

## Contamination and ecological risks of toxic metals in the Hai River, China

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### ABSTRACT

Contamination of trace metals in urban rivers is a global problem. The objective of this study was to investigate the contamination and ecological risks of trace metals in the Hai River, which receives substantial effluents from the Beijing-Tianjin region. A total of 43 surface water and sediment samples were collected and analyzed for physicochemical properties and toxic elements. The concentrations of Cd, Cr, Ni, Pb, Cu and Zn in the river water met the Chinese environmental quality grade I standards for surface water. The average total concentrations of Zn, Cr, V, Cu, Pb, Ni, Co, Sc and Cd in the sediments were 144.2, 77.5, 70.1, 46.1, 40.1, 29.6, 12.4, 9.9 and 0.26 mg/kg, respectively. The enrichment of Cd, Cr, Cu, Pb and Zn in the sediments was influenced by anthropogenic activity, as indicated by the enrichment factor (EF) and multivariate analysis. The concentrations of Co and Cr in the sediments were predominantly in residual fractions, while relatively higher amounts of Cd, Pb, Zn and Cu were in non-residual fractions. There was a significant correlation between concentrations of Cd, Co, Cr, Cu, Pb and Zn in non-residual fractions and their corresponding EF values ( $P < 0.01$ ). This result further demonstrated that anthropogenic inputs were an important contributor to metal enrichment. The high bioavailability of Cd and poor bioavailability of Co and Cr in the sediments were demonstrated by the single extraction procedure. The significant correlation between concentrations of Cd, Cu, Pb and Zn in non-residual fractions and their concentrations in EDTA-extractable fractions was established ( $P < 0.01$ ). This result indicates that single-step EDTA extraction may represent a useful procedure for assessing the bioavailability of Cd, Cu, Pb and Zn in riverine sediments. The ecological risks of the metals studied in the Hai River sediments were at a low level, dominated by the risks imposed by Cd.

### 1. Introduction

Trace metal contamination of aquatic systems has become one of the most severe environmental problems in both developed and developing countries (Schwarzenbach et al., 2006). Increased urbanization is associated with physicochemical changes in rivers, such as the elevated concentrations of trace metals (Peltier et al., 2008; Wong et al., 2006). Trace metals transported to river systems are mainly incorporated into riverine sediments (Gibbs, 1973). Therefore, riverine sediments have been widely used as environmental indicators of the present contamination characteristics of trace metals (Chen et al., 2016; Lin et al., 2008; Singh et al., 2002; Wang et al., 2011; Wu et al., 2011).

Elevated concentrations of trace metals in riverine sediments have been reported in rivers worldwide (Lin et al., 2013a; Ouattara et al., 2014; Peltier et al., 2008; Wu et al., 2016). Multivariate analysis is widely used to assess the interrelations and sources of metals (Lin et al., 2013b; Tang et al., 2013; Zhang et al., 2017). In general, the trace metals in sediments are present in different chemical forms, with each

possessing different chemical stabilities and bioavailabilities (Peijnenburg et al., 2007; Tessier et al., 1979; Usero et al., 1998). Lithogenic and pedogenic trace metals often occur in more stable fractions, while anthropogenic trace metals are usually associated with more liable fractions (Kabata-Pendias and Mukherjee, 2007; Li et al., 2015). The partitioning of trace metals in various sediment fractions is influenced by many factors, such as organic matter (Morel and Hering, 1993), carbonate minerals (Han and Banin, 1999), iron oxides (Wang et al., 2011) and metal concentrations (Li et al., 2015). Sequential extraction is a procedure widely used to assess the chemical speciation of trace metals in sediments (Peijnenburg et al., 2007; Tessier et al., 1979). In addition, single extraction is a procedure widely applied to assess the bioavailability of trace metals in sediments (Brun et al., 2001; Garrabrants and Kosson, 2000; Luo et al., 2012; McGrath, 1996; Miličević et al., 2017; Peijnenburg et al., 2007; Rao et al., 2008).

The Hai River Catchment is one of the most developed and densely populated regions in China (Tang et al., 2013; Zhang et al., 2017). Water bodies in the Hai River receive a large fraction of the Beijing and

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Tianjin Municipalities' sewage treatment plant (STP) effluents (Gao and Chen, 2012; Pernet-Coudrier et al., 2012). It is reported that Beijing discharges an annual average of  $29.5 \text{ m}^3 \text{ s}^{-1}$  raw or treated wastewater into the Hai River system (Zhang and Brown, 2005). In the past 40 years, nutrient concentrations in the Hai River have massively increased, mostly because of the discharge of untreated sewage from megacities (Pernet-Coudrier et al., 2012). In addition, Beijing and Tianjin have one of the most intensive livestock and poultry production industries (Li et al., 2010; Xiong et al., 2010). Several studies investigated the contamination of heavy metals in the sediments of the Tianjin section of the Hai River (Liu et al., 2006; Yuan et al., 2004). However, the chemical forms, bioavailability, contamination, and ecological risks of toxic metals in the Hai River need to be further investigated and clarified.

Discharge of municipal and animal production sewage effluent into the Hai River may lead to the accumulation of toxic metals in the river sediment. Further, anthropogenic input of toxic metals into the river may change their original partition in sediment and pose potential risks to aquatic ecosystems and human health due to their accumulation in food webs. Therefore, the objectives of this study were to investigate the contamination of toxic metals in both surface water and sediments, to identify the geochemical fractions of these metals in the sediment, and to assess the metals' potential ecological risks to the Hai River.

## 2. Materials and methods

### 2.1. Study area

The Hai River ( $39^{\circ}4' - 40^{\circ}8' \text{ N}$ ,  $116^{\circ}0' - 118^{\circ}0' \text{ E}$ ) is located in the northern part of China (Fig. 1). This river has a length of 240 km, flowing from Beijing Municipality to Tianjin Municipality. The river is characterized by a semi-arid, semi-humid continental monsoon climate, with a mean annual precipitation of approximately 550 mm (Guo and Shen, 2015). Heavy industrial development and rapid urbanization have caused significant pollution in aquatic systems in this region (Tang et al., 2013; Zhang et al., 2017).

### 2.2. Sample collection and analysis

A total of 43 water and sediment samples were collected in the Hai River in November 2016 (Fig. 1). The water samples were filtered

through  $0.45\text{-}\mu\text{m}$  Teflon filters, acidified with concentrated nitric acid to a  $\text{pH} < 2$ , and stored in polystyrene bottles. The sediment samples were placed in the acid-rinsed polyethylene bags and later transported to the laboratory, where they were freeze-dried, ground, homogenized, passed through a 100-mesh sieve, and stored in polyethylene bottles.

For total metal analysis, the sediments were digested with  $\text{HNO}_3\text{-HF-HClO}_4$  (Lin et al., 2008). The concentrations of Cr, V, Mn, Ti, Al, Fe, Ca, Mg, Na and K in the extracts were measured with an inductively coupled plasma atomic emission spectrometer (ICP-AES) (IRIS Intrepid II, Thermo Electron), and the concentrations of Cd, Co, Cu, Ni, Pb, Sc, and Zn were measured with an inductively coupled plasma mass spectrometer (ICP-MS) (X Series II, Thermo Electron). The accuracy of these sediment analysis methods was assessed according to the analyses of the elements in the certified reference materials GBW07450 (National Institute of Metrology, Beijing, China). The analytical errors were  $-2.5$  to  $-1.6\%$  for Al,  $2.7\text{--}5.6\%$  for Ca,  $-3.4\text{--}2.5\%$  for Cd,  $-2.9\text{--}2.6\%$  for Co,  $-5.1\text{--}4.8\%$  for Cu,  $-6.3$  to  $-4.0\%$  for Fe,  $-8.3$  to  $-7.9\%$  for K,  $-3.3$  to  $-2.5\%$  for Mg,  $-1.2\text{--}1.7\%$  for Mn,  $-1.2\text{--}0.9\%$  for Na,  $-1.9\text{--}1.6\%$  for Ni,  $-2.5\text{--}1.9\%$  for Pb,  $-1.6\text{--}1.2\%$  for Sc,  $-3.9$  to  $-3.0\%$  for Ti,  $-9.2\text{--}1.2\%$  for V and  $-2.7$  to  $-0.9\%$  for Zn ( $n = 3$ ).

The Tessier method of sequential extraction was used to analyze the chemical properties of toxic metals in the sediments (Tessier et al., 1979). The following fractions were extracted from 1 g of sediment: F1, exchangeable fraction (8 mL 1 M  $\text{MgCl}_2$ , shaken for 1 h); F2, bound to carbonates (8 mL 1 M  $\text{CH}_3\text{COONa}$ , shaken for 6 h); F3, bound to iron and manganese oxides (20 mL  $\text{NH}_2\text{OH}\cdot\text{HCl}$ , shaken at  $96^\circ\text{C}$  for 6 h); F4, bound to organic matter (3 mL  $0.02\text{HNO}_3$  + 5 mL  $30\% \text{H}_2\text{O}_2$ , shaken at  $85^\circ\text{C}$  for 3 h; 3 mL  $30\% \text{H}_2\text{O}_2$ , shaken at  $85^\circ\text{C}$  for 3 h; 5 mL  $\text{CH}_3\text{COONH}_4$ , shaken for 0.5 h); and F5, residual fraction (total digestion of the residue by the mixture of concentrated  $\text{HF-HClO}_4\text{-HNO}_3$ ). The concentrations of Cd, Co, Cr, Cu, Pb, and Zn in each extract were measured using ICP-MS. The recoveries of the sum of each fraction compared to the total concentration were in the range of  $96.8\text{--}109.1\%$  for Cd,  $101.4\text{--}118.2\%$  for Co,  $89.7\text{--}104.1\%$  for Cr,  $87.3\text{--}102.7\%$  for Cu,  $85.9\text{--}112.6\%$  for Pb, and  $88.8\text{--}106.8\%$  for Zn ( $n = 25$ ).

The bioavailability of metals in the sediments was assessed using the single extraction method (Peijnenburg et al., 2007). The EDTA extraction procedure was applied to assess the potential bioavailable metals by mixing 1 g of sediment with 10 mL  $0.05 \text{ M EDTA}$  and shaking it for 1 h at  $25^\circ\text{C}$  (Garrabrants and Kosson, 2000; Milićević et al., 2017). The concentrations of Cd, Co, Cr, Cu, Pb, and Zn in the extracts were

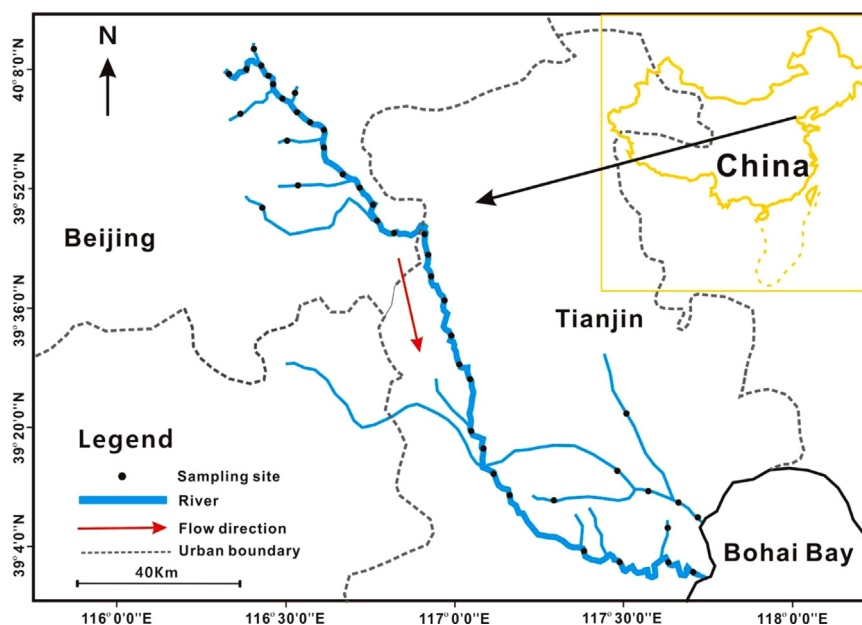


Fig. 1. Locations of sampling sites in the Hai River, China.

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