

# Removal of Phenol from Contaminated Kaolin Using Electrokinetically Enhanced In Situ Chemical Oxidation

PRAPISALA THEPSITHAR AND  
EDWARD P. L. ROBERTS\*

School of Chemical Engineering and Analytical Science,  
University of Manchester, P.O. Box 88,  
Manchester M60 1QD, U.K.

The application of electrokinetic treatment to phenol contaminated soil combined with the addition of a chemical oxidant (permanganate) to the catholyte has been evaluated. It was found that the counter-current flow of the phenol (by electroosmosis) and permanganate (by electromigration) leads to a rapid oxidation of the phenol in the soil. After 5 days of treatment over 90% of the phenol (initial concentration 1 g kg<sup>-1</sup> dry soil) in a 15 cm long container of kaolin was removed by electrokinetic treatment with a voltage gradient of 1 V cm<sup>-1</sup> and an initial concentration of KMnO<sub>4</sub> of 9 g L<sup>-1</sup> in the catholyte. This level of treatment was achieved with an electrical energy consumption of less than 15 kWh per m<sup>3</sup> of soil treated.

## Introduction

Phenol is listed as a 'priority' contaminant in the United States (1) and the U.K. (2). It occurs in industrial waste from the coal-gas, coal-coking, petroleum, and pharmaceutical industries as well as in wastes from processes involving the use of phenol as a raw material (3, 4). According to the U.S. EPA Annual Status Report (2004), 172 of 863 Superfund Projects in the United States during 1982–2002 involved soils contaminated with nonhalogenated semivolatile organic compounds including phenol. The available remediation techniques for soil contaminated with phenol include bioremediation, incineration, soil washing, soil vapor extraction, solidification/stabilization, and landfill (5, 6).

In situ chemical oxidation by potassium permanganate has been reported to be an effective soil remediation method for the removal of organic contaminants including phenol (7, 8). Potassium permanganate is an oxidizing agent with a unique affinity for organic compounds containing hydroxyl groups such as phenol (7). In the presence of sufficient permanganate, phenol can be completely mineralized to carbon dioxide (CO<sub>2</sub>) (9). Complete oxidation requires 15.7 g of KMnO<sub>4</sub> for 1 g of phenol, although the presence of other compounds may increase the amount of potassium permanganate required (10, 11). The efficiency of phenol oxidation by potassium permanganate is not strongly affected by the pH of the media, and the reactions are effective for pH 3–10 with an optimum pH range of 7–10 (8, 11). The process requires the supply of sufficient oxidant to all parts of the contaminated zone. Research and demonstration activities have explored approaches of oxidant delivery including

permeation by lances, flushing by groundwater wells, and using hydraulic fractures filled with oxidant (12). Oxidant supply is particularly difficult when the soil has a low permeability, especially clay.

The electrokinetic process is one of the most effective techniques for the movement of substances in low permeability soils. The process involves the movement of water (electroosmosis), ions (electromigration), and charged particles (electrophoresis) between two electrodes under an applied DC electric field. Early laboratory studies of electrokinetic processes for phenol removal focused on electroosmosis with or without a purge solution containing species such as NaCl, NaOH, or surfactants (13–17). The process has also been demonstrated for the movement of anions in soil (18–20) and iodide delivery to enhance the removal of mercury (21).

In this study, the electrokinetic process is used to deliver an oxidant to enable in situ chemical oxidation. The removal of phenol from contaminated soil using this process involves the advection of phenol by electroosmosis, electromigration of permanganate ions, and phenol oxidation by permanganate. Three types of experiment are reported: electrokinetic delivery of permanganate to uncontaminated soil; electrokinetic treatment of soil contaminated with phenol; and electrokinetically enhanced in situ chemical oxidation of phenol by permanganate.

## Materials and Methods

**Experimental Setup.** The apparatus used (see Figure 1) comprised two parts: the electrokinetic cell and the electrolyte circulation systems. The electrokinetic cell included a tubular sample vessel, porous dividers, and electrode compartments containing the electrodes. The sample tube was made of clear Perspex, with an inside diameter of 5 cm and a length of 15 cm. The tube was constructed in five sections for accurate separation of samples at the termination of each experiment. Porous carborundum separators were used between the electrode compartments and the sample, to ensure that the soil sample was retained within the sample vessel. Each electrode compartment had a volume of approximately 400 cm<sup>3</sup> and was fitted with a vent. A stainless steel cathode and dimensionally stable (iridium mixed metal oxide) anode were used, each with an area of 38.5 cm<sup>2</sup> and connected to a DC power supply. The circulation system comprised reservoirs, pumps, and a balance to measure the electroosmotic flow.

**Soil Specimen.** Kaolin was used as a model low permeability soil. Equal masses of dry kaolin and water or phenol solution (1000 mg L<sup>-1</sup>) were mixed using a mechanical stirrer for 2 h. A total of 520–525 g of soil was loaded into the test cell using a tamper. A sample of the soil was retained to determine the initial phenol concentration, water content, and soil pore fluid properties. The soil specimen initially contained around 50% water, 1000 mg phenol kg<sup>-1</sup> dry soil, and the initial pH and conductivity of the soil pore fluid were 3.5–3.6 and 850–920  $\mu$ S cm<sup>-1</sup>. The low pH suggests there was significant proton adsorption on the kaolin particles used.

**Methods.** Three series of experiments were performed: evaluation of electrokinetic permanganate delivery; removal of phenol using the electrokinetic process alone; and removal of phenol using the electrokinetically enhanced oxidation process. A potential gradient of 1 V cm<sup>-1</sup> was applied across the cell during all experiments which is typical for electrokinetic treatment as it is considered a safe maximum for practical application. During each experiment the electroosmotic flow and electrical current were monitored. Upon

\* Corresponding author phone: +44 (0)161 306 8849; fax: +44 (0)161 306 9321; e-mail: edward.roberts@manchester.ac.uk.

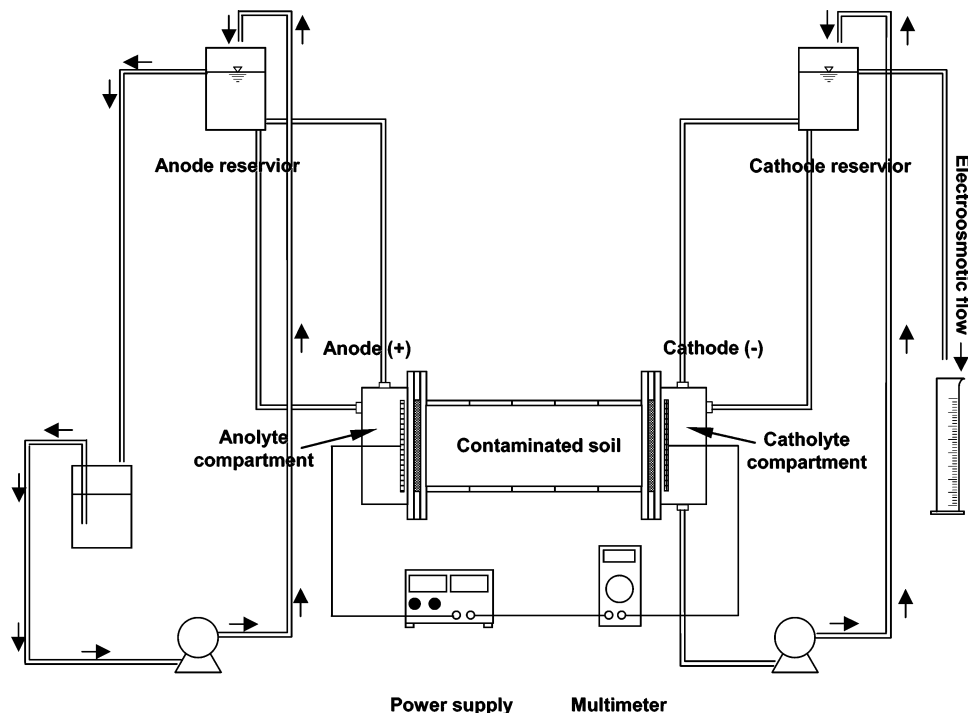


FIGURE 1. Schematic diagram of the laboratory apparatus.

completion of the experiment, the soil was separated into five sections (0–3 cm, 3–6 cm, etc.). Each section was analyzed for phenol, water content, pore fluid pH and conductivity, and, where appropriate, manganese content and the pore fluid permanganate concentration. The pore fluid was separated by centrifugation followed by filtration. The permanganate concentration in the pore fluid was measured by spectrophotometry at a wavelength of 545 nm (22). The manganese was extracted from the soil sample using nitric acid solution at a pH of 2.0 followed by inductively coupled plasma atomic emission spectroscopy.

Phenol was extracted from the soil samples using dichloromethane. The soil was mixed with deionized water and dichloromethane (in a mass ratio 2:3:12) for 1.5 h, and the organic phase was analyzed for phenol by gas chromatography with a flame ionization detector. Although more vigorous extraction conditions could be used, the recovery achieved (>90%) was sufficient for the purposes of this study.

## Results and Discussions

**Permanganate Delivery.** The phenomena responsible for the movement of permanganate ions through the soil specimen include electromigration, diffusion, and electroosmotic advection. Figure 2 shows the distribution of permanganate in the soil after electrokinetic treatment with permanganate in the catholyte. The results show that permanganate ions migrated through the soil specimens from the cathode to the anode when the potential gradient was applied across the cell. Permanganate ions had migrated from the cathode through the soil specimen and reached the region in the middle of the cell after 24 h. Increasing treatment time was observed to increase the concentration of permanganate in the soil which could be detected throughout the soil after 72 h.

Assuming that the transport of permanganate is primarily by electromigration (23), the time for permanganate migration through the soil specimen can be estimated as follows

$$t = \frac{L}{u_{\text{MnO}_4^-} \tau n \nabla(-E)} \quad (1)$$

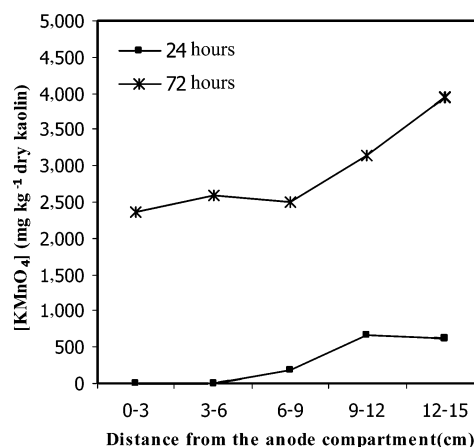
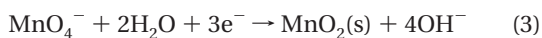


FIGURE 2. The distribution of  $\text{KMnO}_4$  concentration in the soil specimens after the electrokinetic process of 24 and 72 h with permanganate delivery.

where  $t$  is the time required for permanganate ions to travel from the cathode to the anode,  $L$  is the length of the test cell (15 cm),  $u_{\text{MnO}_4^-}$  is the ionic mobility of permanganate, and  $\nabla(-E)$  is the applied potential gradient. Values for the porosity ( $n$ ) and tortuosity ( $\tau$ ) of kaolin have been reported in the literature (25), with typical values of 0.4 and 0.5, respectively (16). Some of the permanganate had migrated 6–9 cm from the cathode compartment after 24 h (Figure 2). Using eq 1, the ionic mobility of permanganate in the kaolin can be estimated to be  $35\text{--}52 \times 10^{-5} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ , which is slightly lower than the reported value at infinite dilution (and in the absence of kaolin) of  $63.5 \times 10^{-5} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$  (24). The rate of transport based on the measured electroosmotic flux was around 1 order of magnitude smaller than the electromigration rate and in the opposite direction. These results confirm that the permanganate transport is primarily by electromigration.

Permanganate may also be reduced at the cathode by the reactions shown in eqs 2 and 3.



After treatment, the cathode was coated with a thin layer of dark brown particles, assumed to be manganese dioxide. The concentration of potassium permanganate solution decreased from the initial concentration of  $6000 \text{ mg L}^{-1}$  to around  $5500 \text{ mg L}^{-1}$  after 24 h and to around  $5000 \text{ g L}^{-1}$  after 72 h. Thus excess potassium permanganate must be provided, or the cathode could be placed in a separate compartment although this would add to the complexity of the process.

For uncontaminated soil, lower soil pHs were observed with permanganate compared to experiments in the absence of permanganate. This was probably caused by a reduction in the rate of electromigration of hydroxide from the cathode, as the higher conductivity associated with the permanganate led to a lower potential gradient in this region. The apparent electroosmotic permeability with permanganate ( $1.7 \pm 0.2 \times 10^{-5} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ , calculated using the Helmholtz-Smoluchowski equation using the electroosmotic flow volume) was slightly lower than that obtained in the absence of permanganate ( $2.6 \pm 0.3 \times 10^{-5} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ ). This was probably due to a higher ionic strength of the soil pore fluid leading to reduction in the thickness of the double layer.

The energy consumed by the electrokinetic process with permanganate ( $5 \text{ kWh m}^{-3}$  and  $40 \text{ kWh m}^{-3}$  for 24 and 72 h of treatment, respectively) was higher than that consumed by the process without permanganate ( $4.6 \text{ kWh m}^{-3}$  for 24 h of treatment). This was due to the higher conductivity of the pore water and consequently higher current.

#### Phenol Removal Using Electrokinetic Remediation.

Figure 3 shows the distribution of the residual phenol across the soil specimens after a range of electrokinetic treatment times, in the absence of permanganate. As the operating time increased the residual phenol in the soil decreased, as expected. However, the soil specimen showed a slight accumulation of phenol in the middle of the cell after 120 h of electrokinetic treatment, probably due to the formation of cracks (a few millimeters in thickness and up to 5 cm in length) perpendicular to the electric field and 8–10 cm from the anode, leading to discontinuity of the flow path. The percentage of phenol removed is plotted as a function of operating time in Figure 4. The results show that the amount of phenol removed gradually increased with increasing operating time, although the rate of removal appears to decrease with time. This was probably due to the lower electroosmotic flow rate observed after a few days of treatment.

The electroosmotic flow was toward the cathode in all of the tests, and there was no measurable flow within the first few hours of treatment. The electroosmotic flow rate increased during the first few days and then decreased to around one-quarter of its maximum value, due to changes in the soil pore fluid properties during the electrokinetic process. The apparent electroosmotic permeability coefficient was in the range  $1.1 \pm 0.2 \times 10^{-5}$ – $1.9 \pm 0.2 \times 10^{-5} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$  and was consistent with values for kaolin reported in literature ( $1 \times 10^{-5}$ – $10 \times 10^{-5} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ ) (26).

During the electrokinetic process, the characteristics of the soil pore fluid (i.e., pH and conductivity) and water content varied with time. Figure 5 shows the distribution of pH of the soil pore fluid across the soil specimen upon the completion of each test. The pH of pore fluid strongly influences the zeta potential of soil and may affect the speciation of phenol in the soil. The removal of organic contaminants including phenol from contaminated soil is significantly dependent on the electroosmotic flow rate (16, 27). The zeta potential of the soil controls the direction and the rate of the electroosmotic flow (28). At low pH, the

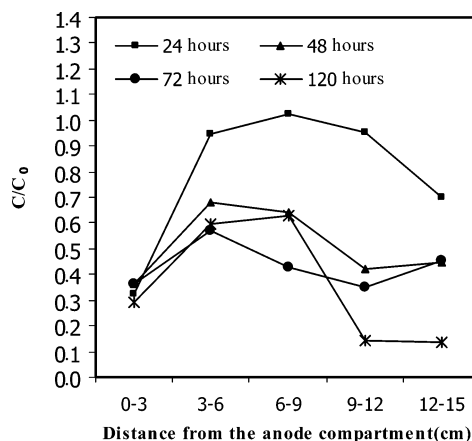


FIGURE 3. The distribution of the residual phenol across the soil specimens upon the completion of the electrokinetic process for 24, 48, 72, and 120 h.

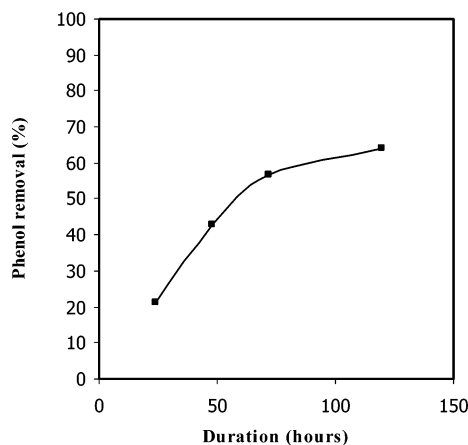


FIGURE 4. The percentage of phenol removed from contaminated kaolin using the electrokinetic process as a function of the operating time.

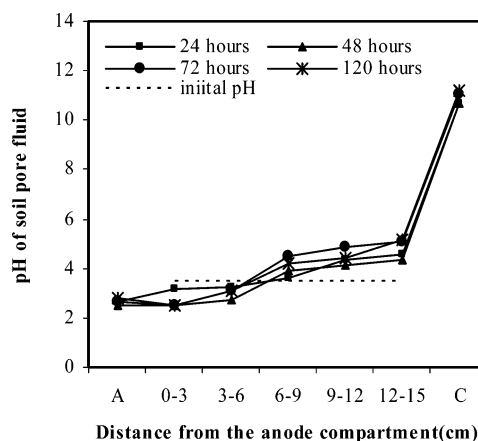


FIGURE 5. The distribution of the soil pore fluid pH across the soil specimens upon the completion of the electrokinetic process for 24, 48, 72, and 120 h.

magnitude of the zeta potential decreases and may become positive below the isoelectric point, which depends on the surface chemistry and varies considerably depending on the kaolin source. The measured isoelectric point of the kaolin used occurred at a pH of 2.0, thus for the pH range measured (2.5–4.6), the zeta potential of the soil particles remained negative, consistent with observed electroosmotic flow toward the cathode.

**TABLE 1. Percentage Phenol Removed,  $\text{KMnO}_4$ , and Energy Consumed per  $\text{m}^3$  of Soil Treated, as a Function of the Treatment Conditions**

duration (h)	initial $\text{KMnO}_4$ concn ( $\text{mg L}^{-1}$ )	percentage phenol removal	$\text{KMnO}_4$ consumption ( $\text{kg m}^{-3}$ )	energy expenditure ( $\text{kWh m}^{-3}$ )
24	0	21	0	4.87
48	0	43	0	7.36
72	0	56	0	8.86
120	0	64	0	11.78
24	6000	39	10.8	4.99
24	9000	40	12.1	4.47
24	12000	52	14.7	5.91
72	9000	75	16.9	10.70
120	9000	91	19.4	14.23

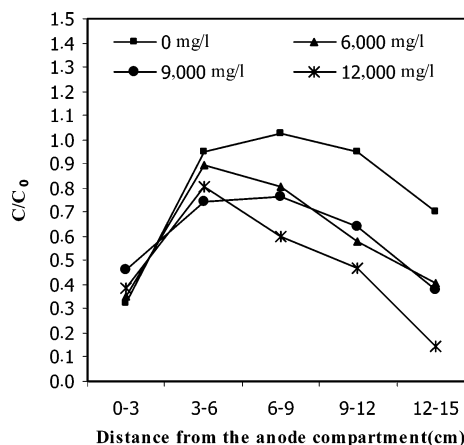
For  $\text{pH} > 9$ , a significant proportion of phenolate ions,  $\text{C}_6\text{H}_5\text{O}^-$  will be present. and this species will migrate toward the anode (28, 29). Although a high  $\text{pH}$  was observed in the catholyte, acidic conditions were observed in the soil pore fluid, so that phenol was expected to exist in nonionic form throughout the specimen.

Table 1 shows the energy required for the removal of phenol from contaminated kaolin using the electrokinetic process. The amount of phenol removed from the soil increased with operating time and energy expenditure. The percentage of phenol removed reached 64% after 120 h of treatment and consumption of  $11.8 \text{ kWh m}^{-3}$ . These results are consistent with those reported by Acar et al., who found that the energy required to achieve 85–95% phenol removal from kaolin by electrokinetic treatment was around 18–39  $\text{kWh m}^{-3}$  (13).

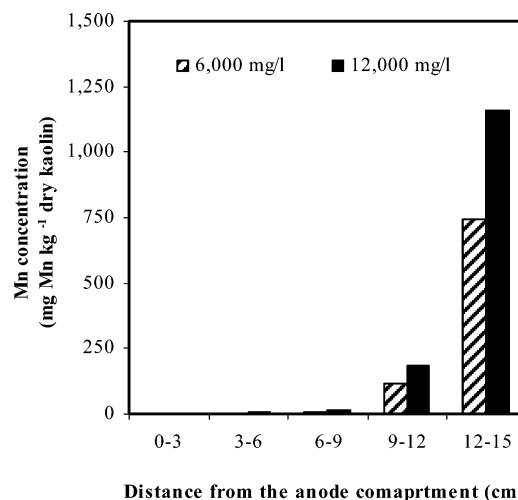
**Phenol Removal Using Electrokinetically Enhanced Chemical Oxidation.** The mechanism of oxidation of phenol by permanganate is complex and sensitive to  $\text{pH}$  (12). Under acidic conditions ( $\text{pH} < 3.5$ ), which occurred near the anode, direct oxidation of phenol is expected to occur, with formation of  $\text{Mn}^{2+}$ , which may subsequently be oxidized to  $\text{MnO}_2$  in the presence of excess permanganate. Under the slightly milder acidic (or neutral) conditions which are also observed to occur in the sample, direct oxidation with formation of  $\text{MnO}_2$  is expected. Very close to the catholyte membrane (or in the catholyte) alkaline conditions may lead to the formation of hydroxide radicals (by reaction between hydroxide and permanganate) which subsequently oxidize the phenol. In all cases, phenol oxidation is expected to lead to the formation of intermediates which will undergo further oxidation by permanganate. It is likely that in some parts of the cell there was insufficient permanganate for complete mineralization, but this possibility was not investigated in this study.

In all of the experiments where phenol and permanganate were present, a brown precipitate (taken to be  $\text{MnO}_2$ ) was observed in the region near the cathode. As the treatment time increased this precipitate was observed at increasing distance from the cathode. Where phenol was not present, no precipitate was observed within the sample. Since the electroosmotic advection resulted in the movement of phenol from the anode toward the cathode, while electromigration of permanganate occurred from the catholyte toward the anode, the main reaction zone was at the cathode end of the cell, consistent with the observation of the precipitate in this region.

Figure 6 shows the distribution of residual phenol across the specimens after 24 h of treatment for a range of initial concentrations of permanganate in the catholyte. Figure 7 shows the manganese content of the soil after treatment with initial permanganate concentrations of 6000 and 12 000  $\text{mg L}^{-1}$  in the catholyte. The residual phenol concentration in the soil closest to the cathode was significantly lower when



**FIGURE 6. The distribution of the residual phenol across the soil specimens upon completion of the tests with variation of initial concentration of  $\text{KMnO}_4$  used in the cathode compartment.**



**FIGURE 7. The manganese content in the soil after treatment where the initial  $\text{KMnO}_4$  concentrations in the cathode compartment were 6000 and 9000  $\text{mg L}^{-1}$ .**

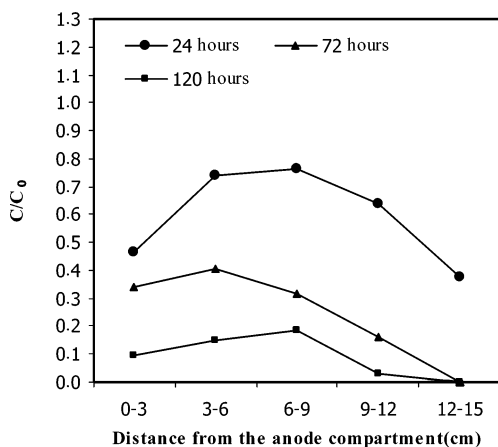
the highest concentration of potassium permanganate was used in the catholyte.

The manganese content of the soil in the area closest to the cathode increased with the concentration of potassium permanganate used in the catholyte. This finding suggested that phenol oxidation by permanganate took place in this area. Phenol removals of 39, 40, and 52% were obtained after 24 h of treatment with 6000, 9000, 12 000  $\text{mg L}^{-1}$   $\text{KMnO}_4$  in the catholyte (Table 1).

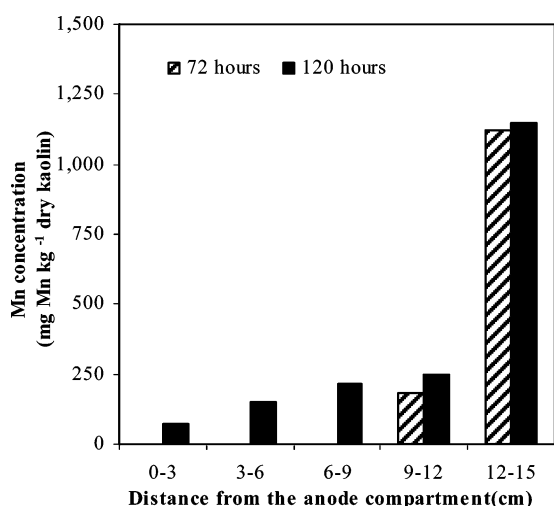
Although the production of the  $\text{MnO}_2$  precipitate could lead to a lower hydraulic permeability, the apparent electroosmotic permeability in these experiments ( $1.6 \pm 0.2 \times 10^{-5}$ – $2.4 \pm 0.3 \times 10^{-5} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ ) was similar to that obtained in experiments using electrokinetic treatment alone ( $1.9 \pm 0.2 \times 10^{-5} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ ). The generation of  $\text{MnO}_2$  does not appear to have a significant effect on the electroosmotic permeability of the soil and consequently the transport of phenol.

The energy required for the electrokinetic process with permanganate delivery and the percentage of phenol removed are shown in Table 1. The experimental results indicate that the energy required for the electrokinetic process with and without potassium permanganate were not significantly different, although with a high concentration of potassium permanganate the energy expenditure increased. The electrokinetic process in conjunction with chemical oxidation achieved higher phenol removals.





**FIGURE 8.** The distribution of the residual phenol across the soil specimens upon the completion of the tests with the initial  $\text{KMnO}_4$  concentration of  $9000 \text{ mg L}^{-1}$  in the cathode compartment with variation of the operating time.



**FIGURE 9.** The manganese content in the soil after treatment where the initial  $\text{KMnO}_4$  concentration in the cathode compartment was  $9000 \text{ mg L}^{-1}$  and the operating time was 72 or 120 h.

The distribution of residual phenol across the specimens after electrokinetic treatments of increasing duration with an initial catholyte  $\text{KMnO}_4$  concentration of  $9000 \text{ mg L}^{-1}$  are shown in Figure 8. The manganese content in the soil after treatment under these conditions is shown in Figure 9. The results show that an increase in the operating time resulted in lower phenol concentrations in all of the sections of the specimen. After 72 and 120 h of treatment the phenol content close to the cathode was diminished to below the detection limit. Significant concentrations of manganese were present in the soil up to 6 cm from the cathode after 72 h and throughout the soil specimen after 120 h.

In the area near the anode the percentage of phenol removed was around 71% after 120 h of electrokinetic treatment in the absence of permanganate (refer to Figure 3), while after the same treatment time with permanganate around 90% of the phenol was removed in this region. These results and the detection of manganese near the anode indicate that oxidation of phenol took place throughout the soil specimen after a few days of treatment. Hence, the increasing operating time led to an increase in the size of the oxidation zone.

The phenol removal achieved by the electrokinetic treatment with chemical oxidation and the associated electrical energy and mass of  $\text{KMnO}_4$  used is summarized for a range of operating conditions in Table 1. The amount of phenol

removed significantly increased with increasing operating time when permanganate was present in the catholyte and the increasing operating time resulted in higher energy expenditure. As expected the consumption of  $\text{KMnO}_4$  increased with both  $\text{KMnO}_4$  concentration and treatment time. The consumption of  $\text{KMnO}_4$  is relatively high, and the cost associated with the  $\text{KMnO}_4$  used is much higher than that of the electrical energy. Assuming that the  $\text{KMnO}_4$  achieved complete oxidation of the phenol that was removed, around 45–55% of the  $\text{KMnO}_4$  was consumed by reduction at the cathode. This loss could potentially be recovered or eliminated by use of an ion exchange membrane. Further work is needed to establish the extent of phenol oxidation achieved and the amount of  $\text{KMnO}_4$  that is consumed by the phenol oxidation reaction in order to evaluate the feasibility of the process.

Only  $14.2 \text{ kWh m}^{-3}$  of electrical energy was required to remove 90% of the phenol from the contaminated soil after 120 h of treatment, for an initial phenol concentration of  $1000 \text{ mg kg}^{-1}$  dry kaolin. The results show that electrokinetics can provide an effective method for delivering permanganate for oxidation of organic contaminants, with a very small cost in terms of electrical energy consumption. Further experiments are needed to determine the optimum operating conditions to achieve a desired level of treatment.

It is clear that the energy cost is relatively low, but the shorter treatment time and higher removal achieved will lead to a reduced treatment cost when the chemical oxidation and electrokinetic treatment processes are combined. In conclusion, electrokinetically enhanced chemical oxidation by permanganate offers a new approach for the treatment of contaminated soil containing organic contaminants able to be oxidized by permanganate (including phenol, benzaldehyde, and polynuclear aromatic hydrocarbons) in low permeability soils. In addition, this process would also be applicable to hydrophobic organic contaminants, which cannot be removed by electroosmotic advection.

## Acknowledgments

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