



# Heavy metal partitioning of suspended particulate matter–water and sediment–water in the Yangtze Estuary



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## HIGHLIGHTS

- Temporo-spatial phase partition of heavy metals in the Yangtze Estuary was studied.
- Higher salinity resulted in larger partition coefficients ( $K_p$ ) in the north branch.
- No specific phase transfer of heavy metals existed during the flood and ebb tides.
- DOC and particle size had obvious opposite correlations with  $K_p$  for most metals.
- Metal speciation and  $K_p$  correlations can reflect phase transfer ways of heavy metals.

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## ABSTRACT

The partitioning of ten heavy metals (As, Cd, Co, Cr, Cu, Hg, Ni, Pb, Sb, and Zn) between the water, suspended particulate matter (SPM), and sediments in seven channel sections during three hydrologic seasons in the Yangtze Estuary was comprehensively investigated. Special attention was paid to the role of tides, influential factors (concentrations of SPM and dissolved organic carbon, and particle size), and heavy metal speciation. The SPM–water and sediment–water partition coefficients ( $K_p$ ) of the heavy metals exhibited similar changes along the channel sections, though the former were larger throughout the estuary. Because of the higher salinity, the  $K_p$  values of most of the metals were higher in the north branch than in the south branch. The  $K_p$  values of Cd, Co, and As generally decreased from the wet season to the dry season. Both the diagonal line method and paired samples *t*-test showed that no specific phase transfer of heavy metals existed during the flood and ebb tides, but the sediment–water  $K_p$  was more concentrated for the diagonal line method, owing to the relatively smaller tidal influences on the sediment. The partition coefficients (especially the  $K_p$  for SPM–water) had negative correlations with the dissolved organic carbon (DOC) but positive correlations were noted with the particle size for most of the heavy metals in sediment. Two types of significant correlations were observed between  $K_p$  and metal speciation (i.e., exchangeable, carbonate, reducible, organic, and residual fractions), which can be used to identify the dominant phase-partition mechanisms (e.g., adsorption or desorption) of heavy metals.

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## 1. Introduction

One of the major pathways through which pollutants are transported to oceans is riverine input (Zhou et al., 2003; Duan et al., 2014). During this process, the distribution of pollutants

between dissolved and suspended particulate matter (SPM) and sediment phases will be modified in estuaries (Paucot and Wollast, 1997). An estuary is a complex system governed by tidal action and river flow. The mixing of salt water and freshwater constitutes a dynamic environment (Hierro et al., 2014). The behavior of pollutants in these areas is significantly influenced by flocculation, organic and inorganic complexation, adsorption, sediment resuspension, etc. (Fang and Wang, 2006; Duarte et al., 2014). When the environmental conditions change, pollutants may be desorbed and suspended from the sediment and cause secondary pollution (Bai

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et al., 2011; Wang et al., 2016), which can result in more complex phase partition characteristics of the pollutants in estuaries. The inconstant phase transfer in estuaries has drawn increasing attention of academicians.

In the past decade, partitions of heavy metals in estuaries have been widely investigated (Duarte et al., 2014; Hierro et al., 2014). Heavy metal pollution in estuaries has caused serious environmental problems worldwide because of its toxicity, wide range of sources, non-biodegradable properties, and accumulation behaviors (Li et al., 2013). However, no study has comprehensively analyzed the multiphase partition of heavy metals for SPM–water and sediment–water in the Yangtze Estuary. The Yangtze Estuary is one of the largest estuaries in the world; its mouth is approximately 90 km wide (Yan et al., 2013). Located in an area with one of the highest population densities and fastest developing economies in China, the Yangtze Estuary has suffered heavy metal contamination (Feng et al., 2004); thus, the partitioning of heavy metals in this estuary requires further investigation (Wang et al., 2014).

The partition coefficient ( $K_p$ ) between sediment and water or between SPM and water has been widely used to analyze the migration and transformation of pollutants in field or lab tests (Lu and Allen, 2001; Zhao et al., 2013; Montuori et al., 2015). In these studies, the partition coefficient was often combined with other influential factors (such as pH, temperature, and organic matter) to predict the transfer trend of pollutants. Among these factors, salinity and SPM concentration had the greatest effects and various influencing characteristics were determined for these factors (Fang and Lin, 2002; Duarte et al., 2014). Moreover, other chemical and influential factors (such as dissolved oxygen and pH) have been studied (Paucot and Wollast, 1997; Hierro et al., 2014). However, because of the limitations associated with sample collection, the roles of changing hydrologic seasons and tides have been rarely studied. Additionally, it was reported that heavy metal speciation played an important role in the partition behaviors but this topic has also been rarely studied.

Therefore, in this study, the  $K_p$  values of SPM–water and sediment–water for ten heavy metals (As, Cd, Co, Cr, Cu, Hg, Ni, Pb, Sb, and Zn) at 16 sampling sites in 7 channel sections of the Yangtze Estuary were comprehensively investigated during flood and ebb tides and during the wet, normal, and dry seasons. The effects of hydrologic seasons and tides on the phase transfer of heavy metals were also investigated. Furthermore, correlation analysis was used to determine the roles of certain influential factors (SPM concentration, dissolved organic carbon (DOC), and particle size) and heavy metal speciation. The main purpose of the study was to describe the temporo-spatial changes in the phase-transfer behaviors of the above-mentioned ten heavy metals for the Yangtze Estuary.

## 2. Materials and methods

### 2.1. Study area and sample collection

The 16 sampling sites, the 7 channel sections (X stands for Xuliujing; N1–N3 stand for stations upstream, midstream, and downstream of the north branch; and S1–S3 denote stations upstream, midstream, and downstream of the south branch), and the location of the Yangtze Estuary in China are shown in Fig. 1. The samples of water, SPM, and sediment were collected at the 16 sampling sites in July 2013 (wet season), October 2013 (normal season) and February 2014 (dry season). The water samples were collected from the surface and bottom layer and were mixed in borosilicate glass bottles while SPM deposits were collected from several barrels of water (about 100 L per sample) by lockable barrel (25L) from different depths and the sediment was collected using

homemade collectors. Except for the sediment sampling, all the work was conducted during the flood and ebb tides. Prior to the analysis, the water samples were filtered through a pretreatment fiberglass membrane (pore size 0.45  $\mu\text{m}$ ) and stored at 4 °C. The SPM and sediment samples were naturally air-dried, and stored in –20 °C before analysis.

### 2.2. Measurements

The treatment of the samples and the measurement of the heavy metals were conducted at the Institute of Geophysical and Geochemical Exploration (IGGE) at the Chinese Academy of Geological Sciences, which is certified by the China National Accreditation Board for Laboratories. The metal sequential extraction procedures for the SPM and sediment samples followed the classical five-step method proposed by Tessier et al. (1979). The method was briefly described as follows:

- Step 1 Exchangeable fraction (EXC) Two grams soil was extracted with 20 mL of 1.0 mol/L  $\text{MgCl}_2$  in a Teflon centrifuge tube for 2 h at 25 °C and pH = 7 with continuous agitation, then centrifuged for 20 min at 4000 rpm/min. The wash water was added to the supernatant. The mixture was filtered through a 0.45  $\mu\text{m}$  membrane and deionized water was added to make a volume of 25 mL for measurement. The residue was retained for the next steps. The same centrifugation–decantation–washing procedure was also used after each of the following steps.
- Step 2 Carbonate bound fraction (CARB) Residue from exchangeable fraction was extracted with 20 mL of 1.0 mol/L NaOAc (pH = 5) for 5 h at 25 °C with continuous agitation, then centrifuged for 20 min at 4000 rpm/min.
- Step 3 Fe–Mn oxide fraction (Fe–Mn) Residue from the carbonate fraction was extracted with 20 mL of 0.04 mol/L  $\text{NH}_2\text{OH}/\text{HCl}$  in 25% acetic acid for 5 h at 25 °C with continuous agitation, and then centrifuged for 20 min at 4000 rpm/min.
- Step 4 Organic matter bound fraction (OM) Residue from the Fe–Mn oxide fraction was extracted with 3 mL of 30%  $\text{H}_2\text{O}_2$  and 2 mL of 0.02 mol/L  $\text{HNO}_3$  for 2 h at 85 °C and pH = 2 with occasional agitation; additional 3 mL  $\text{H}_2\text{O}_2$  was added again with occasional agitation for 3 h; and 10 mL of 3.2 mol/L  $\text{NH}_4\text{OAc}$  in 20%  $\text{HNO}_3$  was added for 30 min at 25 °C with continuous agitation, then centrifuged for 20 min at 4000 rpm/min.
- Step 5 Residual fraction (RES) Residues from the organic fraction were together digested with 3 mL  $\text{HNO}_3$ , 1 mL HF and 1 mL  $\text{HClO}_4$  for 5 h, and then added with another 1 mL  $\text{HNO}_3$ . Digestion was conducted in a water bath at 80 °C for 16 h”

The total metal contents were determined using the sums of the metal contents for every extraction procedure. The concentrations of As, Hg, and Sb in the water samples and the filtrates of the sequential extraction procedure were measured via hydride generation atomic fluorescence spectrometry (HG-AFS, IGGE), whereas the concentrations of Cd, Co, Cr, Cu, Ni, Pb, and Zn were measured using high-resolution inductively coupled plasma mass spectrometry (HR-ICP-MS, Thermo).

The water flow velocities and SPM concentrations were measured with an acoustic Doppler current profiler (ADCP, SonTek, River Surveyor S5, USA). The total organic carbon (TOC) in the water samples was measured via a total organic carbon analyzer (Elementar, Germany). The particle sizes of the SPM and sediment were analyzed using a laser particle size analyzer (0.25–1400  $\mu\text{m}$ ) (Microtrac S3500, USA).

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