Predicting the Capacity of Powdered Activated Carbon for Trace Organic Compounds in Natural Waters

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The purpose of this research was to develop a simple method for predicting the powdered activated carbon (PAC) capacity for micropollutants in natural water. The herbicide atrazine and the odor-causing compound 2-methylisoborneol served as target compounds. Isotherm data from experiments conducted with a constant initial adsorbate concentration and varying adsorbent doses exhibited nonlinearity in both single- and multi-solute systems. However, at any given adsorbent dose, the PAC capacity for the micropollutant in multi-solute systems was directly proportional to the initial micropollutant concentration. Using the ideal adsorbed solution theory (IAST), an equation was derived that validated the experimentally observed direct proportionality between PAC capacity and initial micropollutant concentration at a given adsorbent dose. The results of this study show that the PAC dose to remove any amount of micropollutant from natural waters can be estimated without the use of mathematical models from a single isotherm experiment conducted in the natural water of interest, provided that the initial target compound concentration is sufficiently low.

Introduction

Powdered activated carbon (PAC) adsorption processes in drinking water treatment can effectively control problems related to trace organic substances such as taste- and odorcausing compounds, volatile organic compounds, and synthetic organic compounds including pesticides. As a result, the use of PAC in drinking water treatment has gained popularity because (i) PAC can be applied only when it is needed, e.g., during a pollution episode such as elevated pesticide concentrations during spring runoff or a chemical spill in a river; (ii) PAC can be fed in many drinking water treatment plants following a relatively small capital investment; and (iii) PAC is cheaper than granular activated carbon (GAC). However, with increasingly more stringent legislation

pertaining to the control of micropollutants, it has become essential to develop tools that allow professionals in the drinking water industry to design effective PAC adsorption processes and to evaluate their feasibility.

To predict the removal of trace organic compounds from natural water by PAC, both equilibrium and kinetic parameters need to be determined. While the kinetic parameters that describe the rate of trace organic compound adsorption from natural waters can easily be determined with the pseudo-single-solute homogeneous surface diffusion model (1, 2), the quantification of the competitive effect of BOM on the equilibrium parameters of trace organic compounds has been difficult to date.

Mathematically describing adsorption equilibria in multisolute systems of known and unknown composition has been the subject of many research studies (1, 3-15). The simplest approach to date that describes competitive adsorption equilibria in systems containing micropollutants and natural organic matter treats the competing background organic matrix as one hypothetical compound, the equivalent background compound (EBC) (10). Based on single-solute isotherm parameters for the target compound and isotherm data for known initial concentrations of the target compound in natural water, the parameters describing the adsorption characteristics of the EBC can be determined by solving the equations of the ideal adsorbed solution theory (IAST) as an inverse problem (11). Using the EBC method, Najm et al. successfully predicted adsorption equilibria of 2,4,6-trichlorophenol and metazachlor in different natural waters (10). Qi et al. also applied the EBC method successfully to model atrazine removal from Illinois groundwater by PAC (1). Although the EBC method has been applied successfully, the experimental and mathematical modeling efforts required for model calibration are substantial. Therefore, the principal objective of this research was to develop a simple method for the capacity prediction for trace organic compounds in the presence of natural organic matter. The following analysis of isotherm data and the IAST led to results that greatly simplified the capacity prediction for micropollutants in the presence of competing BOM.

Experimental Section

Organic-Free Water and Natural Water. Organic-free water was obtained by deionizing and distilling Urbana, IL, tap water. The water had a conductivity of $\leq 0.2~\mu\text{S/cm}$, and the dissolved organic carbon (DOC) concentration was $\leq 0.3~\text{mg/L}$. Single-solute isotherm experiments were conducted in this water at sufficiently high initial target compound concentrations (about $1000~\mu\text{g/L}$) such that competition due to the remaining DOC was not observed.

Illinois groundwater from a well located in Urbana, IL, served as a natural water source. The pH of this water was 7.3, and the DOC concentration was 3 mg/L.

Adsorbents. Two activated carbons were tested: PICA A PAC (PICA, Levallois, France), a thermally activated coconut shell-based activated carbon, and PICA B PAC (PICA, Levallois, France), a chemically activated wood-based activated carbon.

Adsorbate. Experiments were conducted with ¹⁴C ring-labeled atrazine (Ciba-Geigy, Greensboro, NC, and Sigma Chemical Company, St. Louis, MO) to facilitate quantification of herbicide concentrations at trace levels. To analyze samples for atrazine, 2.5-mL aliquots were mixed with 18 mL of scintillation cocktail (Ecoscint, National Diagnostics, Manville, NJ) in scintillation vials. The emitted radiation was then measured in a liquid scintillation analyzer (Tri-

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TABLE 1. Comparison of Atrazine and EBC Solid-Phase Concentrations for PICA B PAC

	initial atrazine concentration		ratios	
PAC dose	36.0 μg/L	8.3 μg/L	36.0/8.3 = 4.34	
10 mg/L	$q_{\mathrm{Atra}} = 12.93 \mu \mathrm{mol/g}$	$q_{Atra} = 3.011 \mu mol/g$	12.93/3.011 = 4.29	
	$q_{\rm EBC} = 87.0 \mu \rm mol/g$	$q_{\mathrm{EBC}} = 87.0 \mu \mathrm{mol/g}$		
100 mg/L	$q_{\text{Atra}} = 1.655 \mu \text{mol/g}$	$q_{\text{Atra}} = 0.381 \mu \text{mol/g}$	1.655/0.381 = 4.34	
	$q_{\rm EBC} = 8.70 \mu \rm mol/g$	$q_{\rm FBC} = 8.70 \mu \rm mol/g$		

Carb Model 1600CA, Packard Instrument Company, Downers Grove, IL). For a specific activity of 7.8 mCi/mmol atrazine or greater, a detection limit of $0.1\,\mu\text{g/L}$ was realized without sample concentration.

Isotherms. The bottle point isotherm technique as outlined by Randtke and Snoeyink (16) was employed to determine the equilibrium capacity of PACs for trace organic compounds. In an isotherm experiment, different amounts of carbon were added to amber glass bottles. Subsequently, a known volume of solution containing the target compound was transferred to the isotherm bottles, which were then placed on a shaker table. Screening studies conducted over a 3-week period showed that equilibrium was reached within 1 week of agitation. Upon equilibration, the remaining liquidphase concentration of the trace organic compound was measured, and the solid-phase concentration on the PAC was calculated via a mass balance. Three blanks were included with each isotherm experiment to evaluate adsorbate losses by mechanisms other than adsorption. No losses were detected in the blanks over the 1-week equilibration time.

Results and Discussion

Model Simplification. As a starting point for modeling competitive adsorption equilibria in systems involving nonlinearly adsorbing micropollutants and background organic matter (BOM), the ideal adsorbed solution theory (IAST) was employed. The competing BOM was represented by a single, hypothetical compound, the equivalent background compound (EBC). Thus, the system was modeled as a bi-solute system, in which the two adsorbates were the trace organic compound and the EBC. Incorporating the Freundlich isotherm equation ($q = KC^{1/n}$) into the IAST, the following pair of equations are obtained for each PAC dose in a bi-solute system:

$$C_{1,0} - q_1 C_c - \frac{q_1}{q_1 + q_2} \left(\frac{n_1 q_1 + n_2 q_2}{n_1 K_1} \right)^{n_1} = 0$$
 (1)

$$C_{2,0} - q_2 C_c - \frac{q_2}{q_1 + q_2} \left(\frac{n_1 q_1 + n_2 q_2}{n_2 K_2} \right)^{n_2} = 0$$
 (2)

where $C_{i,0}$ is the initial concentration of adsorbate i (μ mol/L), q_i is the equilibrium solid-phase concentration of adsorbate i (μ mol/g), C_c is the PAC dose (g/L), and K_i and $1/n_i$ are the single-solute Freundlich isotherm constants for adsorbate i.

The subsequent model simplification assumes that compound 1 is the trace organic compound and that compound 2 is the EBC. Solving eqs 1 and 2 poses a problem because the single-solute Freundlich isotherm constants of the EBC (K_2 and $1/n_2$) and the initial concentration of the EBC (C_2 ,0) are not known; in addition, the EBC solid-phase concentration (q_2) has to be computed for each PAC dose. To calibrate the EBC model, a nonlinear least-squares optimization routine is required to obtain the EBC characteristics K_2 , $1/n_2$, and C_2 ,0 (11, 12, 14). However, analysis of experimental data and mathematical modeling results obtained in the course of this study showed that the PAC capacity for a micropol-

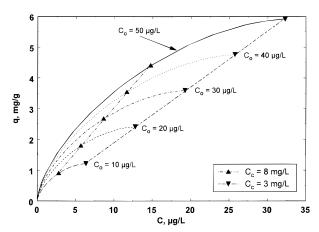


FIGURE 1. EBC model results for the adsorption of atrazine on PICA B PAC from Illinois groundwater. Nonlinear curves depict atrazine isotherms for initial atrazine concentrations in the range of 10-50 μ g/L. Linear trends depict the proportionality between PAC capacity and initial atrazine concentration for PAC doses of 3 and 8 mg/L.

lutant in natural water was proportional to the initial micropollutant concentration at any given PAC dose. This proportionality was observed even though nonlinear isotherms were obtained from experiments conducted with a given initial micropollutant concentration and varying PAC doses. Figure 1 summarizes these observations by depicting EBC model results for the adsorption of atrazine from Illinois groundwater on PICA B PAC.

For nonlinear isotherms, the proportionality between PAC capacity and initial micropollutant concentration at any given PAC dose is consistent with the IAST when the following assumptions hold: (i) the EBC solid-phase concentration (q_2) is much greater than the solid-phase concentration of the target compound (q_1) ; and (ii) the Freundlich exponents $1/n_1$ and $1/n_2$ are similar, i.e., both fall into the range between 0.1 and 1. Incorporation of the above assumptions into eq 1 leads to the following expression for the solid-phase concentration of the target compound, q_1 :

$$q_{1} = \frac{C_{1,0}}{C_{c} + \frac{1}{q_{2}} \left(\frac{n_{2} q_{2}}{n_{1} K_{1}} \right)^{n_{1}}}$$
(3)

Equation 3 demonstrates the direct proportionality between q_1 and $C_{1,0}$, provided that the solid-phase concentration of the EBC (q_2) at a given carbon dose (C_c) is not affected by trace levels of the target compound. To illustrate that the presented assumptions are valid in systems where a micropollutant competes with BOM for adsorption sites on the activated carbon surface, IAST results for atrazine adsorption on PICA B PAC in Illinois groundwater are summarized in Table 1. For this example, the Freundlich exponents for atrazine and the EBC were equal $(1/n_1=1/n_2=0.65)$. Table 1 compares the predicted solid-phase concentrations for the EBC with those for atrazine at PICA B PAC doses of 10 and 100 mg/L and initial atrazine concentrations of 36.0 and 8.3 μ g/L. At an initial atrazine concentration of 36 μ g/L, the

TABLE 2. Single-Solute Freundlich Isotherm Parameters

ausorbeni	target compound	single
PICA B	atrazine	K = 2
PAC	$MW = 215.68 \mu\text{g}/\mu\text{mol}$	1/n =
PICA A	atrazine	K=9
PAC	$MW = 215.68 \mu g/\mu mol$	1/n =
Hydrodarco B	atrazine	K=4
PAC ^a	$MW = 215.68 \mu g/\mu mol$	1/n =
Watercarb	2-methylisoborneol (MIB)	K=2
PAC ^b	$MW = 168 \mu\text{g}/\mu\text{mol}$	1/ <i>n</i> =

^a From refs 12 and 13. ^b From ref 15.

solid-phase concentrations of the EBC were 5-7 times as large as those of atrazine, and at an initial atrazine concentration of 8.3 μ g/L, the solid phase concentrations of the EBC were 23-29 times as large as those of atrazine. Thus, the IAST results verified the first assumption, especially as the initial atrazine concentration decreased. Furthermore, Table 1 shows that EBC solid-phase concentrations at a given PAC dose were unaffected by changes in the initial atrazine concentration. Having shown that the assumptions necessary for the derivation of eq 3 were met, Table 1 further illustrates that the ratios of atrazine solid-phase concentrations were equal to the ratio of the initial liquid-phase atrazine concentrations. In other words, the results of the IAST demonstrated that the PAC capacity for atrazine was directly proportional to the initial atrazine concentration at a given PAC dose, a result consistent with eq 3.

The direct proportionality between PAC capacity and initial target compound concentration further implied that percent removal of the target compound at a given PAC dose is independent of the initial target compound concentration. The latter was shown by incorporating the mass balance for the trace organic compound ($C_{1,0} = C_{1,\mathrm{eq}} + q_1 C_c$) into eq 3, which led to an expression that related the dimensionless equilibrium liquid-phase concentration of the target compound to the applied PAC dose:

$$\frac{C_{1,\text{eq}}}{C_{1,0}} = \frac{\frac{1}{q_2} \left(\frac{n_2 q_2}{n_1 K_1}\right)^{n_1}}{C_c + \frac{1}{q_2} \left(\frac{n_2 q_2}{n_1 K_1}\right)^{n_1}} \tag{4}$$

IAST results further showed that the equilibrium liquidphase concentration of the EBC was negligible at large PAC doses. Therefore, the equilibrium solid-phase concentration of the EBC at large PAC doses can be approximated by

$$q_2 \simeq \frac{C_{2,0}}{C_c} \tag{5}$$

Incorporation of eq 5 into eq 4 led to the following relationship between the remaining liquid-phase concentration at equilibrium and PAC dose:

$$\frac{C_{1,\text{eq}}}{C_{1,0}} = \frac{\frac{1}{C_{2,0}} \left(\frac{n_2 C_{2,0}}{n_1 K_1}\right)^{n_1}}{C_c^{n_1}}$$
(6)

Equation 6 shows that a log—log plot of $C_{1,eq}/C_{1,0}$ versus PAC dose (C_c) should result in a straight line with slope $-n_1$ at sufficiently large PAC doses. As discussed below, IAST model results verified that the assumptions incorporated into eq 6 hold, provided that the initial concentration of the target compound is sufficiently low and that the PAC dose is sufficiently large.

single-solute Freundlich isotherm parameters

 $K = 223.2 \text{ (}\mu\text{mol/g)}\text{(}L/\mu\text{mol)}^{1/n}$ 1/n = 0.648 $K = 937.4 \text{ (}\mu\text{mol/g)}\text{(}L/\mu\text{mol)}^{1/n}$ 1/n = 0.443 $K = 489.3 \text{ (}\mu\text{mol/g)}\text{(}L/\mu\text{mol)}^{1/n}$ 1/n = 0.440 $K = 21.19 \text{ (}\mu\text{mol/g)}\text{(}L/\mu\text{mol)}^{1/n}$ 1/n = 0.492

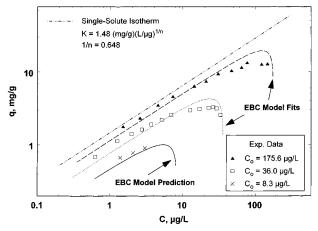


FIGURE 2. Atrazine isotherms on PICA B PAC in organic-free water and in Illinois groundwater.

Isotherm Data. Figure 2 depicts the singe-solute atrazine isotherm for PICA B PAC along with atrazine isotherm data in Illinois groundwater at initial concentrations of 175.6, 36.0, and 8.3 μ g/L. Table 2 shows that the single-solute atrazine isotherm was nonlinear as expressed by a Freundlich 1/nvalue of 0.65. Isotherm nonlinearity is typically observed for the adsorption of micropollutants on activated carbon surfaces (8), and competitive adsorption is important in multi-solute systems as a result (10, 11). As depicted in Figure 2, the initial concentration dependence and the resulting shift of the atrazine isotherm data in Illinois groundwater to lower solid-phase concentrations substantiated that BOM competed with atrazine for adsorption sites. In the presence of a constant initial BOM concentration, lowering the initial atrazine concentration rendered atrazine less competitive, leading to lower atrazine capacities. Atrazine isotherms in natural water remained nonlinear when isotherm experiments were conducted with a constant initial atrazine concentration and varying PAC doses.

To illustrate the validity of the EBC model as a starting point for the development of eqs 3 and 6, Figure 2 further depicts EBC model fits and predictions for the atrazine isotherms in natural water. Isotherm data collected at initial concentrations of 175.6 and 36.0 μ g/L were used to calibrate the EBC model. Good agreement between the experimental data and the resulting model fits were obtained as shown in Figure 2. After the EBC model was calibrated, it was possible to predict the capacity of PICA B PAC for atrazine at lower initial concentrations in Illinois groundwater. For an initial atrazine concentration of 8.3 $\mu g/L$, Figure 2 compares the predicted isotherm with experimental data, which were closely matched by the EBC prediction. For an initial atrazine concentration of 8.3 μ g/L, the atrazine capacity of PICA B PAC at an equilibrium liquid-phase concentration of $1 \mu g/L$ was about three times lower in Illinois groundwater than in organic-free water, a result that illustrates the importance of

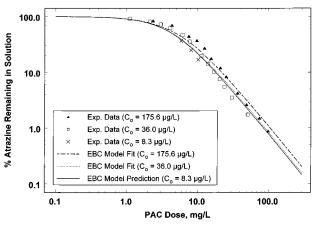


FIGURE 3. Percent atrazine remaining at equilibrium as a function of PAC dose for the adsorption of atrazine from Illinois groundwater on PICA B PAC.

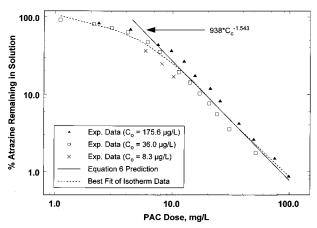


FIGURE 4. Comparison of experimental atrazine isotherm data and simplified model prediction (PICA B PAC in Illinois groundwater).

competition by BOM on the PAC capacity for atrazine in natural waters.

In Figure 3, the isotherm data and EBC model results shown in Figure 2 are represented in a new format by plotting the percentage of the initial atrazine concentration that remained at equilibrium as a function of PAC dose. Figure 3 illustrates that percent atrazine removal for a given PICA B PAC dose was very similar over the tested range of initial atrazine concentrations, a result consistent with eqs 3 and 6 shown above. For example, the predicted equilibrium liquid-phase concentrations of atrazine in Illinois groundwater were 45.4% and 44.3% of the respective initial atrazine concentrations of 36.0 and 8.3 μ g/L when a PICA B PAC dose of 5 mg/L was applied. Consistent with eq 6, Figure 3 further depicts a linear relationship between log $C_{\rm eq}/C_0$ and log $C_{\rm c}$ for PAC doses greater than approximately 10 mg/L.

Figure 4 compares the prediction that was made using eq 6 with the experimental data. Equation 6 closely matched the trends observed in the experimental data once the PAC dose exceeded 10 mg/L. It should be noted that the EBC parameters obtained from the nonlinear least-squares optimization routine were required to evaluate eq 6 $(1/n_2 = 0.65, C_{2,0} = 0.87 \,\mu\text{mol/L})$. However, a simple curve fit of the experimental data in Figure 4 produced results that closely matched those of eq 6. From a practical standpoint, the latter approach was considered preferable because it eliminated the need for a mathematical model as well as the limitation of eq 6, which is valid at large PAC doses only.

To test whether proportionality between PAC capacity and initial micropollutant concentration could be observed

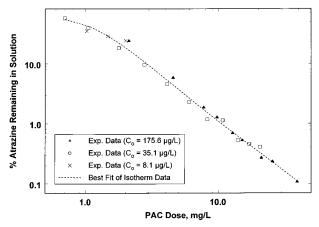


FIGURE 5. Atrazine isotherms on PICA A PAC in Illinois groundwater.

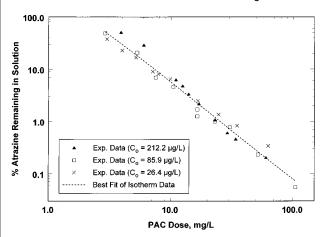


FIGURE 6. Atrazine isotherms on Hydrodarco B PAC in Missouri River water.

with a different PAC in the same solute/solvent system, atrazine isotherms experiments were also conducted with PICA A PAC in Illinois groundwater. Table 2 summarizes the single-solute Freundlich isotherm parameters for atrazine adsorption on PICA A PAC. The single-solute isotherm was nonlinear as indicated by a Freundlich 1/n value of 0.44. As illustrated in Figure 5, the equilibrium data in Illinois groundwater clearly indicate that percent atrazine removal at any given PAC dose was independent of the initial atrazine concentration at the tested initial concentrations even though the isotherms at a given initial atrazine concentration were nonlinear. As before, these results are consistent with the developed simplifications of the IAST for micropollutant adsorption from natural waters on PAC.

Isotherm data for atrazine adsorption from Illinois groundwater on two PACs showed that atrazine removal percentages for a given PAC dose were very similar at different initial atrazine concentrations. To verify that the observed trends were not specific to the atrazine/Illinois groundwater system, isotherm data were analyzed that had been collected in separate research efforts by Greene et al. (12, 13), who investigated the removal of atrazine from Missouri River water at Kansas City, MO, and by Gillogly et al. (15), who studied the removal of 2-methylisoborneol (MIB) from Lake Michigan water at Chicago, IL. Table 2 summarizes the employed PACs and adsorbates as well as single-solute Freundlich isotherm parameters of the target compounds. As on the previously tested PACs, the micropollutant isotherms were nonlinear in both single- and multi-solute systems.

Isotherm data for atrazine and MIB adsorption from natural waters are depicted in Figures 6 and 7, respectively. The figures further substantiate that micropollutant removal

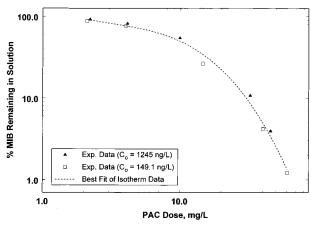


FIGURE 7. MIB isotherms on Watercarb PAC in Lake Michigan water.

percentages at any given PAC dose were essentially constant over the tested initial concentration ranges. For the atrazine data shown in Figure 6, similar removal percentages were observed at a given PAC dose for initial atrazine concentrations that ranged between 26 and 212 µg/L. Similarly, constant removal percentages were observed for MIB at any given PAC dose for initial MIB concentrations of 149 and 1245 ng/L as depicted in Figure 7.

Overall, the results of this research illustrated that, in the presence of competing BOM, the removal percentage of a micropollutant at any given PAC dose was independent of the initial micropollutant concentration, provided that the micropollutant was present at trace levels. This finding for nonlinear isotherms was shown to be consistent with the IAST. Similar removal percentages at different initial concentrations of the target compound were observed for a strongly adsorbing compound (atrazine) as well as for a more weakly adsorbing compound (MIB). In addition, the proportionality between PAC capacity and initial micropollutant concentration was observed for different activated carbons and natural waters. Based on the analysis of the IAST and the obtained experimental data, the initial concentration dependence of the PAC capacity for a trace organic compound in natural water can be estimated without the use of mathematical models once isotherm data have been collected for a micropollutant at a single trace initial concentration in the natural water. Further improvement of the theory is needed to develop a relationship that will permit the calculation of the initial concentration limit below which proportionality between PAC capacity and initial micropollutant concentration is observed. Nonetheless, based on the systems evaluated in this study, the upper concentration limit appeared to exceed micropollutant concentrations typically encountered in drinking water sources. Until a better understanding of the upper concentration limit is obtained, the applicability of the developed approach can be verified in praxis by conducting two isotherm experiments, one at the largest anticipated initial concentration of the target compound and one at a lower initial concentration to test whether percent micropollutant removal is indeed independent of the initial micropollutant concentration at any PAC dose.

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