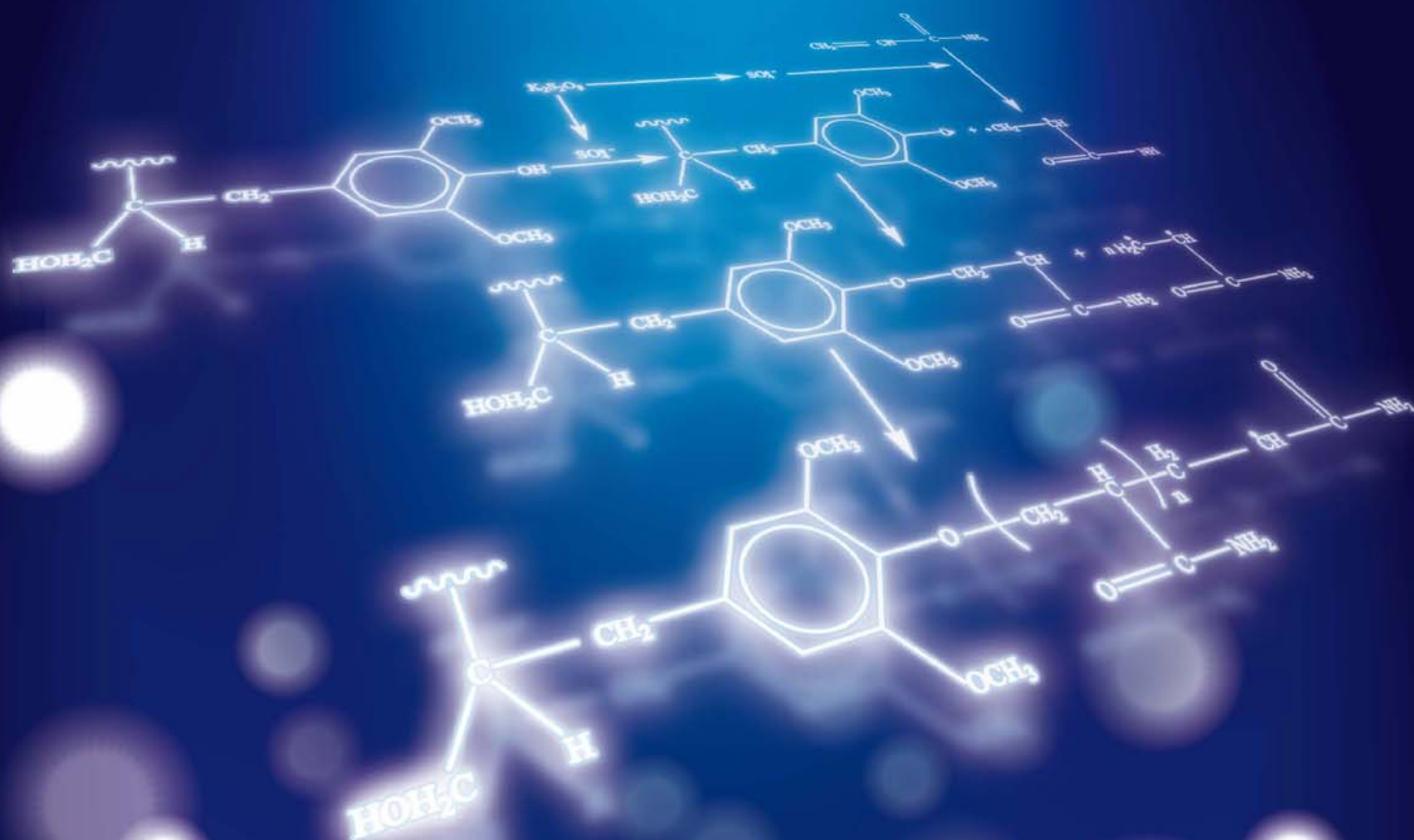


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## Effect of dissolved organic matter on sorption and desorption of phenanthrene onto black carbon

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### Abstract

Sorption and desorption of phenanthrene (PHE) onto black carbon (BC) extracted from sediments were studied in the presence of three types of dissolved organic matter (DOM), including L-phenylalanine (L-PH), peptone and citric acid. The nonlinearity of the sorption isotherms increased in the presence of DOM. The presence of L-PH reduced the sorption capacity and desorption hysteresis because of the solubilization of PHE in L-PH solution. Peptone at 50–500 mg/L also led to a decrease in sorption attributed to solubilization, although the sorbed peptone on the BC surface could slightly increase PHE sorption. Unlike L-PH and peptone, citric acid enhanced the sorption capacity and irreversibility of PHE on BC mainly due to the strong sorption of citric acid on the BC surface. Our results may help to understand the different impacts of DOM on the distribution and transport of PAH in the environment.

**Key words:** DOM; black carbon; phenanthrene; sorption; desorption

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### Introduction

Soil/sediment organic matter (SOM) plays an important role in the sorption, desorption and degradation processes of hydrophobic organic compounds (HOCs), which control the fate, bioavailability and distribution of these contaminants in soils and sediments (Accardi-Dey and Gschwend, 2002). It was reported that there are two domains of physically and chemically heterogeneous SOM, a “soft” domain, including fulvic acids and humic acids in their rubbery states, and a “hard” domain, including kerogen, black carbon (BC) and humic acids in their glassy states (LeBoeuf and Weber, 1997; Weber et al., 1992, Young and Weber, 1995). BC, which is a component of SOM formed during incomplete oxidation of biomass and fossil fuels, is believed to be a super-sorbent because of its high specific surface area and relatively reduced chemical nature (Gustafsson et al., 1997). Indeed, BC materials such as diesel soot and wood char were shown to exhibit sorption capacity several times greater than other forms of natural organic matter (Accardi-Dey and Gschwend, 2002, 2003; Cornelissen and Gustafsson, 2004). For example, environmental BC was reported to explain 49%–85% of

the total Phen sorption on sediments at a concentration of 1 ng/L (Cornelissen et al., 2004; Zhang and He, 2009). Recent investigations also showed that coal and soot particles in fact can explain > 90% of the total sorption for organic compounds (Bucheli and Gustafsson, 2000; Cornelissen et al., 2005). Likewise, BC was responsible for > 80% of the total sorption for PAH, PCB and PCDD by harbor sediments (Lohmann et al., 2005). Therefore, BC plays an important role in the fate of organic contaminants due to its very strong sorption affinity.

Dissolved organic matter (DOM) is one kind of widely existing compounds in soils and sediments, which may interact with water, solid matrix and other contaminants. Tremblay et al. (2005) and Céspedes et al. (2006) reported that DOM can reduce sorption but enhance desorption of HOC due to the competition with organic compounds for the sorption sites of soil/sediment surfaces and the increase in HOC solubility by partitioning them into DOM solutions. However, according to a prior study of Tremblay et al. (2005), the sorption of DOM by solid matrices may increase HOC sorption by partitioning HOC into immobile sorbed DOM. The above two contrasting processes determine the apparent impacts of DOM on the sorption and desorption of HOC in soils and sediments. A number of studies have investigated the influence of DOM on the

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sorption and desorption of organic compounds in soils and sediments. For instance, the presence of tannic acid at 15–100 mg/L produced an increase in 3,4-dichloroaniline and 4-bromoaniline sorption but a decrease in imidacloprid sorption on a typical calcareous soil (Céspedes et al., 2006). Gao et al. (2007) reported that inherent DOM in soils impeded phenanthrene (PHE) sorption, but the impact of exotic DOM extracted from straw waste on sorption was DOM concentration-dependant. Low levels of exotic DOM ( $\leq 28$  mg/L) promoted sorption, but high levels of exotic DOM ( $\geq 52$  mg/L) impeded the sorption of PHE on soils. Moreover, application of DOM extracted from sludge and straw was able to decrease the chlorotoluron sorption and increase its desorption capacity from soils (Song et al., 2008). As reported by Li et al. (2005), addition of DOM extracted from organic fertilizers reduced the sorption of naphthalene and chlorpyrifos by soils. However, little information is available on the effect of DOM on the sorption and desorption of HOC onto BC, which may help to predict the fate and transport of HOC in contaminated soils and sediments, especially for those with high BC content.

The aims of this study are to: (1) examine the sorption of DOM (L-phenylalanine (L-PH); peptone; citric acid) on BC isolated from a sediment sample, and (2) compare the effects of DOM on the sorption capacity and desorption hysteresis of PHE onto BC.

## 1 Materials and methods

### 1.1 Chemicals

PHE (> 98%, Aldrich Chemical Co.) was selected as a representative HOC in the sorption and desorption experiments. The aqueous solubility  $S_w$  at 25°C is 1.29 mg/L, and  $\log K_{ow}$  is 4.57 (Mackay et al., 1992). Three types of dissolved organic matter (L-PH, peptone and citric acid) were purchased from Sigma Chemical Company and used without further treatment. The molecular weight of L-PH, citric acid and peptone are 165 g/mol, 192 g/mol and > 2000 g/mol, respectively. Stock solutions of PHE were prepared in high performance liquid chromatography (HPLC)-grade methanol. Methanol concentrations in the aqueous solutions were always less than 0.2%, a level at which methanol has no measurable effect on sorption (Wauchope and Koskinen, 1983). L-PH, peptone and citric acid solutions were dissolved in deionized water.

### 1.2 Sorbent

One surface (0–20 cm) sediment sample was collected from the Hunhe River in the Daliaohe River water system, China, in May 2006. After collection, the sediment was freeze-dried (FD-1A, China), passed through a 100-mesh sieve and stored in a brown glass bottle. The extraction of BC was performed upon treatment with a combustion method at 375°C (Gelinas et al., 2001). Briefly, carbonates

in the sediment were first dissolved in 1 mol/L HCl for 24 hr. Then, the residues were demineralized with 1 mol/L HCl and 10% HF for 5 days, which was repeated four times. Finally, the residual fraction was heated in a muffle furnace at 375°C for 24 hr under a constant air flow of 200 mL/min. The BC sample was washed with deionized water, freeze-dried, passed through a 100-mesh sieve, and used for sorption and desorption experiments. Fourier transform infrared spectroscopy spectrum,  $^{13}\text{C}$  NMR spectrum and scanning electron microscopy analysis of the BC sample was performed in the previous study (Zhang and He, 2010).

### 1.3 Sorption and desorption experiments

Sorption isotherms of PHE and DOM onto the BC sample were conducted in replicates in 50 mL glass tubes with Teflon-lined caps.  $\text{CaCl}_2$  solution of 0.01 mol/L and 200 mg/L  $\text{NaN}_3$  were added to stock solutions to minimize biological activity (Cornelissen and Gustafsson, 2004; Kang and Xing, 2005; Wen et al., 2007). The ratios of water to solids (20 mL:5 mg) were adjusted to achieve 30%–80% sorption of PHE. Our preliminary test showed that apparent sorption equilibrium was reached in less than 7 days. The vials with BC and initial PHE or DOM solutions (20 mL) were shaken at 125 r/min at  $(25 \pm 0.5)^\circ\text{C}$  for 7 days and then centrifuged at  $4000 \times g$  for 30 min to separate solid and aqueous phases. PHE in the supernatant was filtered through a 0.45  $\mu\text{m}$  nylon membrane and analyzed by HPLC. The sorbed amounts were computed from the difference of the initial and final solute concentrations. Two replicates for each point were used in all sorption experiments. Control reactors prepared similarly but with no sorbent were run simultaneously to assess loss of solute to the reactor during sorption. Results showed that average system losses were consistently less than 4% of the initial concentration, indicating that microbial degradation and volatilization during sorption and uptake by the glass walls were negligible. To determine the effect of DOM on PHE sorption onto BC, L-PH, peptone and citric acid was added to the solutions at initial concentrations of 50, 100 and 500 mg/L for L-PH and peptone, and 50 and 100 mg/L for citric acid. The sorption process and analysis were the same as the isotherm experiments.

Desorption experiments were conducted immediately after the adsorption experiments. 10 mL of the centrifuged supernatant was removed and replaced with the same volume of fresh 0.01 mol/L  $\text{CaCl}_2$  and 200 mg/L  $\text{NaN}_3$  solution. The mixtures were then re-equilibrated for 24 hr at  $(25 \pm 0.5)^\circ\text{C}$ . Subsequent separation of soil from the aqueous phase and analysis were conducted as described above. These steps were repeated five times consecutively.

### 1.4 Phenanthrene solubilization in DOM

Solubilization of PHE in L-PH, peptone and citric acid solutions with a range of concentrations of 0–500 mg/L

was performed in 50 mL glass tubes with Teflon-lined caps. Duplicate vials with 30 mL DOM solution at a given concentration containing excess PHE were shaken at 200 r/min for 48 hr at  $(25 \pm 0.5)^\circ\text{C}$ . The tubes were then centrifuged at 5000 r/min for 30 min to separate the solid PHE that did not dissolve. The supernatant was diluted and analyzed for the concentration of PHE by HPLC.

### 1.5 Determination of PHE and DOM

PHE concentrations were determined using a reversed phase HPLC (C18 column, 4.6 mm  $\times$  25 mm, Waters, USA) with a fluorescence detector (model Waters 474, UV excitation/emission wavelengths at 292/366 nm). Isocratic elution was performed at a flow rate of 1.0 mL/min using MeOH:water volume ratio (95:5) as the mobile phase. L-PH and peptone were quantified by UV absorption at 257 nm and 280 nm, respectively. Citric acid concentration was determined as described in the reference (Chen et al., 2010) using HPLC.

### 1.6 Sorption model

The Freundlich empirical model was used to fit the equilibrium sorption and desorption data:

$$q_e = K_F C_e^n \quad (1)$$

$$\log q_e = \log K_F + n \log C_e \quad (2)$$

where,  $C_e$  (mg/L) is the liquid phase concentration,  $q_e$  (mg/kg) is the solid phase concentration,  $K_F$  ((mg/kg)/(mg/L)<sup>*n*</sup>) is the sorption capacity-related parameter, and  $n$  is the isotherm nonlinearity index.

The hysteresis index (HI) for the sorption-desorption isotherm is calculated (Barriuso et al., 1994):

$$\text{HI} = n_{\text{des}}/n_{\text{ads}} \quad (3)$$

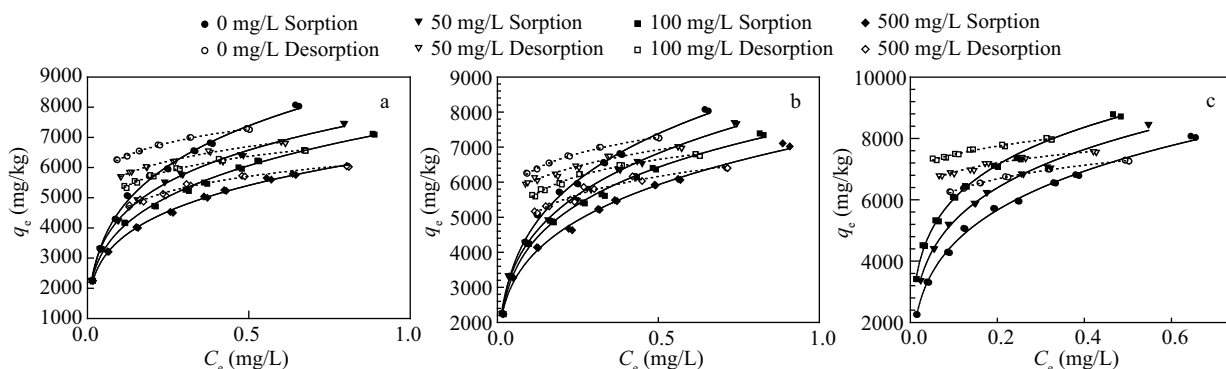
where,  $n_{\text{ads}}$  and  $n_{\text{des}}$  are the Freundlich constants of the adsorption and desorption isotherms, respectively. The fitting was processed using SigmaPlot 2000 (SPSS Inc.) and statistical analysis was performed using SPSS 11.0 (SPSS Inc.).

## 2 Results and discussion

### 2.1 Influence of DOM on the sorption of PHE

The influence of L-PH, peptone and citric acid on the sorption and desorption isotherms of PHE onto BC is given in **Fig. 1**. All sorption and desorption isotherms were nonlinear with the Freundlich parameters listed in **Table 1**. The sorption nonlinearity of PHE onto BC increased in the presence of DOM. The  $n$  values of PHE sorption reduced from 0.330 to 0.258, 0.278 and 0.259, when the added concentration increased from 0 mg/L to 500 mg/L for L-PH and peptone, and to 100 mg/L for citric acid, respectively. This is different from the results in a prior study. Pan et al. (2007) reported that a H<sub>2</sub>O<sub>2</sub>-treated soil coated with dissolved humic acid showed more linear sorption for PHE. The main sorption mechanisms of PHE onto BC were adsorption and pore filling (James et al., 2005). The increasing nonlinearity in this study suggests that the apparent sorption is not just the combination of PHE sorption on sorbed DOM and BC. The sorbed DOM may modify surface characteristics of the BC, such as heterogeneity, organic carbon content, hydrophobicity and functional groups (Chi and Amy, 2004; Gao et al., 2007).

Due to their different chemical structures, molecular sizes and concentrations, the three types of DOM (L-PH, peptone and citric acid) exhibited different effects on the sorption capacity of PHE onto BC. The data in **Fig. 1a** and **Table 1** showed that the presence of L-PH decreased the sorption of PHE onto BC. The  $K_d$  value (at  $C_e = 0.05$  mg/L) of PHE decreased from 70374 to 69250, 64496 and 59759 mL/g with L-PH concentration increasing from 0 to 50, 100 and 500 mg/L, respectively. Similar to L-PH, the sorption of PHE onto BC was also reduced in the presence of peptone (**Fig. 1b**). When the concentration of peptone increased from 0 to 50, 100 and 500 mg/L, the  $K_d$  value (at  $C_e = 0.05$  mg/L) of PHE decreased from 70374 to 69370, 66072 and 62571 mL/g, respectively. Moreover, for both L-PH and peptone, the  $K_d$  values of PHE decreased more sharply at low DOM concentrations ( $C_e < 100$  mg/L). When the added concentration increased to 500 mg/L, PHE sorption was slightly reduced. However,

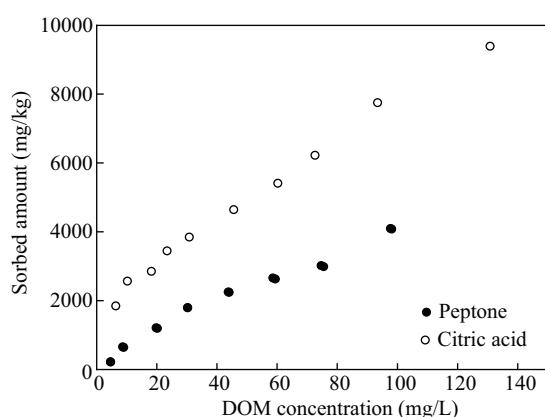


**Fig. 1** Sorption and desorption isotherms of PHE onto BC in the presence of L-PH (a), peptone (b) and citric acid (c).

**Table 1** Freundlich sorption and desorption parameters of PHE onto BC in the presence of different DOM

DOM (mg/L)	Sorption											HI	
	<i>n</i>	$K_F$	$R^2$	<i>N</i>	$K_d$ (mL/g)			<i>n</i>	$K_f$	$R^2$	<i>N</i>		
					$C_e = 0.05$ mg/L	$C_e = 0.1$ mg/L	$C_e = 0.5$ mg/L						
Control	0	0.330 ± 0.005	9456	0.992	20	70374	44230	15046	0.091 ± 0.003	7747	0.996	12	0.28
L-PH	50	0.282 ± 0.004	8059	0.991	20	69250	42100	13256	0.103 ± 0.002	7185	0.990	12	0.36
	100	0.275 ± 0.004	7350	0.992	20	64496	39020	12149	0.118 ± 0.003	6914	0.989	12	0.43
	500	0.258 ± 0.005	6472	0.998	18	59759	35730	10824	0.135 ± 0.005	6269	0.990	12	0.52
Peptone	50	0.297 ± 0.003	8444	0.989	20	69370	42613	13746	0.089 ± 0.004	7343	0.963	12	0.30
	100	0.292 ± 0.003	7923	0.987	20	66072	40447	12943	0.111 ± 0.005	7180	0.984	12	0.38
	500	0.278 ± 0.004	7195	0.993	20	62571	37934	11868	0.128 ± 0.002	6718	0.978	12	0.46
Citric acid	50	0.281 ± 0.005	9953	0.992	20	85782	52114	16383	0.063 ± 0.003	7990	0.965	12	0.23
	100	0.259 ± 0.006	10776	0.990	20	99202	59355	18010	0.051 ± 0.002	8423	0.963	12	0.19

*N* is the number of data;  $K_d$  is the concentration dependent sorption capacity coefficient  $K_d = K_F C_e^{n-1}$ ; HI is the hysteresis index. Data are expressed as mean ± standard deviation.

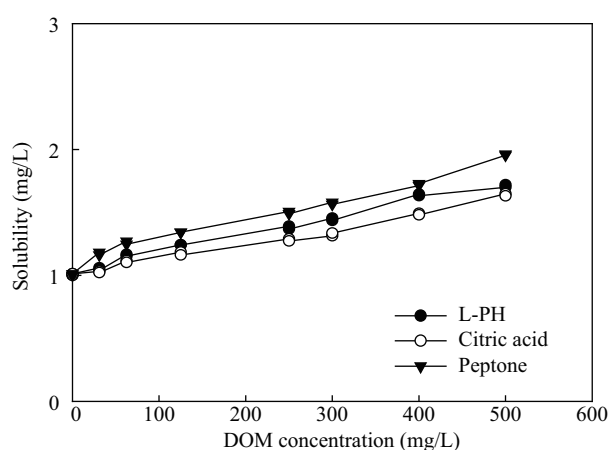
**Fig. 2** Sorption isotherms of peptone and citric acid onto BC.

compared to peptone, the presence of L-PH resulted in a greater decrease of PHE sorption to BC at the same added concentration. In contrast to L-PH and peptone, the presence of citric acid enhanced the sorption of PHE onto BC. When the added concentration of citric acid increased from 0 to 50 and 100 mg/L, the  $K_d$  value (at  $C_e = 0.05$  mg/L) of PHE was enhanced from 70374 to 85782 and 99202 mL/g, respectively.

In a system with the coexistence of PHE, DOM, BC and water, the following interactions may occur simultaneously: (1) competition for active hydrophobic adsorption sites on the BC surface between PHE and DOM, (2) the sorption of DOM molecules on BC surfaces, and (3) partition of PHE among BC hydrophobic adsorption sites, DOM solutions (i.e. solubilization) and sorbed DOM on BC surface (Tremblay et al., 2005; Céspedes et al., 2006; Gao et al., 2007). The apparent sorption of PHE depends on the extent and competition of each interaction. Therefore, the different effects of DOM (L-PH, peptone and citric acid) on PHE sorption mainly depend on their differences in the above processes.

## 2.2 Sorption of DOM on BC

Sorption isotherms of peptone and citric acid on BC are shown in **Fig. 2**. The sorption capacity of DOM onto BC

**Fig. 3** Solubility of PHE as a function of L-PH, peptone and citric acid concentration.

follows the order: citric acid > peptone. There was no sorption of L-PH on the BC surface. **Figure 3** shows the effects of DOM on the solubility of PHE. The presence of L-PH at 30–500 mg/L increased the solubility of PHE by about a factor of 1.7. Therefore, considering the lack of sorption of L-PH on BC, the solubilization of PHE in L-PH solution was the main reason for the decrease of PHE sorption in the presence of L-PH. Different from L-PH, the sorption of peptone on the BC surface was higher, thereby PHE sorption on sorbed peptone increased. However, the solubility of PHE was greatly enhanced in the presence of peptone (**Fig. 3**). When the peptone concentration increased from 30 to 500 mg/L, the solubility of PHE in peptone solution was about two times higher than that in water. Thus, the strong solubilization of PHE in peptone solution and the competition of peptone and PHE for active hydrophobic adsorption site on the BC surface both reduced the sorption of PHE.

In addition, the presence of peptone leads to a lower decrease in PHE sorption than L-PH. This may be due to the presence of sorbed peptone on the BC surfaces on which more PHE can sorb, resulting in an increase in the sorption of PHE. Compared to peptone, the sorption of

citric acid on BC was much higher, since the molecular size of citric acid is smaller (**Fig. 2**). As reported by Schreiber et al. (2005) and Chen et al. (2005), small DOM molecules are preferentially adsorbed onto activated carbon. This was explained by the better accessibility of adsorption sites in pores for small molecules. The sorbed citric acid on BC can sorb more PHE, which increased the sorption. Moreover, the presence of citric acid at 30–500 mg/L had slight effect on the solubility of PHE (**Fig. 3**). Thus, the strong sorption of citric acid on the BC surface may contribute to the increase in the sorption of PHE onto BC.

### 2.3 Influence of DOM on desorption hysteresis

The HI for the desorption isotherms of PHE from BC in the presence of L-PH, peptone and citric acid are given in **Table 1**. The value of HI reflects the sorption irreversibility of PHE onto BC. In general, a value of HI close to 1 indicates that the desorption process takes place as quickly as sorption does, thus, the sorption is reversible. A value of  $HI < 1$ , however, reveals that the rate of desorption is lower than that of sorption, therefore, hysteresis takes place (Pusino et al., 2004). In this study, the desorption of PHE from BC was hysteretic with  $HI = 0.28$  and the presence of L-PH, peptone and citric acid could affect the desorption hysteresis. **Table 1** shows that the presence of L-PH strongly reduced the desorption hysteresis of PHE on BC. The HI value increased from 0.28 to 0.36, 0.43 and 0.52 with L-PH concentration increasing from 0 to 50, 100 and 500 mg/L, respectively. This indicates that increasing L-PH concentration made the sorption of PHE highly reversible, which is attributed to the partition of PHE to L-PH solution. Similar results were observed for naphthalene sorption and leaching in soils. The presence of DOM caused a reduction in naphthalene sorption and an enhancement in the transport of naphthalene in soil (Li et al., 2005). Similarly, peptone also enhanced the sorption reversibility or reduced the desorption hysteresis of PHE on BC. The HI value increased up to 0.46 with increasing peptone concentration. The increase of the sorption reversibility was mainly due to the solubilization of PHE in peptone solution.

However, compared to peptone, the desorption of PHE was promoted more strongly in the presence of L-PH at the same concentration. This may be explained by the sorbed peptone on the BC surface, which can sorb more PHE and thereby inhibit PHE desorption from BC. Unlike L-PH and peptone, the desorption of PHE was highly inhibited at citric acid concentrations of 50 and 100 mg/L, with HI values decreasing from 0.28 to 0.23 and 0.19, respectively. This may be due to the “cumulative sorption”, the citric acid binding to the BC surface, and the association of PHE with bound citric acid (Gao et al., 2007). Therefore, we can conclude that citric acid can both enhance the sorption capacity and sorption irreversibility of PHE on BC, therefore, effectively making PHE immobile in the

environment, especially for soils with high content of BC materials. But L-PH and peptone may mobilize PHE by removing sorbed PHE to the aqueous phase in contaminated soils and sediments.

## 3 Conclusions

This study reveals that the presence of DOM enhanced the sorption nonlinearity of PHE onto BC, but had different effects on the sorption capacity and reversibility. The apparent sorption and desorption of PHE may be largely due to two contrasting interactions, including the sorption of DOM on the BC surface, which can increase PHE sorption capacity, and the partition of PHE in DOM solution, which may decrease the sorption and promote desorption. With the increase of L-PH and peptone concentration, PHE sorption capacity and desorption hysteresis declined. This may be largely due to the solubilization of PHE in L-PH and peptone solutions. Different from L-PH, the sorption of peptone on the BC surface can increase PHE sorption capacity and inhibit desorption. In contrast, citric acid enhanced the sorption capacity but reduced the sorption reversibility of PHE on BC. Sorbed citric acid on BC surfaces may contribute to the increase of the sorption. The results can provide insight into the PAH distribution in three-phase systems including BC, water and DOM.

### Acknowledgments

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