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Mercury distribution, speciation and bioavailability in sediments from the Pearl River Estuary, Southern China

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ABSTRACT

Surface sediments and sediment cores collected from the Pearl River Estuary (PRE) were analyzed for total mercury (THg) concentrations and speciation using a sequential extraction method. The mobility of Hg in sediments was also assessed using a series of single extraction methods. The surface sediments from the PRE showed slightly elevated levels of Hg, with concentrations ranging from 109 to 453 ng/g. The vertical profile of THg in sediment cores indicated an accelerated input of Hg over the past decades. The organo-chelated and strong-complexed Hg species were the dominant Hg species in the sediments, while the more mobile phases of Hg made up less than 0.5% of THg. Less than 10% of the Hg in the sediments was extracted by single extraction, depending on the extractant employed. Significant relationships were found between the total organic carbon and THg, geochemical speciation, and extractability, indicating the important role of organic matter in controlling the distribution, mobility, and bioavailability of Hg in sediments.

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Mercury (Hg) is a widely distributed pollutant and has been listed as a high priority pollutant by many international agencies because of its persistence in environments and high toxicity to organisms (Jiang et al., 2006). The fate of Hg in environments, especially those distributed in sediments, has received great attention as sediments act both as a major sink for or a potential source of Hg in aquatic environments (Green-Ruiz et al., 2005; Wang et al., 2009). Investigating the concentration and distribution of metals, including Hg, in sediments is one of the most effective ways to evaluate the degree of environmental marine pollution because sediments can be a good indicator of the anthropogenic input of metals and provide historical input records of metals (Pan et al., 2011). Inorganic Hg [Hg(II)] is the dominant species of Hg in sediments. It may be methylated into methyl mercury (MeHg), which is more toxic and bioaccumulative than Hg (II) (Shi et al., 2005; Zhong and Wang, 2008a). Inorganic mercury and methyl mercury in sediments may be bioaccumulated in aquatic organisms and transferred along food chains, eventually posing a threat to humans (Pan and Wang, 2004, 2011).

Metal contamination in estuaries has received great attention because estuaries provide unique habitats for a diversity of species (Benoit et al., 1998). The Pearl River Estuary (PRE), which is the largest estuary in Southern China, is created by the outflows of freshwater to the South China Sea. Over the past few decades, massive economic growth and urban development in the Pearl River Delta region have led to a substantial accumulation of trace metals in the surface sediment and aquatic organisms of the PRE, leading to a serious pollution problem. A few studies on metal contamination in this area have been carried out over the past years (Ip et al., 2004, 2007; Yu et al., 2010). However, the Hg contamination in the PRE has not been well studied, nor has the Hg contamination in

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other coastal areas in China (Wang et al., 2009). A previous study indicated that the concentrations of Hg in surface sediments collected from the PRE and its surrounding coastal area in 2002 ranged from 1.5 to 201 ng/g, with an average of 54.4 ng/g (Shi et al., 2010). However, the severity of Hg contamination in surface sediments appears to be increasing in recent years. An average Hg concentration of 168 ng/g, with values ranging from 20.8 to 369 ng/g, was reported by Shi et al. (2007) who collected surface sediments from the PRE during 2004–2005. Moreover, recent studies reported that the sedimentation rate in the PRE has obviously increased over the last few decades (Ip et al., 2007; Liu et al., 2010; Zhou et al., 2004). A high sedimentation rate can cause a change in the geochemical characteristics of the surface sediments on an annual scale (Liu et al., 2011; Zhou et al., 2004).

Although the total mercury (THg) concentrations in sediments reflect the overall pollution levels, it is insufficient to estimate the environmental impact based simply on the total Hg concentration. Hg is present in different forms that can be bound to various matrix phases. These matrix phases have different mobilities, bioavailabilities, and potential toxicities (Issaro et al., 2009). For example, water-soluble Hg or a species that is loosely adsorbed onto particles is much more mobile in sediments than is mercury sulfide (HgS), which is stable in ore bodies for geologic time spans (Zhong and Wang, 2006a). Thus THg is a poor indicator of the toxicological and environmental hazards in sediments. Except for several reports on THg distribution in the PRE and its coastal areas (Shi et al., 2007, 2010), little information is available on the biogeochemistry of Hg which controls its bioavailability. While Hg concentrations in the PRE have been studied, much less is known about the partitioning of mercury in this area. The geochemical partitioning of Hg could reveal information about the Hg origin and form of occurrence, as well as the proportion of Hg that is chemically mobile and bioavailable (Boszke et al., 2006; Botte et al., 2010; Devesa-Rey et al., 2010).

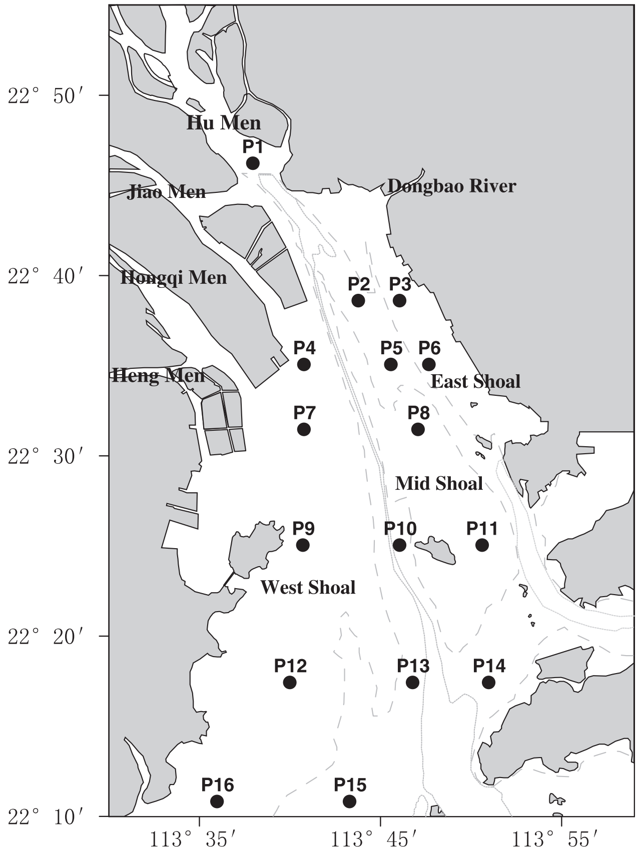

Fig. 1. Sampling sites in the Pearl River Estuary.

To evaluate the overall Hg contamination in the PRE, we studied the spatial and vertical distributions of Hg to assess the historical pollution trends in this area. Surface sediments were also subjected to mercury speciation studies to investigate the biogeochemical characteristics of Hg. Both sequential extraction and single extraction schemes were finally employed to evaluate the bioavailability of Hg in the PRE sediments.

Sixteen surface sediment samples and five sediment cores were collected from different sites in August 2010 in the Pearl River Estuary (PRE) (Fig. 1). Sediment cores, sampled with a gravity corer with an automatic clutch and reverse catcher, were sliced into 2 cm thick slices. There were some perturbations on the sediment surface layers sampled by gravity corer. All samples were placed in clean polyethylene plastic bags and kept in an iced box before being transported back to the laboratory. Large stones and detritus were first removed from the samples which were then freeze-dried. The sediments were ground in a mortar and then passed through a 63 μm mesh so that they would have consistent physical properties.

In order to assess the speciation of mercury in the samples, the five-step sequential extraction developed by Bloom et al. (2003), as summarized in Table 1, was used. The procedure started with the extraction of 0.2 g of dried sediment samples in 50 ml trace metal clean glass vials with Teflon-lined caps. Following each extraction with 20 ml of an extractant, the mixtures were centrifuged at 3000 rpm for 20 min at room temperature. The supernatant was decanted into another clean tube and preserved for further analysis. The residue was re-suspended and washed with the same extractant three times, after which all supernatants were combined as one sample. The supernatant resulting from the first four steps was stored in rigorously cleaned glass bottles with Teflon lining. A BrCl solution, which has been found to be an excellent oxidant and preservative for THg in water samples in EPA method 1631 (Shi et al., 2005), was added until a light yellow color appeared. The residue (F5) was digested with *aqua regia* as indicated below. Because there was no natural reference material to allow determination of the method's accuracy, the method was verified for all samples by calculating the sum of the ratios of Hg distributed in all phases to THg, or ΣHg/THg. These ratios ranged from 82% to 120%, with a mean of 103.4 ± 11.5%.

To evaluate the potential bioavailability of THg in PRE sediments, five selected sediment samples were also analyzed using four different single-selective extractants or extraction methods—1 M HCl, sipunculan gut juices, cysteine, and a toxic characteristic leaching procedure (TCLP) (USEPA, 1992) (Table 2). The sipunculan gut juices were obtained according to the method described by Zhong and Wang (2008b). The sipunculans *Sipunculus nudus* were dissected immediately after being brought to the laboratory. Their gut contents were removed into a centrifuge tube and centrifuged at 2267*g* for 30 min at 4 °C to remove any solid content. The juices were frozen at −80 °C before being used for Hg extraction. After extraction, the mixture of sediments and extractants were separated by centrifuging at 3000 rpm for 20 min. The extracts were preserved with a BrCl solution as above before Hg analysis.

For measurements of THg in sediment samples, 0.25 g of freeze-dried sediments were digested using *aqua regia* (2 mL HNO_3:6 mL HCl) in a microwave digestion system (Speedwave® IV, Germany) with the EPA 7474 program (at 190 °C for 10 min). Residue samples from sequential extraction (F5) were also digested using *aqua regia* following the same procedure. The resulting digested mixture was centrifuged at 3000 rpm for 20 min at room temperature to obtain the supernatant. For analytical quality control, reagent blanks and standard reference materials (IAEA-158, Marine Sediment, IAEA) were digested using the same protocol. The recovery rates ranged from 92% to 107%.

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