ORIGINAL ARTICLE

Role of salinity in the multiphase redistribution of polycyclic aromatic hydrocarbons (PAHs) in sediment suspension

Rufeng $Li^{1,2} \cdot$ Chenghong Feng^{1,2} \bullet Dongxin Wang² · Baohua $Li^2 \cdot$ Lijuan $\text{Hu}^1 \cdot \text{Zhenyao Shen}^1$

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Abstract Sediment suspension in estuarine environment was simulated with particle entrainment simulator device to investigate the phase redistribution characteristics of polycyclic aromatic hydrocarbons (PAHs). The main focus was laid especially on the role of salinity gradient in the static and dynamic release behavior of PAHs via keeping the consistent of other factors (i.e., hydrodynamic condition and sediment component) in the whole experiment. Results showed that salinity facilitated the accumulation of dissolved and particle-bound PAHs in overlying water in sediment pre-standing and suspending period, which certainly increased the adverse ecological effect of PAHs in sediments. The most beneficial value of salinity for PAHs release from sediments was about 20 %. Salinity had more significant impact on the release behaviors of $2+3$ ring PAHs than other individual PAHs species. Salinity significantly changed the diameter and suspending status of flocs via adsorption and charge neutralization, and thus affected the existing status of PAHs in the overlying water. Based on quantitative calculation, salinity was found to have more significant influence on the release of PAHs than the pre-fixed hydrodynamic force.

Keywords Sediment suspension · PAHs · Salinity · Phase redistribution - Estuary - Release mechanism

 \boxtimes Chenghong Feng fengchenghong@hotmail.com

Introduction

Sediment suspension facilitates multiphase redistribution of pollutants in surface water, and has sparked an interest in research community on potential adverse ecological effects (Tremblay et al. [2005;](#page-7-0) Manning et al. [2010](#page-7-0)). The sediments can act as a transient storage compartment for the contaminants, and the sediment suspension will make these compounds release back into the water column and may have the potential to cause adverse toxicity effects on the ecosystems (Hawthorne et al. [2007](#page-7-0); Patrolecco et al. [2010](#page-7-0)).

As a class of well-documented pollutants, polycyclic aromatic hydrocarbon (PAHs) are of great environment concern due to their potential for bioaccumulation, widespread distribution and carcinogenic properties to human and ecosystem health (Li et al. [2012](#page-7-0)). They often easily bind to suspended particles in surface water and subsequently deposit as typical persistent organic pollutants (POPs) in sediments. Therefore, numerous studies investigated the distribution of PAHs in sediments all over the world in the past decades (Qin et al. [2011\)](#page-7-0), and also PAHs are ideal and typical organic compounds to probe their release behavior in sediment suspension.

Sediment suspension especially happens in estuary due to the natural activities (e.g., runoff and tidal current interaction, salt water and fresh water intersection, wind disturbance) and anthropogenic activities (e.g., shipping and fishing) (Brunk et al. [1997](#page-7-0); Xia et al. [2004](#page-7-0); Tremblay et al. [2005](#page-7-0)). In the estuarine area, gradient change of salinity is a typical feature (Tremblay et al. [2005\)](#page-7-0). Under the drive of the ''salting-out effect'', high ionic strengths separate hydrophobic compounds from water to other phases (e.g., solid phase) (Jeon et al. [2011\)](#page-7-0). Meanwhile, the salt-induced flocculation influences the deposition and resuspension of the sediment components (e.g., mineral

State Key Laboratory of Water Environment Simulation, School of Environment, Beijing Normal University, Beijing 100875, China

² Key Laboratory for Water and Sediment Science of Ministry of Education, School of Environment, Beijing Normal University, Beijing 100875, China

composition and organic matters), which are important carriers of PAHs in estuaries (Naudin et al. [1997](#page-7-0)). Therefore, salinity has significant potential for contaminant redistribution between phases in surface water during sediment suspension.

However, according to our knowledge, not all research approved the significant role of salinity in estuaries. In some studies, salt-induced aggregation played a minor role in enhancing the settling of suspended solids (Thill et al. [2001\)](#page-7-0) and salt was even proved to have no obvious effect (Fotopoulou et al. [2011\)](#page-7-0). In sediment suspension, the disruption of sediment cohesion requires sufficient shear stress, and hydrodynamic conditions are often considered as the premise (Evans [1994\)](#page-7-0). Moreover, sediment components are often considered as the main carrier of pollutants and also greatly affect the transportation of contaminants in sediment suspension (Eggleton and Thomas [2004](#page-7-0); Mouhri et al. [2008;](#page-7-0) Wang and Yang [2010\)](#page-7-0). Therefore, obvious controversy still existed on the role of salinity. As for PAHs, even more few studies have yet discussed the impact of salinity on the release behaviors and mechanism of PAHs in sediment suspension (Brunk et al. [1997](#page-7-0); Thill et al. [2001;](#page-7-0) Tremblay et al. [2005](#page-7-0); Fotopoulou et al. [2011](#page-7-0); Oh et al. [2013](#page-7-0)).

Therefore, this study sampled raw sediments at Chaoyangnongchang (31°04'49.42"N, 121°53'29.11"E) in the estuary of the Yangtze River (one of the World's Top 10 Rivers at Risk, World Wide Fund for Nature), and thus kept the consistency of sediment composition and hydrodynamic condition in experiments to probe the impact of salinity gradient on PAHs release behaviors in sediment suspension. The main focus is laid on the impact of salinity on the multiphase redistribution of PAHs. The roles of salinity, hydrodynamic condition and sediment components have also been compared.

Materials and methods

Materials

Sediments were collected from Chaoyangnongchang in the Yangtze estuary using a Van Veen stainless steel grab sampler (Eijkelkamp, Netherlands) and kept at -20 °C

Table 1 Composition of artificial seawater with different salinity

prior to laboratory tests. To probe the impact of salinity, artificial seawater of different salinities [5, 20 and 35 % $(g g⁻¹)$] was synthesized by the prescription in Table 1. The tap water with salinity of ~ 0 % was used as control water. Sodium azide was added as the microbial inhibitor and the concentration was 100 mg L^{-1} (Xia et al. [2013](#page-8-0); Zhou and Broodbank [2014](#page-8-0)).

The sixteen US EPA priority PAHs, i.e., naphthalene (Nap), ace-naphthylene (Acy), acenaphthene (Ace), fluorine (Flu), phenanthrene (Phe), anthracene (Ant), fluoranthene (Flu), pyrene (Pyr), benz[a]anthracene (BaA), chrysene (Chr), benzo[b]fluoranthene (BbF), benzo[k]fluoranthene (BkF), benzo[a]pyrene (BaP), indeno[1,2,3 c,d]pyrene (InP), dibenzo[a,h]anthracene (DahA), and benzo[g,h,i]perylene (BghiP) were analyzed (Li et al. [2012](#page-7-0)). The standard mixtures [PAH Solution Mix, 0.2 mg mL⁻¹ in CH₂Cl₂:MeOH(1:1)] were obtained from Beijing J&K Chemical United Win Technology Limited. The working standard solutions $(1 \text{ mg } L^{-1})$ were prepared by diluting appropriate volumes of the standard mixture with methanol (HPLC grade, Merck, Germany). Other solvents and reagents included acetone (HPLC grade, Tedia, USA), dichloromethane, n-hexane (HPLC grade, Dikma, USA), silica gel (200–300 mesh) and anhydrous sodium sulfate (analytical grade, Beijing Chemical Reagent Co., China). Silica gel and anhydrous sodium sulfate were baked in a muffle furnace at 450 \degree C for 4 h, kept in a sealed desiccator prior to use. Water was obtained from a Milli-Q system (Millipore, Bedford, MA, USA) with a specific resistivity of 18.2 M Ω cm. 0.45 µm glass fiber filters (Whatman, England) used to collect suspended particles were cleaned by baking at 450° C for 4 h. Glass wares were prepared as follows: cleaned in an ultrasonic cleaner (KQ-502B, Kunshan Ultrasonic Instruments, China); then heated at 450 $^{\circ}$ C in a muffle furnace for 4 h and rinsed with acetone, n-hexane.

Sediment suspension experiments

The designed particle entrainment simulator (PES) device, described in detail in our previous study (Feng et al. [2007\)](#page-7-0) illustrated Fig. [1](#page-2-0) was used to perform sediment suspension. Before experiment, the sediment (2.43 kg) was fully mixed to maintain the consistence of the physicochemical

Fig. 1 Schematic view of the particle entrainment simulator (PES) used in the sediment suspensions

characteristics throughout the whole experiment. For each group of sediment suspension experiment, 2.43 kg of the homogenized sediment was transferred into the apparatuses. Then, 1.4 L of artificial seawater of certain salinity degree was poured into apparatuses. The organic glass tank was positioned to the PES unit and the perforated grid was lowered into the water. After 20 h pre-standing, 100 mL of overlying water were taken as control sample to measure PAHs release flux from sediment. Afterwards, based on the optimized hydrodynamic condition in our previous study (Feng et al. [2007\)](#page-7-0), the PES was adjusted to 750 r min^{-1} corresponding to 0.5 N m^{-1} shear level to make the sediments suspended and the motor was opened to shake for 12 h. The distance of the grid and sediment–water interface was 5 cm. 100 mL of overlying water samples was collected at 0.5, 1.5, 3.5, 6.5 and 12 h. Then 100 mL of water with the same salinity of initial water was added after sampling to maintain volume of the overlying water. Each sample was immediately filtered using $0.45 \mu m$ glass fiber filters (Whatman, England) to separate water and particle fraction. 5 mL of water samples was put in clean glass bottle and refrigerated for determination of pH and dissolved organic carbons (DOC). The left 95 mL water was used to determinate the concentration of dissolved PAHs in

water phase. Concentrations of total suspended solids (TSS) and PAHs absorbed on the suspended particles were measured via the freeze-dried particles which were filtered out from the water samples.

Analytical methods

PAHs in suspended particles including each glass fiber filter were extracted using hexane and acetone (1:1 v/v) by an accelerated solvent extractor (ASE-300, Dionex, USA) and then were concentrated to 1–2 mL by a rotary evaporator (RV 05, IKA, Germany). The concentrations were further purified by silica gel and anhydrous sodium sulfate, and the detailed progress can be found in our previous study (Li et al. [2012](#page-7-0)).

PAHs concentrations were analyzed by high-performance liquid chromatography (HPLC) (Dionex Utimate 3000, USA) with UV and fluorescence detectors, which is a classical analytical method to quantify PAHs in environmental matrixes (Gratz et al. [2000;](#page-7-0) Capelo et al. [2005](#page-7-0)). The injection volume was $20 \mu L$, and the temperature of the C18 column (Varian, 250 mm \times 4.6 mm, 5 µm) was 30° C. The gradient process was performed by the method of Feng et al. ([2007\)](#page-7-0) that 75 % methanol at the beginning and followed by a 17 min linear gradient to 100 % methanol for 30 min. A linear gradient (1 min) at starting conditions followed by a 7 min pre-run to achieve equilibrium to each subsequent run. Acenaphthylene, which was weakly fluorescent, was quantified by UV detector at 229 nm. Sediment particle size and DOC were measured by Laser Particle Size Analyzer (Microtrac S3500, USA) and Total Organic Carbon Analyzer (Elementar, Germany), respectively.

Quality assurance and quality controls

Strict quality control was operated in the experiment. The particle samples and water samples were spiked with the mixed PAHs standard solution, then extracted, purified, and analyzed in the same way as the samples. The six parallel experiments indicated that recoveries for the 16 PAHs were 74.9–120 % from the particles samples and 60–100 % from water samples. The relative standard deviations (RSDs) of the 16 PAHs were all below 10 % for the particles samples and below 7.46 % for water samples. Additionally, the detection limits for the 16 PAHs ranged from 0.098 to 0.191 μ g L⁻¹ on water samples, and from 0.34 to 4.05 ng g^{-1} on a sample dry mass basis, respectively. Each series of experiments was accompanied by two blank experiments and method blanks operation showed no detectable amounts of PAHs.

Results

Physicochemical characteristics of raw sediments and the overlying water

The background contents of the sixteen PAHs in the raw sediment were analyzed and listed in Table 2. The total PAHs content is 544.2 ng g^{-1} dry weights, which is at the relative low level among estuaries influenced greatly by human activities in the world (Kwach et al. [2009;](#page-7-0) Garcia et al. [2010](#page-7-0)). In this study, clay was defined as materials with particles smaller than $2 \mu m$. Silt size particles fall in the range $2-63 \mu m$, and sand from 63 μ m to 2 mm (Li et al. [2012](#page-7-0)). It can be seen that the sediment is mainly composed of silt size particles and the mean diameter (i.e., d_{50} size) is 15.7 μ m (Table 2).

The concentration changes of suspended solids (TSS) and dissolved organic carbons (DOC) in the overlying water of different salinities with suspension time were analyzed in Fig. 2. The TSS concentrations generally decrease with the increase of salinity and suspension time, though a slight peak concentration is observed under the salinity of 20% (Fig. 2a). When the salinity increases from 0 to 5 %, more significant decreases (about 54.4–75 % for different suspension time) of the TSS concentrations are observed.

Unlike the TSS fractions, the DOC concentration shows an overall increase with salinity in Fig. 2b, although the percent content of TOC in raw sediment is only 0.47 %. However, peak concentrations are also observed under the salinity of 20 $\%$. And the concentration of DOC decreased with salinity rise when salinity is higher than 20 ‰, which agrees with previous research (Plavsic et al. [2012\)](#page-7-0).

Dissolved PAHs in the overlying water

Concentration changes of individual $(2+3$ rings, 4 rings, and 5+6 rings) and total PAHs (Σ PAH) dissolved in the overlying water of different salinities are shown in Fig. [3.](#page-4-0)

It can be inferred that the ranges of Σ PAH concentrations are 1094–2333,1576–3637, 3326–4104 and 1645–3174 ng L^{-1} for the salinities of 0, 5, 20 and 35 ‰, respectively. The highest concentration is observed under the salinity of 20 %. The lowest concentration is found in fresh water (i.e., the salinity of 0%).

In every sediment suspension in Fig. [3](#page-4-0), the concentration of Σ PAH increases firstly and then decreases. However, the peak concentrations and the occurrence time of the inflection points are different among the four sediment suspensions. In every sediment suspension experiment, two typical release periods are designed in this study, i.e., the sediment pre-standing period (denoted as the gray rectangles at 0 h in Fig. [3,](#page-4-0) i.e., static release period) and the following sediment suspension period (denoted as the blank rectangles at 0.5, 1.5, 3.5, 6.5 and 12 h in Fig. [3](#page-4-0), i.e.,

Fig. 2 Changes of the suspended solids (TSS, a) and dissolved organic carbons (DOC, b) concentrations in the overlying water of different salinities with suspension time

Fig. 3 Concentration changes of different rings and total 16 PAHs dissolved in the fresh water (a salinity 0%) and the salt water (**b** salinity 5 $\%$ ₀; **c** salinity 20 $\%$ ₀; **d** salinity 35 $\%$ ₀) at different

dynamic release). It can also be seen that the mass of the dissolved PAHs released at the former period are approximately 42–85 % of those detected in the later sediment suspension period.

As for the individual PAHs, the concentration change trends are coincident with that of Σ PAH. The 2, 3, 4-rings PAHs constitute the main composition of Σ PAH and play the key role in the concentration changes of Σ PAH due to their high contents in the raw sediments. Relatively, higher concentrations of 2, 3-rings PAHs are observed in the overlying water. The concentrations of 5, 6-rings PAHs are very low and even not detected mostly in Fig. 3.

Particle-bound PAHs in the overlying water

The concentration changes of particle-bound PAHs were also analyzed and described in Fig. [4](#page-5-0). The particle-bound Σ PAH concentrations in salt water are generally higher than those in the fresh water. The highest particle-bound Σ PAH concentrations are observed under the salinity of 20 %. Very slight increase or decrease trends of particlebound RPAH concentrations with suspension time could also be detected under different salinities.

suspension time; the *gray rectangles* represent the release concentration of PAHs at the static release period

(d)

(b)

For individual particle-bound PAH species, 2, 3-rings PAHs also constitute the main composition of Σ PAH. The concentrations of individual PAHs follow the order: 2, $3\text{-rings} > 4\text{-rings} > 5$, 6-rings (Fig. [4\)](#page-5-0).

Discussions

Impact of salinity on PAHs release from sediments

In sediment suspension, PAHs may desorb from sediment component (e.g., mineral composition and organic matter) and entrained into overlying water (Ghosh et al. [2003](#page-7-0)). This study discussed two typical PAH release patterns, i.e., the static release (at 0 h, after the 20 h pre-standing of sediments) and the following dynamic release (i.e., the hydrodynamic force-driven suspension of sediments). In the static release, no hydrodynamic disturbance existed, but high concentrations of dissolved PAHs were still observed (shown by the gray rectangles in Fig. 3). The main driving force should be the ''concentration difference'' (Ghosh et al. [2003](#page-7-0); Zheng et al. [2013\)](#page-8-0). Ionic competition on particle surface might also be taken into consideration, since

Fig. 4 Concentration changes of different rings and total 16 PAHs bounded on the suspended matters with suspension time in the fresh water (a salinity 0%) and the salt water (b salinity 5 %; c salinity

seawater contents high concentrations of anions and cations that exercise competition on particle surfaces and lead to removal on some weakly bound inorganic and organic compound from solid particles. As for the dynamic release, hydrodynamic force entrained sediments into the overlying water (Feng et al. [2007](#page-7-0)), and thus resulted in the changes of physicochemical characteristics of sediment such as organic carbon content and redox state which facilitated the quick increase of the dissolved PAHs concentrations at the initial stage (shown by the blank rectangles in Fig. [3](#page-4-0)) (Komada and Reimers [2001;](#page-7-0) Shen [2009](#page-7-0)). The following decrease means the existence of dynamic equilibrium driven by various factors (e.g., hydrodynamic disturbance, particle size composition and redox potential) in the dynamic release period (Zheng et al. [2013](#page-8-0)). Phase transfer of PAHs via re-adsorption also occurred simultaneously (Fotopoulou et al. [2011;](#page-7-0) Jeon et al. [2011](#page-7-0)), which could be inferred from the comparison of the later decrease of dissolved PAHs concentrations in Fig. [3](#page-4-0) with the slight increase of the particle-bound PAHs concentrations in Fig. 4.

20 %; d salinity 35 %); the insets represent the concentration multiple times of particle-bound PAHs to those in raw sediment

In static release, significantly higher concentrations of the dissolved PAHs were observed in saline water (i.e., gray rectangles in Fig. [3](#page-4-0)b–d) than that in fresh water (i.e., gray rectangles in Fig. [3a](#page-4-0)). Similar phenomenon also existed in the following dynamic release in Figs. [3](#page-4-0) and 4. Either the dissolved (Fig. [3b](#page-4-0)–d) or the particle-bound PAHs (Fig. 4b–d) have significantly higher concentrations in saline water than those in fresh water (Figs. [3](#page-4-0)a, 4a, respectively). The high concentration of particle-bound PAHs should be mainly ascribed to the re-adsorption of the dissolved PAHs onto suspended particles. This can be approved by the phenomenon that the concentration of particle-bound PAHs in the overlying water was significantly higher (over ten multiple times, as shown in the insets of Fig. 4) than that of the PAHs in the raw sediment. And higher multiple times were found in salt water.

However, salinity exerted different impact on the release behaviors of individual PAHs species. Higher multiple times were observed for the low-ring PAHs species, which was ascribed to the difference in the binding capacity of particles with different rings PAHs. Generally, high-ring

PAHs with big molecular weight are more strongly bound by particles, while low-ring PAHs has higher water solubility which led to less partitioning in particles (Chiou et al. [1998;](#page-7-0) Chen et al. [2004](#page-7-0)).

Different salinity also exhibited inequable impact on the release behaviors of PAHs. In the static and dynamic release, peak concentrations of the dissolved and particlebound PAHs were both observed under the salinity of 20 %. Similar distribution was also found for the changes of TSS (Fig. [2](#page-3-0)a) and DOC (Fig. [2](#page-3-0)b). The slight increase of DOC in seawater could be explained by the increase in salinity since seawater can encourage the release of organic carbon from sediment (Zhao et al. [2011](#page-8-0)). The abovementioned experimental results coincide well with the special phenomenon (i.e., Turbidity Maximum) in the area of the similar salinity (the average salinity of 22.82%) in Yangtze Estuary (Shen et al. [2008](#page-7-0)), where is the intersection place of fresh water and sea water (Wang et al. [2013](#page-7-0)).

Action mechanism of salinity

Up to date, no study has proved the direct reaction of saline ions with PAHs. The transportation of PAHs in surface water were closely related to the co-existing organic matters, mineral particles, and so on (Ahrens and Depree [2004](#page-7-0); Yang et al. [2008\)](#page-8-0). In this study, relative high correlation of the TSS and DOC fractions, especially the latter, existed with PAHs species. Stronger linear correlations (most $R > 0.9$) were observed between dissolved PAHs and DOC at the initial stage of sediment suspension. Most likely, salinity changes the physicochemical characteristics of water environment during sediment suspension (Brunk et al. [1997;](#page-7-0) You et al. [2010](#page-8-0)), thereby affects the affinity of PAHs to particles and their water solubility (Tremblay et al. [2005](#page-7-0)).

Generally, particles in raw sediments carry negative charge (Hunter and Liss [1979](#page-7-0)). Saline ions with positive charges encouraged the aggregation and deposition of the suspended matters via charge neutralization (Liu et al. [2007\)](#page-7-0), and thus resulted in the concentration decrease of TSS in Fig. [2](#page-3-0)a and particle-bound PAHs in Fig. [4b](#page-5-0) under the salinity of 5 %. Further increase of salinity from 5 to 20 ‰ changed the electrical property of suspended matters via the adsorption of saline ions, and thus inhibited their sedimentation. As a result, more and more organic matters and fine-particles accumulated in the overlying water and formed the active flocs which facilitated the accumulation of PAHs as particle-bound PAHs (Fig. [4](#page-5-0)c). Thus, peak concentrations of TSS and particle-bound PAHs were all observed under the salinity of 20 %. Meanwhile, as shown in Fig. [2b](#page-3-0), slight increase of DOC with salinity was also observed. The DOC fraction enhanced the water solubility of PAHs and competed with particles to sorb PAHs and thus keep PAHs in the dissolved phase (Tremblay et al.

[2005](#page-7-0)). Therefore, peak concentrations of DOC and dissolved PAHs were also observed under the salinity of 20 %. Similar phenomena were also reported in previous research study (Fotopoulou et al. [2011](#page-7-0)).

Role of salinity

However, higher salinity (i.e., 35 %, excessive charges introduced by salinity) facilitated the deposition of suspended materials again (Stumm [1992](#page-7-0)). The bulk mass flocs were more beneficial for the deposition of TSS, DOC and PAHs. Therefore, it can be concluded that different salinities may result in different phase redistributions of TSS, DOC, and PAHs in sediment suspension. The inflection point of salinity degree should be approximately 20 %. The discrepancy of the roles of salinity among many previous studies should take the salinity degree in their research into consideration (Fotopoulou et al. [2011](#page-7-0); Jeon et al. [2011\)](#page-7-0).

If the concentration of the dissolved PAHs at the static release period in fresh water (i.e., the gray rectangle in Fig. [3](#page-4-0)a) was taken as the base value or background value, it can be inferred from the other blank rectangles in Fig. [3a](#page-4-0) that the hydrodynamic disturbance resulted in onetime increase of dissolved PAHs concentrations. However, as inferred from the gray rectangles in Fig. [3](#page-4-0)b–d (i.e., the static release period), salinity can cause 1.5–3.5 multiple times increase of the mean concentrations of dissolved PAHs. From this aspect, salinity exerted more significant impact than hydrodynamic disturbance. Certainly, the higher mean concentrations of particle-bound PAHs (0.1–5) multiple times higher) in salt water in Fig. [4](#page-5-0)b–d than that in Fig. [4a](#page-5-0) also proved the conclusion. The dissolved PAHs concentrations at the dynamic release period in Fig. [3](#page-4-0)b–d (i.e., the blank rectangles) were observed to be 1.5–4 multiple times of the background value in Fig. [3](#page-4-0)a, which should be ascribed to the coupled effect of the three factors (e.g., salinity, hydrodynamic disturbance, sediment component). Moreover, it can also be easily inferred that the coupled effect was a typical additive effect and salinity facilitates the release of PAHs from sediment into overlying water.

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Compliance with ethical standards

Conflict of interest No conflict of interest.

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