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Enhancement of TCE Attenuation in Soils by Natural Amendments

ZUHAL OZTURK, YELENA KATSENOVICH, BERRIN TANSEL, SHONALI LAHA, LAWRENCE MOOS, AND MARSHALL ALLEN

Applied Research Center and Civil and Environmental Engineering Department, Miami, FL, USA

The objective of this study was to identify low-cost natural amendments that could be used as carbon sources and sustain a bioactive zone to promote biodegradation of TCE in contaminated shallow groundwater. The natural amendments were compared based on their geophysical characteristics as well as TCE adsorption capacities. The amendments studied included low-cost natural and agricultural materials such as eucalyptus tree mulch, pine bark mulch, muck from the Florida Everglades, SRS wetland peat, commercial compost, and peat humus. These natural substrates have relatively high organic fractions that can retard the movement of TCE while serving as carbon sources. Batch sorption studies were conducted to determine the sorption and retardation characteristics of the amendments for TCE. The experimental results were analyzed in relation to the geophysical characteristics of the amendments and compared with those of natural soils.

Keywords Sorption, retardation factor, bioactive barriers, Freundlich model, TCE, soil amendments

Introduction

Bioactive barriers are subsurface layers constructed from mixtures of soil and soil amendments to support the growth of bacteria that are capable of degrading organic contaminants. Soil amendments are added to improve the physico-chemical as well as geophysical properties of soils (i.e., nutrient value, carbon content, pH adjustment, porosity, and density). The amendments suitable for use in bioactive barriers can improve the soil characteristics by providing the necessary conditions to sustain the microbial population capable of biodegrading organic contaminants while reducing their mobility in the soil media.

Trichloroethylene (TCE) is one of the most frequently detected organic compounds in groundwater and soils in the US (Azadpour-Keeley et al., 1999). Table 1 presents the TCE sorption characteristics of different soil media reported in the literature. Due to its hydrophobic nature, TCE is easily adsorbed onto media with high organic content. Therefore, soils and soil amendments which are rich in organic carbon have been considered for use to retard the movement of TCE in the subsurface (Mouvet et al., 1993; Kassenga et al., 2003; Lee and Batchelor, 2004). Sorption phenomenon is one of the major mechanisms that affect the transport of hydrophobic organic contaminants in the groundwater. The sorption

Address correspondence to Berrin Tansel, Civil and Environmental Engineering Department, Florida International University, Miami, FL 33176, USA. E-mail: transelb@fiu.edu

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Soil media	K_d (L/kg)*	K_{oc} calculated using K_d (L/kg)	Fraction of Organic Carbon \mathbf{f}_{or} (%)	Concentration Range (ppm)	Model*	Reference
Corrotant anoul	000	60.03	0.12	10-3 1	1 :====	Monnist at a1 (1002)
	60.0	C7.60	C1. 0	10 -1	LIIICAI	INIOUVEL EL AL. (1993)
Loamy soil A2 horizon	0.11	68.75	0.16	10^{-3} -1	Linear	Mouvet et al. (1993)
Coventry sandstone	0.11	57.89	0.19	10^{-3} -1	Linear	Mouvet et al. (1993)
Sandy soil C1 horizon	0.23	121.05	0.19	10^{-3} -1	Linear	Mouvet et al. (1993)
Sandy soil Ap horizon	0.44	129.41	0.34	10^{-3} -1	Linear	Mouvet et al. (1993)
Loamy soil Ap horizon	0.68	147.83	0.46	10^{-3} -1	Linear	Mouvet et al. (1993)
Balloy alluvium	0.50	102.04	0.49	10^{-3} -1	Linear	Mouvet et al. (1993)
Calcareous soil A/C horizon	0.67	85.90	0.78	10^{-3} -1	Linear	Mouvet et al. (1993)
Calcareous soil Ap horizon	2.51	209.17	1.20	10^{-3} -1	Linear	Mouvet et al. (1993)
Natural humus	33.90		N/A	0-200	Linear	Khandelwal and Rabideau
	(41.70)				(Freundlich)	(2000)
Soil-bentonite (SB)	0.53		N/A	0-200	Linear	Khandelwal and Rabideau
	(0.61)				(Freundlich)	(2000)
Soil-bentonite + natural	2.43		N/A	0-200	Linear	Khandelwal and Rabideau
humus (MSB)	(4.17)				(Freundlich)	(2000)
Natural wetland soil	32	139.13	23	N/A	Linear	Kassenga and Pardue (2001)
Synthetic peat mixture	13.5	46.55	29	N/A	Linear	Kassenga and Pardue (2001)
Latimer peat + sand	2.88	13.27 or	21.7	N/A	Linear	Kassenga et al. (2003)
	(5.71)	26.31	(mass averaged		(Freundlich)	
			value)			
Latimer peat $+$ sand $+$ bion	4.62	15.93 or	29	N/A	Linear	Kassenga et al. (2003)
soil	(10.97)	37.83	(mass averaged		(Freundlich)	
			value)			
Silawa soil	0.91	131.88	0.69	4 to 140	Linear	Lee and Batchelor (2004)

*Values in parenthesis indicate the numbers estimated using Freundlich adsoption model.

 Table 1
 Commarison of TCE Sorntion Characteristics of Different Soil Media

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capacity of the subsurface materials for specific contaminants affects both the mobility and the rate of biodegradation of the contaminant in the subsurface. The sorption capacity of the subsurface materials depends on their organic content and the geophysical characteristics (Allen-King et al., 1996; Khandelwal and Rabideau, 2000).

The sorption potential of a material for a specific contaminant can be described in terms of the partition coefficient, which is defined as the ratio of the contaminant concentration in the sorbed phase to that in the aqueous phases. Soils with organic carbon fractions higher than 0.1% have been observed to exhibit a high degree of sorption for hydrophobic organic compounds (Benker et al., 1998). The partitioning characteristics of organic contaminants between solid and liquid phases are commonly described by either the Freundlich adsorption model or the linear sorption equation, which is a special case of the Freundlich model (US EPA, 1991).

The Freundlich adsorption model can be expressed as:

$$S = K_F \cdot C_w^N \tag{1}$$

where,

S: contaminant mass sorbed on to the solid phase (mg/kg)

 K_F : distribution coefficient (L/kg)

 C_w : concentration in solution (mg/L)

N: constant (Freundlich isotherm exponent)

This equation can be simplified to a linear form when the value of N approaches unity as follows:

$$S = K_d \cdot C_w \tag{2}$$

where,

 K_d : the distribution coefficient for the linear sorption model

In this study, to distinguish the values of the distribution coefficients between Freundlich model and the linear sorption model, K_F and K_d notation has been used, respectively.

The relationship between K_d and the soil organic carbon fraction, f_{oc} can be described as:

$$K_d = f_{oc} \cdot K_{oc} \tag{3}$$

where,

 K_{oc} : Organic carbon content normalized distribution coefficient [L/kg] f_{oc} : Fractional organic carbon content of the sample (dimensionless)

For the linear form of the equation, the retardation factor (*R*) is a function of soil bulk density (ρ_b), porosity (θ), and distribution coefficient (K_d) and is defined by the following equation:

$$R = 1 + K_d \cdot \rho_b / \theta \tag{4}$$

Similarly, the retardation factor for the Freundlich equation can be written as:

$$R = 1 + K_F \cdot \rho_b / \theta \tag{5}$$

The objective of this study was to identify suitable low-cost natural soil amendments capable of attenuating TCE in contaminated shallow groundwater while serving as carbon

sources. The natural amendments investigated included high-organic content materials such as peat, humus, wetland muck, wood mulch and agricultural wastes, including compost, and other non-toxic, readily available solid substrates. These amendments consist primarily of carbon, hydrogen, and nitrogen and include lignin and cellulose that can serve as sorbents to retard the movement of TCE, as well as carbon sources for bacterial growth. The TCE adsorption characteristics of the natural amendments were studied and compared with those of natural soils.

Materials and Methods

The soil amendments used in batch sorption experiments were selected based on the analytical results for their total organic carbon (TOC) content and nutrient value (i.e., nitrogen and phosphorus). Amendments selected for in-depth analyses included a commercial organic humus (Southland Inc., North Carolina), wetland peat obtained from the Savannah River Site (SRS) (from Upper Three Runs Creek, South Carolina), pine bark mulch (Rick's Produce, Aiken South Carolina), muck from Florida Everglades (from Everglades agricultural area, South Florida), commercially available peat humus (Greenleaf Products, Haines City, Florida), organic compost (Greenleaf Products, Haines City, Florida), and eucalyptus tree mulch (Aaction Mulch, Inc., Florida).

TCE stock solution containing 750 mg/L TCE was prepared in methanol by using commercially available pure TCE (Fisher Scientific). The artificial groundwater stock solution was prepared using deionized water according to the solution characteristics provided by Strom and Kaback (1992) as presented in Table 2. The dissolved oxygen content of the artificial groundwater stock solution after preparation was 2 ± 1 mg/L. No adjustments were made to dissolve oxygen level of the artificial groundwater. All chemicals were reagent grade and obtained from Fisher Scientific and Aldrich Chemical Co., Inc.

For the batch sorption experiments, 1,000 grams of each amendment sample were oven dried at 50°C for 2 days. The dried sample was blended for 5 minutes and sieved using sieves No. 20 and No. 100 to prepare a sample with a particle range of 0.15 mm–0.85 mm for the batch sorption experiments. The particles larger than 0.85 mm and smaller than 0.15 mm were discarded. This particle range (0.15 mm–0.85 mm) was determined to be similar to the soil characteristics at the SRS site determined by sieve analysis. Using particles with similar size distribution to that of the site soils allowed uniform mixing with the soil samples.

For sorption studies, 1 ± 0.01 gram (dry weight) of the amendment sample was placed in a 60 mL bottle and the bottles were filled 2/3 full with artificial groundwater. The samples were mixed for 2 days by a shaker for saturation. After two days, the bottles were checked for air bubbles in the solution and on the surface of amendment particles. If any air bubbles

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Compound	Concentration (mg/L)
$CaCl_2 \cdot 6H_2O$	5,477
Na_2SO_4	1,073
$MgCl_2 \cdot 6H_2O$	3,094
KČI	400
NaCl	2,653

Table 2
Composition of Artificial Groundwater Stock Solution

were observed, the samples were placed back onto the shaker for additional mixing and observed twice daily for their saturation. The saturation time was determined by monitoring the bottles until there were no air bubbles forming. After it was observed that there were no air bubbles forming, the bottles were stirred for an additional 12 hours to ensure complete saturation of the amendment samples. The saturation time varied from 2 to 4 days depending on the amendment. After saturation, artificial groundwater was added with zero headspace and the bottles were covered with Teflon tape. To avoid any loss of TCE during its injection to the bottles, an appropriate volume of supernatant, equal to the TCE stock volume to be injected to the bottles, was extracted from each bottle punching through the Teflon tape already placed on them and then through the same punch the TCE stock was injected to the bottles. After TCE addition, the bottles were immediately closed with their caps and sealed with Teflon tape. Blank samples with only artificial groundwater and TCE spikes were also prepared to measure the extent of TCE sorption onto the bottle walls and caps and extent of TCE loss during sample collection. Each batch sorption test and blanks were conducted in triplicate. The batch sorption experiments were conducted on a reciprocating shaker maintained at 20°C (room temperature) and 100 rpm for 48 hours (U.S. EPA, 1991). To evaluate the sorption characteristics of the amendments in their natural or commercial state, the amendment samples were not autoclaved. Following the mixing period, the bottles were centrifuged at 2000 rpm at 10°C for 15 minutes according to the test procedure by Brigmon et al. (1998), and the supernatant was transferred to 40 mL VOC vials for TCE analyses.

To minimize error during sample handling and to determine the most efficient method of transferring supernatant from the 60 mL bottles to 40 mL analytical vials, a sample handling comparative study was conducted prior to the sorption experiments. Several methods for transferring the supernatant sample to 40 mL vials were evaluated. These methods included manual transfer, negative pressure direct transfer, and positive pressure direct transfer methods. The analytical results showed 61.8 %, 88.8 % and 99.5 % recovery for TCE by manual transfer, negative pressure direct transfer, and positive pressure direct transfer methods, respectively. Based on these results, positive pressure-direct transfer method, the vial cap was left slightly open to release air from the bottle while transferring the supernatant from the centrifuged samples as shown in Figure 1.

TCE concentration in the samples was analyzed by a purge and trap gas chromatography/mass spectrometry (GC/MS) method based on EPA methods SW5030B and SW8260B. Analyses were performed on a Varian Star 3400cx GC/ Varian Saturn 2000 MS coupled with a Tekmar Dohrmann 3100 sample concentrator. The samples were introduced to the concentrator using an Archon purge and trap auto sampler with 5 mL injection volume from the 40 mL glass vials. A VOCARB 3000 trap was used with the program setting as desorption time of 4.00 min at desorption temperature of 180°C, purge time of 2.5 min, bake time of 10.00 min at bake temperature of 180°C. Helium was used as the carrier gas. Analytical separations were carried out on a WCOT Fused Silica Select 624CB capillary column (30 m length \times 0.25 mm diameter, DF-1.4 μ m). The temperature program was set as 32°C for 2 min., then temperature was increased at 8°C/min to 190°C and 1 min final holding time.

Water content of the amendments was determined gravimetrically. The pH of the pore water (pHw) was measured by adding deionized water at a soil:water ratio of 1:1. The samples were stirred for 15 minutes and allowed to settle for 15 minutes before taking the pH measurements. Bulk density of the amendments was determined by gravimetric method (Blake and Hartge, 1986a). Particle density was determined by using the pycnometer method



Figure 1. Experimental set-up of positive pressure-direct transfer method.

(Blake and Hartge, 1986b). The porosity (θ) of the amendments was calculated from the experimental measurements of bulk and particle densities using the following equation (Danielson and Sutherland, 1986):

$$\theta = \left(1 - \frac{\rho_b}{\rho_p}\right) \tag{6}$$

where,

 θ : porosity ρ_b : bulk density (g/cm³) ρ_p : particle density (g/cm³)

The total nitrogen and TOC analyses of the amendments were conducted prior to TCE contamination. Total carbon and total nitrogen content of the amendments were determined using a Perkin Elmer 2400 CHNS Analyzer (Perkin Elmer Instruments LLC, Shelton, Connecticut). Total organic carbon was determined according to the EPA Method 9060 using a Shimadzu SSM-5000A TOC Analyzer.

Results

The characteristics of the natural amendments as determined experimentally are presented in Table 3. The pH_w values were within neutral range except for pine bark mulch and SRS wetland peat, which exhibited acidic conditions. The low pH of SRS wetland soil was due to the low alkalinity of the site soils and presence of decaying vegetation (dominated by pine and oak), which typically yielded acidic biodecomposition products.

Figure 2 compares the bulk densities and porosities of the amendments. SRS wetland peat and muck from Florida Everglades had similar bulk densities and porosities (bulk density of about 0.6 g/cm³ and porosity of about 0.66). The eucalyptus tree mulch and pine bark mulch had similar characteristics with relatively low bulk densities and high porosities

Soil/Soil Amendment	Bulk Density (g/cm ³)	Particle Density (g/cm ³)	Porosity	Total Carbon (mg/g)	Total Organic Carbon (mg/g)	% f _{oc}	Total Nitrogen (mg/g)	pH _w
Commercial Compost	0.94	2.30	0.59	129.10	108.70	10.87	3.00	7.35
SRS Wetland Peat	0.62	1.85	0.66	363.10	350.90	35.09	15.90	4.88
Eucalyptus Tree Mulch	0.28	1.54	0.82	456.60	404.30	40.43	15.10	7.27
Southland Organic Humus	0.89	2.39	0.63	61.77	61.66	6.17	2.60	7.48
Muck from Florida Everglades	0.61	1.81	0.66	345.50	277	27.70	21.80	7.16
Peat Humus	1.18	2.32	0.49	102.10	86.50	8.65	0.80	7.17
Pine Bark Mulch	0.33	1.55	0.79	449.09	448.88	44.90	13.20	4.74
SRS Compost	0.69	2.24	0.69	127.74	127.06	12.70	N/A	6.50

 Table 3

 Results of Geophysical Characteristics of Soil Amendment Materials

(bulk density of about of 0.3 g/cm^3 and porosity of about 0.8). Peat humus had relatively high bulk density and low porosity (bulk density of about of 1.18 g/cm^3 and porosity of about 0.5). Total carbon and total nitrogen contents of the amendments are presented in Figure 3. The total carbon contents of the amendments were between 61.66 mg/g and 448.88 mg/g, and total nitrogen content ranged from 0.80 mg/g to 21.80 mg/g. Both the eucalyptus tree



Figure 2. Comparison of bulk densities and porosities of the soil amendments.



Figure 3. Comparison of total carbon and total nitrogen contents of the soil amendments.

mulch and pine bark mulch had high carbon content while commercial compost and peat humus had relatively low carbon levels.

Figure 4 compares the total organic carbon contents and the organic carbon partition coefficients (K_{oc}) of the natural amendments for TCE. The SRS wetland peat had high carbon content with relatively high partition coefficient, which makes it a good candidate for use as an amendment in TCE contaminated areas. However, pH adjustment would be needed to support a viable environment for use as a bioactive barrier due to the acidic nature



Linear

Figure 4. Comparison of total organic carbon and TCE-organic carbon partition coefficients of the soil amendments as estimated by the linear model.

of SRS wetland peat (pH value of about 4.9). The near neutral pH value, high porosity, and high organic carbon content of Eucalyptus tree mulch make it a good candidate for use as a soil amendment in bioactive barriers. However, the organic carbon partition coefficient of the Eucalyptus tree mulch was relatively small.

Table 4 compares the linear and Freundlich isotherm equations based on the results of the sorption experiments conducted with each amendment. Muck from the Florida Everglades and SRS wetland peat sorption data gave an adequate fit with both models. Even though SRS wetland peat and Eucalyptus tree mulch had very similar carbon contents as TOC (350.90 mg/g and 404.30 mg/g, respectively), SRS wetland peat had significantly higher partition coefficient for TCE than that of Eucalyptus tree mulch due to differences in the nature of the organic matter in each material. Figure 5 presents the sample linear and Freundlich isotherms that gave adequate fit for the experimental data.

The organic carbon partition coefficient of the amendments did not show a correlation with the fraction of organic carbon (f_{oc}) as shown in Figure 6. This may be due to the differences in the forms of organic carbon present in the amendments. For example, both the Eucalyptus tree mulch and pine bark mulch had high total organic carbon with relatively low affinity for absorbing TCE. On the other hand, the organic carbon present in SRS wetland peat, muck from Florida Everglades, commercial compost, and peat humus had relatively high affinity for TCE. Figure 6 also presents the organic carbon partition coefficients of natural soils and peat-sand mixtures as reported by other researchers (Mouvet et al., 1993; Kassenga and Pardue, 2001; Kassenga et al., 2003). On the other hand, the distribution coefficients determined by the linear sorption model (K_d) correlated well with the fraction of organic carbon present in each amendment as shown in Figure 7. The SRS wetland peat had a significantly high K_d value for its organic carbon fraction, which may be due to the

Soil Amendment	Linear Equation (1)	Freundlich Equation (2)
Commercial Compost	y = 8.4441x	$y = 6.8437 x^{0.6678}$
	$r^2 = 0.6392$	$r^2 = 0.7674$
SRS Wetland Peat	y = 86.425x	$y = 59.188 x^{0.7446}$
	$r^2 = 0.7612$	$r^2 = 0.8736$
Eucalyptus Tree Mulch	y = 12.328x	$y = 12.458x^{1.0552}$
	$r^2 = 0.605$	$r^2 = 0.6954$
Muck from Florida	y = 22.486x	$y = 21.272x^{0.9664}$
Everglades	$r^2 = 0.9226$	$r^2 = 0.8917$
Peat Humus	y = 7.0605x	$y = 6.2754x^{0.9898}$
	$r^2 = 0.5791$	$r^2 = 0.5708$
Pine Bark Mulch	y = 11.736x	$y = 7.8601 x^{0.4808}$
	$r^2 = 0.2431$	$r^2 = 0.7214$
SRS Compost	N/A	$y = 6.8496x^{0.4612}$
-		$r^2 = 0.7799$
Southland Organic Humus	N/A	N/A

 Table 4

 Sorption Isotherm Equations of Soil Amendments

(1) Eq. 2; (2) Eq. 1.



a. Linear isotherms fitted for selected amendments





Figure 5. Linear and Freundlich isotherms that gave adequate fit for the experimental data.



Figure 6. Comparison of TCE-organic carbon partition coefficients of the soil amendments in relation to the organic carbon fraction.

presence of organic decay products which show higher affinity for TCE. All the amendments showed significantly high K_d values in comparison to natural soils. Based on the K_d values, peat humus, compost, natural wetland soil, Everglades muck, mulch and SRS wetland peat would increase TCE sorption potential of the site soils.



Figure 7. Comparison of TCE sorption potentials of the soil amendments in relation to the organic carbon fraction.

		Linear		Freundlich			
Soil Amendments	K _d (L/kg)	K _{oc} (L/kg)	R	K _F (L/kg)	K _{oc} (L/kg)	R	
Commercial Compost	8.44	77.64	14.45	6.84	62.93	11.90	
SRS Wetland Peat	86.43	246.30	82.19	59.19	168.68	56.60	
Eucalyptus Tree Mulch	12.33	30.50	5.21	12.46	30.82	5.25	
Muck from Florida Everglades	22.49	81.20	21.78	21.27	76.79	20.66	
Peat Humus	7.06	81.62	18.00	6.28	72.60	16.11	
Pine Bark Mulch	11.74	26.15	5.90	7.86	17.51	4.28	
SRS Compost	N/A	N/A	N/A	6.85	53.94	7.85	
Southland Organic Humus	N/A	N/A	N/A	N/A	N/A	N/A	

 Table 5

 Distribution Coefficients K_d, K_F, K_{oc} and Retardation Factors (R) of Soil Amendments

As shown in Table 5, when the linear sorption model was used, the SRS peat had the highest adsorption coefficient (86.43 L/kg) with a retardation factor of 82.19 while peat humus had the lowest (7.06 L/kg) with a retardation factor of 18. The Freundlich model gave similar results in identifying amendments with high adsorption potential. When the Freundlich model was used, the adsorption coefficient of the SRS peat was 59.2 L/kg with a retardation factor of 56.6 and the adsorption coefficient of the peat humus was 6.28 L/kg with a retardation factor of 16.11. The Southland organic humus had the lowest TOC (61.66 mg/g) among the amendments studied. Neither the linear nor Freundlich isotherm produced an adequate fit for the Southland Organic Humus. For the SRS compost data, only the Freundlich isotherm produced an adequate fit.

Conclusions

This study was conducted to investigate the suitability of natural materials for use as amendments in construction of permeable bioactive barriers to retard movement of TCE in contaminated soils. The ability of different soil/soil amendment mixtures to adsorb TCE, and their geophysical characteristics such as bulk density, particle density, porosity and TOC, were evaluated. In general, the TCE soil-water distribution coefficients increased with increasing organic carbon content of the amendments.

Peat humus showed suitable properties for use as soil amendment; however, it had relatively low porosity (0.49). Pine bark mulch had the highest TOC among the amendments evaluated; however, it exhibited acidic characteristics. Eucalyptus mulch had a high TOC level similar to pine bark mulch and was less acidic. Eucalyptus tree mulch and the two compost materials showed favorable characteristics in terms of TOC, pH_w, and K_d values in comparison to the other amendments studied.

The linear model was adequate for Eucalyptus mulch, SRS peat, Everglades muck and peat humus within the TCE concentration range studied (0.1–0.8 mg/L TCE). The TCE adsorption characteristics for compost, SRS compost and pine bark mulch showed adequate fit for the Freundlich model. The SRS peat had the highest retardation factor while both the Eucalyptus tree mulch and pine bark mulch had the lowest retardation factors for TCE.

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