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Removal of trichloroethylene in reduced soil columns

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Abstract

A continuous soil column experiment was conducted to investigate reductive dechlorination of trichloroethylene (TCE) in soil system reductively manipulated by three types of reductants (Fe(II), dithionite, and Fe(II) + dithionite (combined treatment of Fe(II) and dithionite)). The soil column reduced by Fe(II) + dithionite has the greatest bed volumes (51.8) treated to breakthrough indicating that the combined treatment of Fe(II) and dithionite is more effective for the reductive dechlorination of TCE in the reduced soil column than the separate treatment of Fe(II) or dithionite. The measured bed volumes to breakthrough in control and treated soil columns were similar to the estimated bed volumes based on the result of batch kinetic experiment, differing by a factor of 0.96-1.02. The relative concentration of bromide (non-reactive tracer) reached the approximate value of 1 between 0.87 and 1.03 bed volume. C₂ hydrocarbons (acetylene, ethylene, and ethane) were observed as transformation products in the effluents of soil columns treated by the reductants. However, no chlorinated intermediates were observed at the concentrations above detection limits throughout the experiment. Chloride was observed in the effluents of soil columns reduced by dithionite and Fe(II) + dithionite.

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1. Introduction

Abiotic reductive dechlorination of chlorinated organics by natural reductants such as reduced natural organic matters (NOMs) [1], soil minerals [2,3] and sediments [4] has been characterized to develop remedial technologies to treat the contaminants in natural and engineered systems. Natural attenuation, in situ bioremediation, enhanced pump and treat systems, and in situ redox manipulation are examples of emerging remedial technologies under development to date. In situ redox manipulation, based on the abiotic reductive dechlorination, is an attractive alternative because it removes the contaminants efficiently without extensive changes at the contaminated sites. Some experimental studies have shown the viability of reduced soil zone to treat the groundwater and soil contaminated with chlorinated solvents and heavy

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metals in in situ permeable barrier formed by the injection of an external reductant (dithionite) [5,6]. It has been reported that the reactivity of reduced soil zone is due mainly to reduced natural organic matters, sulfides, and reactive chemical compounds (iron and manganese) on the surfaces of soil minerals. These activated natural reductants in the reduced soil zone can treat the contaminants subsequently passing through the treated zone. Some field-scale experimental studies have shown that in situ redox manipulation by dithionite injection is very effective in remediation of Cr(VI) and carbon tetrachloride [5,7,8]. It has been reported that hexachloroethane, tetrachloroethylene, trichloroethylene (TCE), and uranium can be reductively degraded in the soil zone reduced by dithionite [4,9,10]. A recent batch kinetic research investigating the effect of reductant type on the reactivity of reduced soil has shown that the soil sequentially treated with Fe(II) and dithionite is most reactive for the reductive dechlorination of chlorinated ethylenes [11]. Lee et al. have reported that Cr(VI) can be effectively immobilized to Cr(III)

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in the soil columns reduced by Fe(II), dithionite, and a combination of Fe(II) and dithionite (Fe(II) + dithionite) and that the oxidized soils during the reduction can be continuously regenerated by the repeated addition of reductants [12]. However, no significant research has been conducted to improve the reactivity of reduced soil zone using different types of reductants for the redox manipulation of continuous groundwater flow system contaminated with chlorinated organics.

The goal of this research is to investigate the reductive dechlorination of chlorinated ethylene in the soil zone reduced by different types of reductants. A column reactor was used for the continuous flow experiment so that the experimental results can evaluate the ability of batch kinetic data to predict the performance of reduced soil for the reductive dechlorination in the continuous flow system. TCE was used as a representative chlorinated organic and Fe(II), dithionite, and Fe(II) + dithionite were used as reductants to improve and evaluate the reactivity of reduced soil. The experimental results would provide basic understanding to be applied to the development of new abiotic remediation technology and novel knowledge to develop more reactive soil zone for the treatment of subsurface system contaminated with chlorinated organics.

2. Experimental

2.1. Materials

Soil collected from the top 20-40 cm near College Station, TX is an alluvium of the Brazos River and is classified as Silawa loamy sand [13]. It was brought to the laboratory without any efforts to maintain the original redox state. Soil samples were air-dried in the atmosphere for 14 days and screened with 0.425 mm sieve. X-ray diffraction (XRD) analysis semi-quantitatively estimated the composition of the clay mineral fraction with Rigaku automated diffractometer using Cu K α radiation from 0 to 70° 2 θ at the scan speed of $1^{\circ} 2\theta \min^{-1}$. It includes 20–25% quartz (4.26 and 3.34 Å), 40-50% kaolinite (7.1 and 3.56 Å), and 30-40% mica (10 and 5 Å). The silt and sand fractions include quartz with small amount of feldspar. Table 1 shows the distribution of particle size analyzed by the pipette method [14]. Some important physico-chemical characteristics of the soil samples were measured and are summarized in Table 2. The soil contains approximately 0.5 mg/g Fe(II), 6.26 mg/g total iron, and 0.69% organic carbon. The measured reductive capacity of soil for Cr(VI) was 0.38 µeq/g.

Table 1 Particle size (mm) distribution of Silawa soil

Table 2	
Characteristics of Silawa soil	

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Surface area ^a (m ² /g)	14.0
Organic carbon ^b (%)	0.69
Fe(II) ^c (mg/g)	0.5
$Fe(III)^{c}$ (mg/g)	5.76
Reductive capacity for Cr(VI) ^d (µeq/g)	0.38
pH ^e	6.1

^a Specific surface areas of soil minerals and soil were measured by the ethylene glycol monoethyl ether (EGME) method [14].

^b Organic carbon content was determined by dry combustion method [15]. ^c Fe(II) and total iron in soil were measured by modified 1,10phenanthroline method [16]. A reducing agent (10% hydroxylamine) was added to measure total iron. Fe(III) in soil was determined by subtracting Fe(II) from total iron.

^d Reductive capacities of soil for Cr(VI) were measured by a standard method developed by Lee et al. [12].

^e Standard soil analysis method was used to measure the pH of the soil. Deionized water was added to the soil (mass ratio = 1:1) and equilibrated for 1 day [15].

Chemical reagents and soil samples were prepared in an anaerobic chamber (Coy Laboratory Products Inc.) filled with 95% N₂ and 5% H₂. All chemicals used for this research were American Chemical Society (ACS) or higher grades and used as received. Stock target organic solution was prepared by diluting TCE in methanol. Hexane was used as an extractant and 1,2-dibromopropane (1,2-DBP) was used as an internal standard for gas chromatograph (GC) analysis. Deaerated deionized water (ddw) was prepared by deoxygenating nano-pure water ($18 M\Omega cm$) with N₂ for 2 h and then with the mixed gases in anaerobic chamber for 12 h. Fe(II) solution (0.6 M) was prepared by adding an exact amount of FeSO₄·7H₂O into ddw and dithionite solution (0.1 M) was prepared by adding an exact amount of Na₂S₂O₄ into 0.4 M K₂CO₃. Tris buffer (0.05 M) was prepared by adding exact amounts of Tris[hydroxymethyl]aminomethane and Tris[hydroxymethyl]aminomethane hydrochloride to ddw. Standard TCE (0.03 mM) and bromide (40 mg/l) solutions were prepared by adding exact amounts of stock target organic solution and KBr to Tris buffer, respectively.

2.2. Experimental methods

The column reactors (1.5 cm diameter \times 30 cm length) were equilibrated with anaerobic atmosphere for 2 days and packed to a uniform bulk density of 1.55 \pm 0.05 g/cm³ and total porosity of 0.45 \pm 0.05 with Silawa soil. After plugging each end with Teflon adapters, the columns were flushed

Size and content Size (mm)	Sand					Silt		Clay			
	2.0–1.0	1.0-0.5	0.5-0.25	0.25-0.10	0.10-0.05	Total 2.0-0.05	0.02-0.002	Total 0.05-0.002	< 0.0002	Total <0.002	
Content (%)	0.3	0.6	15.5	46.0	18.7	81.1	5.5	12.2	3.7	6.7	

with anaerobic atmosphere to remove air pockets. The soil in the columns was then equilibrated with Tris buffer solution for 5 days at the flow rate of 0.07 ml/min with a peristaltic pump in the anaerobic chamber. Three types of reductants were used to reduce Silawa soil after the equilibration with Tris buffer. Fe(II) and dithionite solutions were introduced into the buffer-saturated soil columns at the Darcy velocity of 2.6 cm/h with the peristaltic pump for four bed volumes, respectively. In the case of combined treatment of Fe(II) and dithionite, Fe(II) solution was initially introduced into the column for two bed volumes and then dithionite solution was introduced for two bed volumes at the same velocity. The reduced soil in the columns was washed by re-introducing Tris buffer solution to column inlets to remove residual Fe(II) and dithionite sorbed on the soil for two bed volumes after the full drain of reductants. The reductive dechlorination of target chlorinated organic in the reduced soil columns was investigated by introducing standard TCE solution to the column inlets at the same Darcy velocity under the room temperature (22.5 \pm 0.5 °C). A self-collapsible bag was used as a feeding reservoir to prevent the partitioning of TCE to its headspace. Standard bromide solution was also introduced to the columns as a non-reactive tracer to determine the physical characteristics of soil columns. The effluents from outlets of control column (no treatment with reductants) and reduced soil columns were collected at the regular sampling times to monitor the concentrations of target organic, transformation products, and tracer in the effluents.

2.3. Analytical methods

TCE and its chlorinated transformation product (1,1dichloroethylene, (1,1-DCE)) were measured by Hewlett-Packard (HP) GC 5890 equipped with an electron captured detector and combination of DB-5 and DB-5MS columns (J&W Scientific). The temperature condition of GC for the measurement of TCE and 1,1-DCE was well described everywhere [2,3,11]. A 50 μ l of aqueous sample was extracted with 1 ml extractant (hexane with 0.025 mM 1,2-DBP) at each sampling time. An aliquot amount of extractant was automatically injected into the GC inlet at the split ratio of 35:1.

Other chlorinated transformation products (i.e., *cis*dichloroethylene (c-DCE), *trans*-dichloroethylene (t-DCE), and vinyl chloride (VC)) were measured by HP GC 6890 equipped with a flame ionization detector (FID) and DB-VRX column (J&W Scientific). The temperature program and operation condition of GC were also well described everywhere [2,3,11]. A 1 ml sample of effluent was transferred to a 2 ml vial and allowed to equilibrate for 2 h at room temperature. An aliquot amount of gas-phase headspace sample was injected into the GC inlet at the split ratio of 5:1. The aqueous concentrations of the transformation products were calculated using dimensionless Henry's law constants [17,18]. C₂ hydrocarbons (i.e., acetylene, ethylene, and ethane) in the effluents were identified by HP GC 6890 equipped with a FID and GS Alumina column (J&W Scientific) following the same headspace procedure above. The operation condition of GC for the measurement of C_2 hydrocarbons was well described everywhere [2,3,11].

An aliquot of effluent filtrate (filtered by $0.2 \,\mu\text{m}$ membrane filter) was introduced into the injection port of Dionex ion chromatograph equipped with AS9-HC column and conductivity detector to analyze bromide and chloride. A 10 mM of Na₂CO₃ solution was used as an eluent and flow rate was kept at 1 ml/min during the analysis.

3. Results and discussion

The removal of chlorinated ethylene in reduced soil was investigated by monitoring the concentrations of TCE and transformation products in the effluents from the reduced soil columns and control column. Figs. 1–4 show the concentration profiles of target organic and transformation products in the effluents of the columns. The relative concentration $(C_{\text{TCE}}/C^0_{\text{TCE}})$ of TCE in the effluent reached approximate value of 1 at 22.1 (control), 34.1 (Fe(II)), 39.4 (dithionite), and 79.4 bed volumes (Fe(II) + dithionite), respectively. This result indicates that the soil treated with Fe(II) + dithionite is the most effective for the removal of TCE in the reduced soil column. The similar result has been reported in a batch regeneration experiment using soils reduced with Fe(II), dithionite, and Fe(II) + dithionite [11]. In contrast to the treatments of soil by the separate addition of Fe(II) and dithionite, the



Fig. 1. Relative concentrations of TCE, bromide, chloride in the control column.



Fig. 2. Relative concentrations of TCE, bromide, and chloride in the soil column treated by Fe(II).



Fig. 3. Relative concentrations of TCE, bromide, chloride in the soil column treated by dithionite.

combined treatment could improve the reductive capacity of soil by adding reactive chemical compounds (i.e., Fe(II) and sulfide), reducing redox-active chemical compounds in the soil (i.e., NOM, iron-bearing soil minerals, iron and manganese complexes sorbed on the surface of soil), and forming reactive soil minerals. It has been known that ironbearing soil minerals such as iron sulfide (pyrite), iron hydroxide (green rust), and iron oxide (magnetite) can reductively degrade chlorinated organic compounds in natural and engineered systems [2,3]. The color of soil in the column turned to black after the combined treatment. Blackcolored precipitates were observed on the soil particles, which are assumed to be reactive iron sulfides. An XRD analysis needs to be conducted to confirm the identity of blackcolored precipitates. The reductive capacity of dithionitetreated soil for TCE (0.281 µmol/g) estimated by modified Langmuir-Hinshelwood kinetic model was greater than that of Fe(II)-treated soil (0.276 µmol/g) but the difference between them is not significant [11]. Although the reductive capacity does not consider the sorption of target compound on the surface of soil, this result can explain the reason why the bed volume of dithionite-treated soil is slightly greater than that of Fe(II)-treated soil. The reductive capacity of Fe(II) + dithionite-treated soil for TCE has not been measured nor estimated by kinetic models to date. However, based on the



Fig. 4. Relative concentrations of TCE, bromide, and chloride in the soil column treated by the combination of Fe(II) and dithionite.

experimental results reported [11,12] and obtained in this research, it can be assumed to be greater than the reductive capacity of soil reduced by Fe(II) and dithionite, separately.

The removal of TCE in a control column seems to be significantly influenced by the sorption of TCE on the surface of soil. The soil includes 0.69% organic carbon so that the target organic can be easily adsorbed on the soil surface. TCE could be also removed by the reductive dechlorination caused by the intrinsic reactivity of soil in the control column. Fe(II) content of Silawa soil is equivalent to 1.4 times the stoichiometric amount required to fully reduce the initial concentration of TCE to ethylene. However, no experimental evidence (i.e., C2 hydrocarbons, chloride, and chlorinated intermediates in effluent) was observed to support the reductive dechlorination of TCE in the control column. The transformation products may be produced at concentrations under detection limits (c-DCE: 0.2 mg/l, t-DCE: 0.08 mg/l, 1,1-DCE: 0.23 mg/l, VC: 0.26 mg/l, and chloride: 0.04 mg/l) during the reaction and/or adsorbed on the soil surface. An extraction of soil with non-polar solvent was not conducted to investigate TCE and transformation products sorbed on the soil surface after the full drain of standard TCE solution at this study.

The removal of target organic in a soil column treated with Fe(II) is similar to that treated with dithionite. The dithionitetreated soil column has greater bed volume to reach the relative TCE concentration of 1 than Fe(II)-treated soil column, however the difference is not significant. C₂ hydrocarbons were observed in effluents from the reduced soil columns. Acetylene started to be observed in the effluents at 35 bed volumes (Fe(II)-treated column and dithionite-treated column) and at 20 bed volumes (Fe(II) + dithionite-treated column), respectively. The formation of acetylene was continuously monitored in the reduced soil columns until the reaction finished (55 and 88 bed volumes). Ethylene was observed in effluents at 45 bed volumes (dithionite-treated column) and at 25 bed volumes (Fe(II) + dithionite-treated column). However, the formation of ethylene stopped in 5 and 10 bed volumes, respectively. Ethylene was not observed in effluent from Fe(II)-treated soil column. Further reduction to ethane was only observed in effluent from Fe(II) + dithionite-treated column at 30 bed volumes. The concentration of chloride in effluents was monitored to investigate the reductive dechlorination of target organic in soil columns. Chloride was observed in the effluents of soil columns treated with dithionite and Fe(II) + dithionite after 47 and 65.1 bed volumes, respectively. However, their relative chloride concentrations do not exceed 0.06. The concentration of chloride corresponding to initial TCE concentration (C^{0}_{TCE}) was calculated by assuming that TCE (0.03 mM) is fully transformed to ethylene and chloride (1.65 mg/l) without any loss of TCE due to the partitioning to solid and gas phases. No chloride was observed in the effluents of control and Fe(II)-treated soil columns at the concentration above detection limit throughout the column experiment. The chloride un-recovered may be sorbed on the soil surfaces and/or form surface complexes. No sequential chlorinated intermediates (DCEs and VC) that could be found in the biodegradation process were observed in all effluents at concentrations above detection limits. This suggests that TCE may be reductively dechlorinated to nonchlorinated C₂ hydrocarbons in soil columns via a reductive elimination pathway (TCE \Rightarrow chloroacetylene \Rightarrow acetylene \Rightarrow ethylene \Rightarrow ethane). Unstable intermediates, such as chlorinated acetylenes may be produced but not detected in the effluents during the reductive elimination of TCE.

Breakthrough of bromide and TCE observed in the control and reduced soil columns showed that concentrations of TCE were attenuated by a factor of 20-50 compared to those of bromide. Breakthrough curves for TCE were retarded, while no significant retardation was observed in breakthrough curves for bromide. The relative concentration of tracer $(C_{\rm Br}/C_{\rm Br}^{0})$ in the effluent of each column reached approximate value of 1 between 0.87 and 1.03 bed volume. In contrast to a breakthrough curve for TCE observed in the control column, those in the reduced soil columns have slopes. This may be due to the longitudinal dispersion caused by the heterogeneity of soil treated by different types of reductants. The bed volume corresponding to the measured TCE breakthrough time of each column was 21.4 (control), 29 (Fe(II)), 31 (dithionite), and 51.8 (Fe(II) + dithionite), respectively. It was determined by assuming that TCE breakthrough occurs when the relative TCE concentration reaches 0.5. The bed volume corresponding to the estimated TCE breakthrough time was calculated using the result of batch kinetic experiment [11]. It is 21.6 (control), 28.5 (Fe(II)), 31.6 (dithionite), and 54.2 (Fe(II) + dithionite), respectively. The estimated bed volumes to breakthrough are very similar to the measured bed volumes to breakthrough obtained in this research, differing by a factor of 0.96-1.02. The amount of TCE removed in a batch reactor was calculated (TCE mM/g soil) and then converted to the bed volume corresponding to TCE breakthrough time. The result indicates that batch kinetic data for the reductive dechlorination of chlorinated ethylenes by soil can be used to approximately estimate the performance of soil column for the reductive dechlorination of chlorinated organics in a continuous flow system.

4. Summary and conclusions

The removal of chlorinated organic in reduced soil zone was demonstrated by a continuous flow experiment in which TCE was reductively dechlorinated throughout the soil column reactors reduced by three different types of reductants. The soil column reduced by Fe(II) + dithionite has the greatest bed volume treated to TCE breakthrough followed by those of dithionite, Fe(II), and control (i.e., Fe(II) + dithionite > dithionite > Fe(II) > control). The highest soil reductive capacity by the combined treatment may be due to the addition of reactive Fe(II) and sulfide and/or the formation of reactive iron sulfides. The estimated TCE breakthrough time based on the result of batch kinetic experiment was very similar to that measured in each column, which indicates that simple batch experiment can be used to approximately estimate the performance of reduced soil zone for the removal of chlorinated organics in a continuous flow system. C_2 hydrocarbons and chloride were observed in the effluent as transformation products but chlorinated intermediates were not observed at concentrations above detection limits. This indicates that TCE may be reductively transformed to non-chlorinated compounds by a reductive elimination. This result would be an advantage over the biodegradation of chlorinated organics producing more toxic sequential chlorinated transformation products.

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