NATURAL ATTENUATION OF TRICHLOROETHYLENE IN WETLAND SOIL

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ABSTRACT

The Hemispheric Center for Environmental Technology (HCET) is supporting the application of natural attenuation to the cleanup of the U.S. Department of Energy's Savannah River Site (SRS). SRS contains a number of groundwater plumes contaminated with organics, metals, and radionuclides as a result of past waste disposal practices. SRS is aggressively pursuing the use of monitored natural attenuation (MNA) and enhanced natural attenuation as remedial options for many of these sites [1]. In support of this objective, HCET is conducting studies of various aspects of natural or nature-based remedial strategies. One of these activities involves natural attenuation of trichloroethylene (TCE) in wetland soil. In this study, batch and column tests were performed to investigate TCE adsorption onto wetland soil, measure the effects of the soil on pore water pH and oxygen/reduction potential, and study the biodegradation of TCE in groundwater in contact with wetland soil. The results indicate that complete biodegradation of TCE can occur as groundwater flows upward through wetland soil.

INTRODUCTION

Historic waste handling and disposal techniques in the early years of the U.S. Department of Energy's Savannah River Site (SRS) have resulted in soil and groundwater contamination by trichloroethylene (TCE) and related compounds. At several SRS groundwater seepage locations, the contaminated groundwater flows through natural wetland systems before discharging into surface water. Natural attenuation of TCE in these environments has been found to reduce TCE concentrations [2,3]; however, where existing wetlands are inadequate, or nonexistent, TCE

attenuation rates are insufficient to reduce residual TCE concentrations to levels that are safe for discharge to adjacent water bodies. The Hemispheric Center for Environmental Technology (HCET) is investigating the mechanisms that degrade TCE in wetlands in an effort to increase the attenuation of TCE prior to its discharge into surface water. Studies were conducted to develop a more complete understanding of natural attenuation processes and mechanisms at work in a natural or constructed wetland that could remove contaminants from groundwater. This paper presents the results of the first phase of this multi-year study.

Extensive research has been conducted on natural attenuation of chlorinated solvents in groundwater aquifers and other environments, including wetlands. During natural attenuation, chlorinated solvents are removed from the water phase by physical or biological processes, dechlorinated to less chlorinated daughter products, or completely dechlorinated to ethene, ethane, or CO_2. This attenuation results from multiple mechanisms including biodegradation, plant uptake, volatilization, and sorption [4]. At many sites, however, physical, chemical, and biological conditions do not fully support these mechanisms. Either the conditions in the aquifer are not suitable, or the travel time of the contaminants in the subsurface is too short to permit complete degradation of the contaminants. The characteristics of a wetland system, including its microbial, physical, and chemical makeup, create an ideal environment for TCE attenuation through biodegradation and other processes. TCE biodegradation occurs under anaerobic conditions by reductive dechlorination, either as a cometabolic process or as a growth-linked respiratory process known as dehalorespiration [5]. Results from the Aberdeen Proving Ground indicate that natural attenuation rates increase dramatically as contaminated groundwater passes through wetland systems. Rate constants of 30-40 $years^{-1}$ have been observed for the dechlorination of TCE in wetlands as opposed to 1-4 $years^{-1}$ typically observed in aquifers [6]. Organic sediments retard the movement of contaminants relative to groundwater flow, resulting in longer retention times for the contaminants in the wetland soil where biodegradation and plant uptake may destroy or remove the contaminants [7]. The objective of this project was to investigate the mechanisms responsible for attenuation of TCE in a wetland environment in order to identify ways of enhancing the attenuation process where existing wetlands are unable to completely remove TCE.

MICROCOSM STUDIES

Microcosm studies were carried out to quantify adsorption effects and pH adjustment strategies. The wetland soil studied was a mixture of 70% wetland soil obtained from SRS and 30% commercial topsoil. This resulted in wetland soil with approximately 3.3% organic matter. The organic matter content of SRS wetland soil varies widely, ranging from 5-48%. Thus, the experiments conducted in this project were conducted on soil with relatively low organic content. The control soil was unwashed sand excavated from natural deposits in the vicinity of SRS. The sand contained less than 0.5% organic content. Table I shows the characteristics of the test wetland soil, sand and several SRS wetland soil samples.

Table I. Soil Analysis Results

	pH	Organic Matter (%)	Total Phosphorous (µg/g)	Total Nitrogen (mg/g)	Total Carbon (mg/g)
Experimental Soils					
Wetland soil	4.27	3.29	254.8	2.6	21.9
Sand	5.04	0.53	43.1	BD	4.5
SRS Wetland Samples					
Upper Three Run peat layer	5.02	47.81	476.4	19.5	319.
Upper Three Runs subsurface	4.73	24.21	113.4	12.5	232.3
TNX Area, 14-16"	4.44	21.55	537.1	5.0	65.4
CMP Area surface	4.86	5.97	312.9	2.9	38.1
CMP Area, 24"-30"	4.89	4.97	129.4	2.8	43.6

Artificial groundwater (AGW), formulated to simulate the low alkalinity groundwater at SRS, was used through this experiment. The AGW composition was based on a recipe developed at the Savannah River Environmental Laboratory [8]. This recipe contains only naturally-occurring cations including calcium, sodium, potassium, and chloride and surface anions. The AGW consisted of 5.5 mg/l $CaCl_2.6H_2O$, 1.1 mg/l Na_2SO_4, 3.1 mg/l $MgCl_2.H_2O$, 0.4 mg/l KCl, and 2.7 mg/l NaCl. No alkalinity was added, resulting in an unbuffered, slightly acidic solution.

Adsorption Studies

The objective of the adsorption studies was to determine the TCE sorption capacity of the experimental soils and the potential for desorption of TCE. Adsorption isotherms were measured using a traditional batch adsorption test. The microcosms consisted of 60 ml dark glass bottles fitted with rubber septum tops. A known amount of dried soil was placed in each bottle, with the remainder being filled with artificial SRS groundwater. Varying amounts of TCE stock solution, prepared in methanol, were then added to each bottle using a gas-tight syringe. Appropriate controls, without soil, were also prepared. After mixing on a shaker table for 48 hours and centrifuging to separate soil from the water, samples of the water were removed and analyzed for equilibrium TCE concentration. To measure the amount of TCE desorbed, following collection of the water samples, the bottles were filled with TCE-free water and again mixed for 24 hours. A second water sample was then collected and analyzed for TCE. The concentrations of TCE and its degradation products were determined by the purge & trap gas chromatography/mass spectrometry (GC/MS) method, according to U.S. Environmental Protection Agency (USEPA) methods SW5030B and SW8260B (USEPA 1998). A PerkinElmer Clarus 500 gas chromatograph/flame ionization detector (GC/FID) analyzed ethene with Supelco Carboxen 1010 PLOT 30mx0.53mm column.

The slope of the linear isotherms generated for these materials (not shown) were used to estimate partition coefficients of 29.2 L/Kg for the soil and 5.0 L/Kg for the sand. Using these values, as well as values for bulk density and porosity determined experimentally for the two soils, the retardation factor for movement of TCE through the soil was estimated at 50 for the soil and 13

for the sand. The higher retardation factor for wetland soil is thought to result from the much higher organic content of the wetland soil. This finding implies that wetland soil, which typically has a very high organic-content, will adsorb large amounts of TCE and slow the movement of TCE dramatically.

The desorption measurements indicated that 20-30% of the TCE adsorbed onto the soil was released back into the TCE-free water within 24 hours. Thus, the adsorbed TCE will be released from the soil if water-phase concentrations decrease, disturbing the equilibrium. This finding indicates that adsorbed TCE remains available to soil bacteria, allowing for continued biodegradation of adsorbed material.

pH Adjustment Studies

The groundwater at SRS is naturally low in alkalinity and, as a result, has a relatively low pH, in the 5.0-6.0 pH unit range. Biodegradation rates are sensitive to solution pH, with the optimum range at 7-7.5 pH units [9]. Thus, studies were carried out to identify the effect the test soils would have on the pH of groundwater. In addition, studies were completed to identify the required dosage of limestone (calcium carbonate) necessary to adjust and maintain the soil pH close to the optimum range.

To measure the effects of the experimental soils on pH, 5 grams of the air-dried sand and wetland soil were mixed with 5 ml of distilled water and stirred vigorously for 30 minutes and the pH was measured. The vials were then capped and allowed to sit. Soils were again stirred and the pH recorded after an additional 30 minutes, then 60 minutes later, and again on the following day (19 hours later).

The experimental soils were found to increase the pH, but not enough to raise it to its desired value of 7.0-7.5. When the soils were added to AGW at a pH of 4.8, the pH of the sand increased by 0.1 units within the first 60 minutes of flooding and then remained stable at around pH of 5.0. The wetland soil increased the pH to 5.5 and thereafter remained stable.

The effects of buffering by calcium carbonate ($CaCO_3$) were then examined. The mass of $CaCO_3$ added to a known mass of wetland soil was varied to determine pH-buffering curves for oxidized soil and anaerobic soil. The pH of each mixture was then monitored over a period of several days to determine the equilibrium pH value achieved by each $CaCO_3$ concentration.

These studies indicated that a pH between 7.0 and 8.0 could be achieved with varying amount of $CaCO_3$. The response of the oxidized and anaerobic topsoil tested was similar, with the addition of higher $CaCO_3$ amounts causing a slightly greater increase in pH. It appears that a 2% addition of $CaCO_3$ (dry weight) to wetland soil will buffer the pH of the porewater at a value of approximately 8.0 regardless of aerobic or anaerobic conditions. A stable pH within the optimum range of 7.0-7.5 could not be achieved with this approach.

MESOCOSM STUDIES

Continuous flow mesocosm studies were carried out to measure the rate of TCE degradation under steady-state conditions. The mesocosms consisted of two 15cm diameter open top glass columns one of which, Column A, was filled with wetland soil and the other, Column B, with

sand. The sand-filled Column B served as a growth-inhibited control column. Since the columns were open to the atmosphere, the control column could not be made sterile; however, to inhibit biodegradation, only distilled water with 1.0 mg/L (7.6 micromoles/Liter) TCE added, was pumped through this column. Column A was supplied only with AGW with the same amount of TCE added. Feed solutions containing TCE were placed in flexible Teflon bags with no headspace to prevent loss of TCE during storage. The columns were monitored over several months to determine the physical and chemical conditions inside and to measure the magnitude of TCE attenuation mechanisms including biodegradation, adsorption, and volatilization. Figure 1 is a sketch of one of the experimental columns. The system was operated in an upflow mode with a hydraulic detention time of approximately 15-20 days after initial acclimation. The daily flow rate was approximately 500-600 ml/day. The water exited the column through a porous outlet port located approximately 10 cm below the top of the soil column.

Column Start-up and Operation

The start-up of the column followed a three-step process. Step one involved slowly pumping clean artificial groundwater (Column A) or distilled water (Column B) through the columns to saturate the column's soil, to stimulate biological activity, and to generate reducing conditions in Column A. Step 2 involved the introduction of TCE into the column as rapidly as possible to allow for equilibration of adsorption of TCE onto the soil. Step 3 involved reducing the flow to the desired long-term HRT of 15-20 days and monitoring TCE and its daughter products to determine when steady-state conditions would be achieved.

During the start-up process the columns were monitored periodically to determine the conditions within the column. A Yellow Springs Instrument multi-probe was used to measure pH and ORP in the column using a flow-through cell. Periodically, samples were extracted from the inlet tubing, intermediate sampling ports and outlet port for analysis of TCE and its degradation products, cis-1,2-dichlorethyelene (cis-1,2DCE), trans-1,2DCE, 1,1DCE and vinyl chloride (VC) using the GC/MS method discussed earlier.

Tracer Studies

Prior to the start-up of the columns with TCE in the feed solution, a tracer study was performed. The purpose of the tracer study was to compare the actual retention time with the theoretical retention time based on flow rates, soil volume and porosity. A difference in retention time would indicate either short-circuiting via preferential flow paths in the soil, or displacement of pore water from the soil by gas generated during decomposition of organic matter in the soil. The displacement of pore water by gas would decrease the volume of water in the column, resulting in a reduction in the actual hydraulic retention time.

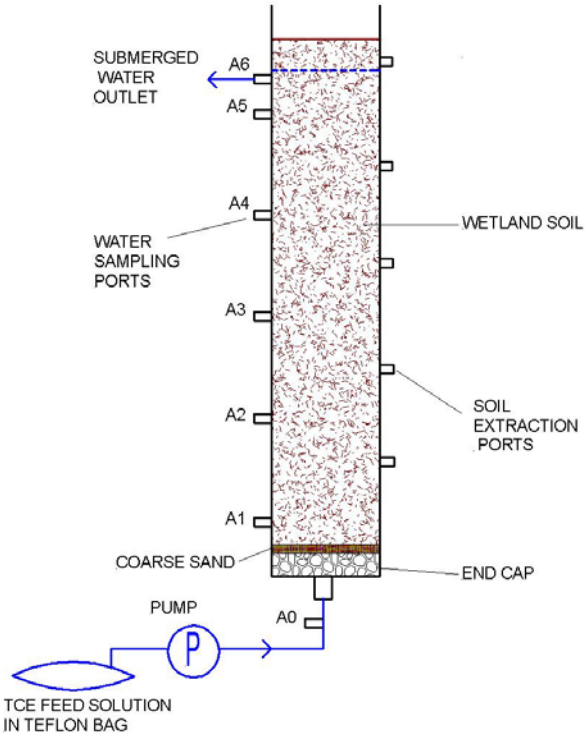

Fig. 1. Sketch of Wetland Column Reactor

The tracer studies were performed by injecting a pulse of sodium bromide solution into the influent at a known time, then periodically analyzing effluent samples for bromide. The resulting effluent bromide curves were used to estimate the mean hydraulic retention time as the time when 50% of the tracer mass had passed the effluent point. The time when the peak tracer concentration passed the effluent point was also noted. These values were then compared to the calculated theoretical retention time, which assumes that all soil pore space is filled with water and no short-circuiting occurs. The sand filled column was found to have a mean HRT of 17.4 days and a time for peak bromide concentration of 15.6, compared to the theoretical retention time of 15.7 days. Thus, little short-circuiting was observed in the sand column. The wetland column; however, had a mean HRT of only 14.0 days and a peak bromide time of 13.1 days, as compared to the theoretical retention time of 17.3 days. The fact that the actual retention time was 3-4 days shorter than anticipated was likely due to gas build-up within the soil column, which was observed throughout the experiment.

Column Monitoring

Routine monitoring of the columns generated large quantities of data that describe the conditions in the two experimental columns.

pH Monitoring Results

Routine monitoring indicated that the pH increased in both columns as the water passed through the soil. The pH of the influent averaged around 6.5. In both columns, the pH had increased at

the first sampling port by 0.5 to 1 pH unit, due to the alkalinity present in the soil. In both columns the pH remained relatively stable, ranging between 7.0 and 7.8, creating conditions ideal for biodegradation to occur without the need to add $CaCO_3$. In both columns the pH often decreased slightly near the soil surface. The alkalinity present in the experimental soils was sufficient to maintain the pH in the optimum range for biodegradation to occur.

ORP Monitoring Results

Figure 2 shows representative profiles of ORP in the two columns. The rapidly decreasing ORP values in Column A indicate that the oxygen was rapidly consumed as the water passed into the wetland soil column, generating mildly reducing conditions within the column. Typical inlet ORP values (measured with respect to a Ag/AgCl reference electrode) were +100 to +150 mV. By the time the water had moved past the first sampling port, approximately 5 cm above the inlet, it had dropped to approximately -50 mV. The middle ports (A2, A3 and A4) exhibited the lowest ORP, with values ranging from -100 to -150 mV. The upper ports, A5 and A6, were often somewhat higher, with values ranging from -25 to -75 mV. The higher ORP of the upper ports is likely the result of oxygen penetration into the upper layers of soil. Even with the oxygen penetration, ORP values were still in the mildly reducing range throughout the column. However, none of the ORP values measured were low enough to indicate that methanogenic conditions existed in the column, which many researches have indicated are necessary for full TCE biodegradation to occur.

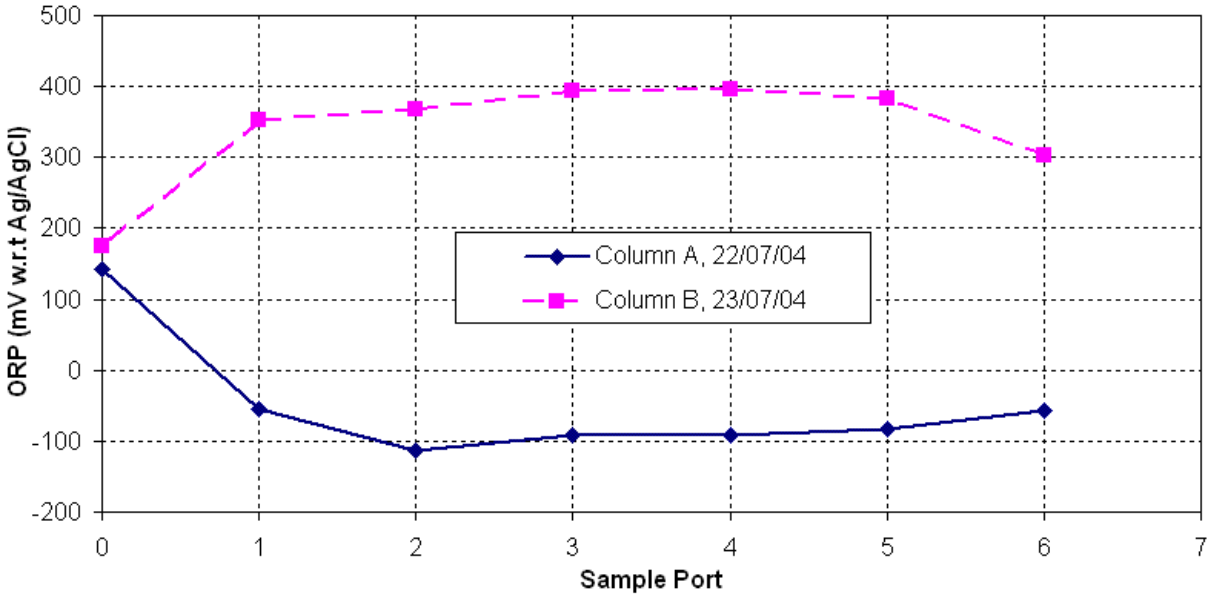

Fig. 2. Monitoring results for ORP in Column A

Monitoring of Column B gave very different results. The ORP values at all of the sampling ports were significantly higher than the ORP in the distilled water influent, which typically ranged from +150 to +220. The ORP in the sampling ports was generally similar to one another and ranged from +150 to +450. The reason for the higher ORP within the column likely the result of oxidized material present in the sand. In any case, it suggests that the reduction in ORP seen in Column A is a result of the organic matter present in the soil.

VOC Monitoring Results

TCE concentrations in Column A varied during the experiment as the bacteria in the column acclimated to its presence and biodegradation began. Figure 3 shows the change in TCE concentrations over time in the inlet, intermediate Ports A1, A2 and A4 and the outlet Port A6. While the inlet concentrations remained relatively steady at approximately 7 to 8 micromoles per liter, the concentrations of TCE in Port A2 first increased, as TCE passed up through the soil and came into equilibrium with the adsorbed fraction on the soil surface. After that, it rapidly decreased from a high of 5 micromoles/liter to essentially nondetectable levels within 40 days after TCE injection began. Port A4 showed a similar pattern, though the maximum TCE concentrations were much lower. At the outlet, TCE concentrations remained very low throughout the experiment. The decrease in TCE from the inlet port to Port A1 is most likely due to adsorption and volatilization from the inlet tubing.

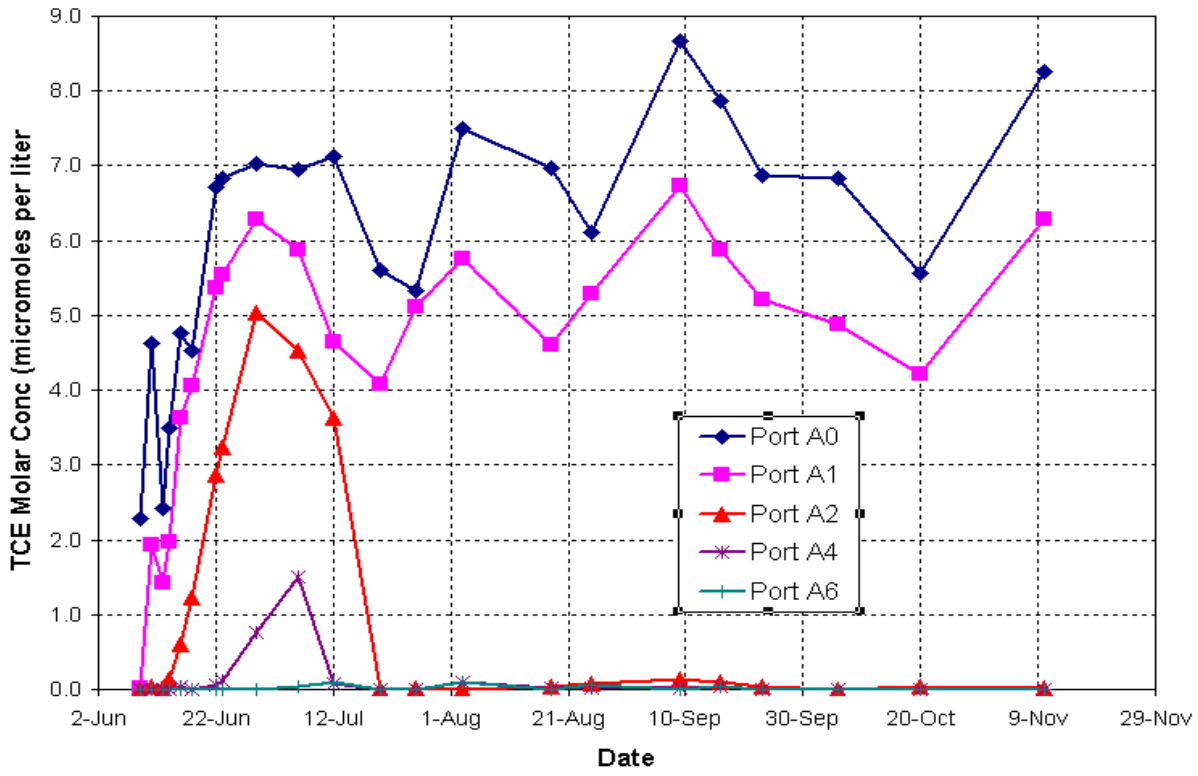

Fig. 3. Results of VOC measurements in Column A

At the time the TCE concentration was dropping within the column the concentrations of the biodegradation products were increasing as a result of sequential reductive dechlorination of the TCE. Figure 4 shows the change in composition of TCE over time at Port A5. TCE was never present at this port in significant concentrations; however, after 80 days, almost the entire molar mass of TCE was observed at port A5 in the form of cis-1,2DCE. Small amounts of trans-1,2 DCE, and 1,1DCE were also present, but the majority was cis-1,2DCE.

Towards the end of the planned monitoring period, a small amount of VC was observed at this port, indicating that the conditions in the column were suitable, if not ideal, for biodegradation

from DCE to VC to occur. To determine if the biodegradation from DCE to VC was limited by the natural bacterial population present in the soil, on 09/10/2004 the column was inoculated with a bacterial culture developed at HCET that has been shown to fully degrade TCE to ethene. As Figure 4 shows, after this date the amount of VC increased dramatically until all of the cis-1,2DCE had been converted to VC with small amounts of other DCE isomers also present. Near the end of the monitoring period the molar concentration of VC was almost three times higher than the molar concentration of TCE entering the column (7-9 micromoles/liter), indicating that, in addition to the TCE in the influent, the TCE adsorbed onto the soil was being degraded and converted to VC. Over time, the VC concentrations are expected to decrease as the stored mass of TCE is removed.

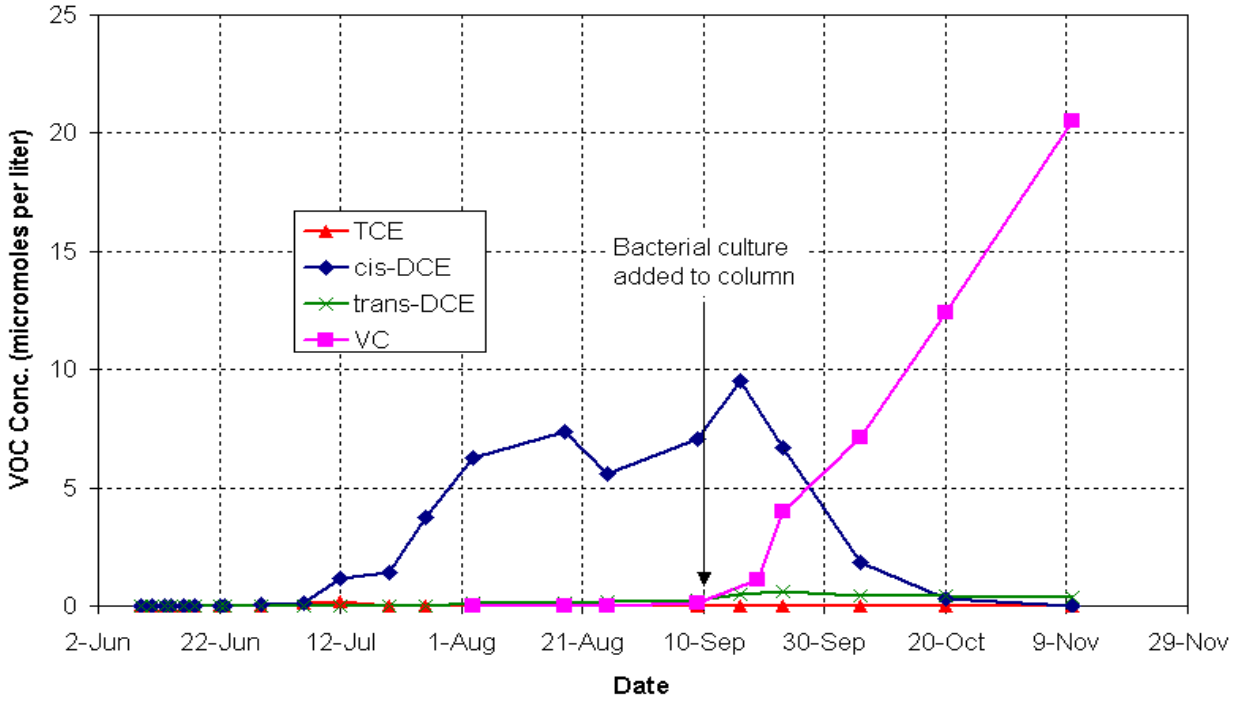

Fig. 4. TCE degradation products in Port A5

Near the end of the monitoring period the VC concentrations in the effluent (not shown) began to decrease, possibly indicating that the final step in the dechlorination reaction, the conversion of VC to ethene, was starting. One sample was analyzed for ethene. Approximately 16.7 micromoles per liter were detected, indicating possible conversion of VC to ethene. Analytical problems prevented the collection of additional ethene data; therefore, additional monitoring will be needed to fully document the complete conversion of TCE to ethene.

Column B, showed little change in TCE concentrations during the experiment, being limited to a slight decrease near the top of the column. No biodegradation products were ever observed, indicating that the sand could not support a TCE-degrading bacterial population. The decrease in TCE near the open top of the column suggests that some TCE was volatilizing through the semisaturated soil layer above the saturated zone. Based on the average concentration of TCE in

the middle of the column, compared to the outlet concentration, approximately 12% of the TCE moving through the column is thought to have volatilized from the column.

Field Experience at SRS

Scientists at several institutions have been studying the biodegradation of TCE at SRS for a number of years. These studies are intended to support the use of natural attenuation as a remedial strategy for contaminated groundwater at several locations. In one such area, the Twin Lakes Wetlands, contaminated groundwater flows upward through wetland soil in a manner similar to the columns studied in this project. Recent analysis of samples of water from 33 shallow wells screened in the wetland sediment and underlying soil indicate that TCE is biologically dechlorinated as the groundwater travels upward through the wetland sediments [10]. Below a depth of 2.4-3 meters TCE is the only significant chlorinated compound observed. At a depth of 1-1.5 meters the samples contained higher levels of cis-1,2DCE than TCE. In one particularly interesting set of wells located within 60 meters of each other, shown in Table II, an indication of sequential dechlorination can be seen. The deep wells in this area contained primarily TCE with small amounts of cis-1,2DCE. The shallow wells contained primarily cis-1,2DCE, indicating conversion of TCE to DCE. Two well clusters, CRP 42 and CRP 43 both showed significant conversion of TCE to DCE in 1 to 1.2 meters of vertical movement through the wetland soil column. One shallow well contained significant amounts of VC and ethene as well as DCE, indicating that at this point complete degradation of TCE was occurring. That fact that only one of five shallow wells contained VC and significant amounts of ethene indicates that biodegradation in the other wells may be only partial, with the degradation chain stopping at DCE. This observation is similar to what was seen in the mesocosm experiments prior to the inoculation of the soil with the TCE-degrading bacteria.

Table II. TCE and Daughter Products in Selected SRS Wells

Well Number	Depth (m)	TCE (μg/l)	cis-1,2DCE (μg/l)	VC (μg/l)	Ethene (μg/l)	ORP (mV)
CRP 42A	1.2	130	960	5(U)	.032	86.9
CRP 42B	1.8	650	680	5(U)	.018	148.9
CRP 43A	1.5	5(U)	54	5(U)	0.10	79.8
CRP 43B	2.4	11	30	5(U)	.034	30.1
CRP 48B	1.8	5.8	300(J)	180	12.0	N.A.
CRP 20CU	16.8	380 (J)	5(U)	5(U)	.290	N.A.
CRP 20CL	24.4	6800(J)	180	5(U)	.045	N.A.

N.A. signifies that the data was not available for these wells, 5(U) designated that none of the material was detected above the 5 μg/l detection limited, and (J) designates that the value was estimated since if falls below the quantitation limits.

The ORP measurements in those wells where ORP was measured were low but positive [11]. Several other wells in the Twin Lakes wetland sampled at the same time exhibited ORP values as low as -108.7mV, though most of the wells were in the range of +100 to –100mV, similar to what was observed in the wetland mesocosms experiments. The conditions in the SRS wetlands appear to be only mildly reducing and not the highly reducing methanogenic conditions often sited as required for complete biodegradation of TCE. However, it appears from the mesocosm results from this experiment, which adequately modeled conditions in an actual SRS wetland,

that complete degradation may be possible in some locations, given the proper bacterial populations.

Another recent study collected data on chlorinated organics in wetland sediment pore water using passive diffusion sampling devices buried up to 1.5 meters deep in the sediment for two to three weeks [3]. After recovery from the sediment, the water inside the sampling devices was withdrawn and analyzed for TCE and daughter products, as well as other organics. These samplers were deployed in the Pen Branch wetlands at SRS. The results indicated that widespread and complete biodegradation of TCE was not occurring; however, in several locations conversion of TCE to cis-1,2DCE was thought to be underway. These findings are consistent with the mesocosm results with exhibited limited conversion of cis-1,2DCE to VC under natural (unbioaugmented) conditions.

CONCLUSION

Batch adsorption studies measured the TCE partition coefficient for wetland soil as 29.2 L/Kg for wetland soil as compared to 5.0 L/Kg for sand, the experimental control, which results in retardation factors of over 50 for wetland soil and 13 for the sand control. This adsorption results in the accumulation of TCE on the soil surface. However, it was found that adsorbed TCE would easily desorb, becoming available to organisms capable of biodegrading TCE, thus removing the accumulated TCE from the soil surface. The wetland soil was found to increase pore water pH only slightly; however, the addition of 2% by weight of $CaCO_3$ was found to raise the pH to approximately 8.0.

The vertical-flow wetland column was found to quickly establish mildly-reducing conditions and successfully degrade TCE to cis-1,2DCE under up-flow conditions simulating a natural wetland. Within a few weeks of TCE introduction, all of the TCE flowing through this column was degraded to cis-1,2DCE with trace amounts of trans-1,2DCE and 1,1DCE. After approximately 80 days of operation a small amount of DCE was being converted to VC. This conversion to VC occurred with only the naturally-occurring soil bacteria originally present. However, enhancing the bacterial population with a known TCE-degrading culture increased the rate of conversion of DCE to VC. The detection of ethene indicates that further conversion of VC to ethene may also be occurring, but more data are needed to confirm this observation. Thus it appears that, even with the relatively low percentage of organic matter in the wetland soil tested, and resulting mildly reducing conditions, the wetland soil column can support the biodegradation of TCE to VC, and possibly to ethene, given the presence of a suitable consortia of TCE-degrading bacteria.

The decomposition of soil organic matter produced significant amounts of gas that accumulated within the soil matrix. The effect of this gas was to reduce the hydraulic retention time, which may reduce the effectiveness of the soil at biodegrading TCE. This effect will likely be more significant at higher organic carbon concentrations.

TCE concentrations measured in the sand-filled control column rapidly stabilized at concentrations close to the inlet concentration of 1.0 mg/L and no TCE biodegradation products were observed. A slight decrease in TCE concentration near the top of this column suggested that volatilization was occurring; however, the volatilization flux was small, 10-15% of the mass

of TCE entering the column each day. Conditions in the control column remained aerobic and oxidizing throughout the study.

The results of the column studies were compared with field investigations recently completed at several natural wetlands at SRS. The equilibrium conditions in the wetland column were similar to conditions measured in an SRS upflow wetland. Sequential dechlorination of TCE as water moved up through the SRS wetland soil was noted. From the results of the mesocosm studies, it appears that the degradation rate of TCE in SRS wetlands, and the subsequent conversion of DCE to VC and then to ethene, would be enhanced by the introduction of a known TCE degrading culture.

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