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Hyoyeol Han[a]; You-Jin Lee[a]; Seong-Hye Kim[a]; Ji-Won Yang[a]
[a] Department of Chemical and Biomolecular Engineering, KAIST, Yuseong-gu, Daejeon, Republic of Korea

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Electrokinetic Remediation of Soil Contaminated with Diesel Oil Using EDTA–Cosolvent Solutions

Hyoyeol Han, You-Jin Lee, Seong-Hye Kim, and Ji-Won Yang
Department of Chemical and Biomolecular Engineering, KAIST,
Yuseong-gu, Daejeon, Republic of Korea

Abstract: The effect of a chelating agent (EDTA), cosolvent (n-propanol), and non-ionic surfactants (Tergitol 15-S-7 and Tergitol NP-10) as additives in the purging solution for electrokinetic remediation of soil contaminated with diesel oil was investigated. It was found that EDTA functioned as both an electrolyte and a desorbent for hydrophobic organic contaminants. Addition of surfactant with EDTA was not effective for diesel oil transport and removal. The addition of n-propanol and EDTA enhanced hydrocarbon removal efficiency, especially for aromatic hydrocarbons. There was no significant enhancement of removal by use of a combination of EDTA, surfactant and n-propanol relative to the use of EDTA and n-propanol together.

Keywords: Cosolvent, diesel oil, EDTA, surfactant

INTRODUCTION

Contamination of soil with petroleum hydrocarbons such as diesel oil is widespread around sites such as petrol stations, industrial areas and military bases. Diesel oil consists of aliphatic and aromatic hydrocarbons with $10 \sim 24$ carbon atoms. These molecules readily adsorb to soil particles due to their hydrophobicity. It is more difficult to remove diesel oil than gasoline from soil because of the low volatility, high viscosity and low solubility

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Address correspondence to Ji-Won Yang, Environmental Remediation, Department of Chemical and Biomolecular Engineering, Korea Advanced Institute of Science and Technology, 373-1 Guseong-dong, Yuseong-gu, Daejeon 305-701, Republic of Korea. E-mail: jwyang@kaist.ac.kr

(about 5 mg/L in water) of the former (1,2). Petroleum oil contamination of soil can cause secondary pollution through the gradual release of contaminants to subsurface water and associated environments.

Electrokinetic (EK) soil remediation is a unique *in situ* technique that can be applied to low permeability soil. Early studies on EK remediation focused on metal and polar organic contaminants, but there is growing interest in the removal of hydrophobic organic contaminants (HOCs) from soil using this technique.

The main mechanism for HOC removal by electrokinetics is electroosmosis (3,4). Excess cations move towards the anode under a direct current, causing electroosmotic flow of soil pore water. Contaminants are solubilized and mobilized by solution in pore water, and are then transported by electroosmotic flow. According to the Helmholtz–Smoluchowski equation (5), $V_{eo} = (D\varepsilon_0\zeta/\eta)\ E_x$, the average velocity of electroosmotic flow (V_{eo}) is proportional to the electrical gradient (E_x), the zeta potential (ζ) and the dielectric constant (D), and inversely proportional to the pore fluid viscosity (η) where the permittivity of vacuum (ε_0) is 8.854×10^{-12} C/V m.

The zeta potential is the only parameter that changes during the EK treatment. It depends on several factors such as the charge on the particle surface and the conductivity of the pore solution (6). In EK removal of HOCs the control of zeta potential is important because the amount of electroosmotic flow increases with zeta potential.

In the EK system, key factors for HOC removal are the solubilization and mobilization capacity of the purging solution and the amount of electroosmotic flow. There have been several approaches to improving solubilization and mobilization of HOCs by addition of reagents to the purging solution (7–9). Surfactants or cosolvents have been used in pump-and-treat remediation to enhance mass transportation of non-aqueous phase liquids (NAPLs) or subsurface sorbed contaminants (10). There have been recent studies of the application of surfactants and cosolvents in EK remediation for the removal of low polarity organic contaminants.

Surfactants can enhance the solubility of organic contaminants in the aqueous phase by solubilizing hydrophobic organic contaminants into micelles. However, the adsorption of surfactant onto soil or soil organic materials can lower the removal efficiency of organic contaminants by adsorbing the organic materials onto hydrophobic phase of surfactant layer. Anionic surfactants show relatively lower adsorption due to their negative charge, but interfere with electroosmotic flow. Cationic surfactants may be toxic to microorganisms in the soil. Non-ionic surfactants are appropriate for the EK process because their neutral charge does not affect electroosmotic flow, and many are biodegradable.

NAPL remediation with cosolvents including alcohols and ethyl acetate has been reported (10). These cosolvents can increase the solubility of HOCs into the aqueous phase, enhancing their removal efficiency. It has been reported that the combined use of a cosolvent and surfactant could be effective in remediation of HOCs because the cosolvent increases the solubility of the surfactant, and reduces retention of the surfactant in the soil.

The use of chelating agents in heavy metal removal has been studied, but they may also have an innovative application in the EK remediation of HOCs from contaminated soil. It was reported that chelating agents enhance electroosmotic flow compared with a common electrolyte such as $CaCl_2$ (11). Adsorption of a negatively charged chelating agent onto soil particles and the dissolution of minerals or ions that suppress the original negative charge of clay can cause the zeta potential to increase (12). Chelating agents also desorbed HOCs by changing the phase properties of the soil organic materials (13).

In this study we investigated the effect of addition of a chelating agent, cosolvent and surfactants as purging solution additives on EK remediation of soil contaminated with diesel oil.

MATERIALS AND METHODS

Materials

Tergitol 15-S-7 and Tergitol NP-10 were used as non-ionic surfactants, n-propanol was used as a cosolvent, and EDTA (ethylenediaminetetraacetic acid tetrasodium salt dehydrate) and NaCl were added as an electrolyte. It was expected that EDTA would act as a HOC desorbent (13,14).

Soil was sampled from a petrol station in Busan, Korea. The air-dried soil was sieved (2 mm sieve size) and artificially contaminated by commercial diesel oil. After 2 years of aging, the total petroleum hydrocarbon (TPH) concentration of the soil sample was about 6800 ppm. Properties of the soil are listed in Table 1.

Batch Test for Screening of Additives

Preliminary batch tests were performed to select a suitable cosolvent and non-ionic surfactants. Soil (5 g) was mixed with about 5 g of anhydrous Na_2SO_4 in a 50 mL glass vial, and 25 mL of cosolvent and/or surfactant solution was added. Then the vial was vertically shaken for 3 h at 30 rpm to facilitate diesel desorption and solubilization.

Table 1. Soil properties

Property	Result
Water content (%)	
Particle size fraction (%) (ASTM D422)	
Sand	90
Silt	3.7
Clay	6.3
pH (ASTM D4927)	7.07
Organic Content (%) (ASTM D2974)	3.07

Diesel removal was determined by analyzing the residual soil diesel oil content. To separate the soil and solution the vials were centrifuged for 10 min at 2000 rpm. About 5 g of anhydrous Na_2SO_4 was added to the separated soil to remove moisture. To extract the residual diesel from the soil, 25 mL of n-hexane was added, the vial was vertically shaken for 12 h at 30 rpm and centrifuged for 10 min at 2000 rpm, and the n-hexane phase was removed. The extraction procedure was repeated, and the n-hexane fractions were combined and condensed to 5 mL in a rotary evaporator. Silica gel (0.5 g) was added to the extract to remove suspended particles and analytical interferences.

The extract was analyzed by high performance gas chromatography (HPLC) using a HP 6890 series II GC-FID equipped with a HP-5 capillary column with helium as the carrier gas (flow rate 1 mL/min). A 2 μL extract sample was injected at 50°C and held at this temperature for 2 min. The temperature was then increased at a rate of 10°C/min up to 320°C, and held at this level for 20 min. The injector and detector temperatures were 300°C and 320°C, respectively.

Electrokinetic Testing Procedure

The soil contaminated with diesel oil was mixed with deionized water in a stainless steel pan then put into a soil chamber (4 cm × 4 cm × 10 cm). The soil was packed firmly into the chamber and pressurized manually.

At the start of the EK procedure, the anode chamber was filled with solution and the cathode chamber was filled with deionized water. Dimensionally-stable anode electrodes (DSA®; 4 cm × 4 cm × 10 cm; DOES Co. Ltd, Korea) were used at the anode and the cathode. A constant current (10 mA) was applied for the duration of EK procedure. The electrical potential gradient and the volume of electroosmotic flow were measured daily. After completion of the procedure the soil in the chamber was divided into six portions which were used for residual diesel oil analysis. To assess diesel oil removal we used the same procedure as that used in the batch test for analysis of the residual concentration of diesel oil in the soil.

The residual diesel oil in soil was analyzed using a Waters HPLC equipped with a Petro XL column and a PDA detector. The analyses were performed at 210 nm (aliphatic hydrocarbons) and 254 nm (aromatic hydrocarbons), and n-hexane was used as the mobile phase with a flow rate of 1.0 mL/min (15).

To assess the effectiveness of the chelating agent as an electrolyte and HOC desorbent, EK experiments were undertaken with treatments comprising

1. 0.005 M NaCl,
2. 0.005 M EDTA, and
3. 0.01 M EDTA (tests 1–3, respectively; Table 2).

The effectiveness of the cosolvent and surfactants was tested in 2 sets of EK experiments with treatments comprising of

1. EDTA,
2. EDTA + non-ionic surfactant,
3. EDTA + cosolvent, and
4. EDTA + a non-ionic surfactant +cosolvent.

Table 2. Conditions of electrokinetic test

Set	Test	Solution	Current (mA)	Duration (d)
1	1	NaCl 0.005 M	10	14
	2	EDTA 0.005 M	10	14
	3	EDTA 0.01 M	10	14
2	4	EDTA*	10	25
	5	EDTA* + 10 g/L Tergitol 15-S-7	10	25
	6	EDTA* + 50% n-propanol	10	25
	7	EDTA* + 10 g/L Tergitol 15-S-7 + 50% n-propanol	10	25
3	8	0.005 M EDTA	10	25
	9	0.005 M EDTA + 10 g/L Tergitol NP-10	10	25
	10	0.005 M EDTA + 50% n-propanol	10	25
	11	0.005 M EDTA + 10 g/L Tergitol NP-10 + 50% n-propanol	10	25

*Concentration changed from 0.005 M to 0.01 M from the 11th day.

In the first set of experiments (tests 4–7, respectively; Table 2) Tergitol 15-S-7 was used as the surfactant, and the initial EDTA concentration was 0.005 M. However, the concentration of EDTA was doubled to 0.01 M from the 11th day of the procedure due to failure of the electroosmotic flow. In the second set of experiments (tests 8–11, respectively; Table 2) Tergitol NP-10 was used as the surfactant, and the EDTA concentration was 0.005 M. The conditions and treatments in the EK experiments are shown in Table 2.

RESULTS AND DISCUSSION

Preliminary Batch Test for Screening of Additives

Tergitol 15-S-7 and Tergitol NP-10 were selected as non-ionic surfactants because of their effectiveness in removal of diesel oil from soil (Fig. 2). n-Propanol was selected as a cosolvent because it showed good efficiency in diesel oil removal in the preliminary batch tests (Fig. 3).

Figure 4 shows the results of preliminary batch tests to investigate the effect of EDTA on removal of HOCs from soil. NaCl showed no significant diesel oil removal because the solubility of the oil was very low. EDTA solutions of 0.005 M and 0.01 M showed 35% and 38% HOC removal, respectively. These results imply that EDTA desorbed organic contaminants from the soil. Yang et al. (13) reported that chelating agents can desorb HOCs from soil, and proposed a mechanism involving chelating agent removal of metal ions, such that organic macromolecules bound to the mineral surface via the metal ions are released into the aqueous phase. The removal of metal ions, which function as cross-linking

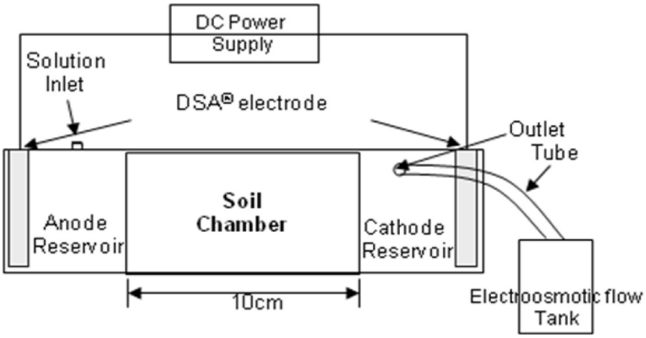


Figure 1. Schematic diagram of electrokinetic reactor.

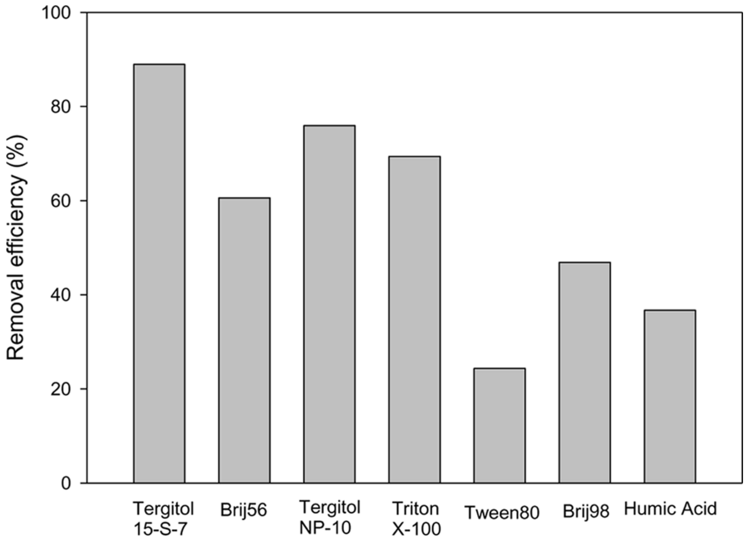


Figure 2. Diesel oil removal efficiency in batch tests of non-ionic surfactants and humic acid.

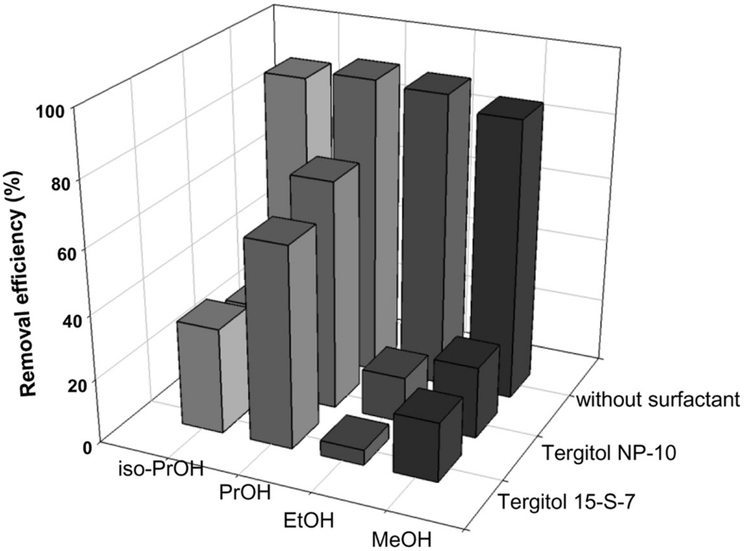


Figure 3. Diesel oil removal efficiency in batch tests for cosolvent selection.

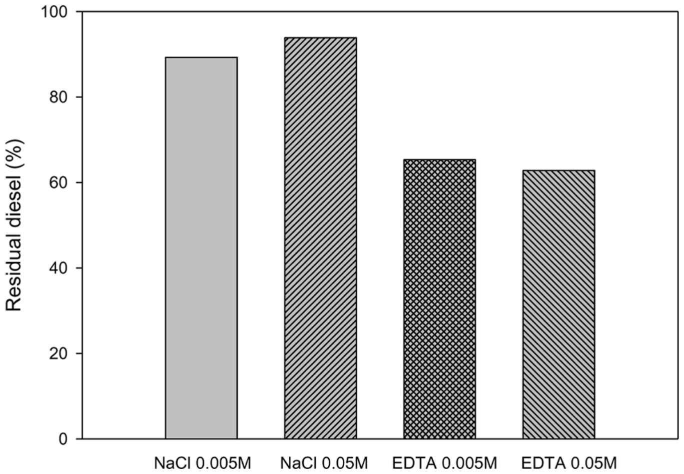


Figure 4. Diesel oil removal efficiency in EDTA and NaCl batch tests.

agents in soil organic matter (SOM), changes the surface properties of SOM to a state whereby HOCs can more easily diffuse into solution.

Effect of the Chelating Agent

After initiation of the EK procedure, the electrical potential gradient decreased during the first day but thereafter increased gradually (Fig. 5). When the gradient plateaued, the electrical potential gradient in the NaCl treatment was about 8 V/cm, while in the 0.005 M and 0.01 M EDTA treatments the electrical potential gradient was reduced by 3 ~ 4 V/cm. It is assumed that, with EDTA as an electrolyte, the electrical resistance was reduced because the multivalent charge of EDTA ($EDTA^{4-}$) enhanced the electrical conductivity of the solution and the soil compared to NaCl. The electrical potential decreased with increased EDTA concentration.

Figure 6 shows that the greatest electroosmotic flow occurred with 0.01 M EDTA. Excess ions in the EDTA solutions could increase electroosmotic flow by raising electrical conductivity of the soil and pore solution. It has been reported that adsorption of a negatively charged chelating agent onto soil particles, and consequent restoration of the original clay negative charge by the removal of minerals or ions, increased the zeta potential, enabling enhanced electroosmotic flow (12). Electroosmotic flow was also positively correlated with pH of electroosmotic flow: with EDTA the pH was higher (10–12) than with NaCl (8–10).

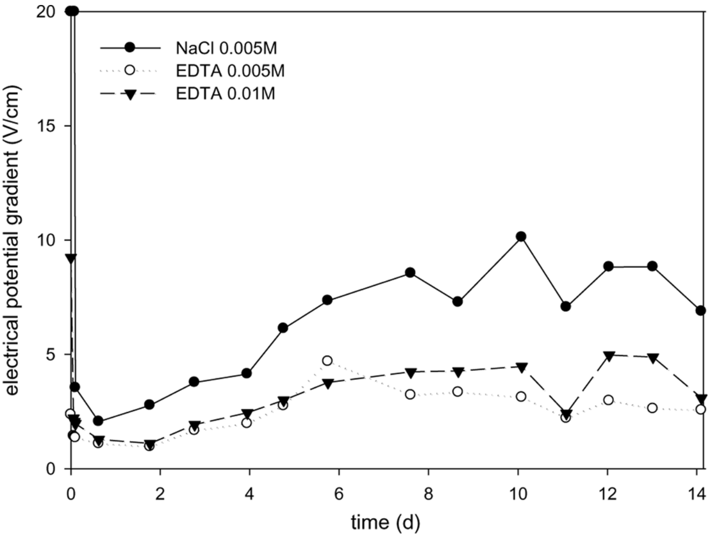


Figure 5. Electrical potential gradients in electrokinetics experiments with EDTA and NaCl.

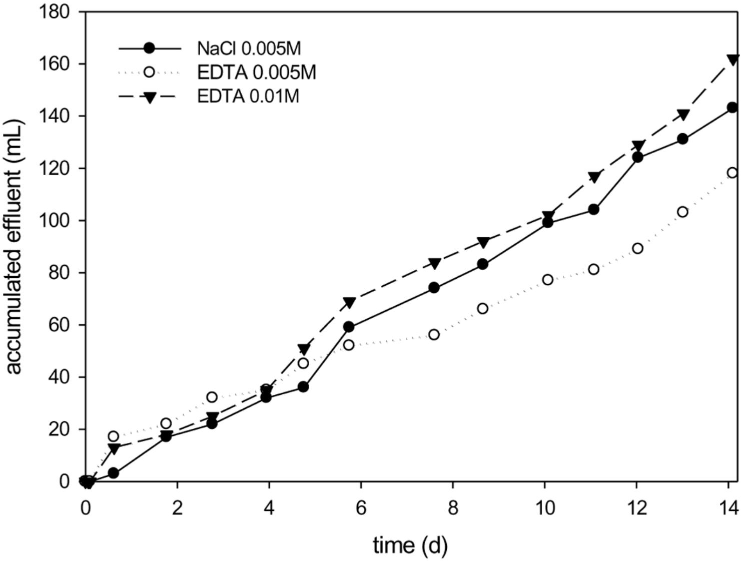


Figure 6. Accumulated electroosmotic flow in electrokinetics experiments with EDTA and NaCl.

Soil pH after EK was low near the cathode (pH 3–4) and increased toward the anode (pH 7–8) in each experiment. However, in these EK tests there was no difference in electroosmotic flow between 0.005 M EDTA and 0.005 M NaCl. Figure 7 shows the residual diesel oil in soil after using chelating agents as an electrolyte and HOC desorbent. Poor

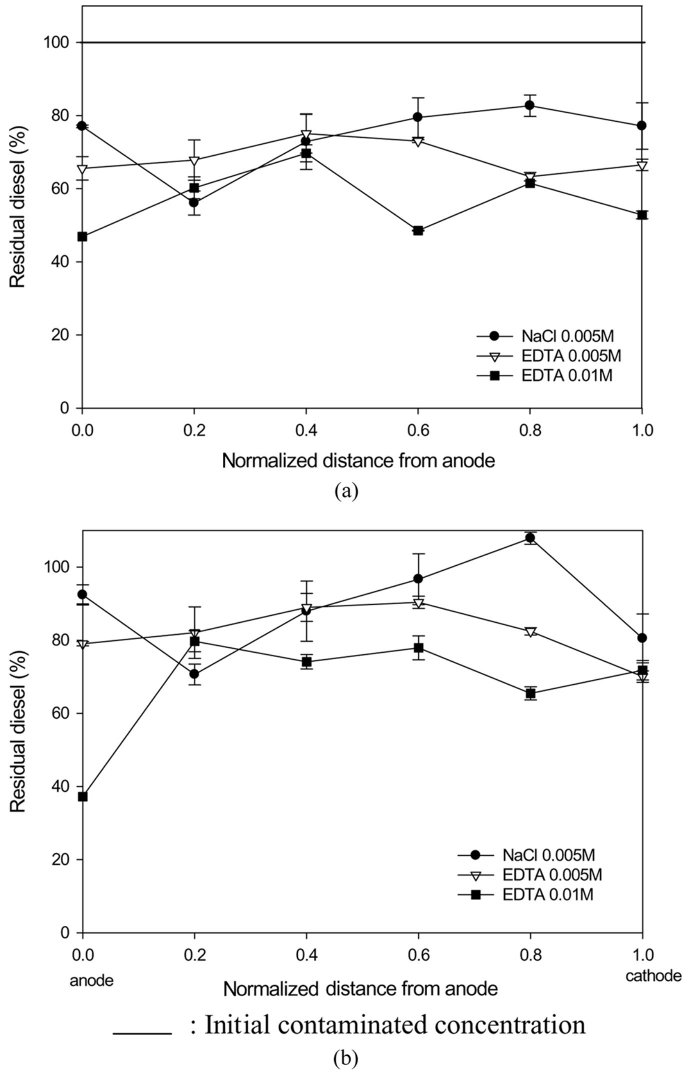


Figure 7. Residual diesel oil in soil after electrokinetics experiments with EDTA and NaCl: (a) aliphatic hydrocarbons and (b) aromatic hydrocarbons.

transport and removal occurred with NaCl due to the low solubilization and mobilization of HOCs in water, with only 33% of aliphatic hydrocarbons and 18% of aromatic hydrocarbons being removed. It seems that HOCs weakly adsorbed onto soil particle surfaces were washed out by the electroosmotic flow. The removal efficiency of aromatic HOCs, which are relatively easily adsorbed, was lower than that of aliphatic HOCs. Deposition of aromatic HOCs occurred in the region of the cathode.

The use of EDTA resulted in greater transport and removal of HOCs than with NaCl, and the removal efficiency increased with increasing EDTA concentration. With 0.01 M EDTA, 42% of aliphatic contaminants and 31% of aromatic contaminants were removed after 14 days of EK operation. It was found that desorption of HOCs was enhanced by EDTA in the EK system, as shown with the batch removal.

The Effect of Cosolvent and Non-Ionic Surfactants

The electrical potential gradient (Fig. 8) tended to decrease at the start of the procedure, and then increased with time during the experiment. In the treatments with EDTA and the surfactant, the electrical potential gradient gradually increased. It is assumed that the electrical resistance of the solution and soil increased because the non-ionic surfactant carries no charge.

In treatments involving n-propanol as cosolvent, the electrical potential gradient increased markedly with fluctuations up to a high value, whereas in treatments lacking n-propanol the electrical potential gradient remained steady or gradually increased. The initial fluctuation of electrical gradient in the presence of n-propanol can be explained by alcohol reactions with soil minerals or electrochemical reactions in the electrokinetics such that the electrical potential gradient was unstable. A high electrical potential gradient could be caused by the low dielectric constant of n-propanol.

Figure 8 shows the accumulated electroosmotic flow during EK remediation. The flow varied depending on the EDTA concentration. In the experiment where the EDTA concentration was increased from 0.005 M to 0.01 M on the 11th day of operation, the electroosmotic flow increased markedly (Fig. 9a). The pH of the electroosmotic flow was about 9–13, and showed the same tendency in each set of experiments.

The addition of the surfactants Tergitol 15-S-7 (Fig. 9a) or Tergitol NP-10 (Fig. 9b) reduced the electroosmotic flow. The viscosity of the solution was approximately 1 mPa s. This indicates that the addition of surfactant did not affect the viscosity of the solution. The zeta potential of soil particles declined more in solutions containing surfactant than in

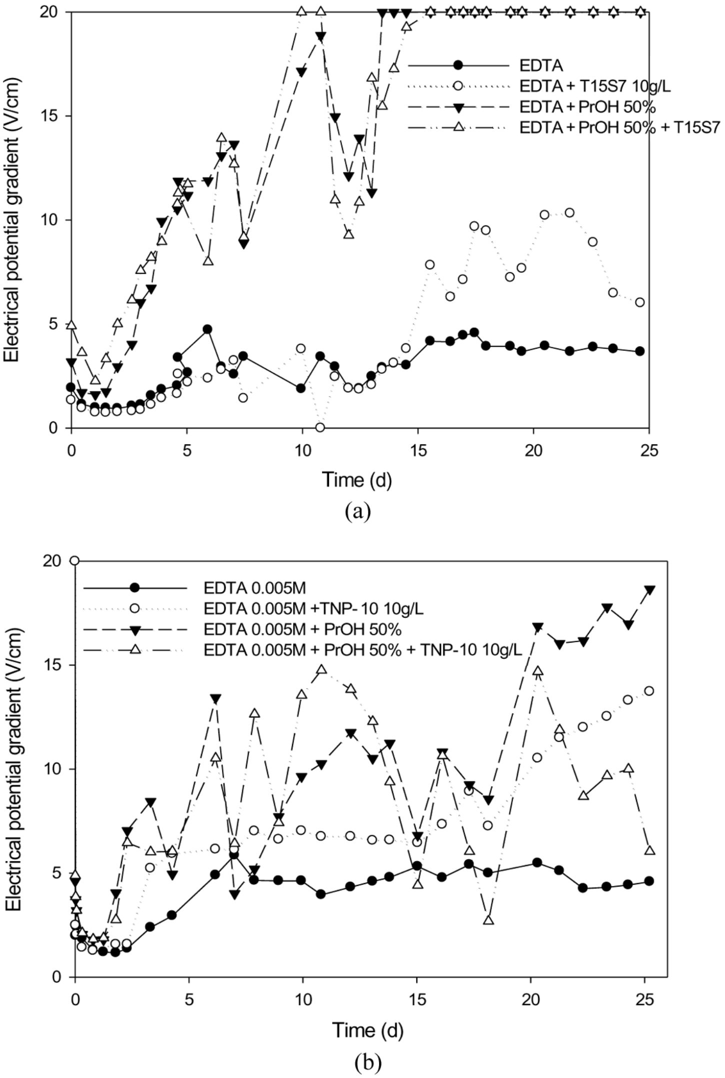
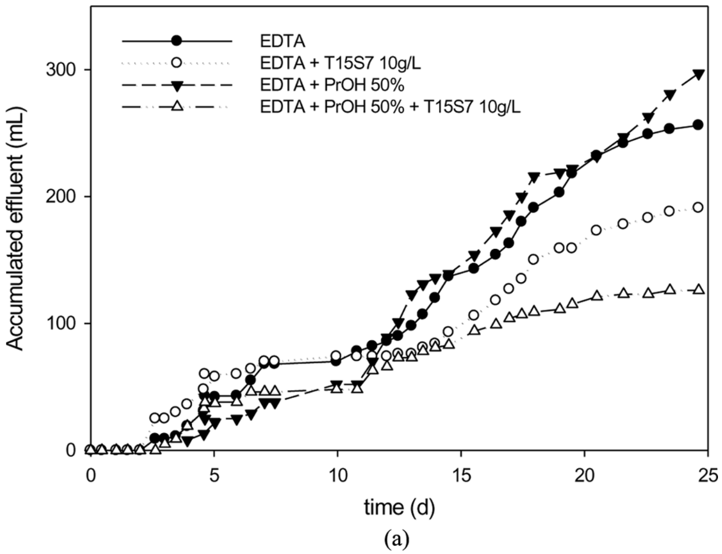


Figure 8. Electrical potential gradient in electrokinetics experiments with cosolvent and surfactant: (a) experiment 1, (b) experiment 2.

EDTA solution alone. It appears that electroosmotic flow, which is proportional to zeta potential and inversely proportional to viscosity, declined because of a reduction in the zeta potential in that solution. However, little effect of non-ionic surfactant solutions on the zeta potential has been in previous studies (16).



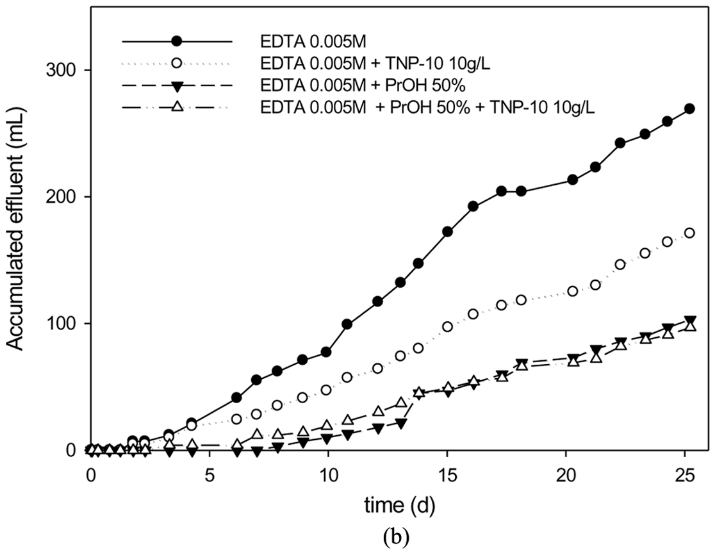


Figure 9. Accumulated electroosmotic flow in electrokinetics experiments with cosolvent and surfactant: (a) experiment 1, (b) experiment 2.

The presence of n-propanol significantly reduced electroosmotic flow compared with EDTA alone. It seems that the low dielectric constant of n-propanol directly affected the electroosmotic flow, and may have had an indirect effect by decreasing the dissolution of minerals and metal ions. An increase in viscosity to 2.5 mPa · s with the addition of cosolvent also contributed to a reduction in electroosmotic flow.

Figure 10 shows residual diesel oil in soil after EK treatment. With EDTA alone (tests 4 and 8), there was significant removal of both aliphatic and aromatic hydrocarbon contaminants. In tests where the EDTA concentration was changed from 0.005 M to 0.01 M (test 4), 59% of aliphatic hydrocarbons and 33% of aromatic hydrocarbons were removed. In test 8 (EDTA concentration 0.005 M), 48% and 45% of aliphatic and aromatic compounds were removed, respectively. The removal of aliphatic hydrocarbon contaminants was slightly higher than that of aromatic contaminants. Similar results were reported by Subramaniam et al. (14).

The mobilization of contaminants near the anode region was improved with the use of 10 g/L Tergitol 15-S-7 (Fig. 10a). It is likely that near the anode, where the strongest interactions occur among the solution, contaminant and soil, the surfactant caused a synergistic effect between enhanced diffusivity by EDTA and HOC micelle formation. However, no actual contaminant removal occurred because of contaminant deposition in the middle section. It seems that surfactant adsorption onto soil particles hindered contaminant diffusion from the SOM. In the presence of 10 g/L of Tergitol NP-10 (test 9; Fig. 10b), contaminant deposition was not observed but both aliphatic and aromatic contaminant removal efficiencies were lower than for EDTA alone (Figs. 10c, d).

The addition of n-propanol (tests 6 and 10) resulted in improved removal of aromatic contaminants, but had little effect on removal of aliphatic contaminants. The addition of n-propanol contributed to the removal of aromatic contaminants by increasing the solubilization and mobilization of HOCs. It is likely that the electroosmotic flow is an important factor in the removal of aliphatic hydrocarbons, which are relatively soluble and less well adsorbed to soil than aromatic hydrocarbons. The reduced electroosmotic flow caused by the addition of n-propanol was not sufficient to enhance mobilization and transport of aliphatic hydrocarbons.

No contaminant deposition was observed in the presence of 10 g/L surfactant and 50% n-propanol (tests 7 and 11). It is likely that surfactant adsorption did not occur because n-propanol addition reduced surfactant adsorption onto soil particles by enhancing surfactant solubilization. Alcohols also prevent surfactant adsorption onto soil particles by filling soil surface sites (4). With Tergitol 15-S-7 and n-propanol (test 7), 69% and 81% of aliphatic and aromatic contaminants were removed, respectively. With Tergitol NP-10 and n-propanol (test 11), the removal efficiency of aliphatic contaminants was 52%, and that of aromatic contaminants was 52%. The combination of the surfactant and n-propanol

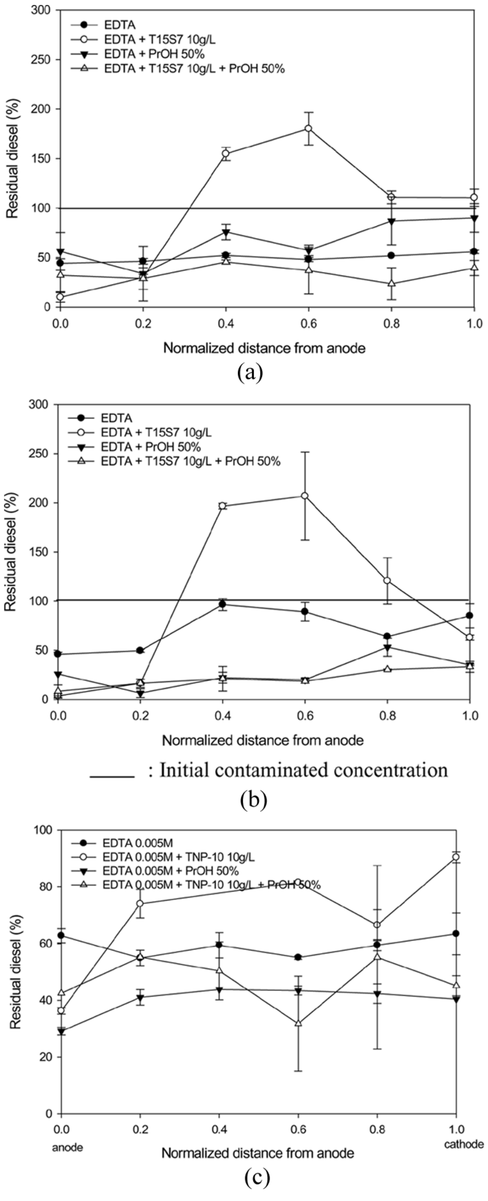
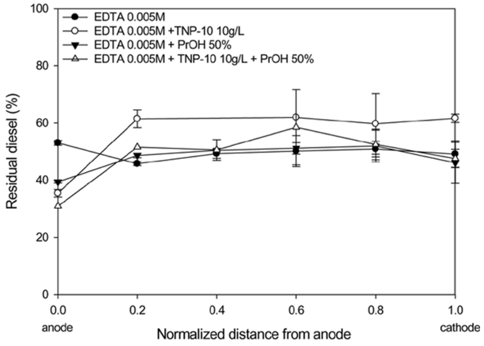


Figure 10. Residual diesel oil in soil after electrokinetics experiments with cosolvent and surfactant: (a) experiment 1; aliphatic hydrocarbons, (b) experiment 1; aromatic hydrocarbons, (c) experiment 2; aliphatic hydrocarbons, and (d) experiment 2; aromatic hydrocarbons.



(d)

Figure 10. Continued.

did not markedly enhance the removal efficiency relative to n-propanol alone. If the main mechanism of HOC removal by cosolvent is mobilization, the addition of the surfactant may be ineffective in enhancing diesel oil removal. St-Pierre et al. obtained similar results (17). Another explanation for reduced electroosmotic flow is that insufficient synergetic interactions occur among the solution, soil and contaminant, such that enhanced HOC solubilization by n-propanol, and micelle formation by the surfactant, does not occur.

CONCLUSIONS

The electrokinetic removal of diesel oil from soil was investigated using agents (EDTA, n-propanol, Tergitol 15-S-7 and Tergitol NP-10) to enhance contaminant solubilization and mobilization.

EDTA alone resulted in significant enhancement of removal of aliphatic and aromatic hydrocarbon contaminants, because EDTA changed the soil surface properties enabling HOCs to easily diffuse into solution. Aliphatic hydrocarbons were more readily desorbed than aromatic hydrocarbons.

A combination of surfactant and EDTA resulted in less removal and poorer transport of contaminants than EDTA alone, as the surfactant apparently caused contaminant deposition. Tergitol NP-10 showed better performance than Tergitol 15-S-7, the latter being associated with significant deposition.

With the combination of n-propanol and EDTA, the low dielectric constant of n-propanol and the increased viscosity significantly reduced the electroosmotic flow. However, the removal efficiency was enhanced (especially for aromatic contaminants) because the addition of n-propanol increased the solubilization and mobilization capacity. Enhanced electroosmotic flow can improve diesel oil removal from soil by increasing interactions among the solution, contaminant and soil, and is needed to optimize the cosolvent concentration for effective electrokinetic removal of diesel oil using a cosolvent.

There was insignificant enhancement of contaminant removal using a combination of surfactant, n-propanol and EDTA relative to the combined use of n-propanol and EDTA. When the main mechanism of HOC removal is by mobilization with the cosolvent, the addition of the surfactant is not effective.

The application of chelating agents, such as EDTA, to electrokinetics is worthy of further investigation as they function as electrolytes, HOC desorbents, and metal contaminant chelating agents. The addition of a cosolvent can significantly enhance the removal of HOCs. The results of this study suggest that HOCs and/or metal contaminated sites can be remediated using electrokinetics in combination with a chelating agent and cosolvent.

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