Preliminary Study on Treatment of Soil Enriched in Chromite Ore Processing Residue by Electrokinetics

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Abstract: The effectiveness of the electrokinetic (EK) process in treating soil enriched in chromite ore processing residue (Cr–soil) was investigated. An electric gradient of 1.2 V/cm was applied to induce the movement of electrolyte solution in the soil cell for 17–43 days. Simulated rainwater was used as EK processing fluid. Results show low removal efficiency of total Cr (2–7%) because the insoluble form of chromite dominates in the Cr–soil. However, more than 95% of leachable Cr(VI) can be removed by controlling the pH of the processing fluid at 9.0 during experiments. The removal of Cr from Cr–soil was characterized by the high Cr(VI) concentration collected at the anode, and the presence of Cr precipitates collected at cathode. The electric energy requirement under all experimental conditions varied from 1.3 to 5.8 kW h/m^3. Results indicated that the EK is an effective process in mitigating the leaching of Cr(VI) from Cr–soil.

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Introduction

The production of chromate in Hudson County, N.J. created approximately 2–3 million tons of chromite ore processing residue (COPR) during the first half of the century [Montclair Environmental Management Team (MEMT) 1970]. The residue has been used as fill material for highway constructions, parks, schools, industrial and factory sites, and even residential areas; thereby causing soil and groundwater contamination. More than 400 chromium-contaminated sites have been identified in the surrounding region. Researchers have reported that a considerable amount of soluble Cr(VI) was reported to leach from the chromium contaminated soil derived from this residue (Weng et al. 1994, 2001, 2002; James 1996) posing a pollution problem. The occurrence of aqueous Cr(III) in New Jersey Cr–soil is due to the acidification of COPR by acid precipitation that is commonly found around the Mid-Atlantic region (Weng et al. 2002). Among the common forms of chromium found in natural systems, Cr(VI) is the most toxic (Holdway 1988). Remediation of these sites is necessary to reduce public health risk. In the U.S., the current action level of cleanup criteria under the Resource Conservation and Recovery Act corrective action for total Cr is 1,000 mg/kg.

Electrokinetics (EK) involves the application of an electrical field across a porous medium to induce the movement of electrolytic solution and soluble contaminants toward the electrodes. The major contaminant removal mechanisms involved in this process are the advection of electroosmosis (EO) flow driven under an electrical field, the movement of H$^+$ ions generated from H$_2$O electrolysis at the anode advancing through soil toward the cathode, and the migration of charged ions toward the opposite electrodes (Alshawabkeh et al. 1999). This process has been demonstrated to be successful and cost effective in removing both organics and heavy metals from soils with low hydraulic conductivity (Acar and Alshawabkeh 1996; Alshawabkeh et al. 1999; Ho 1999; Weng et al. 2000, 2003; Weng and Yuan 2001).

Several studies have focused on EK remediation of chromium contaminated soils, which are summarized as follows. Haran et al. (1996) have conducted experiments on saturated sand cells loaded with potassium chromate close to the cathode. In their study, graphite electrodes have been used as the inert cathode while pure iron is used as the anode. Results have shown that the hexavalent chromium ions can migrate towards the anode and are reduced to trivalent form by chemical reaction with the anodic dissolution product ferrous iron. Bala et al. (1997) have developed a mathematical model to simulate the ionic migration of Cr(VI) in the EK process. Results of simulation have shown that their model can reasonably predict the chromium concentration profiles in the sand cell after 28 days of electrolysis.

Realizing the importance of soil composition in the electrokinetics remediation, Reddy et al. (1997) have studied the chromium-contaminated glacial till, kaolin, and Na–montmorillonite. The test results have indicated that when soils contain high carbonate buffers, i.e., glacial till, it can retard the development of an acid front, which has resulted in alkaline soil pH throughout the test. When soil had low buffering capacity, such as kaolin and Na–montmorillonite, it favored the development of acid front that results in low pH near the anode and high pH near the cathode. They reported that moderated Cr(VI) adsorption in acidic regions in kaolin resulted in lower Cr(VI) removal than in the glacial till, while high Cr(VI) adsorption in acidic regions of Na–montmorillonite resulted in low Cr(VI) migration. They have also pointed out that the effect of hematite or iron oxide in soils on the removal of Cr(VI) by electrokinetics depends on the mineralogical composition. In soil such as glacial

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till, the presence of iron oxide created complex geochemistry and hindered Cr(VI) removal. However, in homogeneous clays such as kaolin and Na−montmorillonite, the presence of iron oxide did not significantly affect Cr(VI) removal.

Recently, Reddy et al. (2001a) have reported synergistic effect of multiple metal contaminants on the electrokinetic remediation. They claimed that Cr(VI) migration was retarded in the presence of cationic metals, such as Ni and Cd, in both kaolin and glacial till owing to the opposite migration of these contaminants. In order to develop electrode-conditioning strategies on a rational basis in the electrokinetic remediation process, Reddy et al. (2001b) have further studied the forms and distribution of the residual contaminants in soils after electrokinetic treatment. Results of sequential extractions showed that the speciation of contaminants in soils changed significantly in the exchanged and soluble fractions. They have also indicated that the exchangeable Cr(III) and soluble Cr(III) in kaolin decrease near the anode and increase near the cathode, whereas exchangeable and soluble Cr(VI) increase near the anode and decrease near the cathode.

In a study of removal of artificially Cr(III) contaminated clayey soil (1,950 mg/kg) by electrokinetics, Weng and Yuan (2001) have reported that about 30–40% of total Cr(III) is removed using ethylenediaminetetraacetic acid, citric acid, and sodium dodecylsulfate as processing fluids and around 20% for tap water. They have pointed out that the EK treatment using citric acid processing fluid is the most promising method studied.

Albeit extensive EK process has been studied over the last 2 decades, very limited data are available to describe the EK phenomenon in the treating of soil enriched in COPR (Cr–soil). In this study, we investigated the effectiveness of the EK process in treating Cr–soil to minimize the leaching of Cr. Effects of processing time and with/without the controlled pH of processing fluid on the mobility of Cr were studied. After EK treatment, the leaching of Cr(VI) in this Cr-containing soil was evaluated using simulated rainwater as leaching solution. The results of this study will provide useful information for the establishment of effective remediation plans for COPR contaminated sites.

Materials and Methods

Cr−Soil Sample

Top soil sample enriched in COPR was collected from Liberty State Park, Hudson County, N.J. In order to provide a uniform size distribution, the soil aggregates were broken by a wooden mallet or by hand. After it was air dried, the soil was sieved to less than 1 mm in diameter. An average particle size of 250 μm was obtained at d_{60} by American Society of Testing Materials (ASTM) standard sieve analysis. This sieved soil sample was then used in all experiments. Table 1 lists selected properties of the Cr–soil sample. A soil pH of 8.2 was measured in 10^{-2} M CaCl$_2$ solution at a 1:1 soil to solution volume ratio with a pH meter (Model 3500 digital pH meter, Beckman, Irvine, Calif.). The procedures for the determination of soil pH followed ASTM method D4972-89. A pH$_{zpc}$ (zero point of charge) of 6.8 for Cr–soil particles was determined by a zeta-meter (Laser Zee Model 500, Pen Kem Inc., Bedford Hills, N.Y.). The specific surface area of Cr–soil sample was 22 m^2/g, determined by the BET−N$_2$ gas sorption method using a Quantasorb surface analyzer Model QS-7 (Quantachrom Co., Greenvale, N.Y.).

Table 1. Selected Properties of Soil Enriched in Chromite Ore Processing Residue

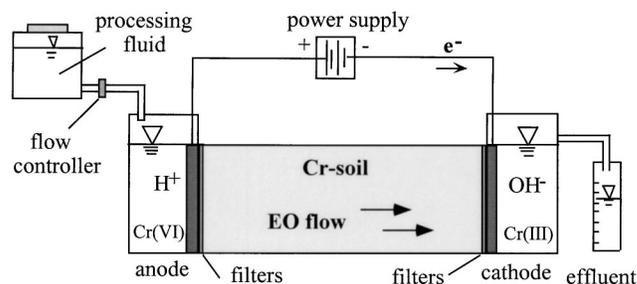| Parameters	Values
Total Cr (mg/g)	25.3
Leachable Cr(VI) (mg/g)[a]	0.23
Soil pH	8.2
Porosity	0.3
Bulk density (g/cm^3)	1.85
Average particle size (μm)	250
Specific surface area (mg^2/g)	22.0
pH$_{zpc}$	6.8

[a]Determined by a 2 day batch of experiments with simulated rainwater.

Analytical Methods

Cr(VI) in the solution was analyzed by reaction with diphenylcarbohydrazide and then the absorbance of the purple product was measured at a wavelength of 540 nm [United States Environmental Protection Agency (USEPA) 1992]. Total Cr was determined by oxidizing the Cr(III) to Cr(VI) with potassium permanganate (Huang and Bowers 1978). The amount of Cr(III) was determined by the difference between the concentration of total Cr and Cr(VI). The total chromium concentration in the soil was analyzed following acid digestion. The soil sample was predigested in 250 mL of concentrated HNO$_3$ and water mixture at a volume ratio of 1:1 for 1 h at 60−80°C. This pretreatment was followed by the addition of a 25 mL solution of concentrated H$_2$SO$_4$, HClO$_4$, and HF at a volume ratio of 1:2:5 for 6 h.

Electrokinetic Experiments

The EK experiments were conducted using acrylic extruded cylindrical cells (6.8 cm ϕ) consisting of three compartments: anode reservoir (4 cm length), soil cell (10 cm length), and cathode reservoir (4 cm length) (Fig. 1). The volume of each reservoir was 140 cm^3. To separate the soil from the water solution, a set of two nylon meshes (149 μm polypropylene filter, Spectrum, Houston) with a paper filter inbetween (Whatman No. 1 filter, Maidstone, England) was used both in the anode and cathode reservoirs. Two sets of graphite rod electrodes, one set at the anode and the second at the cathode, were installed on each side of the soil specimen right behind the filters. After assembling the cell and filling it with the air-drying Cr–soil (655 g), simulated rainwater was introduced into both anode and cathode reservoirs as the processing fluid. The electrodes were then connected to a direct current power supply (12 V) and generated a constant potential voltage gradient of 1.2 V/cm.

Fig. 1. Schematic of electrokinetic remediation process

Fig. 2. Recipe for preparation of simulated rainwater

Fig. 3. Current density as function of time

Preparation of simulated rainwater of the Mid-Atlantic coastal region by following the standard reference methods of the National Bureau of Standards (Koch 1986), is shown in Fig. 2. The pH value of this simulated rainwater prepared is 4.3. Three EK experiments were performed and the operating conditions are listed in Table 2. Except for Test 1, the solution in the reservoir was pumped dry and replaced with new processing fluid at the end of each day's operation. In Test 3, the processing fluid was maintained at pH 9.0 ± 0.1 by adding 1 M NaOH in the anode reservoir and 1 M HCl in the cathode reservoir with a pH controller (New Brunswick Sci., Edison, N.J.). A stirring bar was placed in the bottom of reservoirs to provide uniform mixing. During the EK process, parameters such as current, the amount of effluent in the cathode reservoir, pH, and Cr (both Cr(VI) and Cr(III)) in the reservoirs were monitored as a function of time. At the end of the experiment, the soil sample was removed from the cell and sliced into eight or ten sections. Each section was analyzed for soil pH, total residual Cr concentration, the amount of Cr(VI) leached with simulated rainwater in the leaching experiments as described in the next section.

Leaching Experiment

Batch experiments were designed to evaluate the effectiveness of EK treatment in terms of the amount of soluble Cr(VI) leached from Cr–soil samples. The experiments were conducted by shaking a plastic bottle containing 1 g of sliced-soil sample and 100 mL of simulated rainwater on a reciprocating shaker (Eberbach Co., Ann Arbor, Mich.) at 150 cycles per min for 2 days. The equilibrium pH was then recorded and aliquots of the suspensions were taken and filtered with 0.45 μm membrane filter (Supor-450, 25 mm, Gelman Sciences Co., Ann Arbor, Mich.). The concentration of the Cr(VI) and Cr(III) in the supernatant was determined. A blank of Cr–soil sample without EK treatment was also performed.

Results and Discussion

Variation of Current Density

The current density as a function of processing time is depicted in Fig. 3. The current density sharply decreased from 0.085 mA/cm^2 at the beginning of the test to 0.03 mA/cm^2 for Test 1 and 0.02
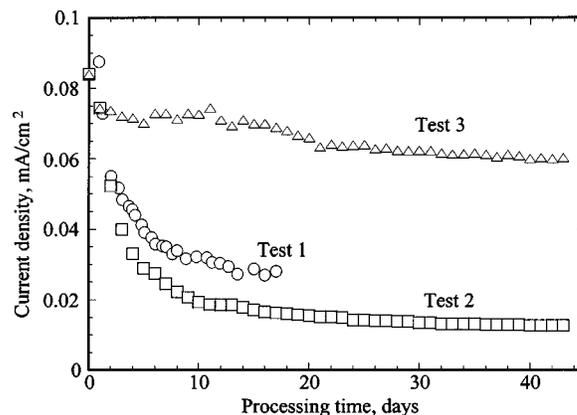
mA/cm^2 for Test 2 in the first 10 days of experiment. With control processing fluid pH at 9.0 in Test 3, the current density decreased from 0.085 to 0.070 mA/cm^2 in the first 2 days, and then continued to decay smoothly to a value of 0.065 mA/cm^2 as the process elapsed. In general, the current density decreases with increasing processing time, then ultimately approaches a steady state. The decrease of current density with time reflects an increasing of electric resistance. The primary reason for the increase of resistance is the formation of metal hydroxide precipitation, such as $Cr(OH)_3(s)$ and $Ca(OH)_2(s)$, on the cathode side, which consequently clogs the soil pores. The drawback of this resistance buildup in the system is that it lowers the EO permeability and eventually retards the transport of Cr ions from the anode toward the cathode end.
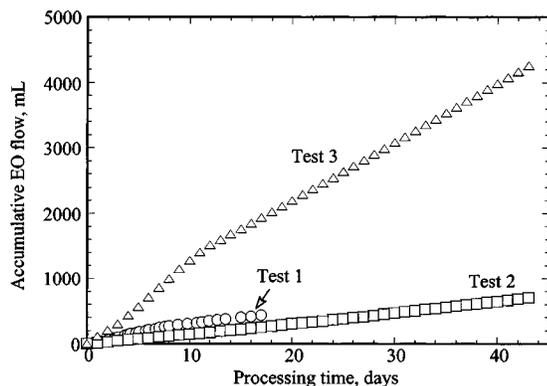
Electroosmotic Permeability

As expected, the direction of EO flow was from the anode to the cathode for all tests. The accumulative volume of effluent (cathode side) versus time is plotted in Fig. 4. The flow rates, given by the slopes of the curves, were approximately 25, 18, and 100 mL/day, respectively, for Tests 1, 2, and 3. The processing fluid with controlled pH in Test 3 presented a higher flow rate than the one without a controlled pH. The higher flow rate observed in Test 3 can be related to the zeta potential of the soil particles. The electroosmotic flow rate, Q_e (mL/day) for a cylindrical soil core of length L and cross-section area of A under a potential difference of V (V), can be described by (Casagrande 1949)

$$Q_e = \frac{\zeta \varepsilon q}{4\eta} \frac{V}{L} A \qquad (1)$$

where ζ, η, ε, and q = zeta potential, viscosity of the fluid, dielectric constant of the fluid, and empirical constant related to the soil physical properties, respectively. Apparently, the EO flow rate is directly proportional to the applied electrical potential gradient,

Table 2. Operating Conditions of Electrokinetic Process and Their Results

Test	Electric gradient (V/cm)	Reservoir pH control	Processing time (day)	Electroosmois permeability $K_e \times 10^{-6}$ (cm^2/V s)	Cr removed (%)	Power expenditure (kW h/m^3)
1	1.2	No	17	6.8	1.9	1.3
2	1.2	No	43	4.4	5.3	1.8
3	1.2	Yes, pH 9.0 ± 0.1	43	26.3	7.1	5.8

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Fig. 4. Accumulative electroosmosis flow in cathode reservoir as function of time

Fig. 6. Soil pH profiles across soil specimen after electrokinetic treatment

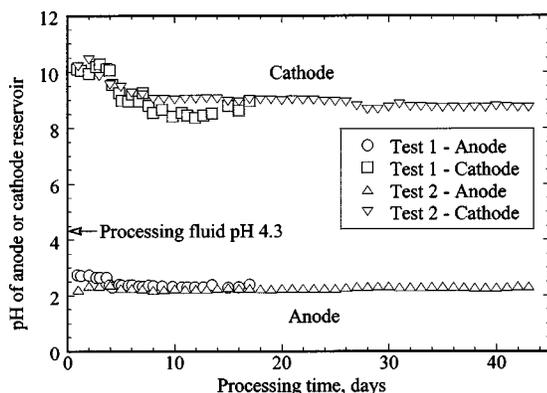zeta potential, and cross-section area of the soil core. In Test 3, the soil pH ($\cong 9.0$) was greater than those tests without pH controlled (average pH$\cong 7.8$), thus a higher zeta potential was expected (pH$_{zpc}$ 6.8) resulting in a higher EO flow rate. Eq. (1) can be rewritten in the form similar to the Darcy's law

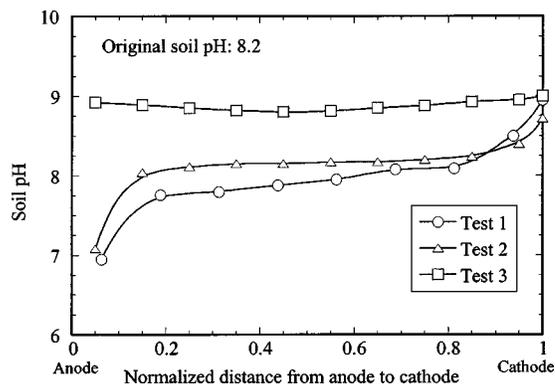$$Q_e = k_e \cdot i_e \cdot A \qquad (2)$$

where k_e and i_e are defined as the EO permeability and the potential gradient, respectively. With a potential gradient of 1.2 V/cm and cross-section area of 36.3 cm^2, the average k_e values according to Eq. (2) were 6.8×10^{-6}, 4.4×10^{-6}, and 2.63×10^{-5} cm^2/V s, respectively, for Tests 1, 2, and 3 (Table 2). The magnitude of K_e is related to the resistance buildup during EK treatment. As described previously, a higher current density exhibited in Test 3 indicated that a rather low resistance occurred in the EK system and, as such, it resulted in a higher k_e value than those of Tests 1 and 2. These obtained K_e values are in good agreement with the literature data of $1.0 \times 10^{-7} - 2 \times 10^{-5}$ cm^2/V s for all soils (Weng et al. 2000, 2001).

Variation of Reservoir pH

The pHs measured in the reservoirs at various time periods are given in Fig. 5. Fig. 5 shows that the pH of anode fluid decreased rapidly from 4.3 to about 2.2. The pH of cathode fluid initially


Fig. 5. Reservoir pH as function of time

increased drastically to 10.0 within the first 4 days, then it remained at around 9.0 for the entire EK process period. The buildup of such strong pH gradient is attributed to the electrolysis of H$_2$O which occurs at the electrodes when an electrical field is applied. The primary electrolysis reactions are
Anode:

$$2H_2O \rightarrow O_{2(g)} + 4H^+ + 4e^- \qquad (3)$$

Cathode:

$$2H_2O + 2e^- \rightarrow H_{2(g)} + 2OH^- \qquad (4)$$
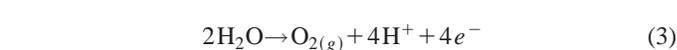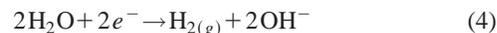
The oxidation of water at the anode produces H$^+$ [Eq. (3)], while the reduction at the cathode generates OH$^-$ [Eq. (4)]. The occurrence of these reactions in the reservoirs can not only affect the operating fluid condition, but can also cause chemical changes in the soil system during electrokinetics. The movement of H$^+$ and OH$^-$ under an applied electric field producing acid and basic fronts in time will change the soil pH drastically during the EK process.

Soil pH Profiles

Fig. 6 shows the soil pH profiles across the soil core as a function of normalized distance from anode for all tests. Since the reservoir pHs were controlled at pH 9.0 for Test 3, after a 43 day EK treatment, the soil pH remained a constant pH value of 9.0. However, in Tests 1 and 2, the soil pHs in the soil core were influenced by the electrolysis reaction that took place when a constant voltage was applied to the system. During the EK process, the acid front generated at the anode advanced towards the cathode. This acid front was neutralized by alkalinity of the soil and the OH$^-$ production at the cathode, which migrated into the soil. In our experiment, the Cr–soil is an alkaline material containing a large amount of Ca–mineral (Weng et al. 1994). Because acidity of the fluid front was buffered, the soil pH along the soil core did not decrease substantially during a 17 day (Test 1) or 43 day (Test 2) EK treatment (Fig. 6). As shown in Fig. 6 the soil pH profiles are close to the original soil pH (8.2) for Tests 1 and 2, except for the soil near the anode and cathode. At the anode side, the soil pH decreased slightly to around 7.0 owing to the movement of acid front and the diffusion of H$^+$ in the vicinity of anode. At the cathode side, the migration of OH$^-$ advanced toward the anode and the OH$^-$ ion concentration differences between the reservoir and soil would make the soil pH hardly decrease even though the H$^+$ ion was continuously swept into this region. Fig. 6 indicates
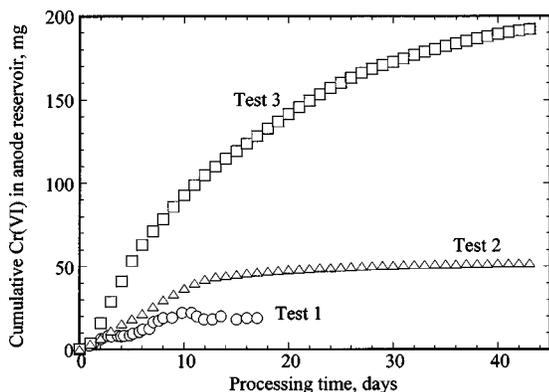
Fig. 7. Cumulative Cr(VI) in anode reservoir as function of time

Fig. 8. Leachable Cr(VI) remaining profiles across soil specimen

that the soil pH near the cathode side was about 9.0 for Tests 1 and 2. Apparently, the movement of H^+ and OH^- under an applied electric field producing acid and basic fronts in time will change the soil pH during the EK process.

Cumulative Cr in Reservoirs

Fig. 7 shows the cumulative mass of Cr(VI) detected in the anode reservoir solution as a function of time. The chromate species occurring in the anode reservoir are expected to be $HCrO_4^-$ and CrO_4^{2-}. The mass of Cr(VI) collected in the anode reservoir was 23, 198, and 52 mg, respectively, for Tests 1, 2, and 3. It was expected that the soluble anionic Cr(VI) would travel toward the anode, while cationic Cr(III) would migrate to the cathode during the EK process. Such phenomena of Cr(VI) migration toward the anode was also reported in other studies (Haran et al. 1996; Bala et al. 1997; Reddy et al. 1997, 2001a,b). Since a basic medium is generated at the cathode, the Cr(III) species will precipitate as hydroxides in the cathode reservoir when the concentration reaches the $Cr(OH)_3(s)$ solubility limit. A high concentration of Cr(VI) was collected at the anode reservoir while low concentration of soluble Cr(III) was observed at the cathode reservoir for all tests (Table 3). Approximately 2, 5, and 7% of total chromium in Cr–soil (25.3 mg/g) was removed in Tests 1, 2, and 3, respectively
(Table 2). The low total chromium removal that can be attributed to the major chromium form in the Cr–soil is chromite, $FeO\cdot Cr_2O_3$ (Weng et al. 1994) which is known to be acid resistant. Thus, the majority of the total Cr in the COPR sample can be considered a nonleachable Cr species that is in the trivalent state.

The Cr removal mechanisms during the EK process are mainly attributed to the ion migration caused by the electrical gradient and the pore fluid convection initiated by the EO flow. Results from Test 2 showed that the largest amount of Cr(VI) was col-
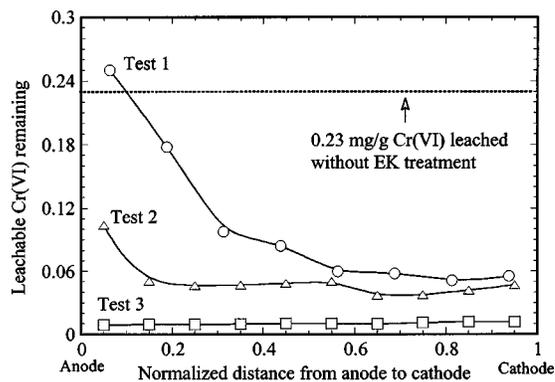
lected in the anode reservoir. This indicated that the convection term was not significant and the ion migration term prevailed in the transport contribution. Among the tests, Test 3 exhibited the largest total chromium removal (7.1%) during the EK process. As shown in Fig. 2, Test 3 had a higher flow rate among these three tests and produced the highest Cr removal across the soil sample. A possible explanation is that the convection and ion migration were both prominent in Test 3. Since the pH was controlled at 9.0 which is not favorable for anionic Cr(VI) adsorption onto soil, the transport of Cr(VI) was not retarded by adsorption. Because a high EO flow rate was observed in Test 3 (100 mL/day), it is possible that some Cr(VI) ions were swept by the flow toward the cathode. Cr(VI) species present in the catholyte can be reduced to Cr(III) by a secondary electrolysis reaction. As shown in Table 3, the amounts of soluble Cr(III) collected in the cathode reservoir were small (<1 mg for Tests 1 and 2 and 2 mg for Test 3). The low amount of soluble Cr(III) present in the cathode was due to the hydroxide precipitation at high pH. Most of chromium removed at the cathode was in the form of Cr(III) precipitates.

Cr Removal

The amounts of Cr(VI) leached with simulated rainwater was determined after a 2 day batch leaching experiment. For the Cr–soil without EK treatment, an amount of 0.23 mg/g of soluble Cr(VI) was leached. Fig. 8 shows the profiles of leachable Cr(VI) remaining after EK treatment. With EK treatment, Test 1 achieved more than 70% removal of leachable Cr(VI) in the region between the mid section and the cathode. An average 80% of leachable Cr(VI) was removed from Cr–soil in Test 2. In Tests 1 and 2, the amounts of Cr(VI) leached near the anode region are high which resulted from the migration of Cr(VI) toward the anode and the favorable adsorption of Cr(VI) onto soil particles under the acidic pH condition. By controlling the processing fluid pH at

Table 3. Mass Balance of Cr Removed in Electrokinetic Experiments

Test	Soil core $[total\ Cr]_{removed}$ (mg)	Anode reservoir $[Cr(VI)]_{collected}$ (mg)	Cathode reservoir	
			$[Cr(III)]_{collected}$ (mg)	$[Cr(III)_{precipitate}]^a$ (mg)
1	320	23	<1	296
2	870	198	<1	671
3	1,180	52	2	1,126

[a]Calculated from $[Cr(III)]_{precipitate} = [total\ Cr]_{removed} - [Cr(VI)]_{collected} - [Cr(III)]_{collected}$.

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9.0 in Test 3, a uniform removal of leachable Cr(VI) throughout the specimen was observed and more soluble Cr was removed than the processes without pH control. As indicated in Fig. 8, more than 95% of leachable Cr(VI) was removed through this process. Obviously, the EK process is effective in reducing Cr(VI) leaching from Cr–soil in situ.

Energy Requirement

Energy expenditure per unit volume of soil processed, *E* (watt-hour/meter³) is given as

$$E = \frac{P}{M} \int VI \, dt \qquad (5)$$

where P = energy expenditure (W h); M = volume of soil (m³); V = voltage (V); I = current (A); and t = time (h). For a constant-voltage EK system, the energy expenditure is directly related to the time integral of the current across the cell. As shown in Table 2, the calculated energy expenditure upon the termination of EK experiments is in the range of 1.3–5.8 kW h/m³. By comparing the energy requirements of Test 2 (1.8 kW h/m³) with Test 3 (5.8 kW h/m³), it reveals that more electric energy is needed for the EK system with controlling reservoir pH at 9.0.

Other costs including those for the electrode materials and installing, power control system, the fluid handling system, and maintenance and operation need to be considered in the scale up cost analysis.

Conclusions

Results show that the total Cr removal efficiency on COPR was very low (2–7%) in all tests because of the insoluble form of chromite dominates in the Cr–soil. Leaching of chromium with simulated rainwater can be drastically reduced after EK treatment. More than 95% of leachable Cr(VI) was removed by controlling the pH of the processing fluid at 9.0 during experiments. The removal of Cr from Cr–soil was characterized by the high concentration of Cr(VI) collected at the anode and by Cr precipitates collected at the cathode. The electric energy expenditure for all experiments ranged from 1.3 to 5.8 kW h/m³, showing that electrokinetics is a cost-effective technology in removing soluble Cr(VI) from Cr–soil. Accordingly, it is suggested that the EK process may be applied at selected sites to minimize the leaching of Cr(VI) with alkaline processing fluid. Remediation efforts may be selected only for those chromium contaminated sites with persistently high levels of Cr(VI) leached, of which they exceed the state cleanup standard or exhibit a high toxic risk to the environment (see Table 3).

In terms of total Cr removal, the EK performance can be further improved by controlling variable operational conditions such as switching electrical polarity, prolonging the processing time, conditioning the processing fluid (such as using citric acid), and increasing the applied voltage. Future studies are needed to monitor the reservoir's ORP in the course of EK testing to obtain information pertaining Cr species in the reservoirs. The amounts of total Cr, Cr(III), and Cr(VI) in soil before and after treatment also need to be investigated further. Large lab-scale, field pilot-scale studies, and the scale up cost analysis are also recommended in future investigations.

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