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Nonideal transport of contaminants in heterogeneous porous media: 10. Impact of co-solutes on sorption by porous media with low organic-carbon contents

M.L. Brusseau^{1,2,*}, G. Schnaar^{1,†}, G.R. Johnson³, and A.E. Russo¹

¹Department of Soil, Water and Environmental Science, University of Arizona, 429 Shantz Building, Tucson, AZ 85721

²Department of Hydrology and Water Resources, School of Earth and Environmental Sciences, University of Arizona, 429 Shantz Building, Tucson, AZ 85721

³Portland State University, OR

Abstract

The impact of co-solutes on sorption of tetrachloroethene (PCE) by two porous media with low organic-carbon contents was examined by conducting batch experiments. The two media (Borden and Eustis) have similar physical properties, but significantly different organic-carbon (OC) contents. Sorption of PCE was nonlinear for both media, and well-described by the Freundlich equation. For the Borden aquifer material (OC = 0.03%), the isotherms measured with a suite of co-solutes present (1,2-dichlorobenzene, bromoform, carbon tetrachloride, and hexachloroethane) were identical to the isotherms measured for PCE alone. These results indicate that there was no measurable impact of the co-solutes on PCE sorption for this system. In contrast to the Borden results, there was a measureable reduction in sorption of PCE by the Eustis soil (OC = 0.38%) in the presence of the co-solutes. The organic-carbon fractions of both media contain hard-carbon components, which have been associated with the manifestation of nonideal sorption phenomena. The disparity in results observed for the two media may relate to relative differences in the magnitude and geochemical nature of these hard-carbon components.

Keywords

nonlinear sorption; competitive sorption; hard carbon; HOCs

1. Introduction

Sorption is one of the primary processes influencing the transport and fate behavior of organic compounds in the environment. An accurate understanding of sorption mechanisms and their impact can often be critical for assessing risk, evaluating bioavailability, and implementing remediation efforts. One key aspect of characterizing sorption processes is determining the potential significance of rate-limited, nonlinear, and hysteretic sorption (e.g., Brusseau and Rao, 1989; Pignatello and Xing, 1996; Luthy et al., 1997), which may be considered nonideal behavior in contrast to the ideal case of instantaneous, linear, non-hysteretic sorption. Over the past two decades, a large body of research has been developed focusing on nonideal sorption and potential causative mechanisms. Attention has recently focused on the condensed or hard-carbon components of soil organic carbon, such as black

*Corresponding author: Brusseau@email.arizona.edu.

†Now with Daniel B. Stephens & Associates, Inc.

carbon and kerogen, as potential sources of nonideal sorption behavior (e.g., Luthy et al., 1997; Xing and Pignatello, 1997; Weber and Young, 1997; Bucheli and Gustafsson, 2000; Jonker and Koelmans, 2002; Ran et al., 2003; Xiao et al., 2004; Cornelissen et al., 2005; Chai et al., 2007; Prevedouros et al., 2008; Johnson et al., 2009; Russo et al., 2010).

Competitive sorption is another potential nonideal sorption phenomenon that may have significant impacts on transport and fate behavior. Several studies have been conducted to examine the impact of co-solutes on sorption of lower-polarity organic compounds by natural soils and sediments (e.g., Lee et al., 1988; McGinley et al., 1993; Xing et al., 1996; Xing and Pignatello, 1997; White and Pignatello, 1999; White et al., 1999; Li and Werth, 2001; Rivett and Allen-King, 2003; Ju and Young, 2004; Zhang et al., 2010; Xiao and Huang, 2011). The majority of such experiments have used porous media with total organic-carbon contents of 1% or greater. Conversely, few have been conducted using media with organic-carbon contents between 0.1 and 1% (McGinley et al., 1993; Li and Werth, 2001; Zhang et al., 2010), and fewer still with values less than 0.1% (Lee et al., 1988; Rivett and Allen-King, 2003).

The objective of this research was to examine the impact of co-solutes on sorption of tetrachloroethene by two porous media with low organic-carbon contents. One medium employed is the Borden aquifer material, collected from the site at which a well-known field study was conducted to examine the impact of sorption phenomena on transport of organic compounds in groundwater (Mackay et al., 1986). The second medium is a sandy soil that has similar physical properties as the Borden material, but a significantly larger organic-carbon content.

2. Materials and Methods

2.1 Materials

Tetrachloroethene (PCE) was used as the model organic compound. In addition, 1,2-dichlorobenzene, bromoform, carbon tetrachloride, and hexachloroethane were used as the co-solutes. Dichloromethane was used to extract sorbed-phase PCE for selected batch-reactors, as discussed below. All chemicals (Aldrich Chemical Co., Inc., Milwaukee, WI) were reagent grade. Relevant properties of the chemicals are presented in Table 1.

Borden aquifer material was collected from the site at which the well-documented natural-gradient organic tracer experiment was conducted (Mackay et al., 1986). The samples were collected from an interval approximately 1.5 – 3 m below ground surface (and below the water table), coincident with the injection interval for the field experiment. Two samples were collected from different locations, with one (used for experiments 1 and 2 in Figure 1) collected adjacent to the injection zone. The properties of the Borden sediment, and its sorption of organic compounds, have been characterized in several studies (e.g., Curtis et al., 1986; Lee et al., 1988; Ball and Roberts, 1991; Ran et al., 2003; Rivett and Allen-King, 2003). Eustis sandy surface soil was collected from the University of Florida campus. The properties and sorption behavior of the Eustis soil have also been characterized in several studies (Brusseau et al., 1989; Nkedi-Kizza et al., 1989; Brusseau et al., 1991; Brusseau, 1993; Russo et al., 2010; Brusseau et al., 2012). Both media were air dried after collection and stored for future use.

Pertinent properties of the media are presented in Table 2. The physical properties of the two media are similar. Conversely, the organic-carbon content for the Eustis soil is approximately ten times larger than that of the Borden material. The geochemical compositions of the organic-carbon fractions of the two media were characterized previously (Russo et al., 2010). The organic carbon for Borden aquifer material comprises a high

percentage (~65%) of kerogen and black carbon, consistent with the results of Ran et al. (2003). The organic carbon in the Eustis media is composed of approximately 37% hard carbon (kerogen and black carbon) and 63% soft carbon (humic/fulvic acids, lipids).

For the Borden experiments, an electrolyte solution was prepared to be representative of the groundwater at the Borden field site, as described by Ball and Roberts (1991). The solution comprised CaCO_3 (100 mg L^{-1}), MgSO_4 (25 mg L^{-1}), NaCl (3.8 mg L^{-1}), and KCl (1.1 mg L^{-1}). An electrolyte solution containing 0.01 N CaCl_2 was used for all Eustis experiments. Aqueous solutions of PCE and the other solutes were made by dilution of pre-prepared stock solutions saturated with the target compounds. The stock solutions were prepared without the use of methanol or other solubilizers.

2.2 Experiment Setup

Three sets of experiments were conducted. The first comprised preliminary batch rate experiments to determine the time required for sorption to attain equilibrium, which was less than approximately 2 d for samples prepared with pulverized media, consistent with prior results (Ball and Roberts, 1991). Equilibration times of 3–5 d were used for all experiments employing pulverized samples, with longer times for those employing unpulverized samples.

The second set of experiments was conducted to examine the impact of a suite of co-solutes on the sorption of PCE for Borden and Eustis media. The concentrations used for the co-solutes were matched to the values used in the Borden natural-gradient field study, as follows: 1,2-dichlorobenzene ($332 \mu\text{g L}^{-1}$), bromoform ($32 \mu\text{g L}^{-1}$), carbon tetrachloride ($31 \mu\text{g L}^{-1}$), and hexachloroethane ($20 \mu\text{g L}^{-1}$). Three sets of triplicate samples were prepared, one set with PCE solution only and no sediment (control), one set with sediment and PCE solution (no co-solutes), and one set with sediment, PCE solution, and the co-solutes.

Based on the results observed for the second set of experiments, a third set of experiments was conducted to further examine the potential impact of co-solute on PCE sorption for the Borden sediment. For this set, 1,2-dichlorobenzene was used as the sole co-solute. Two initial concentrations were used, a low ($100 \mu\text{g L}^{-1}$) and a high (100 mg L^{-1}), with the latter value close to the aqueous solubility of 1,2-dichlorobenzene. Several sets of triplicate samples were prepared similarly to experiment set 2.

2.3 Batch Experiment Methods

Prior to use, the porous media were pulverized using a ball mill to enhance attainment of sorption equilibrium (Ball and Roberts, 1991). In addition, a parallel set of reactors were prepared with unpulverized media to examine the impact of pulverization. No differences in sorption were observed between the two sets of samples. The media were not subjected to autoclaving or other such treatments that may alter properties, and biocides were not employed in the solutions.

Batch reactors consisted of 20 mL flame-sealed glass ampules (Wheaton), which eliminated the use of Teflon and other potentially reactive materials. The ampules were filled with 10–15 g of porous media and approximately 8–10 mL of aqueous solution to minimize headspace. After the ampules were filled, they were immediately flame sealed with an oxygen-natural gas sealing torch (Labconco). A range of four or five initial concentrations (nominal $10 \mu\text{g L}^{-1}$ – 100 mg L^{-1}) were used for PCE. Each concentration was reproduced in several sets of triplicates, along with sediment-free control samples. After sealing, the samples were rotated along the longitudinal axis at 4 rpm using a rotating mixer.

Three sets of ampules for each concentration were sacrificed at selected times to measure PCE concentrations. Prior to sampling, the ampules were centrifuged at 1100 rpm for 30 min to achieve solid-water separation. After sampling, each ampule was oven-dried at 105°C for 24 hours to obtain dry sediment mass. Mass loss of PCE during the experiments via volatilization to headspace and biotransformation was characterized by analysis of the controls for PCE concentrations and by checking selected samples for degradation products. No degradation products were detected, and the PCE concentrations for the controls were typically within approximately 2% of the initial concentrations.

The mass of PCE sorbed to the sediment material was calculated from the change in the mass in aqueous solution, correcting for any volatilization to headspace based on concentrations measured for the control samples. Sorbed mass calculations were verified by subjecting selected samples to solvent extraction with dichloromethane. Mass balances of approximately 90% were achieved, consistent with expected recovery efficiencies associated with standard solvent extraction (e.g., Ball and Roberts, 1991).

2.4 Chemical Analysis

The organic compounds were analyzed by headspace gas chromatography (Tekmar-Dohrmann 7050 coupled with a Shimadzu GC-17A) using either a flame ionization (FID) or electron capture (ECD) detector depending on sample concentration. Chromatographic analysis was done using a glass capillary column (SPB-624, 30-m length, 5- μ m film thickness) with oven temperature programming. Specifically, analysis began with oven temperature set for two minutes at 40 °C, ramped to 150 °C at a rate of 10 °C per minute, and held for two minutes. The injection port temperature was set at 180 °C, with the detectors at 180 °C and 210 °C for the FID and ECD, respectively. Aqueous-phase standards were analyzed every 48 h with check standards and blanks analyzed for quality assurance every 10 to 15 samples. The quantifiable detection limits using headspace GC/ECD were approximately 0.1 μ g L⁻¹. Selected samples were also analyzed using UV-Vis spectrophotometry (QDL = 1 mg L⁻¹) to provide confirmation of analytical results.

3. Results and Discussion

Sorption of PCE by both porous media is nonlinear and is well described by the Freundlich isotherm ($S = K_f C^n$, where S is sorbed-phase concentration and C is aqueous-phase concentration), as shown in Figures 1 and 2. Sorption is moderately nonlinear, with n values of 0.9 and 0.85 for Borden and Eustis, respectively. The magnitude of sorption is significantly greater for Eustis, as would be expected based on its significantly greater organic-carbon content.

For the Borden aquifer material, the isotherms measured with the suite of co-solutes present (1,2-dichlorobenzene, bromoform, carbon tetrachloride, and hexachloroethane) are identical to the isotherms measured for PCE alone (see Figure 1). The regressions obtained with the Freundlich equation are statistically similar for all cases. The impact of varying the concentration of a single co-solute (1,2-dichlorobenzene) from 0 to 100 mg L⁻¹ is shown in Figure 3. Again, the isotherms measured with and without co-solutes present are identical. These results indicate that there was no measurable impact of the co-solutes on PCE sorption for this system.

The impact of a co-solute (trichloroethene, 10 mg l⁻¹) on sorption of PCE by Borden sediment was investigated in a prior study (Rivett and Allen-King, 2003). Competitive sorption effects were reported for lower concentrations of PCE. The impact of a co-solute (p-xylene) on sorption of trichloroethene by Borden sediment was investigated by Lee et al. (1988). They reported no measurable competitive sorption. The difference in results may be

attributed in part to the solutes and co-solutes used in the respective studies. Trichloroethene and PCE are from the same chemical family and are therefore structurally similar, whereas the primary co-solute used herein (1,2-dichlorobenzene) and the co-solute used by Lee et al. are structurally dissimilar from the target solute (PCE or trichloroethene). Xing et al. (1996) examined competitive sorption for a series of compounds, and observed significant competition between structurally similar solutes and minimal competition between structurally dissimilar compounds. In addition, the sediment used for the PCE-trichloroethene study was collected relatively far (~175 m) from the location of the natural-gradient study.

The results obtained for the Eustis soil are presented in Figure 2. In contrast to the Borden results, it appears that there is a reduction in sorption of PCE at lower concentrations in the presence of the co-solutes. Specifically, the K_f value from the Freundlich equation regression changed from 1.8 (1.6–2.0) in the absence of co-solutes to 1.3 (1.1–1.5) in the presence of co-solutes. In addition, the value of the exponent, n , increased from 0.85 (0.83–0.87) to 0.94 (0.87–1.0). These observed changes in K_f and n upon addition of a co-solute are consistent with those observed in prior studies (e.g., Xing et al., 1996; Xiao and Huang, 2011).

The results of the experiments indicate that nonideal (i.e., competitive) sorption was observed for the Eustis soil but not for the Borden aquifer material. These results are consistent with those of Russo et al. (2010), who examined the impact of rate-limited desorption on elution of trichloroethene for several porous media, including the Borden and Eustis materials. They observed that greater nonideal desorption and elution behavior was observed for the Eustis soil compared to the Borden sediment.

As noted previously, attention has recently focused on the hard-carbon components of soil organic carbon as a source of nonideal sorption behavior. For systems wherein this is true, the occurrence and magnitude of nonideal-sorption effects are expected to be mediated by the magnitude and nature of hard-carbon components contained within the porous media. The hard-carbon content for Eustis soil (0.14%) is significantly larger than that of the Borden aquifer material (0.02%). In a prior study, Li and Werth (2001) compared the impact of a co-solute on sorption of trichloroethene for two soils with similar total organic-carbon contents (0.2%) but different magnitudes of hard carbon. Competitive sorption was observed for the one with a larger fraction of hard carbon (0.14%), but not for the one with a lower fraction (0.09%).

The suite of co-solutes used herein was based on those used in the well-known natural-gradient field study conducted at the Borden site (Mackay et al., 1986). In addition, the experiments conducted herein employed concentrations of PCE that span the concentrations of PCE observed in the field study. Specifically, an initial PCE concentration of $30 \mu\text{g L}^{-1}$ was used for the field study, and the lowest aqueous concentrations for the batch experiments are more than ten times lower than this value (Figure 2). One of the major observations of the field study was that transport of PCE was nonideal, in that the PCE plume decelerated with time in contrast to the near-constant velocity observed for the non-reactive tracer plumes (Roberts et al., 1986). This disparity indicates that the magnitude of PCE retardation increased with time and transport distance. The cause of this behavior has been subject to discussion (e.g., Roberts et al., 1986; Brusseau and Srivastava, 1997). One potential cause is competitive sorption, wherein the impacts of such were maximal in the early stage (when all solutes were co-located) and then decreased with time as the individual solute plumes separated during transport (due to chromatographic separation). The absence of competitive sorption observed herein for the Borden media under conditions

representative of the field study suggests that such effects were not a significant contributor to the nonideal transport behavior observed for the field study.

4. Summary

The impact of co-solutes on sorption of PCE by two porous media with lower organic-carbon contents was examined by conducting batch experiments. Sorption of PCE was nonlinear for both media, and well-described by the Freundlich equation. The results indicated that there was no measurable impact of the co-solutes on PCE sorption for the Borden medium. In contrast, there was a reduction in sorption of PCE by the Eustis soil in the presence of the co-solutes. The two media have similar physical properties, but significantly different organic-carbon contents. The organic-carbon fractions of both media contain hard-carbon components, which have been associated with the manifestation of nonideal sorption phenomena. The disparity in results observed for the two media may relate to relative differences in the magnitude and geochemical nature of these hard-carbon components.

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Figure 1.

Isotherm data for sorption of tetrachloroethene by the Borden aquifer material. Single and Multiple refer to the absence or presence of co-solutes (1,2-dichlorobenzene, bromoform, carbon tetrachloride, and hexachloroethane). Regression obtained with Freundlich equation [$S = 0.23 C^{0.9}$, $r^2 = 0.991$, where S is sorbed-phase concentration and C is aqueous-phase concentration].

Figure 2.

Isotherm data for sorption of tetrachloroethene by the Eustis soil. Single and Multiple refer to the absence or presence of co-solutes (1,2-dichlorobenzene, bromoform, carbon tetrachloride, and hexachloroethane). Regression obtained with Freundlich equation [Single: $S = 1.8 C^{0.85}$, $r^2 = 0.998$; Multiple: $S = 1.3 C^{0.94}$, $r^2 = 0.992$], where S is sorbed-phase concentration and C is aqueous-phase concentration.

Figure 3. Isotherm data for sorption of tetrachloroethene by the Borden aquifer material. Impact of the presence of a single co-solute (1,2-dichlorobenzene).

Table 1

Properties of the Chemicals.

Compound	Aqueous Solubility ^a (mg L ⁻¹)	Log K _{ow} ^b	Henry's Constant ^b
Tetrachloroethene	150	2.6	0.59
Bromoform	3190	2.3	0.02
Carbon Tetrachloride	1160	2.7	0.98
1,2-dichlorobenzene	145	3.4	0.08
Hexachloroethane	50	3.6	0.12

^aFrom Verschueren (1996).^bFrom Curtis et al. (1986).

Table 2

Porous Media Properties.

Porous Medium	Sand %	Silt %	Clay %	Organic Carbon Content %	Bulk Density (g/cm ³)	Porosity	Median Grain Diameter d ₅₀ (mm)	Uniformity Coefficient (d ₆₀ /d ₁₀)
Borden	96	2.0	2.0	0.03	1.74	0.34	0.21	1.9
Eustis	96	1.0	3.0	0.38	1.69	0.36	0.27	2.3