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Phenanthrene adsorption by soils treated with humic substances under different pH and temperature conditions

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Abbreviations: CALB – *Candida antarctica* lipase B; CRL – *Candida rugosa* lipase

Abstract

The mobility of phenanthrene (PHE) in soils depends on its sorption and is influenced by either the existing soil humus or exogenous humic substances. Exogenous humic acids (HAs) were added to soil to enhance the amount of soil organic carbon (SOC) by 2.5, 5.0, and 10.0 g kg⁻¹. PHE desorption of the treated soils was determined at two pH levels (3.0 and 6.0) and temperatures (15 and 25 °C). Soil PHE adsorption was related to pH and the type and quantity of added HAs. Humic acid (HA) and fulvic acid (FA) derived from peat had different effects on adsorption of PHE. Adsorption increased at first and then decreased with increasing quantity of exogenous FA. When the soil solution pH (in 0.005 M CaCl₂) was 4.5 or 3.0, the turning points were 2.5 g FA kg⁻¹ at pH 3.0 and 5 g FA kg⁻¹ at pH 4.5. When soil solution pH was 6, the amount of adsorbed PHE was enhanced with increasing exogenous HAs (HA or FA) and amount of adsorption by soil treated with FA was higher than with HA. Adsorption of PHE in the FA treatment at 10.0 g kg⁻¹ was lower than the controls (untreated soil or treatment with HAs at 0 g kg⁻¹) when the soil solution pH was 3.0. This suggests that FA adsorbed by soil was desorbed at low pH and would then increase PHE solubility, and PHE then combined with FA. PHE adsorption was usually higher under lower pH and/or lower temperature conditions. PHE sorption fitted the Freundlich isotherm, indicating that exogenous humic substances influenced adsorption of phenanthrene, which in turn was affected by environmental conditions such as pH and temperature. Thus, exogenous humic substances can be used to control the mobility of soil PAHs under appropriate conditions to decrease PAH contamination.

Introduction

Polycyclic aromatic hydrocarbons (PAHs) are a typical class of persistent organic compounds, which occur in environmental matrices from natural sources such as forest fires or from human activities. They are widely prevalent and recalcitrant to biodegradation. PAHs have been recommended as priority pollutants by the United States Environmental Protection Agency (USEPA)

and are target components for study due to their carcinogenic, mutagenic, and toxic properties (Wilson & Jones 1993; Carmichael *et al.* 1997). Therefore, it is very important to gain some understanding of the detailed geochemistry of PAHs, including their sorption processes.

Sorption processes can substantially influence the fate and transport of PAHs and the design and implementation of effective schemes for remediation of contaminated soils (Luthy *et al.* 1997;

Weber *et al.* 2001). Soil organic matter (SOM) affects the sorption of PAHs. Only if PAHs or their metabolites are bound covalently to soil organic matter are they considered to be an integral part of humus and thus do not represent a danger to the environment (Käcker *et al.* 2002). Therefore the relationship between SOM and PAHs is of major interest. Although several studies have shown an increase in sorption of PAHs with increasing amount of soil or sediment organic matter (SOM) (Chiou *et al.* 1998; Kohl & Rice 1999), little is known about the effects of SOM composition. Humic substances are the dominant organic components in soils (Stevenson 1994) and generally comprise more than 70% of SOM. Sorption of PAHs to humic substances associated with soil or the aquifer solid phase usually inhibits their transport (Laor *et al.* 1998). On the other hand, binding to dissolve humic substances will facilitate desorption from the solid phase and thereby enhance pollutant transport (Rebhun *et al.* 1996). Numerous studies have shown that addition of humic substances enhanced the solubility (Laor *et al.* 1998; MacKay & Gschwend 2001) and leachability (Kim & Osako, 2003) of PAHs. In these experiments humic substances were added after pollutant input. There is little information on the sorption of PAHs in soils associated with exogenous humic substances. Humic substances strongly associate with soil minerals to form an organic coating that allows widely differing chemical interactions with nonpolar compounds (Kubicki & Apitz 1999; Angove *et al.* 2002). It is necessary to study PAH sorption of soils treated by humic substances to obtain the theoretical foundation for the use of humic substances to control soil PAH mobility and reduce the diffusion of PAHs in soils.

The aim of the present work was to obtain information on the role of exogenous humic substances on the capacity of a soil to adsorb PAHs. We have investigated the sorption of PHE on a soil treated with humic substances under different pH and temperature conditions.

Materials and methods

Soil and chemicals

Samples of the 0–20 cm layer of an agricultural soil (Acrisols by UN WRB taxonomy) were col-

lected from the Red Soil Ecological Experimental Station, Institute of Soil Science, Yingtan City, Jiangxi Province, southeast China. The samples were air-dried and passed through a 1-mm sieve. Selected properties were as follows: pH (in H₂O) 5.5, organic matter 8.4 g kg⁻¹, total N 0.71 g kg⁻¹, total P 0.72 g kg⁻¹, and CEC 21.9 mmol kg⁻¹.

Phenanthrene (PHE), >97% purity, was purchased from Fluka Chemicals. A stock solution of 500 mg L⁻¹ PHE was prepared by dissolving in acetone (HPLC grade, TEDIA Company, Inc.). All inorganic reagents used were analytical reagent grade chemicals.

HAs extraction

Relatively pure HA and FA were extracted from a peat from Heilongjiang province, northeast China, according to the procedures recommended (Swift 1996) by the International Humic Substances Society with one modification: after passing through an XAD-8 resin column, the FA solution was passed through XAD-2 resin. A Perkin Elmer CHN S/O Series II 2400 Element Analyzer was used to measure HAs. HAs were ashed by incineration at 750 °C for 5 h after drying to constant weight at 105 °C, then calculated by difference.

The elemental composition (% by weight) of the HA was 55.03 C, 5.74 H, 3.35 N and 1.0 ash. The corresponding values for the FA were 43.52 C, 4.54 H, 1.74 N and 4.1 ash.

HA and FA addition to soil

Three weights (0.050, 0.100 and 0.200 g) of HAs (HA or FA) were dissolved in 20 mL of deionized water, mixed well with 20.0g soil and air-dried for about 10 days at room temperature, then passed through a 1-mm sieve. Addition of HAs increased the soil organic carbon content of the samples by 2.5, 5.0 and 10.0 g kg⁻¹, respectively.

PHE adsorption

Individual adsorption experiments were conducted in triplicate to ensure reproducibility and reliability. Subsamples (1.00 g) of soil were added to 25-mL screw-capped glass centrifuge tubes with 0.005 M CaCl₂ solution. The solution pH was adjusted by adding the minimum amount of 0.2 M NaOH or 0.2 M HCl and after about 15 min

the soil solution pH was 3.0, 4.5, or 6.0. PHE stock solutions in acetone were introduced to the soil-solution systems by syringe. The total volume of the stock PHE solutions was 50 μL . The carrier solvent concentration in the final solution was <3%, rendering its co-solvent effect negligible (Schlautman & Morgan 1994; Lee *et al.* 2003). The PHE concentration in the solutions was 1.0 mg L^{-1} and samples were equilibrated for 24 h in the dark on an oscillating shaker (180 rpm) at room temperature (25 $^{\circ}\text{C}$). After equilibration, the aqueous and solid phases were separated by centrifugation at 3500 rpm for 10 min. PHE volatilization and degradation were found to be negligible during the experimental procedure.

PHE sorption-isotherm experiment

Treated soils with a 10 g kg^{-1} increase in organic C and untreated controls were used. A series of PHE concentrations of (mg L^{-1}) 0.025, 0.050, 0.100, 0.250, 0.50, and 1.00 were prepared. The experiment temperatures were 15 and 25 $^{\circ}\text{C}$. Solution pH was adjusted by adding the minimum amount of 0.2 M NaOH and 0.2 M HCl and after about 15 min the soil solution pH was 3.0. Other procedures were as described above in Section PHE Adsorption.

Extraction and detection of PAHs from water extraction

The equilibrium solution was extracted with dichloromethane. The extraction process employed two 10-mL aliquots of distilled dichloromethane and the extracts were purified by the method of Song *et al.* (1995) as follows. The

extracts were condensed to dryness by vaporizing in a rotary evaporator, 2 mL cyclohexane was added, 0.5 mL of the cyclohexane solution was added to a silica gel column (1.0 g silica gel soaked in hexane for about 15 min before installing the column), then washed with hexane-dichloromethane (1:1) mixture, and finally dried by puffing N_2 and redissolved in acetonitrile. The recoveries of the total masses of PHE in water solution by extraction ranged from 90 to 108%.

The analysis of purified extracts was conducted using a Shimadzu Class-VP HPLC system with computer control. Separations were carried out on a reversed phase column C_{18} (VP-ODS 150 \times 4.6 mm ID, particle size 5 μm), using a mobile phase of water and acetonitrile mixture (1:9 v/v) at a constant solvent flow rate of 0.5 mL min^{-1} . 40 μL extracts were injected by syringe and detected using a fluorescence detector (RF-10AXL). In addition, the chromatograms were monitored using an excitation wavelength of 270 and an emission wavelength of 350 nm. The column temperature was kept constant at 30 $^{\circ}\text{C}$ during each chromatographic run.

Results and discussion

Effect of HAs on PHE adsorption

The adsorption of PHE (1.0 mg L^{-1} aqueous solution) at 25 $^{\circ}\text{C}$ and different pH (3.0, 4.5, 6.0) conditions on the soils is presented in Figure 1, where the amount of PHE uptake by soil is plotted against the amount of HAs added. There were different effects of HAs on soil PHE adsorption under different pH conditions. When the soil

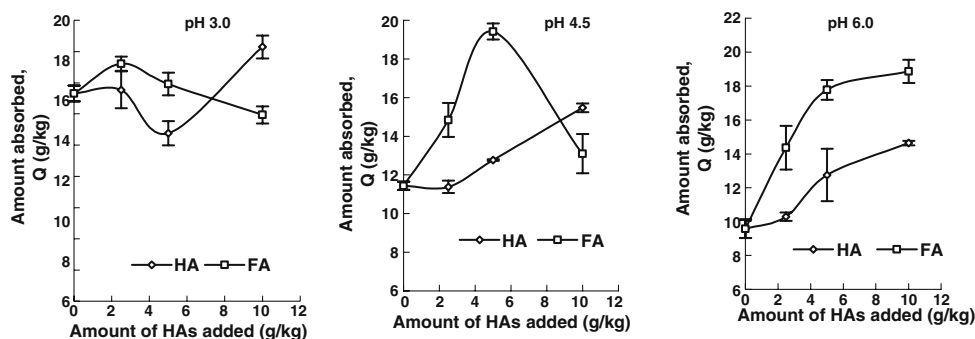


Fig. 1. Adsorption of PHE in differently treated soils.

solution pH was 6.0, the amount of adsorbed PHE was enhanced with increasing exogenous HAs (HA and FA) and adsorption of the soil treated with FA was higher than with HA. These results suggest that addition of exogenous HA to soils may increase the soil surface area and provide additional hydrophobic sites, perhaps changing the amount of PHE adsorption (Conte *et al.* 2001). The maximum adsorption was in the FA 2.5 g kg⁻¹ treatment when the soil solution pH was 3.0 and that of FA 5.0 g kg⁻¹ when the soil solution pH was 4.5. However, maximum adsorption occurred in the HA 10.0 g kg⁻¹ treatment at soil solution pH values of 4.5 and 3.0. Adsorption of PHE in the FA 10.0 g kg⁻¹ treatment was lower than in the HAs 0 g kg⁻¹ treatment when the soil solution pH was 3.0.

FA is soluble at any pH in aqueous solution, thus FA adsorbed by soil could be desorbed at low pH because there was more FA added, and this may have then increased PHE solubility. When the soil solution pH was 4.5, the amount of adsorbed PHE was enhanced with increasing HA except in the 10 g kg⁻¹ treatment and the adsorption capacity of the soil treated with FA was still higher than HA. The adsorption capacities of the soils treated with FA were higher than those of soils treated with HA. PHE distribution in soil FA and HA did not agree with other studies (Kohl & Rice 1998), perhaps because of the different sources of HAs and/or different characteristics of soil native and exogenous HAs. This topic requires further research.

Effect of pH

Adsorption of PHE on the soil under different pH conditions is shown in Figure 2. Figure 2a shows

that the adsorption capacities in all treatments were greatest when soil solution pH was 3.0 (low pH), irrespective of whether the soils were treated with HAs. Adsorption capacities decreased with increasing pH to an extent similar to that observed in other studies (Laor *et al.* 1998; Raber *et al.* 1998). The effect of pH is probably due to the increased polarity of the humic material at higher pH. A more polar humic substance would have a lower affinity for hydrophobic compounds. Schlautman & Morgan (1994) also suggested that the increase in the humic polarity altered its structure and therefore affected its capacity for hydrophobic interactions. At the same time there are other media, such as soil minerals and suspended grains in water, and lower pH may increase the amount of humic substances (especially HA) adsorbed to these media (Laor *et al.* 1998). Adsorption of PHE in the FA treatment was strong at low pH except for the 10 g kg⁻¹ treatment (see Figure 2b), in which PHE adsorption was lower than the control (0 g HAs kg⁻¹) at soil solution pH 3.0. This suggests that FA adsorbed by soil can be desorbed at low pH when there is more FA present, and can then increase PHE solubility when combined with FA. Other studies have given similar results about the effect of HAs on PAH solubility (Lassen & Carlsen 1997; Mackay & Gschwend, 2001).

Effect of temperature

The sorption isotherms of PHE at pH 3.0 are presented in Figure 3, where the amount of PHE uptake by soils (Q) is plotted against the equilibrium PHE concentration (C_e). There was a clear difference in PHE adsorption due to the temperature effect. Adsorption capacities of the treated soils (with the 10 g kg⁻¹ increase in or-

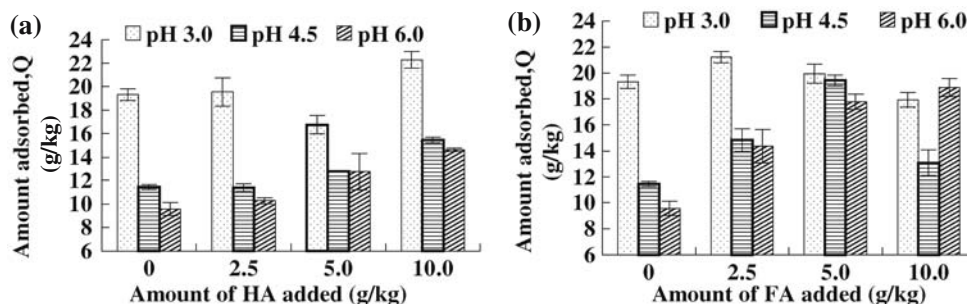


Fig. 2. Adsorption of PHE under different pH conditions.

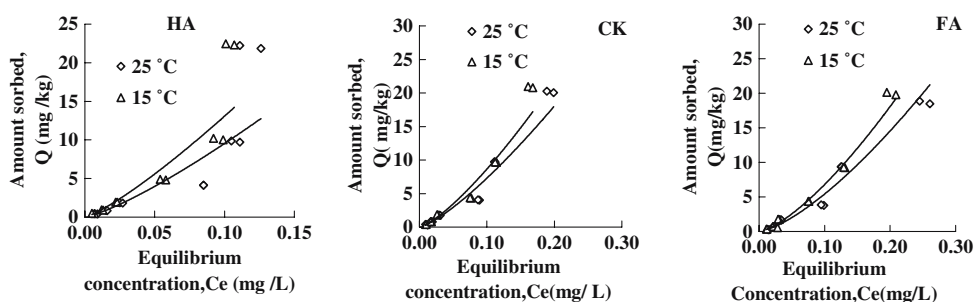


Fig. 3. Sorption isotherms for PHE on soils.

ganic C) and unamended controls were higher at 15 °C than at 25 °C. One explanation is that increased temperature can enhance PHE solubility (Li and Chen 2002), and decrease adsorption. Temperature is a very important factor in the mobility of hydrophobic organic pollutants (HOPs) in a system. The solubility enthalpy (DH), estimated by the Vant Hoff equation (Lodge 1989), of phenanthrene is 5.0 kcal mol⁻¹. The (+) in enthalpy denotes an endothermic reaction, which means that solubility increases as heat enters. Lodge (1989) gave the solubility enthalpy of certain isomers of tetrachlorodibenzo-*p*-dioxins (TCDD), and these were 2, 10, 11.2, and 45 kcal mol⁻¹ for 1,3,6,8-TCDD, 1,2,3,7-TCDD, 1,2,3,4-TCDD, and 2,3,7,8-TCDD, respectively. As a result, it is evident that the solubility of HOPs is an endothermic reaction showing similar enthalpy, although we do not yet know whether enthalpy depends on the molecular weights or octanol–water partitioning coefficients of compounds (Kim & Osako 2003). In addition, increasing temperature can perhaps change characteristics of humic substances such as adsorption to minerals and surface areas that influence PHE adsorption.

The amount of adsorbed PHE were enhanced with increasing of PHE solution concentration over the range of concentrations (0.025–1.0 mg L⁻¹)

studied in this experiment. This indicates that adsorption of PHE on soil has not reached saturation, when the PHE concentration is 1.0 mg L⁻¹.

It is widely found that HOC sorption isotherms are nonlinear and the isotherm nonlinearity depends largely on the properties of the soil or sediment and physicochemical characteristics of the HOC (Kleineidam *et al.* 1999; Xia & Pignatello 2001; Ran *et al.* 2002). The most commonly used model for quantifying HOC sorption equilibrium for soils is the Freundlich model which has the following form: $Q = K_f C_e^n$, where K_f and n are the Freundlich model capacity factor and the isotherm linearity parameter, an indicator of site energy heterogeneity (Huang *et al.* 2003).

Figure 3 shows the sorption of PHE fit to the Freundlich isotherm, and the Freundlich isotherm parameters are presented in Table 1. All isotherms were nonlinear, i.e. $n > 1$. The n values of phenanthrene ranged from 1.227 to 1.4253 (Table 1). The n values of the HA treatment were the lowest among three treatments indicating that soil treated with HA has more heterogeneous sorption sites. Perhaps soil treated by FA also has many heterogeneous sorption sites, but because at pH 3.0 more FA absorbed soil minerals are dissolved, and would then increase PHE solubility and decrease adsorption, then present the less sorption sites. Sorption coefficients (K_f) of phenanthrene ranged

Table 1. Freundlich constants and their coefficient for phenanthrene in soil.

Treatment	15 °C			25 °C		
	K_f	n	R^2	K_f	n	R^2
10 g kg ⁻¹ HA (HA)	220.59	1.227	0.9556	173.31	1.2595	0.9212
0 g kg ⁻¹ HA (CK)	181.85	1.3223	0.9807	147.86	1.3066	0.9700
10 g kg ⁻¹ FA (FA)	175.55	1.4123	0.967	144.24	1.4253	0.9698

from 144.24 to 220.59. It is apparent that K_f values of HA treatment are much larger than control and FA treatments. This may be attributed to the difference in adsorption capacities among the three treatments. HA treatment had higher adsorption capacities than FA treatment and the control. Higher PHE sorption in the humic acid treatment may be due to the stronger affinity to the highly aromatic moieties.

There are numerous reports of sorption not inhibiting but actually enhancing biodegradation of sorbed compounds (Ortega-Calvo *et al.* 1997; Laor *et al.* 1999) because sorbed contaminants were still bioavailable and the presence of surfaces may have stimulated mineralization. In addition, as carbon sources, exogenous humic substances can be used by microorganisms and stimulate microbial activities, thereby increasing contaminant degradation and disappearance. Thus, exogenous humic substances can be used to control soil PAH mobility in appropriate conditions, to decrease PAH pollutant diffusion and increase PAH disappearance from the soil.

Conclusions

Soil PHE adsorption was related to environmental conditions and types and amount of added HAs as follows. Humic acid (HA) and fulvic acid (FA) from the same source had different effects on adsorption of PHE, with adsorption of the soil treated by FA higher than HA in most conditions. The influence of humic substances on PHE adsorption was affected by environmental conditions such as pH and temperature. Adsorption was higher in lower pH and/or lower temperature conditions in most treatments. The sorption of PHE fitted the Freundlich isotherm.

Exogenous humic substances influenced adsorption of phenanthrene, which was affected by environmental conditions. Exogenous humic substances can be used to control soil PAH mobility in appropriate conditions, leading to a decrease in PAH pollutant diffusion.

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