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## Sorption kinetics of naphthalene and phenanthrene in loess soils

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**Abstract** A laboratory study was executed to investigate the effect of surfactants to enhance sorption of polycyclic aromatic hydrocarbon (PAH) contaminants in loess soil. Phenanthrene and naphthalene were chosen as organic contaminant indicators in loess soil modified by the cation surfactant hexadecyltrimethylammonium (HDTMA) bromide. The kinetic behavior of sorption during transport in natural and modified loess soil was studied. The results indicated that sorption rate in the cation surfactant modified loess soils was at least 3 times faster than that of the natural soil. A first-order kinetics model fitted the sorption data well for both soils. The sorption rates of the two organic compounds were related to their primary residual quantity on the soils. The experiments showed that sorption amounts approached constant values approximately within 30 and 90 min for naphthalene and phenanthrene at 298–318 K,

respectively. The rate constants, however, displayed negative correlation with increasing temperature. With changing temperature, the activation energy was calculated at  $-6.196-1.172$  kJ/mol for naphthalene and  $-28.86-15.70$  kJ/mol for phenanthrene at 298–318 K. The results can be used to predict the sorption kinetics of phenanthrene and naphthalene in loess soils, and in a wider perspective, be used to better understand the transport of petroleum contaminants in the soil environment.

**Keywords** Loess soils · Contamination · Naphthalene · Phenanthrene · Sorption kinetics · China

### Introduction

Pollution of soils by petroleum compounds is of major concern as most of these compounds are hydrophobic, persistent, bioaccumulative, and highly toxic. They do not easily disintegrate in the environment, neither are they easily metabolized, and therefore may be

accumulated in the vadose zone or taken up by vegetation. The petroleum compounds, such as polycyclic aromatic hydrocarbons (PAHs), are common pollutants for soil, estuarine waters, and sediments. As a compound group, PAHs have two or more fused aromatic rings that are mutagenic, carcinogenic and teratogenic. For this reason, some PAHs are included in the

Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) Priority List of Hazardous Substances (ATSDR 1995, ATSDR 2001). Furthermore, PAHs from various sources are often retained in the top soils, and due to risks for potential release, it is necessary and important to perform remediation, risk assessment, pollution prevention and groundwater quality risk management. To date, much research has focused on the transport and fate of organic pollutants in soils and application of remedial techniques to deal with these.

Although remediation of contaminated soils is costly and time-consuming, today's *in situ* technologies have become very attractive for treating contaminated soils and groundwater because they create less of a disturbance to the environment and reduce worker exposure to hazardous materials. At potentially contaminated sites, prevention measures need to be undertaken to retard the contaminants in the vadose zone before implementing remediation techniques to avoid groundwater pollution. Natural attenuation is currently considered as one of the most important alternatives to remediate organic contaminants such as petroleum contaminants in soils and groundwater. Among all the natural attenuation mechanisms, sorption is a major process, as the intrinsic mobility of petroleum hydrocarbons is often inversely related to their sorption in soil surfaces (Davis and others 1999). Thus, the release to the environment can be restrained, and the potential risk of their bringing about increased long-term toxic effects to human health can be dramatically minimized. The potential realization of natural attenuation is, however, dependent on many factors such as soil texture, moisture, pH, temperature, residue concentration, organic contents, cation exchange capacity (CEC), co-contaminants, etc. (Pignatello and Xing 1996; Carmo and others 2000; Chiou 2002; Walter and others 2002). Adsorption may also affect other cleaning reactions such as biodegradation, plant uptake, and volatilization, etc., because soil minerals and coating organic matters are the principal sorbents in the soil matrix. It is evident that sorption in natural soils has little impact on the attenuation and fate of the organic contaminants when the distribution coefficient ( $K_d$ ) is small (Pignatello and Xing 1996; Sharma and others 2002). Sorption equilibrium and sorption/desorption kinetics are key processes impacting the subsurface fate and transport of organic contaminants, and therefore, research work is needed to investigate techniques for strengthening the adsorption capacity of natural soils. Temperature is an important factor that has a significant effect on the reaction rates of many exchange processes. Therefore, particular concern must be given to the influence of temperature on adsorption mechanisms on *in situ* remediation where temperatures may vary widely during the day or

the year. For most compounds, sorption quantity decreases with the rise of temperature. However, in some cases equilibrium sorption may be enhanced with increasing temperature or not affected by temperature at all (Luers and Ten 1996). Early studies often treated sorption of organic pollutants on sediments as a rapid equilibrium process that could be described by first-order kinetics for sorption and desorption, but a rapid equilibrium process is unable to efficiently simulate the whole sorption process. Sorption kinetics data always show a rapid original uptake followed by a slow approach to equilibrium. Some studies on sorption kinetics in sediments have identified a fast and a slow period in the sorption process (Karickhoff 1979).

Loess soils cover vast land areas in northwest China where also the number of oil fields has increased greatly in the recent two decades. In many places, the results have been severe petroleum contamination of soils and water resources through spilling of crude oil near drilling location and leakage from storage tanks and pipelines. Loess soils with a low-organic carbon have little sorption capabilities for organic contaminants. Consequently, they have limited potential for retarding organic contamination transport through the unsaturated zone to the groundwater level. Modification techniques with surfactants can be used in the subsurface to improve soil's sorption capacity for hydrophobic organic compounds (HOCs), resulting in a decrease of the mobility of HOC in the vadose zone. While this process naturally occurs as surfactants are introduced into the contaminated subsurface, it might also be a basis for remedial action at contaminated sites. For example, coupling contaminant immobilization with *in situ* biodegradation would provide a comprehensive remediation technology for permanently eliminating target contaminants. There have been some recent studies on the sorption properties and mechanism of PAHs in regards to organobentonite. However, to the authors' knowledge, few studies have concentrated on the sorption kinetics of PAHs in natural loess soils with low CEC and organic matter, or on loess soils modified with surfactants.

The present paper reports findings from a laboratory study with a focus on enhancing the attenuation of PAH contaminants at oil extraction sites where the vadose zone consists of loess soils and daily temperature may vary widely. Herewith, phenanthrene and naphthalene were chosen as indicators of PAHs, and sorption kinetics were studied as a function of temperature in two kinds of soils: natural loess soil and loess soil modified by a cation surfactant, hexadecyltrimethylammonium (HDTMA) bromide. Fused-ring polycyclic aromatic hydrocarbons, naphthalene and phenanthrene, including their substituted derivatives, are regularly found in crude oil and refined oil products. They usually serve as the typical model substrates for studies on the fate,

transport, and metabolism of carcinogenic PAHs (ATSDR 1985; Ohio EPA 2002). Naphthalene and phenanthrene can be removed from the soil surface primarily by evaporation, microbial oxidation and desorption, meanwhile partitioning of these two compounds to soil is generally expected whenever they are released to the soil solution. The objective of this paper is to study the sorption characteristics of naphthalene and phenanthrene in a rapid equilibrium process and their dynamic properties on the basis of changing temperatures. Another purpose is accordingly to investigate possibilities for enhancing the adsorption of petroleum contaminants in the surfactant modified loess soils as an initial technique for restraining transport from the disposal sites prior to removal and/or decomposition.

## Materials and method

### Materials

The loess soil for the experiments was collected from the subsurface at 50–60 cm depth in an uncontaminated area in Lanzhou City, China. The texture corresponds to 18.8% sand, 53.9% silt, 27.4% clay, and 0.1% organic carbon. The CEC of this soil is 3.21 cmol/kg (determined by Atomic Absorption Spectrometry with  $\text{Ca}^{2+}$  as an index cation and a 0.1 mol/L  $\text{BaCl}_2$  solution as the extracting reagent). Phenanthrene and naphthalene were produced by the First Chemical Reagent Factory, Tianjin, China. The chemicals used in this study were all of analytical grade. HDTMA bromide was utilized as the cationic surfactant to modify the loess soil.

### Preparation of naphthalene and phenanthrene solutions

A total of 100.0 mg naphthalene and 50.0 mg phenanthrene were dissolved into the two 500-ml capacity flasks with methanol, respectively. Thus, the base solution concentrations of naphthalene and phenanthrene were 200 and 100 mg/L, respectively. Working solutions were prepared by dissolving the base solution with deionized water into 50 ml, consequently methanol concentration was less than 3%. Therefore, the presence of residual methanol in solution is not expected to affect sorption of PAHs in the solution/soil system. (Moon and others 2003).

### Preparation of HDTMA-modified loess soil

A series of cation surfactant-modified loess soil samples were prepared by coating the natural loess soil sample

with the different aqueous solutions of HDTMA. A total of 500 g of an air-dried soil sample was mixed with 1 L of cationic surfactant solution prior to adding 2 L of distilled water. The mixture was subjected to mechanical stirring at room temperature for 5 h, and the modified soil was then washed repeatedly with distilled water until bromide ions derived from the cation surfactant solution were undetectable as indicated by the 0.01 M  $\text{AgNO}_3$  solution. The above procedures were repeated to modify the soil samples with the various concentrations of HDTMA bromide. Finally, the soil samples were air-dried, passed through a 1-mm sieve, and then stored in glass bottles at room temperature for later use. Modified soils were identified by a prefix that indicated the percent of the loess soil's CEC replaced by surfactant ions followed by the abbreviation for the specific type of cationic surfactant HDTMA. For example, 80 HDTMA indicated that a modified-soil had 80% of loess soil CEC replaced by a hexadecyltrimethylammonium cation ( $\text{HDTMA}^+$ ; Chen and others 2002).

### Sorption procedures

Sorption kinetics was monitored in the batch experiments using naphthalene and phenanthrene as the representatives for PAHs. A total of 5 g of the natural loess soil or modified soil was mixed with 50.0 ml of the two organic solutions with different concentrations in 50 ml Erlenmeyer flasks with glass caps. The flasks were shaken in the dark with a gyratory shaker at 200 r/min at a fixed temperature. After an interval, the flasks were put aside for 10 min and the supernatants were centrifuged at  $3,200\times g$  for 20 min to separate aqueous and solid phase. The concentration of organic compounds in the aqueous phase was determined by RF-540 fluorimetry spectrum. The losses of compounds by photochemical decomposition, volatilization, and adherence to the Erlenmeyer flask were measured in contrast to a blank using shaking and centrifuging without soils added. The mass of phenanthrene or naphthalene sorbed by the natural or modified loess soils was calculated on the basis of reduced solute concentrations in the aqueous phase.

### Analytical method

The concentration of naphthalene and phenanthrene dissolved in the methanol solution was determined by RF-540 fluorimetry spectrum. The stimulation and emission wavelengths for naphthalene were 284 and 331.5 nm, respectively, and 293 and 366.5 nm for phenanthrene, respectively. The determination gap was 10 nm with a scanning speed of 300 nm/min and a registering speed of 30 nm/cm.

## Results and discussion

### Sorption rate

To obtain sufficient kinetic information on soil sorption and to ensure that the sorbed amount of the solute reached its maximum according to previous sorption experiments (Zhou and others 2002), a large number of data were recorded within the first 24 h. A variety of concentrations for naphthalene and phenanthrene in aqueous phase were frequently determined at a temperature of 298 K in an interval to measure sorption rates of the two organic compounds on the natural and modified loess soils. The experimental sorption rate curves of naphthalene and phenanthrene are respectively illustrated in Figs. 1 and 2. It can be seen that, after approximately 30 min for naphthalene and 90 min for phenanthrene, the sorption rate approached constant at 298 K. The sorption rate of naphthalene and phenanthrene on natural loess soils was raised with the increase of the initial concentrations. At a variety of the initial concentrations, the instant sorption rates at 2 min for naphthalene and phenanthrene are correspondently displayed in Table 1, from which it can be seen that the relative values for naphthalene were significantly higher than that of phenanthrene. If the naphthalene and phenanthrene added in the experiments had the same concentration, the sorption amount of naphthalene for the natural loess soil was greater than phenanthrene, corresponding to 17.46  $\mu\text{g/g}$  for naphthalene and 12.59  $\mu\text{g/g}$  for phenanthrene when the initial concentration for both compounds was 2 mg/L. The reason could be addressed to the difference in hydrophobicity (i.e., octanol-water partition coefficient  $K_{ow}$ ), also molecular configurations between two organic compounds as compounds with greater hydrophobicity have slower

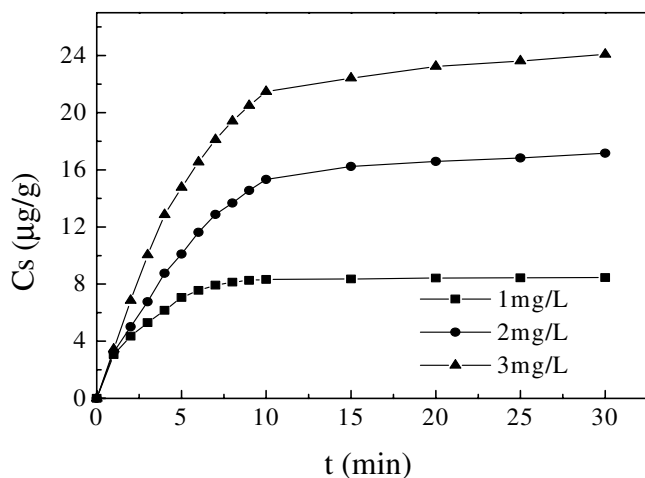


Fig. 1 Sorption rate curves of naphthalene for natural loess soils

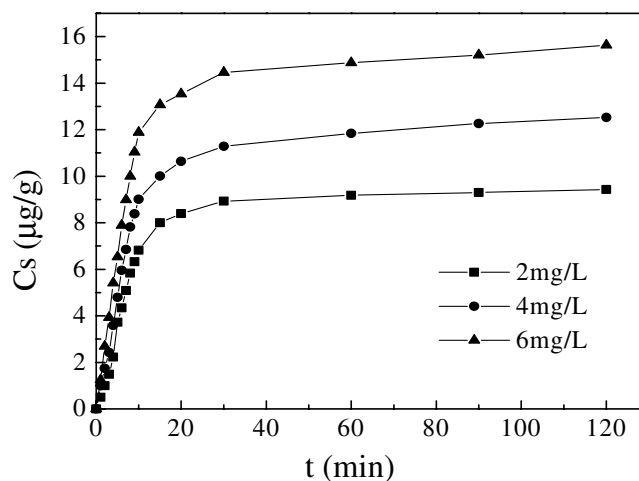


Fig. 2 Sorption rate curves of phenanthrene for natural loess soils

uptake rates in similar soils. Log  $K_{ow}$  value was 4.46 for phenanthrene, but only 3.30 for naphthalene (Yaws 1999); therefore, it gave an explicit explanation for the difference of sorption rates between phenanthrene and naphthalene. These results corroborated the early research results by Karichhoff (1979).

### Surfactant influence

In the experiments, the organic carbon content of the modified loess soils was proportional to the added quantity of the surfactants. The experimental data shown in Figs. 3 and 4 indicate that the sorption rates and relative contents of naphthalene and phenanthrene on the HDTMA-modified loess soils at 298 K were greater than in the natural loess soil for the same conditions, but the presence of surfactant in the modified loess soils did not show any obvious influence on the sorption equilibrium time. The sorption rates and related contents for naphthalene and phenanthrene increased correspondingly with the residual quantity of surfactant in the modified loess soils. At an initial concentration of 2 mg/L, the instant sorption rates for

Table 1 Instant sorption rates for naphthalene and phenanthrene with different initial concentrations at 2 min

Compounds	Initial concentration (mg/L)	Instant sorption rates ( $\mu\text{g/mim}$ )
Naphthalene	1.0	2.175
	2.0	2.505
	3.0	3.424
Phenanthrene	2.0	0.4709
	4.0	0.7798
	6.0	1.330

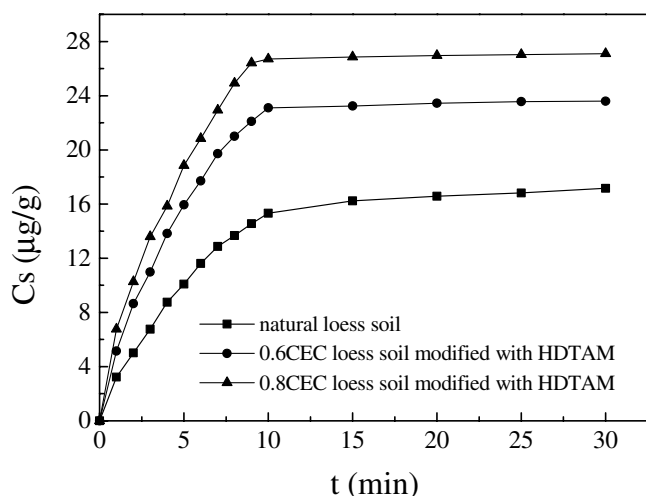


Fig. 3 Sorption rate curves of naphthalene for the HDTMA-modified loess soils

naphthalene in the natural loess soils and two modified soils with 60 and 80 HDTMA were 1.652, 2.309, and 2.671  $\mu\text{g}/\text{mim}$ , respectively, at 10 min; meanwhile, the relative sorption contents were measured as 17.46, 23.59 and 27.09  $\mu\text{g}/\text{g}$ . Similar values for the phenanthrene sorption rates with an initial concentration of 6 mg/L in the three soil samples were 1.228, 2.947, and 4.142  $\mu\text{g}/\text{mim}$ , respectively, at 10 min while the relevant sorption contents were 15.62, 34.53, and 45.39  $\mu\text{g}/\text{g}$ , and sorption rates increased at least 3 times, compared to the natural loess soil. The HDTMA bond is strongly adhered to the negatively charged surfaces of soil minerals. As a result, the organo-modified soils exhibit

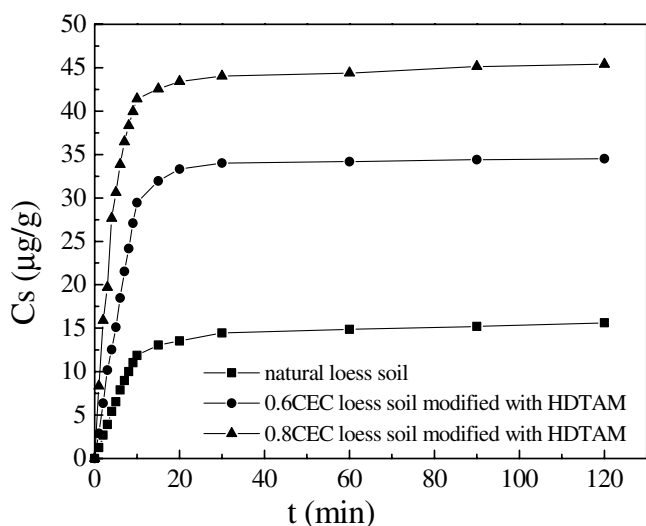


Fig. 4 Sorption rate curves of phenanthrene for the HDTMA-modified loess soils

greatly enhanced sorptive capabilities for organic contaminants. Sorption of nonionic organic chemicals on HDTMA-smectite is essentially due to the solute partitioning into the organic phase created by the large  $\text{C}_{16}$  alkyl chains of HDTMA (Moon and others 2003). Aromatic molecules, depending on their planar shape and delocalized  $\pi$ -bonds, interact strongly with HDTMA, causing a reorientation of the alkyl chains. These solute molecules solvate the cationic ammonium centers and alkyl chains of HDTMA, and also increase the solvency of the HDTMA phase for solute, resulting in greater uptake (Sheng and others 1996).

#### Temperature influence and activation energy

Temperature is an important variable that can dramatically influence the equilibrium and rates of exchange processes. For both organic compounds, sorption rates decreased with increasing temperature. Schwarzenbach and others (1993) stated that temperature had effects on sorption mainly caused by the effect on solubility of the organic compounds. High temperature may result in an apparent increase of aqueous solubility for organic compounds, and thus result in a lower sorption. Figures 5 and 6 show sorption partition coefficients of naphthalene and phenanthrene in the natural loess soil and the dependence on temperature. For naphthalene, the sorption partition coefficient at 318 K ( $K_p = 11.82 \text{ cm}^3/\text{g}$ ) was smaller than 308 K ( $K_p = 25.92 \text{ cm}^3/\text{g}$ ) and much less than 298 K ( $K_p = 32.74 \text{ cm}^3/\text{g}$ ). Similarly, the sorption partition coefficient of phenanthrene at 10 min at 308 K ( $K_p = 1.4181 \text{ cm}^3/\text{g}$ ) was smaller at 318 K ( $K_p = 1.014 \text{ cm}^3/\text{g}$ ), and much less than 298 K ( $K_p = 2.468 \text{ cm}^3/\text{g}$ ). This shows that the relationship between partition coefficient and temperature corresponds to activation energy. The sorption kinetics of organic micropollutants has been shown to occur in at

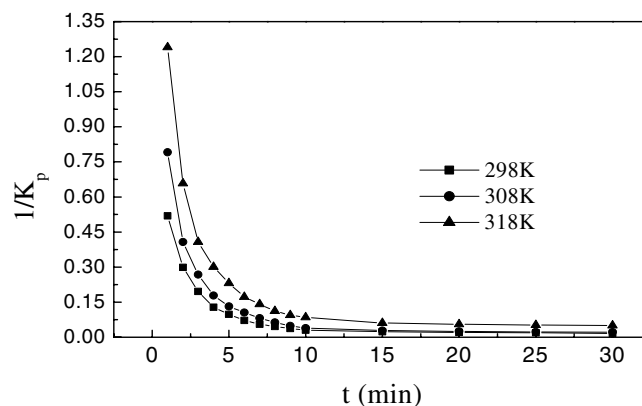
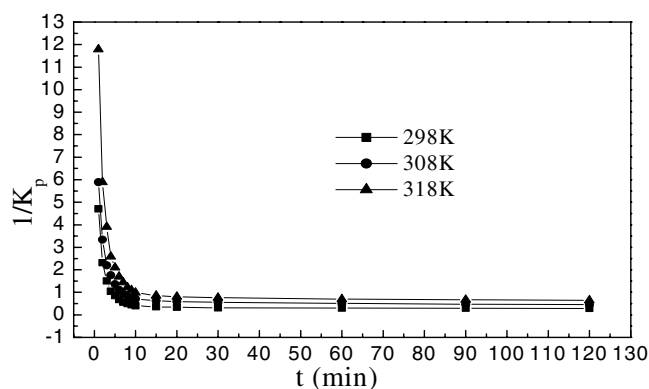


Fig. 5 Partition coefficient curves of naphthalene for natural loess soils at different temperatures



**Fig. 6** Partition coefficient curves of phenanthrene for natural loess soils at different temperatures

least two steps with different rates: a fast process that reaches equilibrium within a few minutes to hours, and a slow process may even take months to years to reach equilibrium. In this paper, the authors focused only on the fast process affected by temperature. The temperature dependence of the sorption process is governed by the equilibrium sorption enthalpy, thus fast equilibrium is probably controlled by equilibrium thermodynamics. If sorption enthalpy is negative, sorption equilibrium will shift to water phase with increasing temperatures.

The temperature dependence of the fast sorption rate can be described by an Arrhenius-type equation:

$$rk = A \exp(-E_a/RT) \quad (1)$$

where:  $E_a$  = activation energy for sorption ( $\text{kJ}\cdot\text{mol}^{-1}$ ),  $A$  = pre-exponential factor ( $\text{min}^{-1}$ ),  $k$  = rate constant of fast sorption ( $\text{min}^{-1}$ )

Eq. (1) can be rewritten:

$$r \ln k = \ln A - E_a/RT \quad (2)$$

Based on the equations above, the activation energy for sorption may be calculated. The experiments verified

that the activation energy of sorption varied from  $-6.196$  to  $-1.172$   $\text{kJ}/\text{mol}$  for naphthalene and from  $-28.86$  to  $-15.70$   $\text{kJ}/\text{mol}$  for phenanthrene at a temperature range between 298 to 318 K. These results indicate a positive temperature dependence for fast sorption rates and implies a low critical limit of the process resulting in low activation energies for sorption.

#### Sorption rate relationships and factors controlling sorption kinetics

Based on the experimental data above, a series of sorption rate relationships were tested depending on initial concentrations. The experimental results showed that sorption rates for naphthalene and phenanthrene in natural and modified soil basically tended to be linear although the initial concentrations varied. The basic equation used is:

$$r - \frac{dr}{r} dt = KC \quad (3)$$

where  $k$  is the sorption rate constant.

Also, several previous studies have found that first-order reaction kinetics fit pesticide sorption processes in soils (Boguslaw and Radoslaw 2002). Table 2 gives fitted kinetic equation constants for the sorption of naphthalene and phenanthrene on the two kinds of tested soils.

For practical applications in the field, it is necessary to understand the factors controlling sorption kinetics. For hydrophobic organic compound sorption, soil particles may be assumed to consist of fine mineral grain and organic matter aggregates. The kinetics of solution-solid exchange could then be described as a radial diffusive penetration of organic pollutants into these porous grain particles based on intra-particle diffusion models (Wu and Gschwend 1986). Sorbate molecules actually diffuse through the pore fluids held in the interstices of natural silt aggregates, and their penetration is "retarded" by microscale partitioning of the

**Table 2** Sorption rate equations and parameters of naphthalene and phenanthrene for natural and modified loess soil at different temperatures

Soil	Compounds	$T/K$	Sorption rate equation	Correlation coefficient	$K/\text{min}^{-1}$	
Natural loess soils	Naphthalene	298	$r = 0.00936C_0 + 0.00966$	0.9976	0.00936	
		308	$r = 0.00863C_0 + 0.00455$	0.9993	0.00863	
		318	$r = 0.00651C_0 + 0.00191$	0.9998	0.00651	
	Phenanthrene	298	$r = 0.00936C_0 + 0.00966$	0.9992	0.00984	
		308	$r = 0.00863C_0 + 0.00455$	0.9961	0.00801	
		318	$r = 0.00651C_0 + 0.00191$	0.9961	0.00473	
Loess soils modified with cation surfactant	Naphthalene	0.6CEC	298	$r = 0.0160C_0 + 0.00991$	0.9838	0.0160
		0.8CEC	298	$r = 0.0185C_0 + 0.0136$	0.9814	0.0185
	Phenanthrene	0.6CEC	298	$r = 0.0240C_0 + 0.00113$	0.9990	0.0240
		0.8CEC	298	$r = 0.0440C_0 + 0.0196$	0.9623	0.0440

**Table 3** Relativity of diffusion sorption rate curves for naphthalene and phenanthrene and relevant parameters

Compounds	Phenanthrene		Naphthalene	
	Line 1	Line 2	Line 1	Line 2
Relativity	0.9864	0.9405	0.9769	0.9276
$k$	0.09301	0.00279	0.04489	0.00102

compounds between essentially mobile (i.e., dissolved in intra-particle pore fluids) and immobile (i.e., in/on intraparticle solids) states of the organic chemicals. With continuous shaking, the particle sizes became smaller, consequently, sorption rates would be gradually increased throughout the experiment by shortening diffusion paths and by increasing exposed particle surface areas. The hole-diffusion, which is the phase partition between intra-particle pore fluids and intra-particle solids, is a slow approach to equilibrium.

All the experimental data were well fitted to the above kinetic diffusion model. The diffuse rate of naphthalene and phenanthrene in aqueous phase was described by the following equation:

$$r - dC/dt = K_f A (C_0 - C_e) = k(C - C_e) \quad (4)$$

where  $K_f$  = coefficient of liquid film,  $A$  = surface of liquid film,  $K_f A = k$ , constant of sorption rate,  $C$  = instant concentration of phenanthrene and naphthalene in solution phase,  $C_e$  = equilibrium concentration,  $C_0$  = initial concentration.

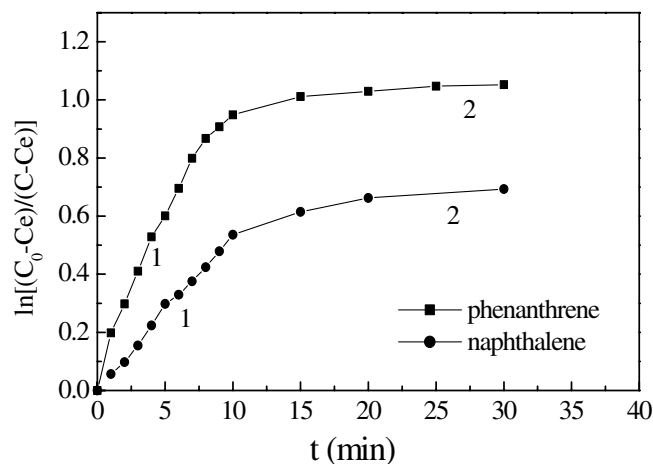
Equation (4) can be rewritten as:

$$\ln[(C_0 - C_e)/(C - C_e)] = K_f A t = kt \quad (5)$$

The relativity of calculated data and related parameter ( $k$ ) in the two parts of each line are listed in Table 3, respectively. The results calculated with diffusion kinetic Eq. (4) are illustrated in Fig. 7 from which the calculated data described a rapid initial uptake on the soil (part 1) followed by a slow approach to equilibrium (part 2), indicating that a fast adsorption for naphthalene and phenanthrene was accomplished within 10 min, but the hole-diffusion process stated the approach to equilibrium after 20 min.

## Conclusions

Soil and aquifer material with low organic matter contents have a very small retardation capability for PAHs; therefore, it is significant to study retardation of cationic surfactant HDTMA bromide treated soil to promote

**Fig. 7** Diffusion sorption rate curves of naphthalene and phenanthrene for natural loess soils

PAHs sorption. The sorption capacity of naphthalene and phenanthrene in the modified loess soils was significantly enhanced and sorption rates were at least 3 times faster than that on the natural loess soil. A first-order kinetic model fitted the experimental data well. The temperature dependent sorption of the two organic compounds displayed a linear relationship on  $\log K_p$  and  $1/T$ . Although the sorption rates of naphthalene and phenanthrene for treated and untreated soils were influenced by initial concentration, the HDTMA-modified loess soil was a much more effective sorbent for naphthalene and phenanthrene in comparison with the natural loess soil for similar reaction conditions. Consequently, soil organic matter (SOM) derived from HDTMA was the predominant phase for the sorption of naphthalene and phenanthrene on the HDTMA-modified loess soils. In this study, temperature was an important component that influenced the equilibrium and rates of sorption, but the effect appeared to be limited. For both naphthalene and phenanthrene, the sorption rate decreased with the increase of temperature varying from 298 to 318 K in the experiments. The results show that treated loess soil can provide a new technique to prevent soil contamination and further deterioration of groundwater aquifers from leaching organic pollutants.

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