Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

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To cite this Article Popov, Konstantin , Kolosov, Andrey , Yachmenev, Val G. , Shabanova, Nadezhda , Artemyeva, Alla , Frid, Alexander , Kogut, Boris , Vesnovskii, Stanislav and Sukharenko, Vladimir(2001) 'A LABORATORY-SCALE STUDY OF APPLIED VOLTAGE AND CHELATING AGENT ON THE ELECTROKINETIC SEPARATION OF PHENOL FROM SOIL', Separation Science and Technology, 36: 13, 2971 — 2982
To link to this Article: DOI: 10.1081/SS-100107640
URL: http://dx.doi.org/10.1081/SS-100107640

SEPARATION SCIENCE AND TECHNOLOGY, 36(13), 2971–2982 (2001)

A LABORATORY-SCALE STUDY OF APPLIED VOLTAGE AND CHELATING AGENT ON THE ELECTROKINETIC SEPARATION OF PHENOL FROM SOIL

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ABSTRACT

The influence of the chelating agent (1-hydroxyethane-1, 1-diphosphonic acid) (HEDPA) on the electroosmotic flow (EF) in natural, sod-podzolic soil contaminated with 0.003 g phenol per 1 g of dry soil was studied. An introduction of a small amount of HEDPA increased the electroosmotic permeability of soil from 4×10^{-5} to 11.2×10^{-5} cm^2/V·s. Up to 80–95% of the phenol

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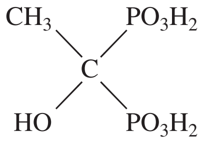
was removed from the soil by the EF during 30 to 50 hours of treatment with the average intensity of the EF remaining constant throughout the tests. The study confirmed that certain hazardous organic contaminants (e.g., phenol) could be successfully extracted from the soils of low permeability by combined application of a direct-current electric field and specific complexing agents.

INTRODUCTION

Electrokinetic (EK) soil flushing is a particularly effective remediation technique for the treatment of soils of very low permeability when traditional pump-and-treat methods of decontamination cannot be employed (1–6). This in situ extraction technology utilizes a direct-current (DC) electric field to transport both organic and inorganic contaminants through the soil pores to a collection zone (7–9). Although this novel technique has been promising and feasible in numerous tests, additional studies of the governing mechanisms of electroosmotic flow (EF) in real soils are necessary for better understanding and optimization of this emerging remediation technique. Further testing is also critical for lowering the costs associated with full-scale field deployment of the technology (4,5,10,11). In this context, a recently discovered phenomenon describing the enhancement of EF in soil by certain chelating agents could be of critical importance (12–14). An objective of the present research was to study cumulative effects of a DC electric field and chelating agent 1-hydroxyethane-1,1-diphosphonic acid (HEDPA) on the remediation of natural soil contaminated with phenol.

BACKGROUND

EK transport refers to all physical and chemical phenomena that occur in soil when an electric field is applied to a soil system. Two major transport mechanisms, which govern the movement of ionic and nonionic species in the soil pores under the influence of an electric field, are electroosmosis and electrolytic migration (7,8). Both take place along with electrolysis of water at the electrode surfaces. In general, electrode reactions generate a strong acidic front at the anode and a basic front at the cathode. These pH extremes at the electrodes greatly affect the pH of the soil and the mobility of contaminants and other ionic species in the pore solution. The EF is the leading mechanism responsible for transportation of nonionic pollutants through the soil. It varies approximately within 1 order of magnitude for different types of soils: approximately 1×10^{-5} cm^2/V·s for light soils to approximately 1×10^{-4} cm^2/V·s for heavy soils; the average mobility of ionic species is approximately 5×10^{-4} cm^2/V·s (3,15). In some cases when the innate EF is too small for cost-efficient removal of the hazardous organic compounds from contaminated soil, some auxiliary enhancement of the EF is required. Based on our previous research, the chelating reagent HEDPA (H_4HEDPA):



might be recommended as an effective agent for enhancement of the EF in soils of low permeability (12–14). It was found that certain chelating agents induce a large increase of EF in soil treated with the complexant in comparison with soil maintained at equilibrium with deionized water. In general, this enhancement depended on the type, concentration, and pH of complexant solution, and the type of soil and voltage applied. The data showed that enhancement of the EF in soil caused by complexant occurred over a wide pH range of complexant solution, but it was much more pronounced at higher pH. Experimental data also revealed that irrespective of the high ionic strength of the liquid phase, the overall negative zeta potential of the soil surface increased to more than 1 order of magnitude due to the specific influence of complexants; this zeta potential increased even at 0.2 mol/L concentrations of complexant. This effect could be the result of a threefold interaction of complexant with the soil surface:

1. Negatively charged complexants and their metal ion complexes could be specifically adsorbed on the surface of clay minerals, silicates, and Fe(Al)/Mn oxides, increasing the negative charge of soil particles.
2. Natural Fe(Al)/Mn amorphous oxides adsorbed onto clay minerals could suppress the original, permanent, negative charge of clay particles. Complexants dissolve these oxides and remove iron (III) and aluminum (III) from the crystal lattice of soil minerals restoring the original negative charge of the clay surface.
3. Complexants are able to mask all multivalent cations present in the liquid phase including the electric double layer. As a result, all multivalent cations are converted into negatively charged complexes and substituted by univalent ones. These univalent complexes are less effective in compression of the diffusion layer; thus, the overall zeta potential of the system is increased.

Although they normally are not associated with the phenomena of electric double layer and zeta potential, processes based on mechanisms no. 1–3 are generally known. The detailed theory of this effect is a matter of further research. The ability of certain complexants to alter electroosmosis in soil evidently offers a unique opportunity to govern remediation technologies (based on that phenomenon) by appropriate choice of the complexant reagent, control of its concentration, and pH parameters.

In our studies, phenol was chosen as a model organic contaminant because it is considered one of the priority pollutants by the U.S. Environmental Protection Agency and recently has been detected in groundwater supplies in certain areas (15,16). Although it is treated as a nonionic compound, in aqueous solutions phenol has a dissociation constant of 1.3×10^{-10} (17). Speciation studies of phenol performed with the SolEq Solution Equilibrium Program (17) indicated that in an aqueous phase below pH 7, phenol is present completely as an uncharged species HL, and it is present as a negatively charged ion L^- at pH > 12. Within the $7 < \text{pH} < 12$ both uncharged and charged species HL and L^- are present. Therefore, on one hand, we should expect that under an electric field gradient of pH < 7, the EF and diffusion would be the exclusive mechanisms of pollutant transport in soil pores, and phenol could be collected in the cathode chamber. On the other hand, within the $7 < \text{pH} < 9$ range, a certain amount of phenol would be driven by electromigration to the anode in a counterflow against electroosmosis. Therefore, the counterflow will render the remediation process to be less than optimally effective.

MATERIALS AND METHODS

Test Soil Selection and Characterization

The soil samples used in this study were collected from the top layer of arable land (0–15 cm) at Moscow site (Pushkinskij District, experimental site of Dokuchaev Soil Institute) and classified as sod-podzolic soil with heavy loam granule content. The sample represented a typical heavy clay soil with the following size fractions: sand 50–40%, silt 10%, and clay 40–50%. The main mineral content of the sample included kaolinite with chlorite (38–48%), mica (24–49%), and smectite (8–21%). Soil humidity at saturation constituted 32%. Other soil properties are given in Table 1.

Soil Preparation

Initial soil pretreatment involved separation of soil mass from large stones, grass remains, and plant roots. Then an approximately 3-cm soil layer was spread over a flat surface and allowed to equilibrate at ambient air for about 2 weeks. In the process of drying, the whole mass was extensively mixed several times. The uniform soil mass (approximately 50 kg) was sieved with a 3-mm sieve and stored at constant humidity. The 286-g sample of dried soil was thoroughly mixed with

Table 1. Sod-Podzolic Soil Properties

Parameter (units)	Value
pH (aqueous)	6.7
pH (KCl)	5.3
Hydrolytic acidity, mg-equivalent/kg of soil	17
Exchangeable Ca, mg-equivalent/kg of soil	160
Exchangeable Mg, mg-equivalent/kg of soil	70
Mobile phosphorus (Kirsanov), mg P_2O_5/kg soil	115
Exchangeable potassium, mg K_2O/kg soil (Maslova)	170
"Free" iron (Jackson), %	0.17
Mobile iron (Tamm), %	0.05
Electroosmotic permeability, $cm^2/V{\cdot}s$	4×10^{-5}
Organic carbon, %	1.2

56 mL of a 1.6%-phenol aqueous solution in a desiccator. After 1 hour, 78 mL of an aqueous HEDPA (3.53 g/dm^3, pH 7) solution was added to the sample so that the soil reached a humidity saturation level. The resulting mixture was equilibrated in a closed vessel for 28 hours. A total of 420 g of the soil/phenol/HEDPA sample was loaded into a soil compartment of Electrokinetic-1 cell. Both electrode chambers were filled with the same HEDPA solutions (pH 7). A nonhermetic (wooden) lid covered the soil compartment of the EK cell to reduce water loss by evaporation.

Electrokinetic Cell Design

Two types of special EK cells were used for the study of EK extraction of phenol from contaminated soil: A large one, E-1 (5 cm × 5 cm × 19 cm), and a small one, E-2 (1.5 cm × 2.0 cm × 11.5 cm). The E-1 cell was made from Plexiglas (see Fig.1) and consisted of 1 soil compartment (approximately 300 g of air-dry soil; approximately 450 g of moisture soil) and 2 electrode chambers (approximately 100 mL each). Both electrode chambers were equipped with special outlets for collection of the electroosmosis effluent. Inert platinum electrodes were used to prevent contamination of the soil and the electrolyte in electrode chambers by corrosion products. The interelectrode distance was 10.5 cm for E-1 and 10 cm for E-2 cells. Temperature, pH, voltage, electric current, and the volume of effluent were monitored and recorded during the experiments. The electrode reservoirs were filled with complexant solution. The manual addition of a corresponding amount of fresh complexant solution compensated for the loss of liquid in the anode electrode compartment caused by the electroosmosis phenomenon. In both E-1 and E-2 cells, soil compartments were separated from elec-

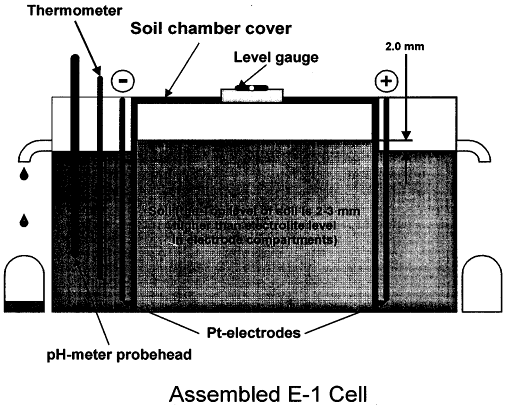


Figure 1. Assembled E-1 cell.

trode solutions by filter paper. The level of soil in the soil compartments was maintained at a level of 2–3 mm higher than the levels of liquid in the electrode chambers to prevent surface overflow of the effluent solution. The design of the soil compartment included cross-sectional hollows on the walls and bottom surface of the cell to prevent leakage of leaching solution. Manual addition of concentrated NaOH (anode) or H_2SO_4 (cathode) solutions compensated for pH changes in the electrode chambers. Upon completion of each experiment, the whole soil sample and aliquots taken from electrode compartments (including effluents) were analyzed for phenol content. A summary of the testing program and processing data for the E-1 experiments is given in Table 2.

Table 2. Summary of Testing Program and Processing Data for E-1 Cell

Parameter (units)	Test 1	Test 2
Soil pH	6–8	6–8
Temperature, °C	20	28
HEDPA concentration, mol/dm^3 (pH)	0.01 (7)	0.01 (7)
Humid soil mass, g	420	400
Voltage gradient, V/cm	2.1	2.1
Effluent, mL	163	235
Effluent pore volumes	1.3	1.8
Average flow, mL/h	5.0	4.5
Electroosmotic permeability, cm^2/V·s	11.2×10^{-5}	10.0×10^{-5}
Electrokinetic treatment time, hours	33	52

Some supplementary experiments were carried out with the E-2 cell, which was equipped with a graphite cathode and a tantalum anode. To imitate a voltage assisted pump-and-treat mechanism, the E-2 cell did not have special outlets for collection of effluent, but instead approximately 2.5 mL of cathode solution was removed every 2 hours and an equal amount of fresh solution was added to the anode compartment. After the EK experiment was completed, the soil sample in the E-2 cell was divided into 4 equal parts: cathode soil (I), 2 middle samples (II and III), and anode soil (IV). These samples, as well as the electrode chamber solutions, were analyzed for pollutant content. A summary of the testing program and the data processing for E-2 experiments are given in Table 3.

Electrokinetic Flushing and Specimen Analysis

All tests were conducted at constant voltage while the current was increasing steadily with treatment time from 20 to 30–50 mA. The phenol concentrations in the soil and in solution aliquots (from the cathode and anode chambers) were analyzed through the use of a UV-VIS spectrometer (Specord M80; adsorption band 270.3 nm). The residual concentration of phenol in soil samples was determined by the hot deionized-water extraction procedure. The pollutant was extracted by deionized water (60°C) during 1 hour at a 1:20 solid-liquid mass ratio. The supernatant and soil were separated by centrifugation and decantation procedures. Independent evaluation of the proposed extraction procedure revealed that this method insured extraction of at least 94% of initial phenol content in the sample with the minimal detectable level at 30 ppm. Changes in the pH of cathode and anode solutions were measured directly in the electrode chambers with a Metrohm pH meter.

Table 3. Summary of Supplementary Testing Program and Processing Data for E-2 Cell

Parameter (units)	Test 3	Test 4
Soil pH		
Initial	6	2
Final cathode	10	2
Final anode	2	2
Temperature, °C	21	21
HEDPA concentration, mol/dm^3 (pH)	0	0.1 (2)
Humid soil mass, g	45	45
Voltage gradient, V/cm	5.0	2.3
Electrokinetic treatment time, hours	120	120

RESULTS AND DISCUSSION

The various effects of applied voltage and complexant on the EF, current, and phenol removal were studied in tests 1 and 2 (cell E-1) and tests 3 and 4 (cell E-2). The results of tests 1 and 2 are presented in Table 4, and results of tests 3 and 4 are presented in Table 5. In all tests, a constant electrical voltage gradient throughout the soil samples was maintained throughout the duration of the experiment. The initial electric current through the cell (approximately 20 mA) decreased slightly (approximately 10%) during the first hour and increased gradually up to 40–50 mA by the end of experiment. Our earlier measurements of the original electroosmotic permeability of the soil (without complexant enhancement) indicated an EF of approximately 2.0 mL/hour and an electroosmotic permeability of 4×10^{-5} cm^2/V·s. This electroosmotic permeability was higher than that reported in the LASAGNA Project: 1.2×10^{-5} cm^2/V·s (1,13). Introduction of HEDPA increased the EF to 4–7 mL/h and the EF depended on the frequency of fresh solution addition (every 20 minutes in the daytime and every 3 hours during the night). Accordingly, the comparison of the empirical electroosmotic coefficients of soil permeability (Tables 1 and 2) shows the increase from 4×10^{-5} cm^2/V·s to 11.2×10^{-5} cm^2/V·s, which was caused by the introduction of HEDPA. These data indicated a significant (nearly 180%) increase in the EK treatment efficiency. The average intensity of the EF must remain constant throughout the whole duration of the experiment, which is contrary to the time-dependent trend that was reported in (15) for kaolinite where no complexants were used. Although in the present tests the overall enhancement of the EF caused by the introduction of complexant was only about one-third of that observed for K-25 soil (12), HEDPA significantly decreased the overall treatment time (13). Thus, use of even a small amount of HEDPA to treat soil significantly intensified the EF and also made the EF time independent.

Table 4. Summary of Phenol Removal Results for E-1 Cell

Parameter (units)	Test 1	Test 2
Initial content of phenol in soil, g	0.70	0.80
Final content of phenol, g		
Cathode + effluent	0.47	0.50
Soil	0.14	0.04
Anode	~0.06	~0.05
Final content of phenol, %		
Cathode + effluent	67	63
Soil	20	5
Anode	~8	~6
Total	88	74
Phenol removal, %	80	95

Table 5. Phenol Content in Specimens for E-2 Cell

Parameter (units)	Test 3	Test 4
Final content of phenol, mg		
Cathode + effluent	13.6	13.5
Soil sections		
I	10.7	4.0
II	3.2	3.7
III	3.3	1.5
IV	5.1	0.5
Anode	4.6	0.3

Accumulation of pollutant in the cathode compartment indicates that the EF is the predominant phenomenon responsible for soil remediation. The significant change in phenol content after first 33–52 hours of treatment is shown in Table 4. The initial phenol concentration was 1700 ppm (Test 1) and 1900 ppm (Test 2) in a humid soil sample. The final concentrations of phenol in treated soils, 300 ppm and 95 ppm, respectively, have been achieved through shorter operating times and for larger mass soil samples than those reported by Acar (15). In part, this result could be attributed to the fact that the phenol concentration in the Acar study (15) (0.5 mg/g of dry mineral) was below the adsorption capacity of kaolinite, while the present research dealt with soils of higher phenol content.

The measured mass balance of contaminant (phenol) in both tests was within 75–90%, which is consistent with the results reported in (15). The observed imbalances could be attributed to several factors:

1. Phenol may have been volatilized during sample preparation and soil treatment. We investigated this possibility in a special blank experiment, where a number of contaminated soil samples was left open at room temperature and analyzed for phenol content after various exposure times. Up to 30% of phenol could be lost within 24 hours, depending on the evaporation surface area.
2. The phenol content in the soil specimens was determined by a hot deionized-water extraction procedure. All the phenol may not have been leached out by this procedure. The corresponding losses could constitute as much as 4–5%.

However, the 10% error was acceptable for the purpose of the present study.

Two additional tests (3 and 4) were carried out under different conditions in the E-2 cell. They provided important data on the final distribution of pollutant throughout the soil sample. Our objective for test 3 was to study the EK soil remediation process without pH corrections of electrode solutions. The data in Tables 3 and 5 show that without pH corrections of electrode solutions, the pH of the immediate layer of soil adjusted to the cathode compartment could increase to a pH of approximately 10–11. Under such high pH conditions, phenol dissociates into species HL and L^- resulting in reversed transport of phenol to the anode compartment via the electromigration transport mechanism. Accordingly, the pH of the immediate layer of soil adjusted to the anode compartment (in the absence of pH adjustment) became very acidic. Thus, when phenol crossed the acid/base "front" in the soil and approached the anode soil layer, its dissociation was suppressed and phenol moved toward the cathode via the EF mechanism. Consequently, the electric field initiates a cyclic (back-and-forth) movement of phenol in the soil sample without its removal. In general, low pH conditions suppress the EF. As a result, the acidified part of the soil becomes partly "inactivated" for the purpose of electroosmotic remediation. Test 3 proved that it was mandatory to maintain adequate pH control of solutions in the electrode compartments to insure efficient EK remediation of soil contaminated with phenol or some other weakly acidic organic compounds. An inappropriate selection of experimental conditions could critically undermine all benefits and advantages of the EK remediation technique.

One of our objectives for test 4 was to use the buffering properties of HEDPA for automatic pH adjustments during the EK remediation process. Test 4 demonstrated that the high pH condition in the cathode chamber could be effectively buffered (neutralized) by complexant solution (HEDPA) thus insuring an efficient decontamination of sod-podzolic soil. The acidic form of HEDPA (pH 1–2) could be relatively harmful for soil, and it should be employed in combination with the sodium salt of HEDPA (Na_2H_2HEDPA; pH 5–7).

SUMMARY AND CONCLUSIONS

Our laboratory research has shown that combined application of a DC electric field and complexant (HEDPA) could be successfully employed for decontamination of natural sod-podzolic soil of low permeability that is contaminated with phenol. It was established that in the presence of HEDPA, electroosmosis was the dominating remediation mechanism. Pretreatment of contaminated soil with HEDPA increased electroosmotic permeability of soils from 4×10^{-5} to 11.2×10^{-5} $cm^2/V\cdot s$ and made the permeability time independent. Up to 80–95% of the phenol was removed within 30–50 hours of treatment. The importance of periodic pH adjustment in both electrode chambers was demonstrated. The study confirmed that the EK remediation of soil contaminated with hazardous, organic contaminants could be significantly advanced by introduction of appropriate complexing agent, which is critical for lowering the costs of full-scale field deployment of this technology.

ACKNOWLEDGMENT

We thank the International Scientific and Technology Center (Moscow, Russia) for financial support of the present research.

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Received March 2000
Revised November 2000

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