

Partitioning Behavior of Trichloroethylene in Cyclodextrin–Water–Granular-Activated Carbon Systems

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Abstract

The purpose of this study was to collect experimental data and develop a mathematical model describing the equilibrium phase distribution of a common organic chemical, trichloroethylene (TCE), among cyclodextrin (CD), water, and granular-activated carbon (GAC). Batch tests and a modified linear regression model were applied to assess the partitioning behavior in this multiphase (CD pseudophase–water–GAC) system. Results showed that CD lowers TCE availability for dissolution in water and adsorption to GAC, demonstrated by reduced TCE partitioning to GAC with increasing CD concentration. Presence of CD does not significantly affect the distribution of TCE between water and GAC. Rescaled Freundlich adsorption isotherms for the GAC–water (CD-free) and GAC–CD solution systems were not significantly different and overlapping normalized isotherms were observed. This is an important indicator that the distribution of TCE within the dissolved and GAC phase is independent of the presence of CD. It further indicates that the distribution within CD–water–GAC systems can be aptly described by standard TCE Freundlich isotherms for water–GAC dual-phase systems and water–CD partitioning coefficients. A linear relationship between the logarithms of apparent Freundlich isotherm coefficients and solubility enhancement factors ($R^2=0.99$) was verified theoretically and experimentally. This study provides important information to optimize the design, assess the performance of GAC treatment systems containing CD solutions, and possibly aid in prediction the lifetime of GAC in a full-scale operation.

Key words: cyclodextrin; granular-activated carbon; trichloroethylene; Freundlich isotherm; solubility enhancement

Introduction

THERE IS A GROWING BODY of evidence suggesting that cyclodextrin (CD) can be an effective solubility-enhancement agent for *in situ* flushing of contaminated soil and aquifer sediments (Wang and Brusseau, 1993; Bizzigotti *et al.*, 1997; McCray *et al.*, 2000). Application of CD to typical groundwater pump-and-treat remediation in an economically feasible manner requires that the extracted pollutants be removed from postflushing solution so that the CD solution can be recycled. Recent pilot-scale studies have demonstrated that pollutant separation from CD solutions can be accomplished by a combination of air stripping and granular-activated carbon (GAC) adsorption (Blanford *et al.*, 2000; Tick *et al.*, 2003; Boving *et al.*, 2006).

While those pilot studies demonstrated the feasibility of removing contaminants from postflushing CD solutions, they did not quantitatively investigate the phase distribution behavior of chemicals in air-stripping systems and GAC treat-

ment beds. Such information is necessary to go beyond the trial-and-error testing of treatments and to develop models that can accurately forecast the performance of systems. In short, the phase distribution of chemicals in relevant multiphase systems must be studied in greater depth in order for development of more cost-effective alternatives for aquifer remediation with CD flushing. The chemical partitioning behavior in air–water (Gossett, 1987) and air–water–CD systems (Kashiyama and Boving, 2004; Gao *et al.*, 2009), which is important for air-stripper design, has been investigated by a number of researchers, and there are, to our knowledge, no published studies of CD-influenced partitioning behavior in CD–water–GAC systems. Hence, there is an unmet need to evaluate the interaction between chemical and GAC in the presence of CD.

The partitioning behavior of chemicals in dual-phase (water–GAC) system is normally described through adsorption isotherms, which can be modeled by the Linear, Freundlich, and Langmuir equations (Giles *et al.*, 1974; Tchobanoglous *et al.*, 2004; Limousin *et al.*, 2007). Extensive studies have been done to determine the adsorption behavior of taste and odor compounds (Gilligly *et al.*, 1998; Cook *et al.*, 2001), organic (Karanfil and Kilduff, 1999; Pelekani and Snoeyink, 1999) and inorganic contaminants (Brasquet *et al.*, 2002; Wilson *et al.*,

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2006) in GAC–water systems. The addition of CD to the aqueous phase, however, introduces a third phase, causing a reduction in the amount of chemicals in the water phase because CD's hydrophobic cavity provides a more stable environment for chemicals than water does (Shirin *et al.*, 2003). In addition, several studies have shown that the presence of solubilizing agent (SA) (e.g., surfactant or CD) reduces the aqueous activity and thereby the driving force for the partitioning of chemicals into gas or solid phase (Tucker and Christian, 1985; Underwood *et al.*, 1995; Cheng and Sabatini, 2007). Mathematical models describing the partitioning behavior of chemicals in air–water–SA system normally treat the CD cavity/micelle as a pseudophase (Vane and Giroux, 2000; Shimotori and Arnold, 2003; Lantz *et al.*, 2005; Gao *et al.*, 2009) or treat the association of chemical-CD as an equilibrium action (Wang and Brusseau, 1993; Connors, 1997; Kashiwama and Boving, 2004).

In this study, the effects of CD on the partitioning behavior of a common chlorinated solvent trichloroethylene (TCE) on a GAC (Norit C Gran) in a three-phase system was evaluated using the pseudophase approach over a range of hydroxypropyl- β -CD concentrations (HP β CD). To our knowledge, this is the first data set on CD pseudophase–water–GAC partitioning yet assembled for this organic pollutant. We have applied a robust experimental design and statistical data analysis method for the acquisition and explanation of partitioning behavior in multiphase system. The objectives of this study were to (1) obtain quantitative descriptions of the effects of CD concentration of TCE adsorption behavior on GAC, (2) to determine TCE equilibrium distribution in multiphase system (CD–water–GAC) as a function of CD concentration, (3) to further investigate the potential correlations between apparent Freundlich adsorption coefficients and the CD solubility enhancement factors. This study provides important information to optimize the design, assess the performance of GAC treatment systems containing CD solutions, and possibly aid in predicting the lifetime of GAC in a full-scale operation. It may also be possible to use the equilibrium adsorption coefficients over a range of CD concentrations as a reference parameter to conduct a dynamic column study for the purpose of investigation of contaminant transport in porous media.

Experimental Protocols

Chemicals

TCE was purchased from Sigma-Aldrich (>99.5% purity). HP β CD (degree of substitution=4.98, average MW 1424 g/mol) was donated by Cargill and used as received (purity wt./wt.: moisture content 5.0%; unmodified CD <0.1%; propylene glycol 0.4%; ash 2.0%; HP β CD 92.6%). NORIT C GRAN (Norit Americas, Inc.) is a chemically activated granular carbon produced by a phosphoric acid activation process applied to coal and used in the food, chemical, and pharmaceutical industries.

Effect of CD on TCE equilibrium phase distribution

Aqueous CD solutions with concentrations of 0, 20, 50, and 100 g/L were prepared by addition of CD powder to the high-purity water. TCE solution (54.32 mg/L) was prepared by dissolving pure TCE into the different CD solutions. For the

batch adsorption isotherm experiments, weighed activated carbon aliquots were placed in seven amber bottles that were subsequently completely filled with TCE-CD solutions with the solid/liquid ratios of 1:20, 1:40, 1:70, 1:90, 1:140, 1:200, 1:280 g/mL. All the bottles were capped immediately using PTFE-faced silicon septa to create headspace-free conditions. Continuous mixing was providing during the adsorption experimental period with a constant agitation speed of 250 rpm through a rotary shaker (Model: LE "Big Bill"; Sybron Thermolyne). A preliminary experiment demonstrated that after 5 days of mixing, the remaining concentration of TCE in aqueous solution changed by <0.1%, which is considered to represent equilibrium adsorption. In this study, a mixing time of 7 days was employed to ensure that equilibrium was achieved. Longer contact times were not used due to concerns regarding GAC particle destruction (Limousin *et al.*, 2007).

At the end of the 7-day equilibration period, mixing was halted and suspended GAC particles were allowed to quiescently settle for 1 h. Supernatant (2 mL) was then transferred into amber vials via a glass, gas-tight syringe. Vials were sealed immediately after subsampling and placed in a Gas Chromatograph (GC) autosampler rack. Analyses were performed using an Agilent 6890 GC fitted with a HP-5MS column using a static headspace technology described in previous study (Gao *et al.*, 2009) and the TCE concentration in aqueous phase was determined from external calibration standard method. One advantage of the static headspace technique is that it does not require direct injection of liquid sample to GC-MS and therefore the presence of CD and the potential trace GAC residue in the liquid phase would not be expected to influence results and would not cause damages to the analytical equipment. Control samples were conducted using the same procedures to monitor the nonsorption losses and the results showed that the nonsorption losses were <0.1%.

Data analysis

Traditional and apparent Freundlich isotherms. For systems containing only water and GAC (Fig. 1A), the phase partitioning of TCE, phase A, is described by the traditional Freundlich isotherm [Eq. (1)], a relationship between the adsorbed concentration C_{AS} (phase S, mg/g) in the GAC and the solute concentration C_{A2} (phase 2, mg/L) remaining in water (Tchobanoglous *et al.*, 2004).

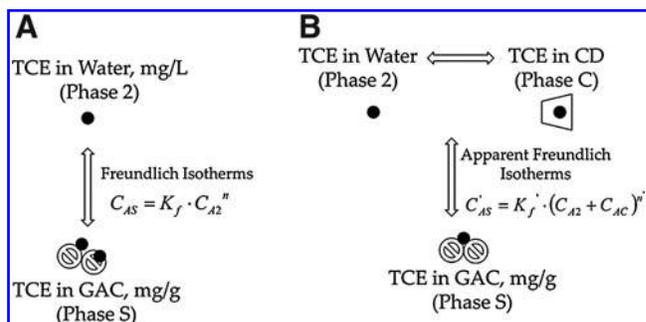


FIG. 1. Scheme of TCE partitioning behavior among phases without (A) and with (B) cyclodextrin. TCE, trichloroethylene.

$$C_{AS} = K_f \times C_{A2}^n \quad (1)$$

where K_f is the conventional Freundlich adsorption coefficient that indicates the adsorption ability and n is a constant indicative of adsorption affinity (Weber and DiGiano, 1996; Mcelroy, 2005). In systems that contain CD (Fig. 1B), an apparent Freundlich isotherm between the TCE concentration in the GAC phase and remaining concentration in the aqueous CD solution is employed [Eq. (2)]. A term is added to the water concentration (C_{A2}) of Equation (1) to account for the chemical present in the aqueous solution that is associated with CD cavities (C'_{A2} , mg/L).

$$C'_{AS} = K'_f \times (C_{A2} + C'_{A2})^{n'} \quad (2)$$

where K'_f is the apparent Freundlich adsorption coefficient and n' is adsorption constant in the presence of CD, C'_{AS} is the adsorbed concentration in the presence of CD (mg/g) and C'_{A2} is the TCE concentration associated with CD cavities (mg/L).

To describe the TCE mass distribution in each phase (CD pseudophase, water, and GAC), the partition coefficient that describes the ratio of the CD-associated and water-solubilized TCE concentrations, K'_{AC2} [Eq. (3)], is adopted from our previous research that analyzed chemical phase distribution among CD, water, and air phases (Gao *et al.*, 2009).

$$K'_{AC2} = \frac{K_{A12}}{K'_{A12}} - 1 \quad (3)$$

where K'_{A12} and K_{A12} are the Henry's law constants with and without the presence of CD. Once the total TCE concentration in the liquid phase ($C_{A2} + C'_{A2}$) was determined via GC/MS analysis and the partition coefficient K'_{AC2} was calculated using Equation (3), the concentration of TCE present in the extra-CD cavity (water-solubilized phase) and the one associated with CD pseudophase can be determined through a mass balance relation.

Modified statistical model on adsorption coefficients. A modified linear regression model was built to evaluate the significance of adsorption coefficients under different CD concentrations. A natural log-transform was imposed to convert the power relationship ($C_{AS} = K_f \times C_{A2}^n$) to a linear model ($\ln C_{AS} = n \ln C_{A2} + \ln K_f$). This model [Eq. (4)] coupled with dummy variables, L_1 , L_2 , and L_3 [Eq. (5)], and interaction terms, I_1 , I_2 , and I_3 [Eq. (6)], was used to evaluate whether the intercept, $\ln K_f$, and the slope, n , are significantly different among the isotherms under different CD concentrations. To be specific, significant parameters for the dummy variables ($L_{1,2,3}$) and interaction terms ($I_{1,2,3}$) indicate significant difference regarding $\ln K_f$ values and n among the isotherms, respectively. The linear regression model and the dummy variables are described below. The model is run via statistical software SAS 9.2 (SAS Institute, Inc.) and the significant values are used to make a conclusion.

$$\ln C_{AS} = \beta_0 + \beta_1 \ln C_{A2} + \beta_2 \times L_1 + \beta_3 \times L_2 + \beta_4 \times L_3 + \beta_5 \times I_1 + \beta_6 \times I_2 + \beta_7 \times I_3 \quad (4)$$

$$\begin{aligned} L_1 &= \begin{cases} 1 \rightarrow C_{CD} = 0 \text{ g/L} \\ 0 \rightarrow \text{others} \end{cases}, \\ L_2 &= \begin{cases} 1 \rightarrow C_{CD} = 20 \text{ g/L} \\ 0 \rightarrow \text{others} \end{cases}, \\ L_3 &= \begin{cases} 1 \rightarrow C_{CD} = 50 \text{ g/L} \\ 0 \rightarrow \text{others} \end{cases} \\ I_1 &= L_1 \ln C_{A2} \\ I_2 &= L_2 \ln C_{A2} \\ I_3 &= L_3 \ln C_{A2} \end{aligned} \quad (5)$$

where $\ln C_{AS}$ and $\ln C_{A2}$ are values of dependent and independent variables, $\beta_{0,1,2,3,4,5,6,7}$ are regression parameters, $L_{1,2,3}$ are dummy variables served as the indicator of the $\ln K_f$ values of the individual isotherms, and $I_{1,2,3}$ are interaction items served as the indicator of the n values.

Correlation between K'_f and E . An empirical relationship between the Freundlich adsorption coefficient, K'_f of activated carbon, and the aqueous solubility S was reported in previous research (Mcelroy, 2005).

$$\ln K'_f = -A \ln S + B \quad (7)$$

where A and B are constants. In systems containing CD, aqueous solubility of chemicals, S , can be estimated by (McCray *et al.*, 2000):

$$S = E \times S_w \quad (8)$$

where E is solubility enhancement factor and S_w is the aqueous solubility. Substitution of Equation (8) in (7) produces an overall correlation [Eq. (9)].

$$\ln K'_f = -A \ln (E \times S_w) + B \quad (9)$$

For a specific chemical, S_w is a constant; therefore, Equation (9) can be rearranged and a linear relationship between $\ln K'_f$ and $\ln E$ is obtained.

$$\ln K'_f = -A \ln E + M \quad (10)$$

where M is a constant that can be determined by plotting $\ln K'_f$ versus $\ln E$.

Results and Discussion

Mass distribution of TCE in multiphase system

The total TCE quantity present in the liquid phase ($C_{A2} + C'_{A2}$) was determined via GC/MS analysis; the individual concentrations in the water and CD pseudophase were calculated by applying the partition coefficient, K'_{AC2} , and the adsorbed quantity was calculated from the mass balance relationship. To illustrate how mass distribution in CD-water-GAC systems changes with CD concentration, a three-dimensional column diagram (Fig. 2) shows the fraction present in each phase. The plots represent systems with the solid:liquid ratio of 1:280. As can be seen in Fig. 2, the amount of TCE initially in the aqueous phase was adsorbed to the GAC phase with the mass fraction adsorbed to GAC of 93%,

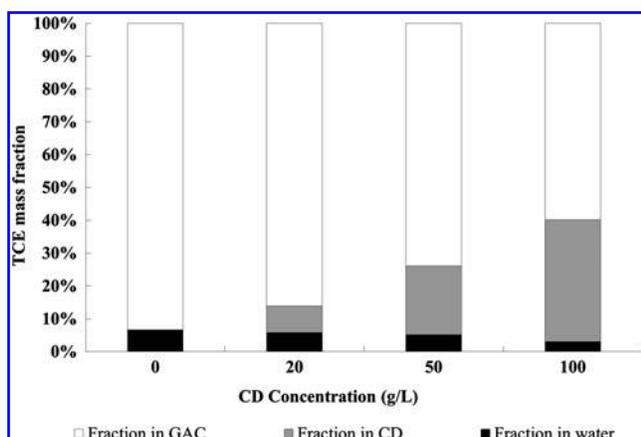


FIG. 2. TCE mass distribution in CD–water–GAC system with solid:liquid ratio of 1:280—water phase (■), CD (■), and GAC (□). CD, cyclodextrin; GAC, granular-activated carbon.

86%, 74%, and 60% for 0, 20, 50, and 100 g/L CD solutions, respectively. As CD concentration increased, the mass fraction in both the GAC and water phase decreases, which can be explained by the hydrophobic property of CD cavity. As the CD concentration increased, more hydrophobic cavities are available for TCE partitioning (Boving and Brusseau, 2000; Kashiya and Boving, 2004). This apparently resulted in a lower fraction of TCE present in the water-dissolved phase, hence, the trend of decrease in fraction adsorbed in GAC phase.

Effect of CD concentration on apparent Freundlich isotherms

The apparent Freundlich isotherm was determined by first measuring the total concentration of chemical remaining in the aqueous phase ($C_{A2} + C'_{A2}$) and then using a mass balance approach to calculate the GAC adsorption concentration C'_{AS} . The apparent adsorption coefficient K'_f and constant n' were then determined by the nonlinear regression of C'_{AS} versus

TABLE 1. FREUNDLICH ADSORPTION COEFFICIENTS (K'_f) AND CONSTANTS (n') IN CYCLODEXTRIN SOLUTIONS

Parameters	Isotherms	No CD	20 g/L CD	50 g/L CD	100 g/L CD
K'_f	Apparent isotherms	7.71	4.99	3.46	1.99
	Rescaled isotherms	7.71	7.64	7.51	7.67
n'	Apparent isotherms	0.48	0.47	0.46	0.49
	Rescaled isotherms	0.48	0.47	0.46	0.49

CD, cyclodextrin.

($C_{A2} + C'_{A2}$). The apparent Freundlich isotherms of TCE are illustrated in Fig. 3, and the tabulated values of the adsorption coefficients are shown in Table 1. As shown, the apparent Freundlich isotherm well described the experimental TCE data, with all correlation coefficients above 0.90 (Fig. 3). Further evidence of the good fit of the Freundlich isotherm can be seen from the results of the statistical analyses (Table 2). The p -values for the constant and $\ln C_{A2}$ of both apparent and traditional isotherms are less than the significance level (0.05), indicating strong power relationship between the aqueous concentration C_{A2} and the adsorption concentration C_{AS} . The p -values for dummy variables (L_1 , L_2 , and L_3) of apparent isotherms are less than critical value (0.05) (the third column of Table 2), indicating significant differences among the K'_f values of apparent isotherms. As can be seen from Fig. 3, the isotherms illustrated a radial spread trend, and the GAC showed greater K'_f value in 0 g/L CD, followed by 20, 50, and 100 g/L CD. This can be readily attributed to the fact that with an increase of CD concentration, a greater amount of TCE partitioning to CD; therefore, there is a decreasing trend in the adsorbed concentration and hence K'_f values (Fig. 3). The p -values for interaction items (I_1 , I_2 , and I_3) of both the apparent and rescaled isotherms, however, are far larger than 0.05. Therefore, as shown in Table 1, there was no

FIG. 3. Effect of CD concentrations on TCE apparent Freundlich isotherm: no CD (×, —), 20 g/L CD (■, ---), 50 g/L CD (▲, - - - -), and 100 g/L CD (●, · · · ·). Discrete dots are experimental data and the lines are shown for isotherm references.

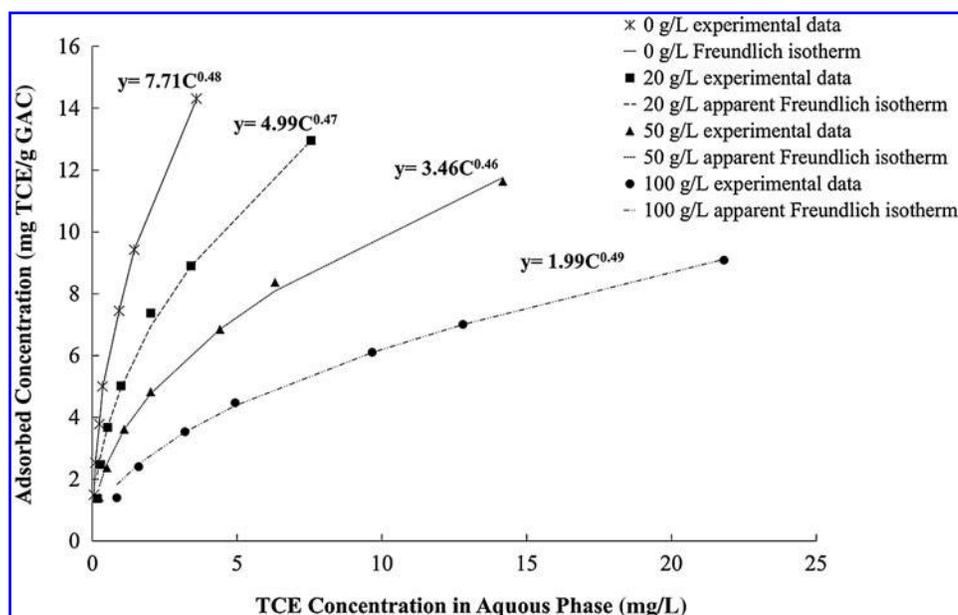


TABLE 2. RESULTS OF SAS LINEAR REGRESSION ANALYSES OF ADSORPTION ISOTHERMS

Apparent isotherms			Rescaled isotherms		
Variables	Regression parameters	p-Values	Variables	Regression parameters	p-Values
Constant	β_0	0.000	Constant	β_0	0.000
$\ln C_{A2}$	β_1	0.000	$\ln C_{A2}$	β_1	0.000
L_1	β_2	0.000	L_1	β_2	0.362
L_2	β_3	0.000	L_2	β_3	0.583
L_3	β_4	0.000	L_3	β_4	0.912
I_1	β_5	0.558	I_1	β_5	0.558
I_2	β_6	0.903	I_2	β_6	0.903
I_3	β_7	0.258	I_3	β_7	0.258

statistically significant differences among the adsorption affinity values, n' , for the isotherms.

Rescaled Freundlich isotherm and significance analysis

The rescaled Freundlich isotherms for 20, 50, and 100 g/L CD solutions were obtained from nonlinear regression of C'_{AS} versus the water phase concentration C_{A2} and a comparison was made with the isotherm obtained from CD-free (0 g/L CD) solutions. The K_f and n values of the rescaled isotherms are shown in Table 1, and the p -values of the statistical anal-

ysis of the comparison are listed in Table 2. Because the p -values of L_1 , L_2 , and L_3 (Table 2, column 6) are far greater than the critical level (0.05), it can be concluded that there is no significant difference between the derived rescaled Freundlich isotherms in CD solutions and the traditional one obtained from CD-free solution. This is an important indication that although the presence of CD can lower the absolute amount of TCE available to water and GAC phases, it however does not alter the relative distribution of TCE between water and GAC, which was supported by the observation of overlapping normalized isotherms (Fig. 4). It further implies that the ratio of water dissolved to GAC adsorbed concentration is aptly described by standard TCE adsorption isotherms for water-GAC dual-phase system. This finding is consistent with the partitioning behavior of chemicals in other multiphase systems (air-water-CD and air-water-surfactant) in which the contaminants are shielded from the solubilized phase by the CD cavity or micelle and the nonshielded portion redistributes in between air-water phases as explained with standard Henry's law (Anderson, 1992; Cheng *et al.*, 2001; Gao *et al.*, 2009).

Correlation between K'_f and E

The plot of apparent Freundlich adsorption coefficient, K'_f , versus solubility enhancement factor, E , and CD concentration is illustrated in Fig. 5. As shown, K'_f is inversely related to E with the natural logarithms linear relationship coefficient of

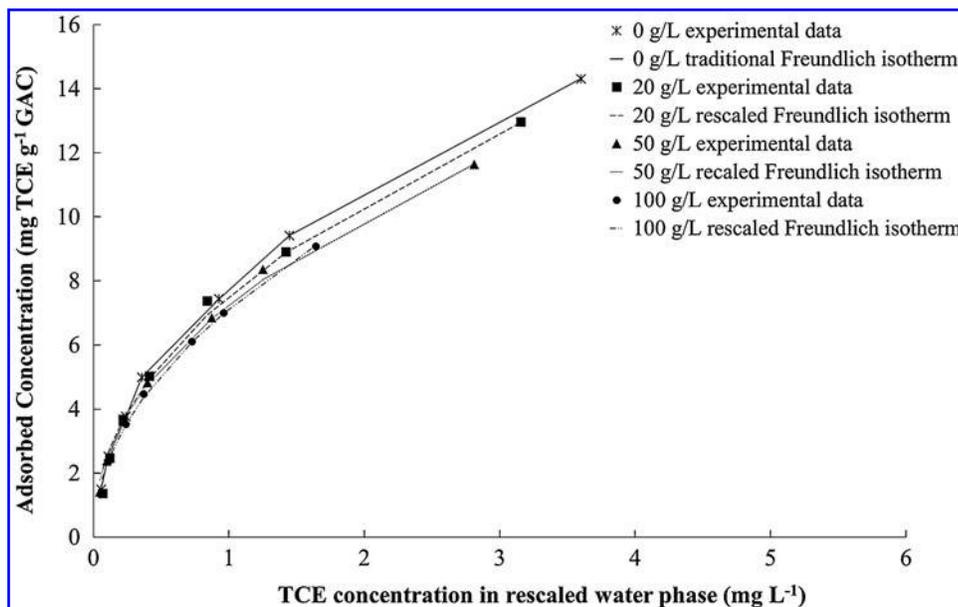


FIG. 4. Comparison of rescaled and CD-free Freundlich isotherms in CD concentrations: no CD (\times , —), 20 g/L CD (\blacksquare , ---), 50 g/L CD (\blacktriangle , - - - -), and 100 g/L CD (\bullet , - · - · -). Discrete dots are experimental data and the lines are shown for isotherm references.

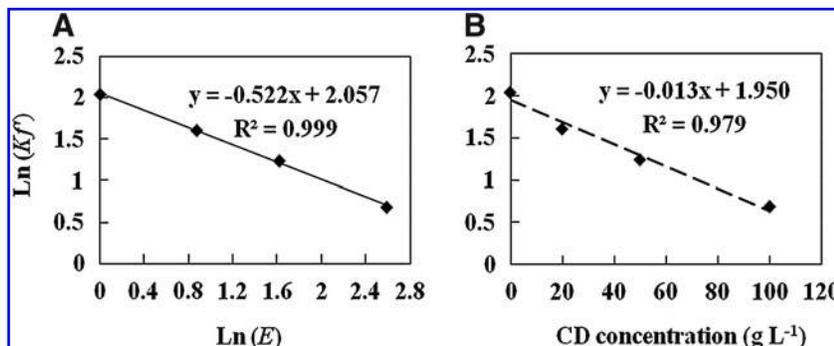


FIG. 5. Correlation of apparent Freundlich coefficients with solubility enhancement factors (A) and CD concentrations (B).

0.999. This can be explained by the energy associated with the adsorption phenomena. K_f' is considered to represent the energy necessary for a chemical to displace a solvent in order to be adsorbed on the adsorbent (Chiou, 2002). For similar solutions under constant conditions, more energy is required to adsorb more chemical due to the increased amount of solvent that must be displaced (McElroy, 2005). For this specific study, a further understanding is that as the CD concentration and solubility enhancement factor increases, more TCE partitions to CD. Therefore, the amount available to adsorb to GAC is decreased and fewer water molecules need to be displaced, resulting in less required energy and smaller K_f' values. This finding can significantly reduce the effort required to accurately estimate isotherm data by using similar correlation between Freundlich adsorption coefficient and solubility enhancement factor during postflushing solution treatments.

Summary

The purpose of this study was to collect experimental data and develop a mathematical model for the equilibrium phase distribution between CD, water, and GAC of a common organic chemical, TCE. The key findings of this study can be summarized as below:

- The presence of CD does not alter the relative distribution of TCE between water and GAC, which was supported by the observation of overlapping normalized isotherms. That is to say, even though some portion of TCE was associated with CD, the rest of TCE can be redistributed between water and GAC phases according to the coefficient obtained from CD-free solutions. This is an important indicator that the relative distribution of TCE between the water and GAC phases is independent of the presence of CD. It further indicates that the distribution of TCE between CD–water–GAC systems can be aptly described by standard TCE Freundlich isotherms for water–GAC dual-phase systems and water–CD partitioning coefficients.
- It however does lower the absolute amount of TCE available to water and GAC phases due to the association of TCE with CD cavities with the evidence that apparent adsorption of TCE to GAC decreased with increasing CD concentration. This implies that absolute amount of TCE would be absorbed less on solid GAC and water phases but associated more in presence of CD. These findings could be important for optimizing the design, assessing treatment performance, and possibly aiding in predicting the lifetime and GAC replenishment rates in full-scale operations.
- Apparent adsorption coefficient, K_f' , decreased as CD concentration increased and adsorption constant, n , was independent of CD concentration. There was a linear relationship between the natural logarithms of K_f' and CD solubility enhancement factor, which is reflective of the mathematical nature of the Freundlich isotherms.

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Author Disclosure Statement

No competing financial interests exist.

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