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Manganese Removal from Spiked Kaolinitic Soil and Sludge by Electromigration

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ABSTRACT

In this paper the feasibility of Mn removal from soils by electromigration is presented. Several experiments were conducted with two kind of samples (saturated and unsaturated) with the aim of understanding the behavior of Mn in a porous medium within an electric field. The unsaturated samples were prepared with properties similar to sludges, and the saturated samples had characteristics similar to a mine soil. An electric field with a constant potential difference of 15 or 30 V DC was applied across the electrokinetic cell (100 mm length, 32 mm diameter), with the sludge sample containing 3 to 5 g Mn/kg dry weight. The system was monitored for the voltage drop, current intensity, electroosmotic flow of liquid through the sample, and the pH of the electrolyte solutions. The test duration ranged from 174 to 218 hours. Metal removal in the unsaturated samples was about 78%, i.e., the Mn removed to the electrolyte so-

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lutions. However, in the saturated soils most of the Mn removed (74%) accumulated in a plain brown layer in the soil, very near the cathode, as $Mn(OH)_2$.

Key Words. Manganese; Electrokinetic; Remediation; Kaolinite; Electromigration

INTRODUCTION

The electrokinetic process is an emerging technology for "in-situ" soil decontamination in which the chemical species, both ionic and nonionic, are transported through the soil to an electrode site in the soil. These products may subsequently be removed from the electrode site via collection systems engineered for each specific application. The electrokinetics refers to the movement of water, ions, and charged particles relative to one another under the action of an applied direct current electric field. In a porous compact matrix of surface-charged particles such as soil, the ion-containing pore fluid may be forced to flow to collection sites under the applied field.

Two mechanisms for the movement of the contaminants are predicted. For constituents of an ionic nature, the transport takes place mainly by ion migration. For nonionic species, the mobilization is by electroosmotic flow. The extraction of contaminants by electrokinetic methods is based on the assumption that the contaminant is in the liquid phase in the soil pores. The benefits of increased proton concentration at the anode due to electrolysis of water are evident in the extraction of metals. The pH contributes to the removal process in two ways: it affects the surface charge characteristics of clay soils (the cation retention capacity of the clay decreases in a low pH environment) and creates suitable environments for metal ions to remain in solution in the bulk pore liquid so that they can be extracted in a feasible manner. In this process the soil pH and metal type were found to influence the removal rather than the electroosmotic flow. Furthermore, electroosmosis does not appear to be the dominant mechanism of the electrokinetic extraction of contaminants when they are present at high concentration (1).

The results of Pamukcu and of other investigators demonstrate that electrokinetic treatment may be a viable solution for a variety of soil contamination types including heavy metals, radionuclotides, organic compounds, and mixed wastes (2).

This work deals with a study of Mn recovery by electromigration from an open cast lignite mine in As Pontes de García Rodríguez (Galicia, Spain), where ENDESA, the most important electric company in Spain, has a 1400 MW installation which burns domestic lignite and imported subbituminous coal.

The uncontrolled release of acid mine drainage (AMD) is perhaps the most serious impact mining exerts on the environment. In addition to low pH and high acidity, acid mine drainage from metal mines often contains dissolved heavy metals in concentrations toxic for living organisms (3).

Most of the Mn in AMD comes from the mine dump where the Mn concentration ranges from 60 to 7000 mg/kg. The concentration is largely dependent on the technique used to build up the dump. Streams from the mine and from the dump pass through a wastewater treatment plant where most of the metals are precipitated, thereby preventing pollution of Eume River. The removal of Mn from the wastewater is quite difficult, which makes it necessary to operate both the hydraulic capacity and chemical addition at extreme conditions (4).

The objective of this work is the assessment of electromigration as a useful technique to remove Mn from soils and sediments. The evolution of pH, electric field, and Mn distribution along the cell was obtained.

MATERIALS AND METHODS

Electrokinetic Cell

The experiments were conducted in a electrokinetic cell based on the model designed by Pamukcu et al. (5) in Lehigh University, USA. A schematic diagram of the experimental setup is shown in Fig. 1: Cylindrical samples of 100 mm length and 32 mm diameter are placed between two porous stones and then an electric field is applied across two electrodes, anode and cathode, located in the electrode chambers. Three auxiliary electrodes allow the field along the different sections of the sample to be measured. The gas produced

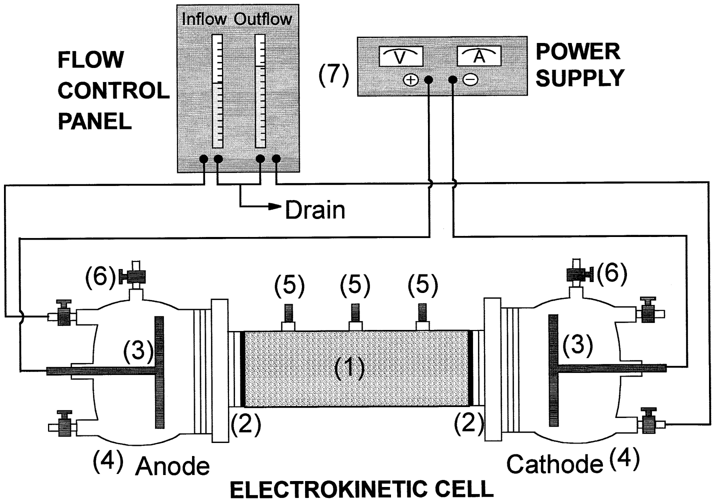

FIG. 1 Schematic diagram of experimental setup: (1) sludge sample, (2) porous stones, (3) electrodes, (4) electrode chambers, (5) auxiliary electrodes, (6) gas valves (7) electroosmotic flow control panel and power supply.

by the electrode reactions is purged from the electrode chambers through a manual valve. The electroosmotic liquid flow through the sample is measured by connecting two burettes to the electrode chambers, which also serve to maintain each end of the test cell at atmospheric pressure.

Consolidometer

A diagram of the compression unit used to create the soil matrixes is shown in Fig. 2. The consolidometer is an apparatus capable of applying small increments of pneumatic stress on the soil slurry poured into the tube from the electrokinetic cell, while allowing the excess fluid of the sample to drain out slowly.

Analytical Methods

The test protocols for chemical extraction and analysis procedures were performed in accordance with EPA Method 3010 (Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FAA and ICP Spectroscopy) and Method 3050 (Acid Digestion of Sediments, Sludges, and Soils). Flame atomic adsorption spectroscopy (AA) was used to analyze Mn (6). In addition, the solid sections from saturated samples were analyzed by x-ray fluorescence.

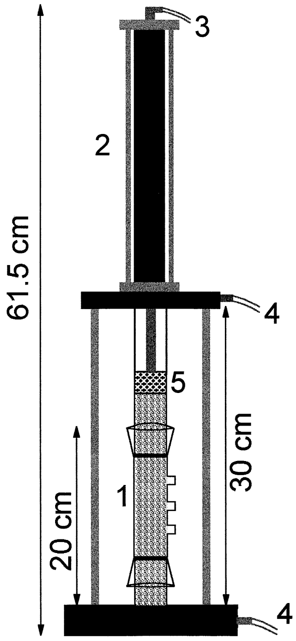

FIG. 2 Consolidometer apparatus: (1) sample tube, (2) pneumatic cylinder, (3) air pressure inlet, (4) drain, and (5) piston.

The soil pH_{H_20} was measured by mixing distilled water and dry soil in a 2.5 mL/g ratio. The pH was measured after 1 hour of contact.

Sample Preparation

Samples of Mn-spiked soil and sludge were prepared by adding a concentrated solution of $MnCl_2$ to kaolinitic clay in a 750 mL/kg ratio. The mixture stood for 24 hours to allow the sorption of Mn to kaolinite, and a slurry of 5 g Mn/kg dry soil was obtained. Kaolinite was selected since it shows a much lower buffering capacity because of its lower cation-exchange capacity compared with other clay minerals (1). Kaolinite P-30 has an average particle size of 3 μm and a specific surface of 13.5 m^2/g. Mineralogy analysis by x-ray diffraction indicated the presence of 85% kaolinite clay, 14% mica, and 1% quartz.

Two kinds of samples were prepared from the slurry: 1) a saturated clay sample, and 2) an unsaturated clay sample with a higher water content.

To obtain the saturated sample the slurry was compacted to a constant density and pressure in the consolidometer unit, which creates homogeneous, near-saturated soil matrixes. The most beneficial aspect of these custom-manufactured units was that at the end of the consolidation process the soil sample could be installed directly in the electrokinetic cell (5). Therefore, handling, such as trimming and placement, did not disturb the soil sample. The applied pressure during the consolidation process was 290 kPa. This resulted in a cylinder of 0.583 g H_2O/g dry weight, with a Mn content of 3.8 g/kg dry weight.

The unsaturated sample was obtained directly from the prepared slurry by adding 33 mL of a 65% HNO_3 solution to ensure all the Mn was in solution. The final sample had a humidity of 1 kg water/kg dry soil, a Mn content of 5 g/kg dry weight, 5460 g/L in pore water, and pH 2.5.

Methodology

Readings of voltage (V) between the anode, the auxiliary electrodes, and the cathode were taken hourly during the tests. Other measurements included the current intensity (mA), electroosmotic flow of liquid through the sludge, and the pH in the electrode chambers (6).

A constant potential difference of 30 V DC or 15 V DC, equivalent to potential gradients of 3 and 1.5 V/cm, were applied to the saturated and unsaturated clay samples, respectively, for periods ranging from 174 to 218 hours.

Upon completion of all experiments, postelectrokinetic soil and liquid samples were obtained for chemical analysis. The soil samples were divided into four equal parts of approximately 20 cm^3. The presence of a brown layer was observed in some of these parts. The parts were subdivided into two parts and

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analyzed separately. These samples were stored at 4°C for analysis of their water content, Mn concentration, and pH. Anode and cathode liquid samples were collected for all tests and analyzed for pH and Mn concentration.

Electromigration Experiments

The electromigration experiments were performed with the two kinds of prepared samples:

1. Experiment 1: Mn-spiked kaolinitic soil (saturated sample) whose behavior should be similar to mine soil and mine dump.
2. Experiment 2: Mn-spiked kaolinitic sludge (unsaturated sample) with properties similar to sediments in the Eume River's reservoir.

The experimental conditions for the experiments are presented in Table 1.

RESULTS AND DISCUSSION

Experiment 1. Unsaturated Clay Sample

An experiment with the unsaturated clay sample was carried out (Experiment 1). A constant difference of 15 V DC was applied to a sample having an initial Mn concentration of 5 g/kg dry weight. The treatment time was 218 hours.

Manganese Distribution

In Fig. 3 the Mn concentration before and after treatment and the final pH of each sample part are shown. It can be observed that the manganese migrated effectively through the soil to the cathode end. The Mn removed from soil was very high, ranging from 70 to 90% (quite homogeneous along the cell) with an average value of 76%. The Mn removed from the soil accumulated in a 60:40 proportion in the cathode and anode solutions, respectively.

TABLE 1
Electromigration Experiments of Kaolinitic Soil and Sludge Spiked with Mn: Experimental Conditions

Experiment	Sample	Potential difference (V)	[Mn] (g/kg dry weight)	Humidity (g H_2O/g dry weight)	Time (h)
1	Unsaturated	15	5	1	218
2	Saturated	30	3.8	0.582	174

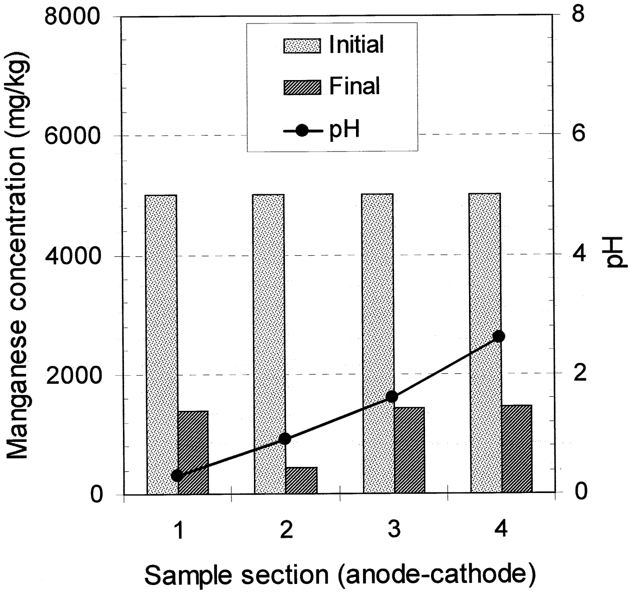

FIG. 3 Mn and pH distribution after electromigration of an unsaturated kaolinitic sample.

The pH remained at a very low value because the hydroxide ions generated at the cathode were not able to cope with the initial proton concentration and the incoming acid front from the anode. When the initial soil pH is low (2 to 3), very little electroosmotic transport occurs and the ionic species are transported efficiently. A reverse electroosmotic flow may result if the pH of the cathode chamber solution is decreased substantially (1). The chemical composition, in particular the pH, of the saturating solution has a significant role in determining the charge on soil particles. It is well known that kaolin clays typically acquire a negative potential (for pH greater than ~3), the magnitude of which increases with the pH. Parks (7) measured the zero point of charge (PZC) of several minerals including aluminosilicates such as kaolinite. The PZC varied from 3.3 to 4.6 depending on the clay source. Below this pH, the clay has a positive potential and the electroosmotic flow is toward the anode. The potential is negative above the PZC, and the electroosmotic flow is in the direction to the cathode (8). This finding has also been observed in this experiment, where 40% of the Mn which accumulated was collected in the anode chamber solution due to the effect of reverse pore fluid advection.

pH Evolution of Electrode Solutions

The evolution of pH in the electrode chambers solutions during the process is shown in Fig. 4. At the beginning the pH was adjusted to 2.5 in both cells by adding HNO_3 to the slurry. After 50 hours the pH of the cathode increased

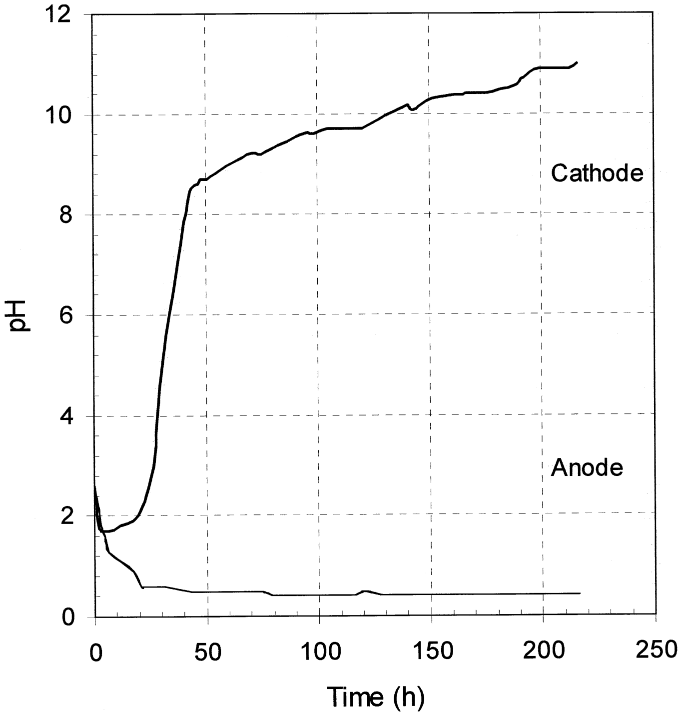

FIG. 4 Evolution of pH in electrode chambers along electromigration of Mn in an unsaturated kaolinitic sample.

dramatically due to the OH^- produced by water electrolysis. As a consequence of the high pH value, most of the Mn removed from the sample precipitated in the cathode chamber (540 mg of precipitate per liter of cathode solution) as Mn hydroxide.

Electric Field Distribution

The current intensity was observed to increase initially and then decrease progressively, as shown in Fig. 5. The maximum value achieved was 39 mA. As expected, the electrical resistance of the sludge sample had an opposite behavior. Initially it maintained a low, constant value, and then it increased from 140 hours until the end of treatment, when the resistance achieved a maximum of 125 Ω because the ions concentration in the sample decreased progressively. These values of the electrical resistance of the sample were calculated by applying Ohm's law to the voltage drop measured between the auxiliary electrodes located at the ends of the sample.

Manganese Speciation

The visual changes observed along the treatment time are presented in Tables 2 and 3. The different Mn species present in the electrode chambers, ei-

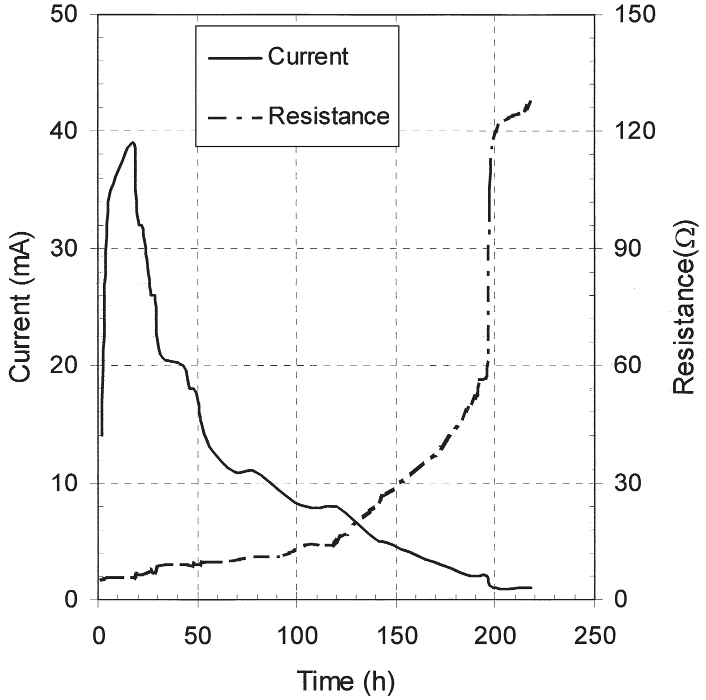

FIG. 5 Current intensity and electrical resistance evolution across the cell along electromigration of Mn in an unsaturated kaolinitic sample.

ther in solution or precipitated over surfaces, were identified by means of a Pourbaix diagram (9).

Experiment 2. Saturated Clay Sample

An experiment with the saturated clay sample was performed (Experiment 2). A constant potential difference of 30 V DC was applied to the sample with

TABLE 2
Speciation in the Anode Chamber along Electromigration of Mn in an Unsaturated Kaolinitic Sample

Time (h)	Visual changes	Species
3	Pale pink solution	MnO_4
17	Pale pink solution	MnO_4^-
	Brown layer on the wall	$MnO_4^- + H^+ \rightarrow MnO_2$
	Cl_2 evolution	$MnO_2 + HCl \rightarrow Cl_2$
		$2Cl^- \rightarrow Cl_2 + 2e^-$
19	Pink-brown and turbid dissolution	
140	Yellow and transparent dissolution	

TABLE 3
Speciation in the Cathode Chamber along Electromigration of Mn in an Unsaturated Kaolinitic Sample

Time (h)	Visual changes	Species
17	White layer on electrode surface	$Mn(OH)_2$
21	Brown-dark green precipitate on front side electrode	MnO
43	Brown colloidal precipitate. pH 8.5	$Mn(OH)_2$
50	Brown colloidal precipitate	$Mn(OH)_2$
	Black-brown precipitate on electrode	$Mn(OH)_2$
70	Transparent dissolution	MnO_2
	Brown precipitate on bottom chamber, pH 9.2	$Mn(OH)_2$
166	Brown-dark precipitate on bottom, pH 10.4	MnO_2

an initial Mn concentration of 3.8 g/kg dry weight. The treatment time was 174 hours.

Manganese Distribution

Figure 6 shows the Mn concentration before and after electromigration treatment, as well as the pH profile of the sample.

As in the previous experiment, a noticeable migration of Mn to the cathode occurred. An average Mn removal from the soil of 74% was attained. Most of the Mn removed accumulated at the cathode end (98%) where a brown layer (due to Mn precipitation) was observed after the electrokinetic remediation. The final pH of each section is indicated in the same figure. The pH change dramatically in a narrow zone close to cathode.

A small fraction of the Mn removed (2%) reached the cathode solution and precipitated as manganese hydroxide in a ratio of 100 mg of precipitate per liter of cathode solution.

The Mn distribution along the cell agrees with the findings of others researchers who reported the behavior of heavy metals within an electric field. Unless it is neutralized by the incoming acid front, the alkaline front generated by electrolysis at the cathode provokes precipitation of most heavy metals as hydroxides. The backmigration and diffusion of the hydroxide ions generated at the cathode may lead to premature precipitation of cations transported to this region (1). The metals vary in their tendency to be adsorbed onto oxidized clays. Recent work on metal adsorption by clay soils shows that there is a good correlation between metal adsorption and soil pH, alkaline saturation, and cation-exchange capacity (10). The cation-exchange capacities of clays are pH-dependent because their surface potential changes. At high pH the zeta potential becomes increasingly negative due to the adsorption of hydroxide ions onto the soil surface, and metal adsorption increases as a result. Part of the metal adsorption with an increased pH is attributed to the adsorption of metal hydrolysis products (MOH^+) by the soil (2).

The soil reaction near the cathode removes charged species and markedly decreases the local conductivity. This creates a small region near the cathode (10–20% of the total medium length) that develops a large electric field (11). When the experiment stopped, this region was observed as a brown layer that measured 12% of the total length.

pH Evolution at Electrode Solutions

The pH of solutions in the electrode chambers increased in the cathodic solution and decreased in the anodic one (Fig. 7). A constant value was achieved very quickly, approximately 24 hours after start-up.

Changes in the electrode chamber solution were observed visually during the experiment. The cathodic solution turned turbid and brownish once the pH value increased from 4.9 to 9, changing to a transparent solution when some amount of the brown material precipitated (Mn hydroxide) at pH 10. At higher values the formation of the precipitate increased and many small gas bubbles

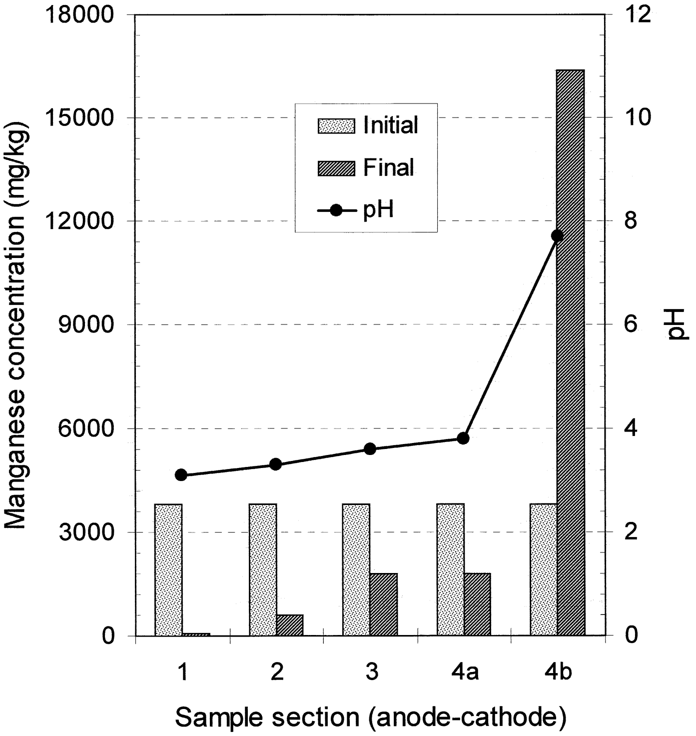

FIG. 6 Mn and pH distribution after electromigration in a saturated kaolinitic sample.

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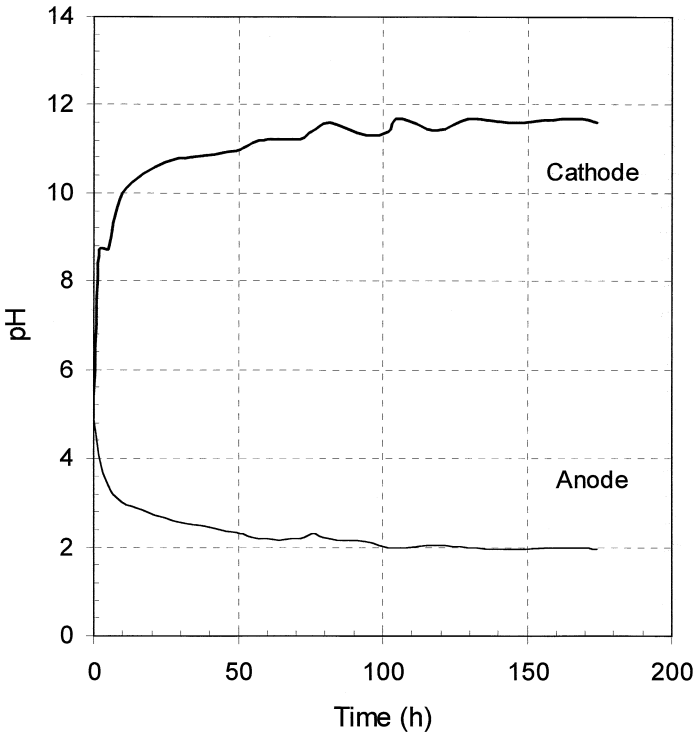

FIG. 7 Evolution of pH in electrode chambers along Mn electromigration in a saturated kaolinitic sample.

(H_2 produced by water electrolysis reaction) were produced in the cathode surface at the moment of maximum current intensity. In the last stages of the experiment, fewer but larger bubbles remained adhered on the cell walls.

The anode solution did not show any significant changes, although it turned yellowish at the last stages of treatment.

Electric Field Distribution

The current intensity was observed to increase and then taper off to a low value, as shown in Fig. 8. The current drop-off occurred approximately 34 hours after the start of testing, the maximum current intensity achieved was 2.83 mA.

Initially, the potential difference of 30 V was distributed as 25 V across the interface sample–cathodic solution, 4.8 V across the interface sample–anodic solution, and 0.2 V across the sample itself. This ratio was maintained throughout the treatment. The main voltage drop in the interface sample–cathodic solution rose to 28.6 V at the end of testing.

These results agree with the electrical resistance developed through the sample and across the interface sample–cathodic solution, which are indicated in Figs. 8 and 9. In the first stages of electromigration treatment, the sample

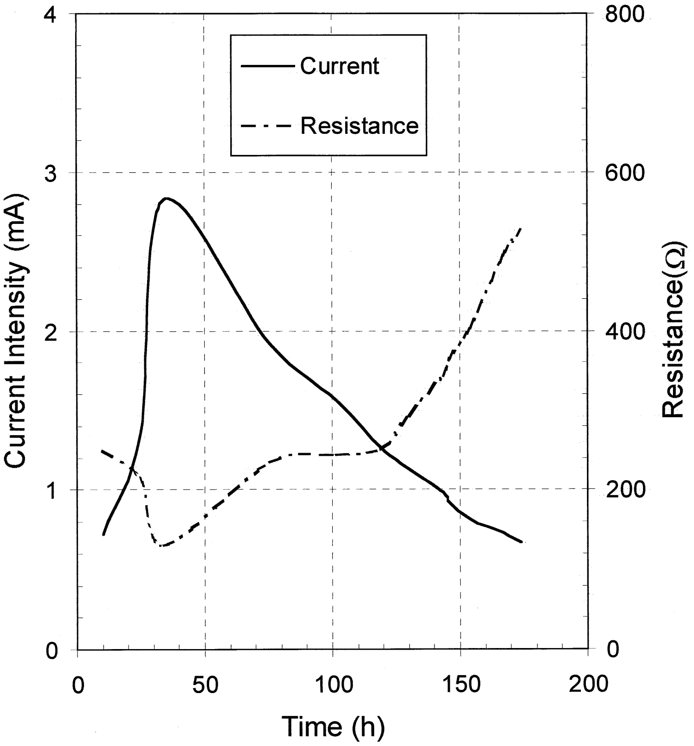

FIG. 8 Current intensity and electrical resistance evolution across the cell along Mn electromigration in a saturated kaolinitic sample.

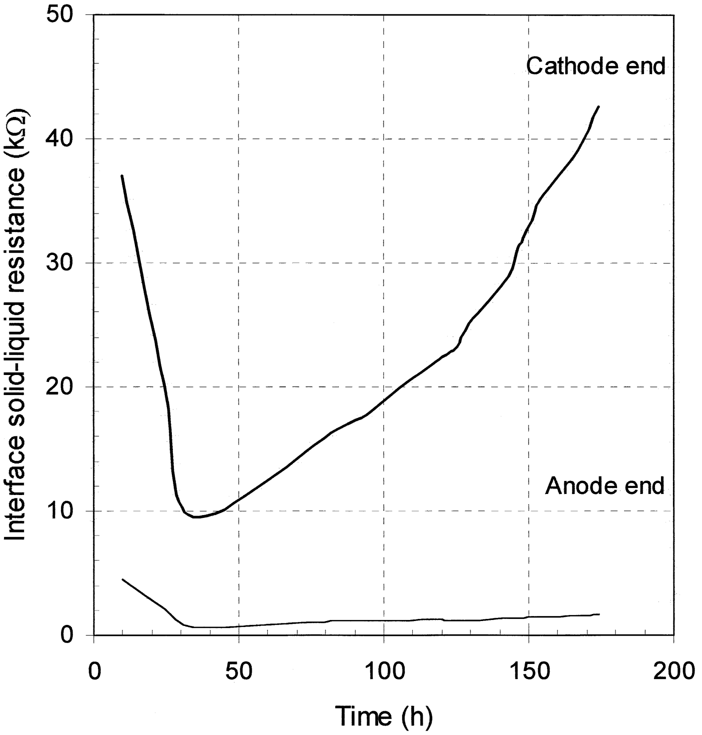

FIG. 9 Electrical resistance evolution across the interface of the sample–electrode solution along Mn electromigration in a saturated kaolinitic sample.

resistance decreases to a low value while the current intensity is rising, then the resistance increases progressively up to 500 Ω in the last stages. Moreover, the interface solid–liquid resistance at the cathode end decreased during the first 34 hours and then progressively increased.

Once the maximum intensity was attained, a brown layer began to appear in the sample near the cathode end. This phenomenon can be explained by Mn precipitation close to the cathode, which clogged the soil pores, hindering further transport of more Mn cations and other species (1). This fact creates a low ionic strength region of low conductivity resulting from reactions that eliminate charge-carrying ions (e.g., H^+ and OH^-) in interactions either with each other or with the soil surface. In addition, this causes an increase in the electric field, which in a feedback effect accelerates the deionization. The early drop in overall conductivity is a result of the local conductivity changes that are driven mainly by electromigration transport. The low ionic region is often associated with a jump in the pH that can change the sign of the charge of the dominant heavy metal species, leading to focusing of the contaminant within the medium. Moreover, low conductivity means that the overall movement of ions by electromigration is slow, so the remediation process takes a great deal of time (11). This underlines that soil pH is of crucial importance for the successful removal of Mn from soil.

CONCLUSIONS

Mn was easily removed by electromigration from kaolinite samples (unsaturated and saturated), obtaining average removal efficiencies of 76 and 74%, respectively, along with different treatment times. The relationships between the different redox/pH conditions developed during treatment and the visual changes in the electrode chambers in the process for the unsaturated sample, permit identification of the different Mn species.

Mn migrated in all samples toward the cathode end and was removed in the cathodic solution. For the saturated sample, the hydroxide ions generated by water electrolysis at the cathode crossed the interface sample–cathodic solution, and therefore the pH of this region increased. This caused Mn accumulation at the cathode end and subsequent precipitation as the hydroxide. This phenomenon was observed as a brown layer that appeared during the electromigration treatment.

If the precipitation of Mn hydroxide is not wanted, it could be avoided by controlling the pH at the cathode by adding acid. A cationic-exchange membrane located between the sample and the cathode could be used to prevent OH^- ions from entering the sample. Another way would be to apply complexing agents to protect the Mn cations from the basic conditions.

The electrokinetic experiments presented in this paper allowed useful information about the behavior of Mn under a transient electric field to be obtained.

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