Effect of sorbed nonylphenol on sorption of phenanthrene onto mineral surface

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ABSTRACT

The effect of sorbed nonylphenol (NP) at low levels on phenanthrene sorption onto the mineral surfaces (organic-removed sediment, kaolinite) and a natural sediment with low organic content was investigated in this study. NP could be sorbed on the sediment and the minerals, and part of sorbed NP interacted with the solid surface stably. At very low concentrations, sorbed NP was observed to inhibit the sorption of phenanthrene on the hydrophilic mineral surfaces (organic-removed sediment and kaolinite) based on the change of phenanthrene apparent solid–liquid distribution coefficients. When the amount of sorbed NP increased to higher levels, enhancement of phenanthrene sorption occurred. Similar result was also obtained in isotherm experiments. On the mineral sorbents contacting with 1.0 mg/L NP solution previously, the K_d values were lower compared with those on the sorbents without sorbed NP. On the sorbents with higher levels of sorbed NP (contacting with 10 mg/L NP solution previously), the K_d values increased, while the isotherm tended to be more nonlinear. Concentrations of sorbed NP determined its orientation on the surface, which could presumably affect the water film above the sorbents, or contribute more adsorption sites for phenanthrene.

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1. Introduction

Sorption of hydrophobic organic chemicals (HOCs) to soils or sediments has a major influence on their transport, bioavailability, and fate in natural aquatic environments. The sorption of non-ionic HOCs from water to solid phase is thought to be primarily due to their interaction with the immobile organic matter. However, for sorbents containing only very small amount of organic carbon (OC), the mineral phase was found to contribute significantly to the sorption of HOCs [1,2]. The sorption of HOCs onto mineral surface is considered to occur presumably by dispersion force onto a water film above the minerals [3]. Even if the interaction is usually weak and non-special, difference of HOCs sorption on different mineral surfaces has also been found [4–6].

Surfactants could enter environments through the seepage of wastewater. Besides, they were also used as washing agents in HOCs contaminated soil remediation [7], because surfactants could enhance the solubility of HOCs in aqueous phase when added as a cosolvent at their critical micelle concentration (CMC). All above might result in a residue of surfactant compounds in the natural water or soil environmental, but usually at relatively low concentrations. Particles modified by surfactants, especially cationic surfactants, have been found to present elevated capability for sorption of HOCs in many studies [8–10], which is mainly caused by partitioning of HOCs into sorbed surfactant phase [11–16]. Most of current studies focused on the modification of sorbents, with the concentrations of surfactants much higher than any currently known environmental level, while few studies reported the competitive influence of surfactants on HOCs sorption on soils [17,18]. However, understandings of the effect of surfactant at low concentrations relevant to environment on the HOCs sorption, especially for that on the hydrophilic mineral surface, are still deficient.

The objective of this study was to investigate the effects of low level sorbed nonylphenol (NP) on the sorption of a representative HOC, phenanthrene onto sorbents with low OC content. NP was selected because of its widespread occurring in the world as an intermediate from degradation of nonylphenol ethoxylates (NPEOs), a widely used nonionic surfactant [19], while NP itself is surface active, with a CMC of about 6×10^{-5} mol/L [20]. Natural low OC content sediment and organic-removed sediment, as well as a clay mineral, kaolinite, were used as the sorbents.

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2. Materials and methods

2.1. Sorbents and reagents

Sediment collected from the Yellow River of China was selected as the natural sorbent in this study, because of its low OC content (0.27%). Sediment was air-dried and homogenized, and then sieved to 38–75 μm. To gain the organic-removed sediment, natural sediment was further treated with H_2O_2 to remove the natural organic matters (NOMs) [21]. The particles of 38–75 μm were then sieved out after washing and drying. Result of X-ray diffraction analysis to the organic-removed sediment showed that the main mineral components were quartz (~30%) and kalialbite (~25%), which is a common precursor of clay mineral (provided by Center Lab of Nankai University). A clay mineral, kaolinite, was also selected as a sorbent, which was purchased from Tianjin Fuchen Chemical Reagent Co., Inc., China. The BET-based specific surface area and porosity of these three sorbents were determined by a surface area analyzer (ASAP2010, Micromeritics Instrument Corporation, USA) using N_2 as the sorbate. OC content was determined by Walkley–Black method [22].

Phenanthrene (standard) and nonylphenol (NP, technical grade) were purchased from Acros Organics (NJ, USA) and Tokyo Chemical Synthesis Ind. Co. Ltd. (Tokyo, Japan), respectively. Acetonitrile, methanol, and water used in this study were of HPLC grade.

2.2. Sorption experiment

Sorption experiments were conducted in triplicates using batch equilibration method. In NP sorption isotherm experiment, 50 μL of NP/methanol stock solution with concentrations ranging from 90 to 1800 mg/L was added into 10 mL glass centrifuge tubes, which contained 500 mg sorbent and 9 mL electrolyte solution, to gain NP solutions at five concentrations (0.5, 1.0, 2.0, 5.0, 10.0 mg/L). The solution matrix contained 1 mmol/L $CaCl_2 \cdot 2H_2O$, 0.1 mmol/L $MgCl_2$, 0.5 mmol/L $Na_2B_4O_9 \cdot 10H_2O$, and 0.3 mmol/L NaN_3 (pH 8.0 ± 0.1) [23]. The tubes were sealed with groundin glass caps and Teflon liners. Then, all tubes were hand-shaken for a few minutes to make them uniformly mixed, before they were horizontally placed on a constant temperature air bath shaker and oscillated for a period of 24 h at 150 rpm and $25 \pm 1\,^{\circ}\text{C}$. Preliminary sorption dynamics studies indicated that this time was sufficient for NP to reach the adsorption steady state, which was in accordance with other kinetic studies of NP sorption [25]. The solids were separated from aqueous solution by centrifugation at 4800 rpm for 20 min, and 8 mL of the supernatant were taken out exactly for HPLC analysis to determined NP concentration. Also the systems without NP added were made, to gain the sorbents without NP sorbed.

After that, 8 mL of electrolyte solution containing 1.00 mg/L phenanthrene was added to the above tubes. Then the tubes were sealed and shaken for 12 h at 150 rpm and $25 \pm 1\,^{\circ}\text{C}$. Apparent steady state of phenanthrene sorption can be achieved for all the three sorbents as indicated by a preliminary experiment. After centrifugation at 4800 rpm for 20 min, the supernatants were analyzed by HPLC. Sorbed NP concentrations on the solids were calculated by differences of amounts of NP in solution, while the NP desorption caused by solution replacement had been accounted for. Amounts of sorbed phenanthrene were also determined, and the apparent solid–water distribution coefficient (K_d^{app}) values of phenanthrene on sorbents with sorbed NP at different concentrations were calculated. The recoveries of control solute samples (without solids) were $98.9 \pm 1.2\%$ for NP and $98.2 \pm 1.0\%$ for phenanthrene.

Moreover, phenanthrene sorption isotherms on non-NP sorbents and sorbents with different amount of sorbed NP were established. To obtain sorbents with two levels of sorbed NP, 500 mg

Table 1
Properties of the sorbents

Sorbents	BET (m^2/g)	Pore volume (10^{-2} cm^3/g)	OC (%)
Natural sediments	4.767 ± 0.0246	1.26	0.27
Organic-removed sediments	2.972 ± 0.0309	0.85	0.06
Kaolinites	8.048 ± 0.1042	4.50	0.08

of the sorbents were equilibrated for 24 h in 9 mL electrolyte solution containing 1.0 or 10.0 mg/L NP. Sorbents without sorbed NP were treated through the same process in clean electrolyte solution. After centrifugation, 8 mL of the supernatants were replaced by 8 mL electrolyte solution containing 7 different concentrations of phenanthrene. The tubes were sealed and shaken for 12 h, and then the supernatants were analyzed after centrifugation.

2.3. Chemical analysis

Waters 1525 high-performance liquid chromatograph (HPLC), with Waters 2475 fluorescence (FL) detector (Waters Company, USA) was utilized for simultaneous analysis of NP and phenanthrene using a Waters C_{18} column (with μBondapak 3.9 mm i.d. × 300 mm × 10 μm, Waters Corp., USA). The mobile phase was acetonitrile: water (80:20, v/v), and the flow rate was 1.0 mL/min. Ex/Em wavelengths in FL were programmed to optimize phenanthrene and NP detection, which is 280/355 nm during 0–5.1 min (for phenanthrene), and 233/302 nm during 5.1–7.0 min (for NP). All measurements were in duplicate, with the uncertainty being generally less than ±5%. The average data were used for discussion. The detection limits were 0.05 mg/L for NP and 0.50 μg/L for phenanthrene.

3. Results and discussion

3.1. Properties of the sorbents

Properties of the sorbents used in this study, including specific surface area, porosity, and OC content, are shown in Table 1. Compared with the natural sediment, BET-surface and pore volume of the sediment decreased obviously after organic-removing treatment, while the OC content of the sediment decreased from 0.27% to 0.06%. This suggests that organic matter on the natural sediment used in this experiment contributes to the BET-surface and the pore volume. The BET-surface and pore volume of kaolinite used in this study are much larger than the other two sorbents. The greater BET-surface of kaolinite should be due to its larger inner surface area.

3.2. Sorption behavior of NP onto the solids

Fig. 1 presents sorption isotherms of NP on the natural sediment, organic-removed sediment, and kaolinite. It was found that the sorption capability of the natural sediment for NP was greater than those of the other two sorbents, which might be due to the NOM in the sediment. For clays and other minerals, surfactants sorption should be mainly due to the adsorption process, which could occur through a number of mechanisms involving ion exchange, ion pairing, hydrophobic bonding, adsorption by polarization of π electrons, and adsorption by dispersion forces, etc. [24]. Nagasaki et al. studied the adsorption of NP on montmorillonite, and attributed the adsorption of NP to the interaction with the Al atoms on the broken-edges of clay sheets [25]. Sorption of NP on an aquifer material with low OC content was also reported, which was according to Freundlich model [26].
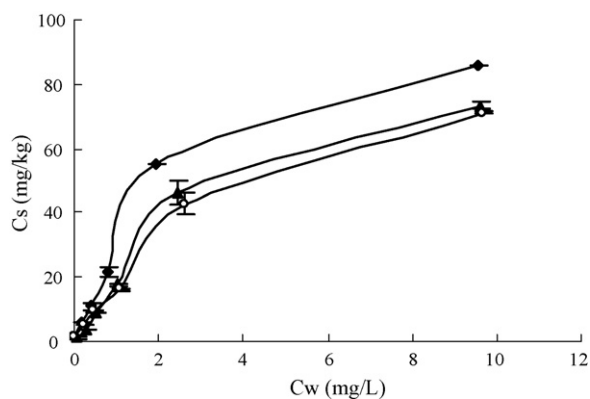
Fig. 1. Sorption isotherm of NP on the natural sediment (♦), organic-removed sediment (▲), and kaolinite (○).

In phenanthrene sorption experiments, parts of pre-sorbed NP returned to solution after the solution replacement (Fig. 2). It is notable that for organic-removed sediment and kaolinite, the interaction of sorbed NP with the sorbent surface at low surface concentration of sorbed NP (Γ_{NP}) (<1 mmol/m²) seems to be stable, with the desorption percent of pre-sorbed NP less than 10%. However, for the natural sediment with low Γ_{NP}, 52–41% of NP desorbed from the solid phase, which might be due to the return of part of NP partitioning in the solid NOM previously. For the three sorbents with NP at relatively higher concentrations, the desorption percent of pre-sorbed NP converged to ∼25%.
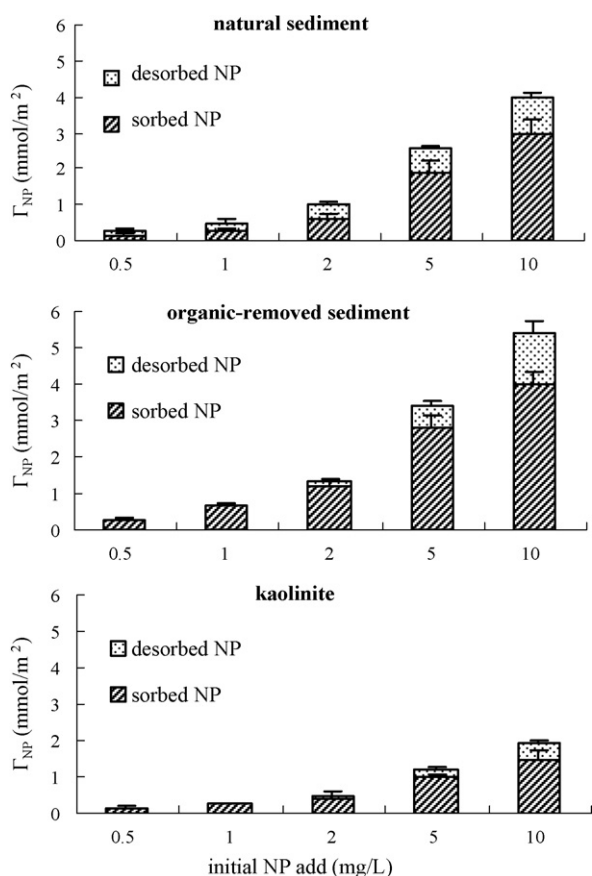

Fig. 2. Surface concentrations of sorbed NP (Γ_{NP}) on the three sorbents in phenanthrene sorption experiment. "Desorbed NP" means the difference of Γ_{NP} caused by solution replacement.
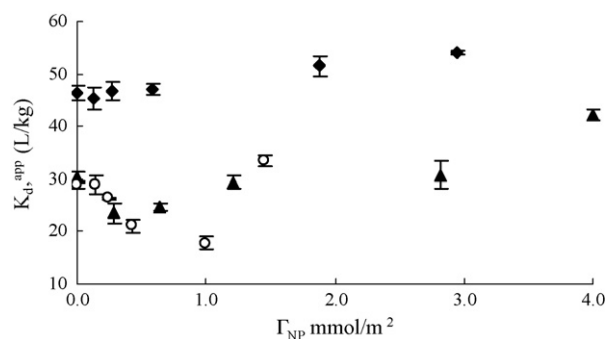

Fig. 3. Apparent solid–water distribution coefficient of phenanthrene on the natural sediment (♦), organic-removed sediment (▲), and kaolinite (○) with different surface concentrations of sorbed NP (Γ_{NP}).

3.3. Sorption behavior of phenanthrene on NP sorbed sorbents

The effects of sorbed NP on the K_d^{app} of phenanthrene on the three sorbents were studied as described formerly, with phenanthrene added at the initial dissolved concentration of 1.0 mg/L. K_d^{app} of phenanthrene on the natural sediment, organic-removed sediment, and kaolinite without sorbed NP were 46.4, 30.3, and 29.0 L/kg, respectively (Fig. 3). For organic-removed sediment and kaolinite with sorbed NP at low Γ_{NP}, K_d^{app} of phenanthrene decreased, and reached a minimum of 24.6 L/kg (for organic-removed sediment) and 17.8 L/kg (for kaolinite), while Γ_{NP} on the mineral surface were 0.65 mmol/m² (for organic-removed sediment) and 0.99 mmol/m² (for kaolinite), respectively. With Γ_{NP} increased continuously, the amount of sorbed phenanthrene began to increase, and finally surpassed the sorption on the three sorbents without NP sorbed, with the K_d^{app} reaching 42.1 L/kg (for organic-removed sediment), and 33.4 L/kg (for kaolinite). Insignificant difference was observed in K_d^{app} of phenanthrene on the natural sediment with low Γ_{NP}, which maintained 46–47 L/kg. However, when the amount of sorbed NP increased to a higher lever (∼3 mmol/m²), K_d^{app} of phenanthrene increased to 54.2 L/kg. It seems that the presence of sorbed NP at low Γ_{NP} inhibit the sorption of phenanthrene on the mineral surfaces (organic-removed sediment and kaolinite), but at higher Γ_{NP}, sorbed NP may increase phenanthrene sorption.

To further validate the effect of sorbed NP on phenanthrene sorption, sorption isotherm experiments on the sorbents with different levels of sorbed NP were done (Fig. 4). Sorbents with low level or relatively higher level of sorbed NP were gained by contact
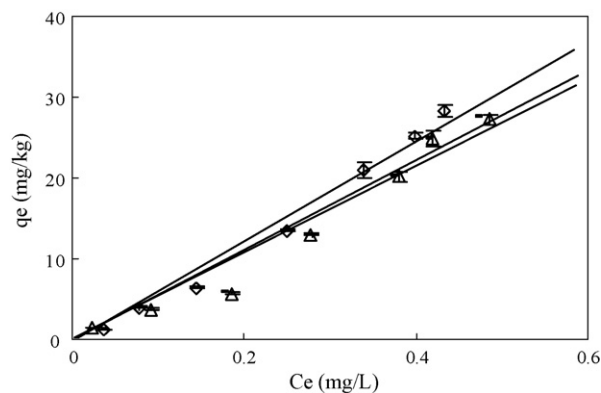

Fig. 4. Sorption isotherms of phenanthrene on the natural sediment without/with sorbed NP. −, sorbent contacting with electrolyte solution previously; △, sorbent contacting with 1.0 mg/L NP solution previously; and ◊, sorbent contacting with 10 mg/L NP solution previously.

Table 2
Linear model parameters of phenanthrene sorption on the sorbents with sorbed NP at different levels

Sorbents		K_d (L/kg)	R^2
Natural sed-i-ment	Non-NP[a]	55.48 ± 2.71	0.99
	L-NP[b]	53.72 ± 2.71	0.97
	H-NP[c]	61.61 ± 2.07	0.99
Organic-removed sed-i-ment	Non-NP	28.49 ± 0.52	0.99
	L-NP	24.45 ± 0.61	0.99
	H-NP	36.20 ± 1.26	0.99
Kaolinite	Non-NP	30.11 ± 0.91	0.99
	L-NP	27.62 ± 1.05	0.99
	H-NP	36.73 ± 1.62	0.98

[a] Sorbents contacting with electrolyte solution previously.
[b] Sorbents contacting with 1.0 mg/L NP solution previously.
[c] Sorbents contacting with 10 mg/L NP solution previously.

with NP solution (1.0 or 10.0 mg/L) previously. As shown, almost all of the isotherms are practically linear. Treating these isotherms in linear form with zero intercept, the linear sorption coefficients (K_d) (L/kg) for the phenanthrene on the different sorbents were calculated through linear regression analysis (Table 2). Coinciding with the foregoing result, the K_d on the natural sediment (55.53 L/kg) was the highest among those on the three sorbents without NP sorbed, which might be partially due to the solid NOM of the sediment. Linear sorption of phenanthrene ($R^2 = 0.99$) were observed on the mineral surface, whereas the K_d values were much lower, which was 28.44 L/kg on organic-removed sediment and 30.10 L/kg on kaolinite, respectively. High-isotherm linearity of phenanthrene onto hydrophilic minerals was also reported in other studies [3], and the sorption mechanism was explained by London forces between the HOCs solute and the water film above the mineral surface.

For the mineral sorbents with low levels of sorbed NP (solids contacting with 1.0 mg/L NP solution previously), lower K_d values (23.44 L/kg on organic-removed sediment and 26.57 L/kg on kaolinite) were gained, which was similar in trend to the foregoing result about K_d^{app}. The inhibition of phenanthrene sorption was hypothesized to be caused by the change of water film due to sorbed NP. The water film on the mineral surface was considered to be formed via the H-bonding of water with surface hydroxyls [27]. For the sorption of surfactants onto the mineral surface, mechanisms including H-bonding, van der Waals interaction, and attraction between the hydrophilic group and the hydrophilic surface were involved [24,28,29], which could probably affect the
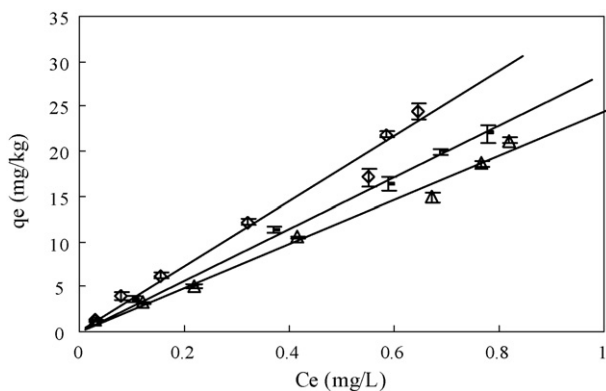

Fig. 5. Sorption isotherms of phenanthrene on organic-removed sediment without/with sorbed NP. —, sorbent contacting with electrolyte solution previously; △, sorbent contacting with 1.0 mg/L NP solution previously; and ◇, sorbent contacting with 10 mg/L NP solution previously.
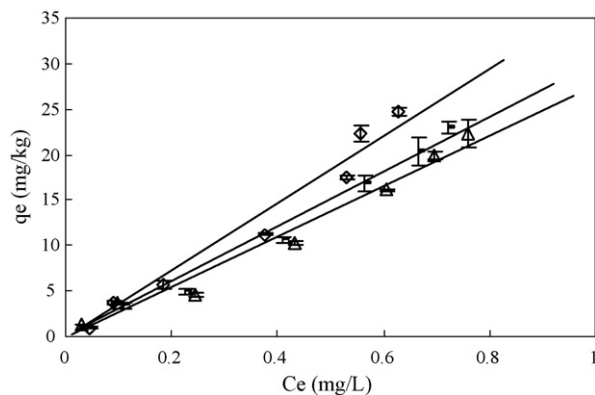

Fig. 6. Sorption isotherms of phenanthrene on kaolinite without/with sorbed NP. —, sorbent contacting with electrolyte solution previously; △, sorbent contacting with 1.0 mg/L NP solution previously; and ◇, sorbent contacting with 10 mg/L NP solution previously.

characteristics of water film (e.g., film surface area). The existing orientation of the surfactants on the solid surface was found to depend on their concentration. When the sorbed surfactant level is low, the adsorbate–adsorbate interactions are negligible because the molecules are far away from each other, and surfactant molecules "pronate" on the sorbent surface sparsely [24]. So, the sorbed NP molecule at low sorbed level could act as a barrier as well as an adsorption competitor for water molecule, which broke the order of sorbed water film [28]. However, sorbed NP seems not to interact with phenanthrene solute directly, because of the lower K_d and good linearity of these isotherms (Figs. 5 and 6; Table 2). For the natural sediment with low level of sorbed NP, no significant difference of K_d value was observed, which might be due to the different mechanism of phenanthrene sorption on solids with NOM.

For the sorbents contacting with 10 mg/L NP solution previously, the Γ_{NP} was calculated to be 2.95 mmol/m² on the natural sediment, 4.00 mmol/m² on organic-removed sediment, and 1.45 mmol/m² on kaolinite, respectively. On these sorbents with sorbed NP at relatively high level, higher phenanthrene K_d of 61.71, 36.13 and 36.70 L/kg were also found. Sorbed surfactants have been reported to enhance the sorption of HOCs in several studies. Edwards et al. proposed that sorbed surfactants enhanced HOCs sorption not only by increasing the OC content of the sorbents [30], but also by acting as an active sorbent for HOCs. With more surfactant molecules sorbing onto the solids, the hydrophobic tails of the sorbed surfactants are displaced from the surface by hydrophobic groups of the adjacent molecules [24]. Hence, the hydrophobic tails of the sorbed surfactants get more chances to interact with the HOC solutes, which could enhance HOC sorption through bridging action [31].

4. Conclusions

At low concentration level, NP could be sorbed stably on sorbents with low OC content, especially on the organic-removed sediment and kaolinite. On mineral surfaces with very low levels of sorbed NP, phenanthrene sorption was somewhat inhibited compared with that on minerals without NP. It was hypothesized to be caused by the change of water film due to sorbed surfactant molecule. But significant difference was not found on the natural sediment with sorbed NP at low levels. When sorbed NP presented at relatively high levels, phenanthrene sorption was enhanced on all the three sorbents, because NP contributed adsorption sites for the solute. This finding implies that a cocontaminant of this sur-

factant even at very low levels could probably affect the sorption of HOCs in the soil or aquatic environment containing solids with low OC content, which related to their mobility, transfer, and health threat.

Acknowledgement

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