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Metals in size-fractionated core sediments of Jiaozhou Bay, China: Records of recent anthropogenic activities and risk assessments



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ABSTRACT

Total contents and chemical speciation of Co, Ni, Cu, Ga, Mo, Cd, In, Sn, Sb, V, W, Tl, Bi and U in size-fractionated (< 32, 32–63 and > 63 μm) core sediments from Jiaozhou Bay were investigated to reveal their responses to anthropogenic activities. Metal contents showed a decreasing trend with increasing grain sizes. However, the loadings of metal fraction on < 32, 32–63 and > 63 μm grain sizes were 16%, 47% and 37%, respectively. Anthropogenic fluxes and enrichment factors of metals in > 63 μm fraction were closely linked to anthropogenic activities, with an obvious increase in upper 27 cm (1998–2015) and a slight decrease in 2009 year. Metals (especially for Cd, Co, Cu and Ni) in > 63 μm fraction were more easily released, with the highest percentage of acid soluble form and lowest residual form. Thus, the size fraction of > 63 μm cannot be ignored.

Metals in sediments are widely concerned because of their toxicity, persistence and non-degradability in the environment (Duan et al., 2010; Irabien and Velasco, 1999; Yang et al., 2012). Cd, Cu, Ni, Tl and their compounds are classified as priority pollutants by the Environmental Protection Agency of China. Sb is in the list of priority pollutants in the United States (Filella et al., 2002). Co, Sn, V and Mo are essential metals for organisms, which are beneficial to normal cell growth when in trace amounts but are toxic when metal contents increase to a high level (Colina et al., 2005). Bi and In are usually called rare metals, with abundances of 0.0085 and 0.250 ppm in earth crust (Taylor and McLennan, 1995). Recently, the rare elements have applications in various alloys, smelting and electronic components, and thus become important pollutants in environment and sensitive indicators for environmental changes due to their low abundance (Kuwae et al., 2013; Tessier et al., 2014; Wei et al., 2011). It is recognized that the biogeochemical behavior and bioavailability of metals strongly depend on their specific chemical forms (Audry et al., 2006; Bacon and Davidson, 2008; Giancoli Barreto et al., 2004). Sequential extraction is a widely used technique for understanding chemical distribution of metals in solid phases (Gleyzes et al., 2002). Among different sequential extraction procedures, the enhanced BCR sequential extraction protocol has been widely adopted (Claudia et al., 2013; Ma et al., 2016; Gleyzes et al., 2002).

Grain size of sediments is one of the most important factors controlling the distribution and toxicity of metals in sediments, which is called “grain size effect” (Windom et al., 1989; Yuan et al., 2011). The choice of grain size fraction to be analyzed was different in different studies. Some researches just selected sediment fraction of < 63 μm to investigate the contamination and biological availability of metals (Borovec, 2002; Chatterjee et al., 2007; Stone and Droppo, 1996). However, anthropogenic metals may also be introduced into marine environment via the large particles (Ajmone-Marsan et al., 2008; Brook and Moore, 1988; Tam and Wong, 2000). In addition, the contents and ecological risks of metals in sediments are not always associated with the finest particles considering the weight proportion of different size fractions (Brook and Moore, 1988). Up to now, limited data are available to compare the environmental information extracted from different size fractions of sediments (Sutherland, 2000).

Jiaozhou Bay is a typical semi-enclosed bay surround by the city of Qingdao, one of the leading cities in Shandong Peninsula Blue Economic Core Area. In the previous studies, only the total contents and distributions of heavy metals in surface or core sediments of Jiaozhou Bay were reported (Dai et al., 2007; Wang et al., 2007). However, the study on chemical speciation of metals and its ecological risks in different size fractions of sediments from Jiaozhou Bay is absent.

In this study, a sediment core C4 in Jiaozhou Bay was collected and

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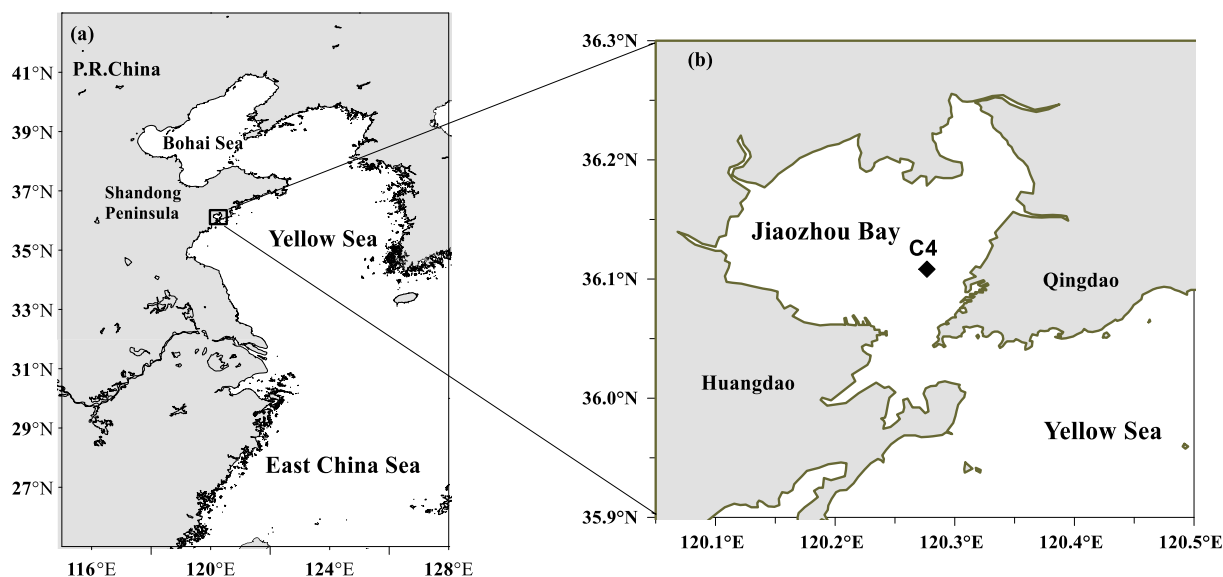


Fig. 1. Location of Jiaozhou Bay (a) and the site of sediment core C4 (b).

the vertical distributions and chemical speciation of Co, Ni, Cu, Ga, Mo, Cd, In, Sn, Sb, V, W, Tl, Bi and U in three size fractions (< 32, 32–63 and > 63 μm) were investigated. Our objectives were to: 1) clarify whether environmental changes and anthropogenic activities could be recorded and reflected by the studied metals in different size fractions of sediments; 2) obtain information about the role of particle sizes and their composition in the control of metal contents and their chemical speciation in sediments; and 3) estimate the ecological risks of metals in different size fractions of sediments.

The sediment core C4 (36°6'29.76"N, 120°16'36.3"E) (Fig. 1) was obtained in July 2015 from Jiaozhou Bay, China. After collected with a PVC coring pipe (10 cm inner diameter), the core (the length of 81 cm) was immediately transferred to the laboratory and stored in a refrigerator at 4 °C for < 24 h before sectioning. Then, the sediment core was cut longitudinally and sliced in 2 cm long sections. All the samples were stored in polyethylene for further particle-size separation.

The sediments were partitioned into three size fractions: < 32 μm , 32–63 μm and > 63 μm . For the separation, aliquots of samples were separated on a nylon sieve for the > 63 μm fraction of sediments by standard wet sieving with filtered sea water. Sediments of the < 63 μm fraction in the filter sea water yielded size fractions of < 32 μm and 32–63 μm using precipitation method according to the Stokes Law (GAQSIQ, 2007). After separation, all the fractions were dried and weighed to calculate the percentage weights of different size fractions.

The modified BCR sequential extraction procedure (Yu et al., 2014) was carried out to determine the chemical speciation of metals in three size fractions (i.e., < 32, 32–63 and > 63 μm). Briefly, about 0.20 g of dried sample was loaded into a 50 ml centrifuge tube. Acid soluble form (F1) was extracted with 20 ml of 0.11 mol·l⁻¹ acetic acid at room temperature for 16 h with continuous agitation. The residue from the first extraction step was subjected to further leaching with 20 ml of 0.5 mol·l⁻¹ hydroxylamine hydrochloride adjusted to pH 2 with HNO₃, and mixture was agitated continuously at room temperature for 16 h to receive reducible form (F2). The residue from the second extraction step was treated twice with 5 ml of 8.8 mol·l⁻¹ hydrogen peroxide adjusted to pH 2 with HNO₃, and mixture was agitated intermittently at room temperature for 1 h and then agitated continuously at 80 °C for 1 h. After cooling down, 25 ml of 1.0 mol·l⁻¹ ammonium acetate, adjusted to pH 2 with HNO₃, was added and the mixture was agitated for 16 h at room temperature for extracting oxidizable form (F3). The residue from the third extraction step was transferred into a closed Teflon digestion vessel, and digested with a mixture of HNO₃-HF-HClO₄

(5:3:2) at 150 °C for 48 h to obtain the residual form (F4). Metal contents of Co, Ni, Cu, Ga, Mo, Cd, In, Sn, Sb, V, W, Tl, Bi, U and Li in digestion solution were determined by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS; SCIEX Elan 5000). Major elements contents of Ti, P, Sr, Ba, Fe and Mn were determined by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES; PerkinElmer Optima 3000). Total organic matter (TOC) contents in sediments were determined using dichromate and concentrated sulfuric acid oxidation method (Nelson and Sommers, 1975).

The total contents of metals were obtained by the sum of F1, F2, F3 and F4. The determination reliability of the total metal contents was tested using standard reference materials, with recovery ranges of 80–104% and 87–120% for GBW07309 and GBW07429, respectively (Table 1). Plastic material was utilized to limit the possible metal pollution thought the sample collection, separation and chemical extraction procedure. All materials were cleaned by soaking with 10% HNO₃, and then they were washed three times with Milli-Q water, and were dried under a closed atmosphere.

The sediment core was dated using ²¹⁰Pb radiometric technology. After three weeks of storage in sealed polythene tubes, ²¹⁰Pb and ²²⁶Ra were determined by the high-resolution gamma-ray spectrometry with a high-purity germanium (HPGe) coaxial detector (Canberra Industries

Table 1

Analytical results of certified and measured values (mg·kg⁻¹) of metals in standard reference materials of GBW07309 and GBW07429.

Element	GBW07309		GBW07429	
	Measured values	Certified values	Measured values	Certified values
Co	12.7	14.4 ± 1.2	16.5	17.6 ± 0.7
Ni	32.4	32 ± 2	39.18	41 ± 1
Cu	30.3	32 ± 2	34	37 ± 2
Ga	11.3	14.0 ± 0.6	18.2	20.5 ± 1
Mo	0.70	0.64 ± 0.11	1.1	0.92 ± 0.07
Cd	0.27	0.26 ± 0.04	0.24	0.21 ± 0.02
In	0.050	0.056 ± 0.09	0.13	0.145 ± 0.021
Sn	2.4	2.6 ± 0.4	4.3	4.5 ± 0.5
Sb	0.83	0.81 ± 0.15	2.0	1.9 ± 0.2
V	93	97 ± 6	104	119 ± 3
W	1.8	1.8 ± 0.2	2.9	2.8 ± 0.2
Tl	0.42	0.49 ± 0.08	–	–
Bi	0.35	0.42 ± 0.04	1.10	1.16 ± 0.06
U	2.3	2.6 ± 0.4	2.8	3 ± 0.2

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