in Table 2. Exp-01 is a control without any enhancing conditions, Exp-02 is a treatment by controlling the anolyte pH at 10, Exp-03 is to examine the electrokinetic treatment using phosphate buffer solutions (pH 10) as the anolyte and catholyte both, and Exp-04 is another treatment similar to Exp-03 except 0.1% NaClO was added in the catholyte. The amount of electroosmotic flow was calculated by measuring the change of anolyte volume. At the end of testing, the DC source was disconnected, the set-up was

Table 2
Experimental design of the electrokinetic treatments ($t = 492$ h)

	Anolyte	Catholyte	Voltage	pH control at 10
Exp-01	Deionized water	Deionized water	20 V	No control
Exp-02	Phosphate buffer	Deionized water	20 V	Anolyte only
Exp-03	Phosphate buffer	Phosphate buffer	20 V and then decreased to 10 V after 350 h treatment	Anolyte only
Exp-04	Phosphate buffer	Phosphate buffer + 0.1% NaClO	20 V and then decreased to 10 V after 160 h treatment	Anolyte only

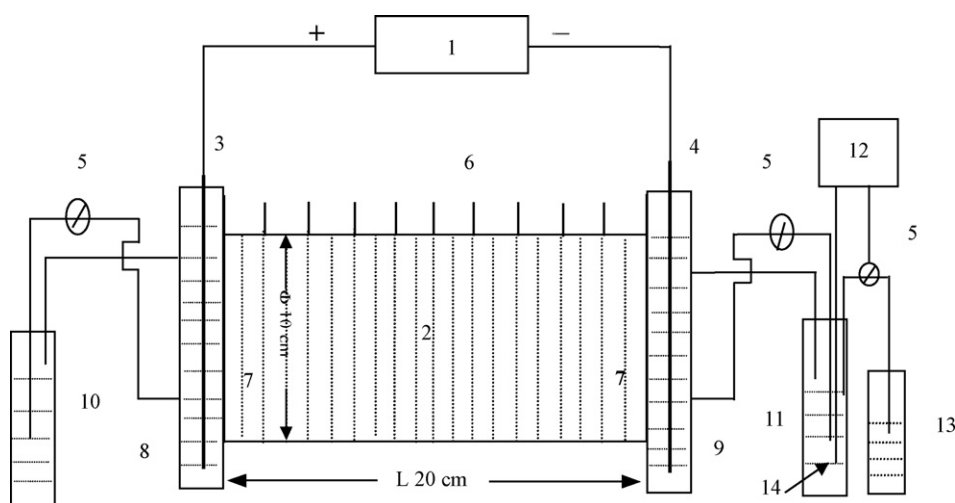


Fig. 1. A schematic of the electrokinetic laboratory apparatus. 1, DC power supply; 2, soil cell; 3, anode; 4, cathode; 5, peristaltic pump; 6, passive electrode; 7, fritted glass membrane; 8, anode chamber; 9, cathode chamber; 10, anodic solution reservoir; 11, cathodic solution reservoir; 12, pH controller; 13, stock solution bottle; 14, pH electrode.

dismantled and the soil column was cut into five equal sections that were labeled S1 to S5 from the anode to cathode.

2.3. Soil subsamples analysis

The pH and EC of soil subsamples after electrokinetic treatments were measured by preparing a slurry of 1:2.5 fresh soil to water ratio and using a pH meter and an EC meter, respectively. The soil subsamples, used for the analysis of total Cr, Cr(VI) and BIOLOG, were air-dried, and ground to pass through a 0.149 mm diameter screen.

The soil subsamples were analyzed by BIOLOG (Hayward, California, USA) to determine the response of soil microbial communities to the electrokinetic treatment. There are 96 reaction wells in the BIOLOG EcoPlates™, which are equally divided into three groups. Each group consisted of one control, four polymers, six amino acids, seven carbohydrates, two amines, nine carboxylic acids, and three phenols. The compositions of the soil microbial communities were analyzed according to the well color change induced by their utilization of various C-sources. The samples for BIOLOG analysis were S1 and S5 in Exp-01, S3 in Exp-03, S2 and S5 in Exp-04. An uncontaminated red soil sample (S0) was used as control.

Soil suspensions were used for BIOLOG analysis. The soil supernatants were prepared as follows: first, 5.0 g of dried fresh soil was placed in a plastic tube, equilibrated with 45 mL of sterilized 0.85% NaCl solution, and shaken for 30 min. The soil solution was then held for 5 min. Five millilitre of the suspension was fetched, diluted in 45 mL of sterilized 0.85% NaCl solution, and added into the BIOLOG ECO test plate. One hundred and twenty-five microliter of the solution was added to each well by an eight-channel injection. After 0, 24, 48, 72, 96, 120, 168 and 240 h, the absorbance was read by a Microtiter-plate reader (Microstation 3, BIOLOG Inc., Hayward, CA) at 590 nm, respectively, and the average well color development (AWCD) was calculated using the equation of $AWCD = \Sigma(C - R)/31$,

where C and R are absorbance in each C-source well and the control, respectively [15]. After 168 h of cultivation, the utilization of various C-sources by soil microbial community was analyzed. The repeatability of the test results was within 95%.

3. Results and discussion

3.1. Electric current and electroosmotic flow

Fig. 2 shows the changes of electric current and electroosmotic flow across the soil column with time in different treatments. The electric current in Exp-01 (no pH control) was relatively low. A similar result was obtained in Exp-02, although the anolyte pH was controlled at 10. However, the current in Exp-03 increased to a high value with treatment time when both the anolyte and catholyte were pH 10 phosphate buffer solution, which is ascribed to the ions in the anolyte and catholyte diffused and electromigrated into the soil column together. The ions broke through the column, and correspondingly increased the current. The results of soil EC were also consistent with this result.

In Exp-04, the electric current was very small at the beginning of the tests. It started to gradually increase after about 200 and 100 h in Exp-03 and Exp-04, respectively. The difference of the time for the initial increase of the electric current in Exp-03 and Exp-04 is due to the presence of NaClO. The increase in the electric current in Exp-04 occurred sooner and was more pronounced than that in Exp-03, because NaClO addition in the catholyte increased its EC.

Due to the significant increase in the electric currents, the applied voltages were reduced from 20 to 10 V in Exp-03 and Exp-04 after 350 and 160 h, respectively. This was not observed in Exp-01 and Exp-02 because water applied in Exp-01 and water and pH buffer solution applied as catholyte and anolyte, respectively, in Exp-02 resulted in small quantity of ions introduced into the soil column.

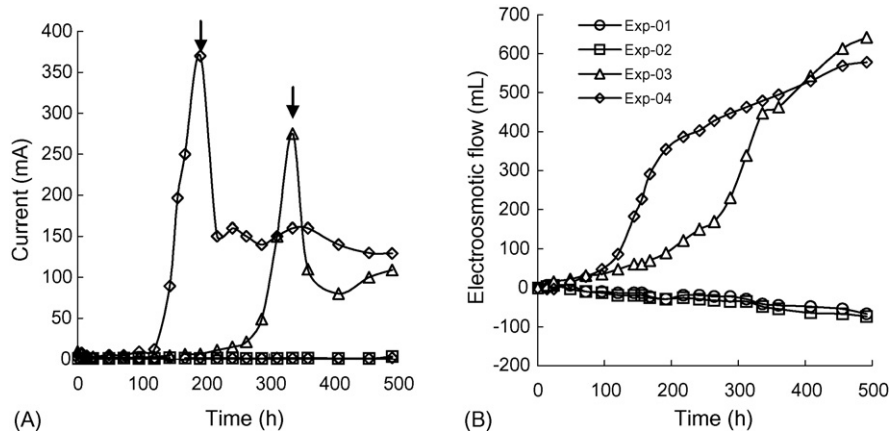


Fig. 2. Change of electric current (A) and cumulative electroosmotic flow (B) with time during electrokinetic treatment. Arrows in (A) means the change of application voltage drop from 20 to 10 V. Positive and negative values in (B) represent the electroosmotic flow towards the cathode and the anode, respectively.

Electroosmosis is affected by soil surface charge, pore liquid ionic strength, and voltage applied [1]. The electroosmotic flux from the cathode towards the anode were small in Exp-01 and Exp-02. As shown in Table 1, the red soil is a variably charged soil (PZC 3.30), and has significantly positive surface charge at low pH value. This is consistent with the previous results [16]. However, the soil surface charge was predominantly negative in Exp-03 and Exp-04 due to the high alkalinity of both the anolyte and catholyte. Accordingly significant electroosmotic flow occurred towards the cathode and the flow rate increased along with the increase of the electric current.

3.2. Soil pH and EC

Fig. 3 shows the pH and EC of the subsamples collected from different electrokinetic experiments. The soil pH varied to different extent compared with the initial conditions because of electrode reactions and pH control of the electrolytes.

In Exp-01, soil pH in the sections 5 (close to the cathode, as S5) and 1 (close to the anode, as S1) increased and decreased, respectively, compared to the initial value, which is due to the formation and transport of H^+ and OH^- ions at the anode and cathode, respectively [2,17]. In Exp-02, the anolyte pH was con-

trolled at pH 10, but soil pH in the S1 increased only slightly compared with the soil pH before treatment. It is ascribed to the high buffer capacity of the red soil for OH^- ions and only limited ions introduced into the soil column, which was affirmed by the low current in Exp-02. The soil pH close to the cathode slightly increased compared to the initial value. When pH 10 phosphate buffer solution was used as the anolyte and catholyte simultaneously (Exp-03 and Exp-04), they increased to high values, suggesting a successful pH control of the electrolytes. Compared with the pH control solution (pH 10), the soil pH after the electrokinetic treatments increased to 12, which is due to the introduction of OH^- ions from the catholyte.

Soil EC after the treatments increased compared to the initial conditions (Fig. 3B). It is ascribed to the transport of ions from electrolytes into the soil as a consequence of pH control of the electrolytes. The soil EC values in Exp-03 and Exp-04 were higher than that in Exp-01 and Exp-02. This tendency was in accordance with the change of electric current, as described above.

The EC profile of the soil subsamples in Exp-01 and Exp-02 decreased from the anode towards the cathode. However, the behaviors were different in Exp-03 and Exp-04 as the conductivity increased from the anode towards the cathode. In Exp-01

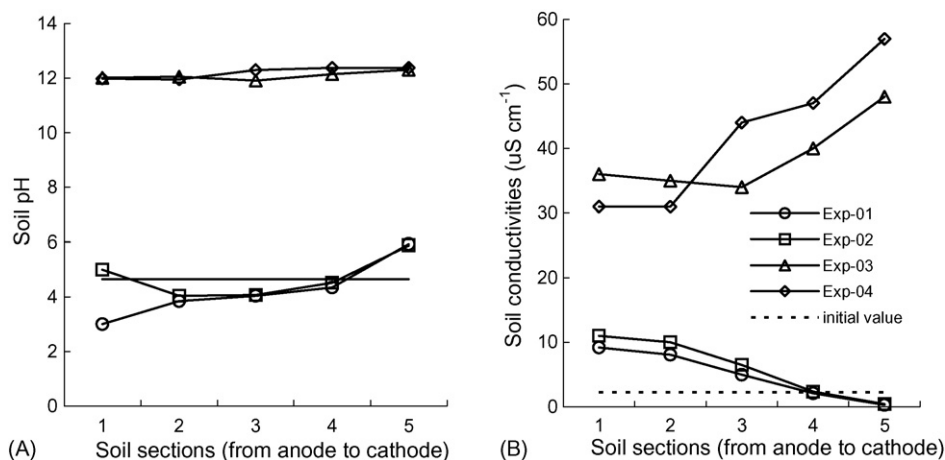


Fig. 3. Soil pH (A) and EC (B) before and after electrokinetic treatment.

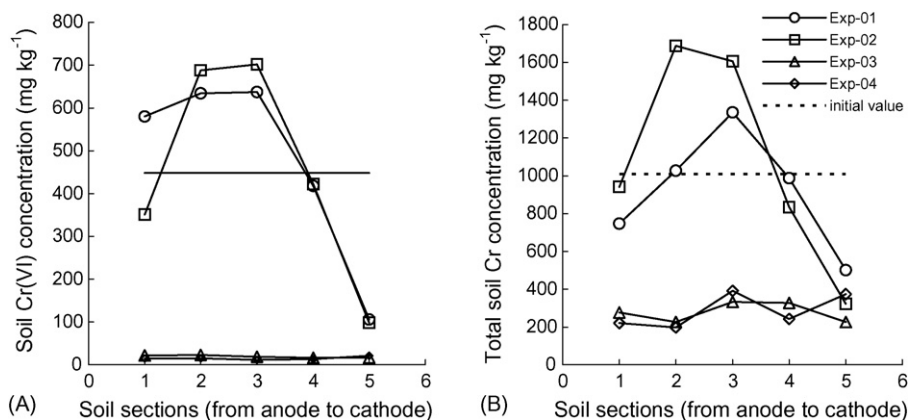


Fig. 4. Concentrations of Cr(VI) (A) and total Cr (B) in the soil subsamples after electrokinetic treatment.

and Exp-02, the OH⁻ ions migrated from the catholyte into the soil column and reacted with cations Cr³⁺, Ca²⁺, Mg²⁺ in soil pore liquid to form metal hydroxide precipitation. The soil EC close to the cathode was correspondingly reduced. However, the EC in the catholyte containing NaClO was higher than that in the anolyte in Exp-04, which resulted in higher soil EC in the sections close to cathode.

3.3. Soil Cr(VI) and total Cr

Fig. 4 shows the final concentration profiles of Cr(VI) and total Cr in the soil subsamples after electrokinetic treatments. In Exp-01 and Exp-02, the distribution of soil Cr showed accumulation and a peak in the middle sections, and the total concentration of soil Cr was low in the sections closed to the cathode. Cr(VI) exists in soil as oxyanions HCrO₄⁻, Cr₂O₇²⁻ and CrO₄²⁻, which migrate towards the anode in the electric field. Therefore, the concentrations of Cr(VI) decreased in the subsamples collected near the cathode and increased in the soil sections close to the anode in both Exp-01 and Exp-02. In Exp-03 and Exp-04, the concentrations of Cr(VI) and total Cr in the soil were significantly lower than initial values.

Table 3 shows the amounts of total soil Cr before and after the electrokinetic treatments. The result indicates that Cr mainly migrates towards the anode reservoir in the form of Cr(VI) and its concentration in the catholyte was very small. While most of the Cr remained in the soil column in Exp-01 and Exp-02, significant amount of Cr migrated and was extracted at the anode as Cr(VI) in Exp-03 and Exp-04.

The mass balance was underestimated in Exp-03 and overestimated in Exp-04. In fact, the underestimation of Cr mass

balance in Exp-03 means that the residual Cr should be higher than our determination because of the difficulty to accurately analyze the remaining Cr in soil column. It also means that there are more Cr residual in the soil column in Exp-03 than that shown in Fig. 4B. So, although the Cr results in Fig. 4 are similar between Exp-03 and Exp-04, the remediation result in Exp-04 is better than in Exp-03. The better results are due to the enhanced oxidation of soil Cr(III) to mobile Cr(VI) when NaClO was introduced into the soil column. In addition, the recovered total Cr(VI) amount in the anolyte in Exp-04 (456 mg) was higher than the initial soil Cr(VI) amount (251 mg). No such significant change occurred in Exp-03. It means that the increased Cr(VI) was due to the oxidation of Cr(III) in soil column during the electrokinetic treatments.

Since the soil was spiked by potassium dichromate, Cr(VI) reacted with the soil components, such as the soil organic matter, to form Cr(III) which precipitates and/or is highly adsorbed in the soil. The soil pH increased and a highly alkaline environment developed across the soil column as a result of the enhanced electrokinetic treatment by using phosphate buffer solution as the anolyte and catholyte both in Exp-03 and Exp-04. Fig. 3 shows that the soil pH was around 12 after the electrokinetic treatment. Under this strongly alkaline environment, Cr(III) will undergo the following reactions:



Cr(OH)₆³⁻ could be written as CrO₂⁻, which can be oxidized to Cr(VI) [14]:

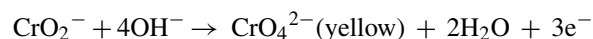


Table 3
Chromium mass balance

Treatment	Initial total Cr (mg)	Initial Cr(VI) (mg)	Cr amount after treatment				Recovery (%)
			Residual soil Cr (mg)	Removal percentage (%)	Anolyte Cr (mg)	Catholyte Cr (mg)	
Exp-01	566	251	524	7.4	31.8	0	98
Exp-02	571	254	550	3.7	59.0	0	107
Exp-03	571	254	159	72	307	0.3	82
Exp-04	566	251	161	72	456	0	109

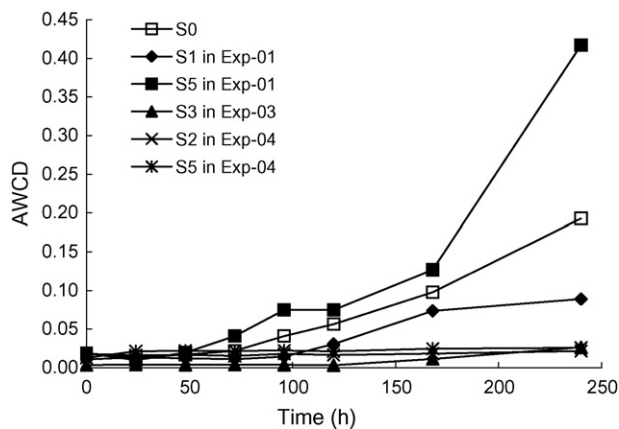
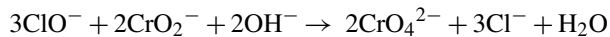


Fig. 5. Change of AWCD of soil subsamples after electrokinetic treatments with cultivation time (S0 is the control).

Under the applied electric field, CrO_4^{2-} will migrate towards the anode, which is why Cr(VI) appeared, in amounts that exceed the initial soil Cr(VI), in the anolyte in Exp-03 and Exp-04.

When NaClO was used as oxidant in the system, more amounts of Cr(III) was oxidized into Cr(VI) causing higher removal of Cr(VI) in Exp-04 than in Exp-03. The effect of hypochlorite on oxidation and extraction of Cr can be described by the following equation:



3.4. Soil microbial functional diversity

As a result of the electrokinetic treatment, changes in the soil environmental conditions, such as pH and Eh, due to electrolysis and amendments will impact the microbial function. However, there are limited studies on the potential impacts of the electrokinetic treatment on microbial culture and behavior. Recently, Lear et al. [18] investigated the effect of electrokinetic treatment on the microbial communities of an uncontaminated soil and reported that such effect was weak.

Fig. 5 shows the change of AWCD of the soil subsamples after the electrokinetic treatments with cultivation time. For the control (S0), the change of AWCD with time was greater than that of S1 in Exp-01, S3 in Exp-03, S2 and S5 in Exp-04, and the order was $\text{S0} > \text{S1 in Exp-01} > \text{S5 in Exp-04} > \text{S2 in Exp-04} > \text{S3 in Exp-03}$. However, the S5 in Exp-01 showed a significant increase with the cultivation period, especially after 168 h of cultivation. Compared with the control, fewer C-sources were utilized in S1 in Exp-01, S3 in Exp-03, S2 and S5 in Exp-04, suggesting that the activities of the microbial community in these soils were reduced. The decreased activities of soil microbial community are possibly ascribed to the big change of soil pH in these soil sections.

The microbial community of S5 in Exp-01 effectively utilized various C-sources. Although S1 and S5 in Exp-01 are soil subsamples collected from the same experiment, their AWCD values differed, which is mainly due to the difference of soil EC and soil pH. The pH values of S0 and S5 in Exp-01 were 4.8 and 5.9, respectively. S1 in Exp-01 has a low pH (3.0), and S3 in

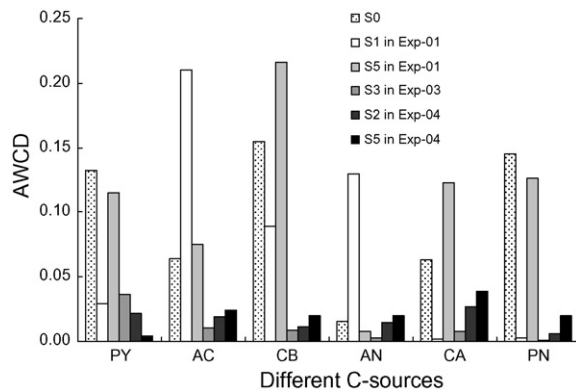


Fig. 6. Utilization of different C-sources by soil microbial community after 168 h of cultivation (PY, polymers; AC, amino acids; CB, carbohydrates; AN, amines; CA, carboxylic acids; PN, phenols).

Exp-03, S2 and S5 in Exp-04 have high pH (varied from 11.9 to 12.4). The ECs of S0, S1 and S5 in Exp-01 were in the range of $28\text{--}92 \mu\text{S cm}^{-1}$. However, the ECs of S3 in Exp-03, S2 and S5 in Exp-04 varied from 3100 to $5700 \mu\text{S cm}^{-1}$, respectively. The differences are significant. Clearly, the changes of soil microbial community are related to the soil pH and EC.

Fig. 6 shows the utilization of various C-sources by the soil microbial community after 168 h of cultivation. Soil subsamples exhibited various capacities to utilize various C-sources. For the control, the soil microbial community mainly utilized polymers, carbohydrates and phenols. For S1, the soil microbial community utilized more amino acids and amines, and less polymers, carboxylic acids and phenols. For S5, the soil microbial community utilized more polymers, carbohydrates and carboxylic acid and less amines. However, the utilization of C-sources by soil microbial community in S3 in Exp-03, S2 in Exp-04, and S5 in Exp-04 were low, especially of polymers, carbohydrates and phenols. These results indicate the treatments Exp-03 and Exp-04 had adverse effect on the soil microbial function. It suggests that electrokinetic treatment had a negative impact on the microbial although contaminants were effectively reduced.

4. Conclusions

The enhanced electrokinetic treatment of the chromium-contaminated red soil is successful for the removal of soil Cr, especially soil Cr(VI), but the functional diversity of soil microbial community decreased when high pH buffer solution and NaClO were applied. Therefore, new enhancing conditions which can not only improve soil Cr removal but also restore the function of Cr contaminated soil simultaneously will be necessary for the practical application of this technology.

Acknowledgements

This research was supported by National Natural Science Foundation of China (20207010) and State Key Laboratory of Soil and Sustainable Agriculture, Institute of Soil Science, Chinese Academy of Sciences. Dr. Manqiang Liu and Miss

Xiaoyun Chen from College of Resources and Environmental Sciences, Nanjing Agricultural University are appreciated for the BIOLOG data analysis.

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