Removal and degradation of phenol in a saturated flow by in-situ electrokinetic remediation and Fenton-like process

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Abstract

In this laboratory study, a sandy loam soil saturated with phenol solution was treated by in-situ electrokinetics-Fenton process incorporated with a permeable reactive wall of scrap iron powder (SIP). The soil was contaminated and saturated with aqueous phenol solution of 90–115 mg/kg in concentration. It was then placed in a soil cell. The soil cell was assembled with an anode reservoir and a cathode reservoir at its ends. A bed of SIP (1.05–32.69 g) was inserted in the soil cell at a distance of 5 cm from the anode reservoir compartment. For the test runs, 0.3% H_2O_2 was used as the anode reservoir fluid, whereas de-ionized water was used as the cathode reservoir fluid. An electric gradient of 1 V/cm was applied to enhance the saturated flow in the soil cell for a period of 10 days. Experimental results have shown that the electroosmotic (EO) flow quantity decreased as the amount of SIP increased. This phenomenon was in good agreement with the results showing the value of EO permeability increased with a decreasing amount of SIP. Results also showed that throughout the test period the cumulative, consumed mass of H_2O_2 in the anode reservoir increased as the amount of SIP decreased. On the other hand, the cumulative, increased mass of phenol in the cathode reservoir was found to increase with a decreasing amount of SIP. Meanwhile, the residual phenol concentration in the soil cell was found to decrease with a decreasing amount of SIP. When 1.05 g scrap iron powder was used, an overall removal and destruction efficiency of phenol of 99.7% was obtained. Therefore, it is evident that an in-situ combined technology of electrokinetic remediation and Fenton-like process is capable of simultaneously removing and degrading the phenol in a saturated flow. © 1999 Elsevier Science B.V. All rights reserved.

Keywords: Remediation; Electrokinetic; Fenton; Iron powder; Phenol

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1. Introduction

Phenols are among the 129 priority pollutants listed by the U.S. EPA. However, compounds of phenol are often used by various industries and found in various consumer goods. Therefore, contamination of soil and groundwater by phenols are common in the world including Taiwan.

According to a U.S. EPA report [1], a majority of the 259 completed North American innovative remediation technology demonstration projects were conducted for cleaning up the sites contaminated by various organic pollutants. Of which, 5% of the above projects employed the technology of chemical oxidation.

It has been reported that Fenton process is very effective for the degradation of various organics in the presence of wastewater and soil [2–8]. Conventionally, H_2O_2 and Fe^{2+} are used in the Fenton process for generating hydroxyl radicals to chemically destruct organic pollutants including phenol. Recently, a Fenton-like reaction using zero-valent iron instead of Fe^{2+} has been found to be effective in degradation of organic pollutants in wastewater and soil as well [9,10]. In addition, zero-valent iron has also been successfully employed as a material in permeable reactive walls for the remediation of contaminated groundwater [11–13]. Electrokinetic remediation (EK process) is an innovative technology that shows lots of promise and is receiving lots of attention worldwide [14–21]. By applying an electric field EK process would create an electroosmotic flow enhancing the removal of subsurface contaminant(s). Therefore, it is likely to combine the concepts of EK process, permeable reactive wall of iron powder, and Fenton-like reaction for the chemical destruction of organic contaminant(s) in the subsurface [22].

The main objective of this work was to determine the feasibility of simultaneously employing EK process and Fenton-like process (designated as ''EK-Fenton process'' in this study) for the remediation of a soil saturated with an aqueous solution of phenol. Therefore, effects of the amount of scrap iron powder used and treatment time on electroosmotic permeability and removal and destruction efficiency of phenol were also studied. The reaction mechanisms of the EK-Fenton process and possible pathways of phenol degradation in this process are worth studying. However, they are beyond the scope of the present investigation.

2. Experimental

2.1. Materials

2.1.1. Soil

The original soil sample was obtained from the topsoil layer (0–30 cm in depth) of a farmland nearby a local petroleum refinery. Tree roots and debris were first removed from the soil sample. After it was dried in the air, the soil sample was sieved to pass through a 10-mesh screen. The fraction finer than 2 mm was properly stored for later analyses and experiments. The soil sample (designated as Soil No. 1) studied was categorized as a sandy loam with various characteristics listed in Table 1.

Table 1
The characteristics of original soil

Item determined	Value
Texture	Sandy loam
Particle size analysis	< 2 μm: 1.97%
	$2 \sim 50$ μm: 23.33%
	$50 \sim 2000$ μm: 74.7%
Density	2.45 g/cm^3
BET surface area	3.94 m^2/g
pH	4.67
Loss on ignition	2.58%
Cation exchange capacity	9.60 meq/100 g
Organic content	1.25%
Total iron content	2622 mg/kg
Point of zero charge	≈ 1.3

2.1.2. Scrap iron powder

The specimen of scrap iron powder was obtained from a local iron processing plant. The scrap iron powder was the residue due to the elongation of iron wires. The only mineral species identified in its X-ray analysis is magnetite (Fe_3O_4).

2.2. Apparatus

Fig. 1 shows a schematic diagram of the experimental set-up for the EK-Fenton process used in this study. It consists of three components: anode reservoir, soil cell (or soil column), and cathode reservoir. These components are made of Pyrex glass. The
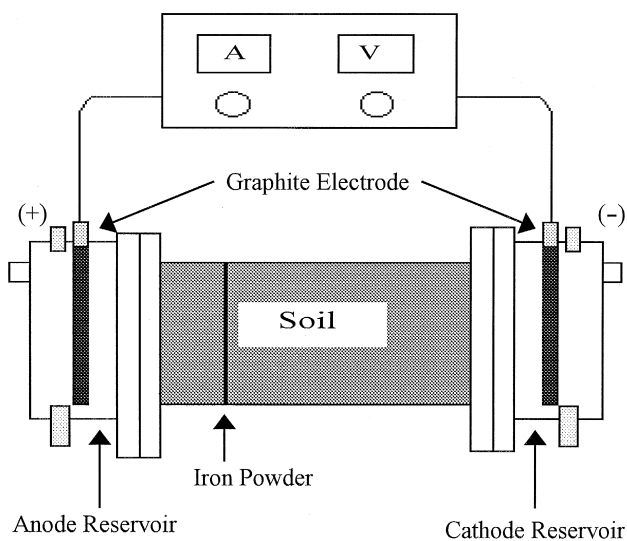

Fig. 1. A schematic diagram showing the experimental set-up for EK-Fenton process.

cylindrical soil cell has a length of 20 cm and a diameter of 5.5 cm. Each electrode reservoir has a length of 5 cm and a diameter of 7.5 cm. The soil specimen saturated with an aqueous solution of phenol was placed in the soil cell. Scrap iron powder was embedded in the soil cell at a distance of 5 cm from the anode reservoir compartment serving as a permeable reactive wall for the Fenton-like reaction. At each end of the soil cell, a set of filter media (a Whatman GF/F fiberglass filter paper was sandwiched in two pieces of nylon clothes) was used to separate the soil particles from entering the electrode reservoir compartments. Graphite electrodes, partly immersed in the electrode reservoir fluids, were connected with a direct-current power supply. The graphite electrode has an electric resistance of 1200 $\mu\Omega$/cm, a flexible strength of 650 kg/cm^2, and a compressive strength of 1300 kg/cm^2.

2.3. Methods

2.3.1. Preparation of the test specimen

In this work, the artificially contaminated soil was prepared by mixing the pretreated, original soil with a phenol stock solution of 800 mg/l. First, 212.5 ml of phenol solution was added to 850 g of soil. The mixture was then agitated for 3 h using a horizontal rotary shaker at 200 rpm. After mixing, the wet soil mass was placed in the soil cell in a vertical position till it was about 90% full. Then it was compacted using a 2-kg weight from above for sample settlement for 12 h. A bed of scrap iron powder of a desired amount (1.05–32.69 g) was inserted. Additional wet soil mass was placed in the soil cell till its fullness. At this stage, a fraction of the soil specimen was obtained for the determination of initial phenol concentration for each test run.

2.3.2. Operating conditions

In Tests 1–7, 0.3% hydrogen peroxide and ASTM Type II de-ionized water were used as the anode reservoir fluid and cathode reservoir fluid, respectively. In these test runs, an electric gradient of 1 V/cm was then applied to the EK-Fenton remediation system for 10 days unless otherwise specified. For the control run and blank run (i.e. Tests 8 and 9), de-ionized water was used in both electrode reservoirs. Detailed experimental conditions are given in Table 2. The control run was carried out for the performance comparison between EK-Fenton process and EK process alone. The purpose of conducting the blank run was to determine the phenol mass loss due to vaporization during the test period. This must be taken into account when the phenol mass balance is determined.

2.3.3. Determination of phenol concentrations in soil specimens

Each soil specimen was subjected to an extraction procedure so that its phenol concentration could be analyzed by gas chromatography (GC). First, 3–4 g of the soil specimen was placed in a glass vial. After 9 ml of methylene chloride was added to the glass vial, it was tightly sealed and horizontally agitated for 1.5 h at 200 rpm. It was set still for a while until two insoluble layers appeared. We then took 2 μl of the lower layer (i.e. methylene chloride layer) and injected it to GC for analysis.

Table 2
The summary of test program for soil remediation by electrokinetics and Fenton-like process

Type of experiment	Test No.	Soil No.	Experimental condition					
			Pollutant	Electric gradient (V/cm)	Anode reservoir fluid	Cathode reservoir fluid	Catalyst	Treatment time (days)
Test runs	Test 1	No. 1	Phenol	1	$0.3\%H_2O_2$	D.I. Water	32.69 g Scrap Iron Powder	10
	Test 2	No. 1	Phenol	1	$0.3\%H_2O_2$	D.I. Water	16.67 g Scrap Iron Powder	10
	Test 3	No. 1	Phenol	1	$0.3\%H_2O_2$	D.I. Water	8.42 g Scrap Iron Powder	10
	Test 4	No. 1	Phenol	1	$0.3\%H_2O_2$	D.I. Water	4.2 g Scrap Iron Powder	10
	Test 5	No. 1	Phenol	1	$0.3\%H_2O_2$	D.I. Water	2.1 g Scrap Iron Powder	10
	Test 6	No. 1	Phenol	1	$0.3\%H_2O_2$	D.I. Water	1.05 g Scrap Iron Powder	10
	Test 7	No. 1	Phenol	1	$0.3\%H_2O_2$	D.I. Water	2.1 g Scrap Iron Powder	13
Control run	Test 8	No. 1	Phenol	1	D.I. Water	D.I. Water	1.05 g Scrap Iron Powder	10
Blank run	Test 9	No. 1	Phenol	0	D.I. Water	D.I. Water	–	10

A Varian Star 3400 CX gas chromatography with a flame ionization detector (GC/FID) and a capillary column (J&W P/N DB-5; 0.53 μm in diameter and 30 m in length) was used in this work for the determination of phenol concentration.

2.3.4. Determination of residual hydrogen peroxide

In this work, residual hydrogen peroxide was analyzed by an iodometric titration method as described below [23]. (a) Eight milliliters of 6 N H_2SO_4, 10 ml of KI, and three drops of 3% ammonium molybdate were added to 5 ml of hydrogen peroxide sample. (b) Next, 0.025 N $Na_2S_2O_3$ was used to titrate the above mixed solution till the yellowish color disappeared. Three drops of starch indicator was then added. (c) The titration was continued with 0.025 N $Na_2S_2O_3$ till the color disappeared, the end point of the titration. (d) The concentration of hydrogen peroxide was calculated.

3. Results and discussion

3.1. Basic concept of EK-Fenton process

The basic concept of EK-Fenton process is an integration of the individual principles of electrokinetic remediation and Fenton process. Normally, during the EK process, the pH in the neighborhood of the anode would decrease to around 3 and the pH in the neighborhood of the cathode would increase to around 11. From an engineering point of view, the application and design of the EK remediation system must take into account these strong acidic and basic conditions. For the Fenton process to be effective, a pH in the vicinity of 3 is required. Therefore, the present study took advantage of the perfect pH condition at the anode reservoir by providing the Fenton reagent in or close to the anode reservoir. In so doing, hydroxyl radicals would be effectively generated and organic pollutant(s) would be chemically oxidized. In this study, for example, the initial pH at the anode reservoir was 6.84 for Test 6. During the test period, the pH at the anode reservoir dropped to 2.65–3.51. This strong acidic condition did fulfil the basic concept and design of EK-Fenton process employed in this work.

3.2. Effect of the amount of scrap iron powder on the cumulative electroosmotic flow quantity

Fig. 2 shows the cumulative electroosmotic (EO) flow quantity for each of Tests 1–6. Experimental results showed that all EO flows had the same flow direction from the anode toward the cathode and the cumulative EO flow quantity decreased as the amount of scrap iron powder increased. By comparing Test 1 (32.69 g scrap iron powder used) and Test 6 (1.05 g scrap iron powder used), their cumulative EO flow quantities are 87 ml and 278 ml, respectively. This phenomenon can be explained by the results shown in Fig. 3. Generally speaking, the average electric current throughout the test period for Test 6 is much greater than that of Test 1. For a constant applied voltage, a lower electric current is due to a higher electric resistance. Hamed et al. [15] also reported that hydroxyl ions generated by the cathode might react with the neighboring cations and
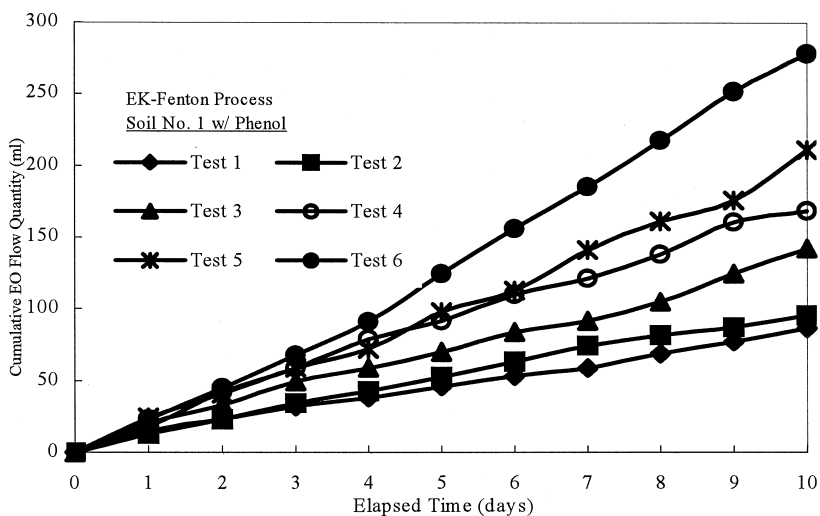
Fig. 2. Cumulative EO flow quantities vs. time for Tests 1–6.

form hydroxide precipitates, which would lower the soil permeability and electric current. The flux of EO flow would then reduce accordingly.

3.3. Effect of the amount of scrap iron powder on the electroosmotic permeability

The effect of the amount of scrap iron powder used on treatment of phenol was also investigated through the determination of the EO permeability (K_e) in each test. Table 3
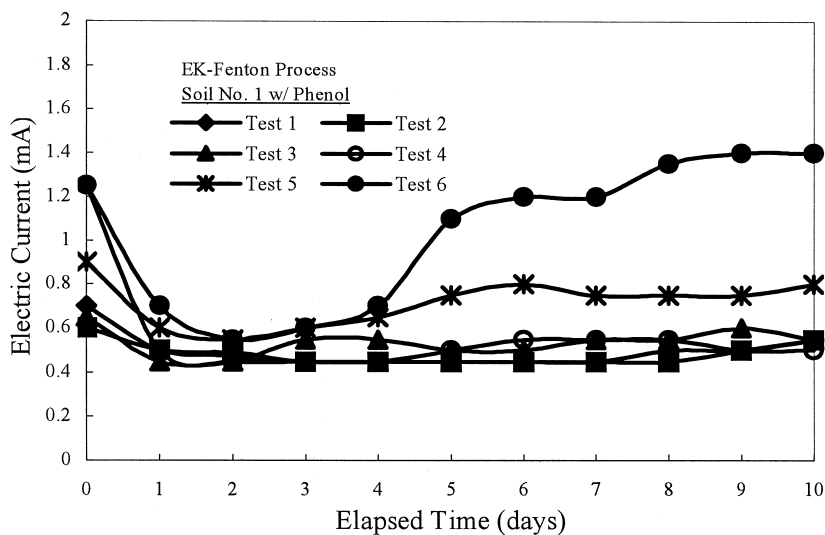

Fig. 3. Electric current vs. time for Tests 1–6.

Table 3
Values of EO permeability (K_c) for Tests 1–8

Test no.	Electric gradient (V/cm)	EO flow rate (ml/s)	Cross section area (cm^2)	EO permeability (cm^2/V-s)
Test 1	1	1.01×10^{-4}	23.75	4.24×10^{-6}
Test 2	1	1.11×10^{-4}	23.75	4.65×10^{-6}
Test 3	1	1.64×10^{-4}	23.75	6.92×10^{-6}
Test 4	1	1.95×10^{-4}	23.75	8.21×10^{-6}
Test 5	1	2.44×10^{-4}	23.75	1.03×10^{-5}
Test 6	1	3.22×10^{-4}	23.75	1.35×10^{-5}
Test 7	1	2.48×10^{-4}	23.75	1.04×10^{-5}
Test 8	1	4.20×10^{-4}	23.75	1.77×10^{-5}

shows that the values of K_c are in the range of 4.24×10^{-6} to 1.77×10^{-5} cm^2/V-s for Tests 1–8. It also indicates that the K_c value increases with a decreasing amount of scrap iron powder used. Normally, a greater removal of pollutant(s) is associated with a greater value of K_c. Comparing the K_c values of Tests 6 and 8, it is obvious that EK process alone has a greater EO permeability than that of EK-Fenton process. However, EK process contributed only removal of phenol, whereas Ek-Fenton process contributed both removal and chemical oxidation of organic pollutant(s). For a longer treatment time of 13 days instead of 10 days, it turned out that Tests 5 and 7 had almost the same K_c value. Acar et al. [16] reported K_c values ranging from 1.40×10^{-5} to 2.90×10^{-5} cm^2/V-s for phenol-contaminated kaolinite treated by EK process using de-ionized water as the processing fluid. Weng et al. [24] also reported K_c values ranging from 5.70×10^{-6} to 1.21×10^{-5} cm^2/V-s for a sandy loam contaminated by phenol using tap water as the processing fluid. For a real clayey soil contaminated with trichloroethylene, K_c values ranging from 5.60×10^{-6} to 1.06×10^{-5} cm^2/V-s were reported by using the Lasagna™ process of bench-top and pilot units [25]. Therefore, the values of K_c obtained in this study are in good agreement with the literature data. Nonetheless, an employment of scrap iron powder did slightly lower the K_c value. Perhaps it is due to the occurrence of certain reactions on the surface of scrap iron powder. The actual determination of these surface reactions is beyond the scope of this study.

3.4. Effect of the amount of scrap iron powder on H_2O_2 consumed in the anode reservoir

The relationships of the cumulative, consumed masses of hydrogen peroxide in the anode reservoir and the treatment time for Tests 1–6 are presented in Fig. 4. It was found that the cumulative, consumed mass of hydrogen peroxide in the anode reservoir increased as the amount of scrap iron powder decreased. It is ascribed to the fact that the quantity of EO flow is greater for the test using a smaller amount of scrap iron powder. Therefore, the anode reservoir fluid (i.e. hydrogen peroxide) in Test 6 would be the most capable one to pass through the permeable reactive wall of scrap iron powder and phenol-saturated soil mass in the cell. In other words, probably the greatest amount of hydrogen peroxide would be catalyzed by scrap iron powder generating the greatest
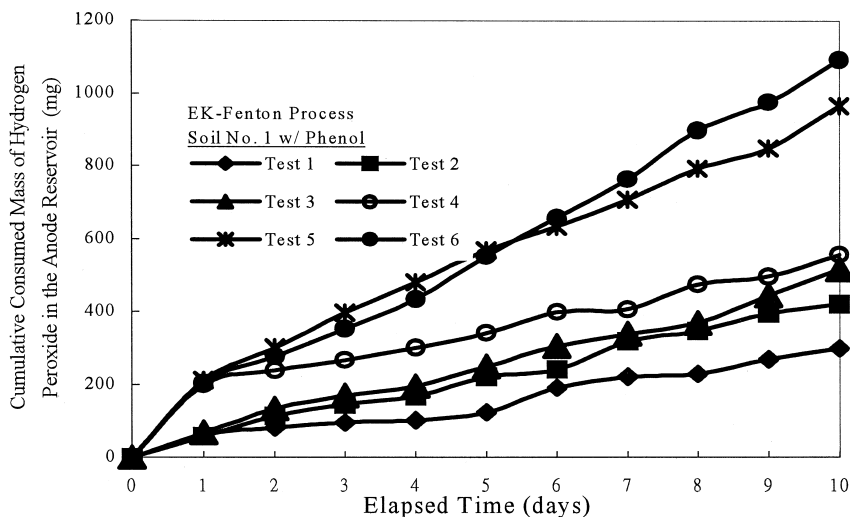
Fig. 4. Cumulative consumed masses of H_2O_2 in the anode reservoir vs. time for Tests 1–6.

amount of hydroxyl radicals in Test 6. For a test period of 10 days, the cumulative, consumed masses of hydrogen peroxide in the anode reservoir for Test 1 and Test 6 are 2.97×10^5 µg and 1.09×10^6 µg, respectively.

3.5. Effect of the amount of scrap iron powder on H_2O_2 increased in the cathode reservoir

Fig. 5 shows the relationships of the cumulative, increased mass percents of phenol in the cathode reservoir and the treatment time for Tests 1–6. The experimental results
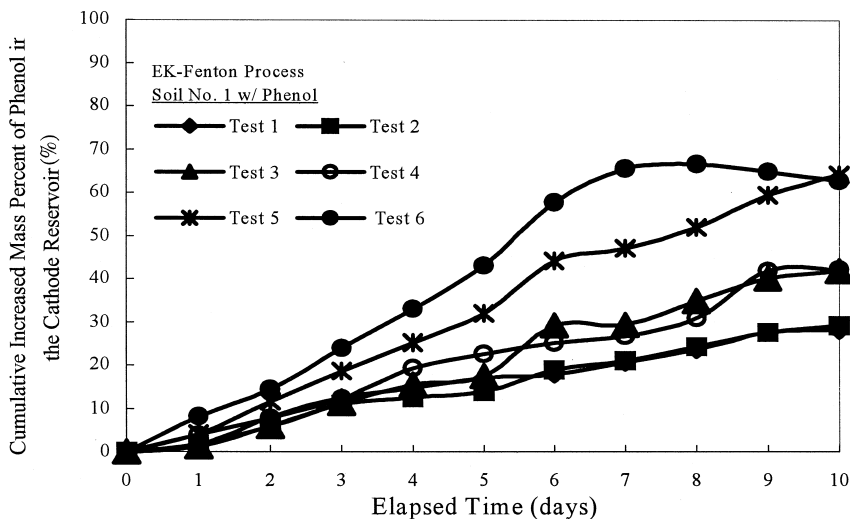

Fig. 5. Cumulative increased mass percents of phenol in the cathode reservoir vs. time for Tests 1–6.

Table 4
Phenol mass balances for Tests 1–9

Test No.	Initial phenol mass in the soil cell (μg)	Residual phenol mass in the soil cell (μg)	Cumulative increased phenol mass in the cathode reservoir (μg)	Phenol mass loss due to vaporization (μg)[a]	Residual phenol fraction in soil (%)[b]	Percent removal (%)[c]	Percent destruction (%)[d]	Percent removal and destruction (%)[e]
Test 1	9.12×10^4	3.93×10^4	2.57×10^4	8.11×10^3	47	31	22	53
Test 2	9.31×10^4	3.79×10^4	2.72×10^4	8.27×10^3	45	32	23	55
Test 3	9.33×10^4	2.95×10^4	3.91×10^4	8.29×10^3	35	46	19	65
Test 4	8.03×10^4	2.42×10^4	3.08×10^4	7.13×10^3	33	42	25	67
Test 5	7.18×10^4	4.68×10^3	4.61×10^4	6.38×10^3	7	70	23	93
Test 6	7.40×10^4	1.82×10^2	4.65×10^4	6.57×10^3	0.3	68.9	30.8	99.7
Test 7	7.61×10^4	4.38×10^2	4.35×10^4	1.13×10^4	0.7	67	32	99
Test 8	7.27×10^4	1.32×10^2	6.60×10^4	6.53×10^3	0.2	99.8	0	99.8
Test 9	8.71×10^4	7.63×10^4	2.98×10^3	7.74×10^3	96	4	0	4

[a] Based on the phenol mass vaporized in the Blank Run, each Test Run must take into account this mass loss accordingly.

[b] Residual Phenol Fraction in Soil = [Residual Phenol Mass in the Soil Cell/(Initial Phenol Mass in the Soil Cell − Phenol Mass Loss Due to Vaporization)] × 100%.

[c] Percent Removal = [Cumulative, Increased Phenol Mass in the Cathode Reservoir/(Initial Phenol Mass in the Soil Cell − Phenol Mass Loss Due to Vaporization)] × 100%.

[d] Percent Destruction = 100% − Residual Phenol Fraction in Soil − Percent Removal.

[e] Percent Removal and Destruction = Percent Removal + Percent Destruction.

indicated the cumulative, increased mass percent of phenol in the cathode reservoir increased as the amount of scrap iron powder decreased. Again, this phenomenon is due to the fact that a greater quantity of EO flow is associated with less scrap iron powder used. In other words, the removal of phenol from the soil cell would be easier and more effective when less scrap iron powder is embedded. The cumulative, increased mass percents of phenol in the cathode reservoir for Test 1 and Test 6 are 28.26% and 62.79%, respectively. For the purpose of remediation, phenol contained in the cathode reservoir of course needs further destruction. The degree of chemical destruction of phenol in the cathode reservoir would depend on the availability of hydroxyl radicals therein.

3.6. Effects of the amount of scrap iron powder and treatment time on phenol removal and destruction

The removal and destruction of phenol in the soil cell and cathode reservoir were evaluated based on the mass balance analysis of phenol of each test. Phenol mass balances for Tests 1–9 are given in Table 4. For the test runs conducted by EK-Fenton process, in general, the less amount the scrap iron powder used, the greater the percentage of phenol removal and destruction. Therefore, Test 6 has the highest overall removal and destruction efficiency of phenol (i.e. 99.7%) and Test 1 has the lowest efficiency (i.e. 53%). This finding is in good agreement with the fact that Test 6 has the greatest EO permeability and cumulative EO flow quantity. However, even in Test 6 only 30.8% of phenol were actually destroyed. It was postulated that a longer treatment time would increase the percentage of chemical destruction of phenol. Accordingly, Test 7 was carried out to study the effect of the treatment time on phenol destruction. By comparing the percent destruction of phenol of Tests 5 and 7, it is evident that an increase of the treatment time by 3 days has increased the phenol destruction from 23% to 32%. The overall removal and destruction of phenol for Test 7 was determined to be 99% instead of 93% for Test 5. Therefore, a longer treatment time would be beneficial to phenol destruction. For the control run of EK process alone (i.e. Test 8), it has the greatest percent removal of phenol. Almost all the phenol mass in the soil cell was completely removed to the cathode reservoir. No degradation of phenol would take place in this case.

4. Conclusions

A sandy loam saturated with an aqueous solution of phenol was treated by electrokinetics-Fenton process incorporated with a permeable reactive wall of scrap iron powder. The research findings are given as follows.
(1) Electrokinetic remediation alone is capable of removing phenol from the soil cell. An incorporation of Fenton-like process with scrap iron powder in the electrokinetic remediation system is beneficial to the chemical degradation of phenol in a saturated flow. An overall removal and destruction efficiency of phenol of 99.7% is achievable.

(2) A greater amount of scrap iron powder embedded in the soil cell would yield a lower value of EO permeability. Therefore, to obtain a higher removal and destruction efficiency of phenol, a less amount of scrap iron powder (e.g. 1.05 g) is preferred.

(3) To obtain a destruction efficiency of phenol of more than 30.8%, a treatment time of greater than 10 days is required.

Acknowledgements

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