



# Phase partitioning of trace metals in a contaminated estuary influenced by industrial effluent discharge<sup>☆</sup>



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## ABSTRACT

Severe trace metal pollution due to industrial effluents releases was found in Jiulong River Estuary, Southern China. In this study, water samples were collected during effluent release events to study the dynamic changes of environmental conditions and metal partitioning among dissolved, particulate and colloidal phases controlled by estuarine mixing. Intermittent effluent discharges during low tide caused decreasing pH and dissolved oxygen, and induced numerous suspended particulate materials and dissolved organic carbon to the estuary. Different behaviors of Cu, Zn, Ni, Cr and Pb in the dissolved fraction against the conservative index salinity indicated different sources, e.g., dissolved Ni from the intermittent effluent. Although total metal concentrations increased markedly following effluent discharges, Cu, Zn, Cr, Pb were predominated by the particulate fraction. Enhanced adsorption onto particulates in the mixing process resulted in elevated partitioning coefficient ( $K_d$ ) values for Cu and Zn, and the particle concentration effect was not obvious under such anthropogenic impacts. Colloidal proportion of these metals (especially Cu and Zn) showed positive correlations with dissolved or colloidal organic carbon, suggesting the metal-organic complexation. However, the calculated colloidal partitioning coefficients were relatively constant, indicating the excess binding capacity. Overall, the intermittent effluent discharge altered the particulate/dissolved and colloidal/soluble phase partitioning process and may further influence the bioavailability and potential toxicity to aquatic organisms.

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## 1. Introduction

With progressive human activities and rapid industrial developments, estuaries in China are now facing increasing metal pollution pressures (Pan and Wang, 2012; Wang et al., 2014). Trace metals are of great concerns because of their toxic effects and potentials for substantial and long-term accumulation in sediments and organisms (Oursel et al., 2013). It is insufficient to assess the environmental risk simply based on total metal concentrations. The partitioning of trace metals in particulate, colloidal and soluble phases and the interactions among these phases will affect the metal bioavailability and the hazard posed (Simpson et al., 2014). In estuary, adsorption and desorption are among the most important factors that control solid/liquid interaction (Fu et al., 2013), and distribution coefficient ( $K_d$ ) has been used to describe the

partitioning behavior. Furthermore, colloids, operationally defined as particles in the size fraction between 1 nm and 0.2  $\mu\text{m}$  (Buffle, 1990), are abundant in aquatic environment and play central role in regulating the concentration, speciation, transport, fate and bioavailability of trace metals (Guo and Santschi, 2007).

Trace metal cycling in estuaries is controlled by a variety of factors including ionic strength, redox, pH and abundance of adsorbing surfaces (Bianchi, 2006). Previously, the relationships between environmental parameters and metal speciation or kinetic partitioning have been examined in both freshwater and seawater systems (Gundersen and Steinnes, 2003; Hatje et al., 2003a,b). Conservative or non-conservative behaviors along salinity gradient varied according to metals and estuary (Sanudo-Wilhelmy et al., 1996; Guo et al., 2000; Wei et al., 2004), suggesting the dynamic conditions in the field and the importance of colloids. When effluent and estuarine water are mixed, redistribution of trace metals to different size fractions may occur (Luan and Vadas, 2015). Studies have reported enhanced coagulation and deposition in a temporally anoxic estuary (Jiann et al., 2005) and higher scavenging of dissolved metals with higher turbidity in a channelized river

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resulting from navigation (Prygiel et al., 2015). For the colloidal carriers, Cu showed high affinity for dissolved organic carbon while Pb was more typically associated with inorganic particles (Vega and Weng, 2013). Following effluent discharge, flocculation and precipitation of considerable portion of metals can also be expected (Simpson et al., 2014).

Jiulong River Estuary is a shallow estuarine system on the southeast coast of Fujian Province. The estuary is characterized by high fluctuation of hydrographic and geochemical conditions due to natural mixing process and anthropogenic activity (Weng and Wang, 2014). Both intermittent effluent discharges and continuous drainage water releases are common in this estuary (Wang et al., 2014), with inputs of industrial contaminants in an unprecedented manner, causing acute and chronic toxicity to organisms in the receiving stream. Previous studies in this area have revealed multi-metal pollution in surface sediments (Liu et al., 2006) and in some bivalves (Wang et al., 2011; Weng and Wang, 2014, 2015), but data on the dynamics of trace metals are limited. A few studies have noticed the environmental impacts of drainage water on localized ecosystems by examining the distribution and fate of metals (Gagnon and Saulnier, 2003; Oursel et al., 2013; Cresswell et al., 2013) or by experimental simulation approaches (Simpson et al., 2014). Nevertheless, how mixing of effluent and estuarine water influences the metal partitioning and bioavailability in such complexed condition remains poorly understood.

The main purpose of the study was therefore to take the advantages of such intermittent metal effluent releases and explore the dynamic changes of metal concentrations in dissolved, particulate and colloidal phases as well as their relationships to physico-chemical parameters. Several hypotheses were proposed, including, the effluent discharge would alter dissolved metal concentration in estuarine process; drastic change of ionic strength, redox and pH could contribute to the metal partitioning; the anthropogenic and newly formed particulates could probably be involved in the mixing scenario; and particulates as well as organic and inorganic binding ligands would influence the deemed mediating role of colloids. To test out those, time-series based samplings on each day were conducted to cover the tidal cycle and the intermittent effluent event (if any). We collected surface and bottom water samples from two sites during the two sampling campaigns. Trace metals including Cu, Zn, Ni, Cr and Pb in different phases were distinguished using filtration and ultrafiltration methods and then quantified. The variations and mechanisms for particulate/dissolved, colloidal/soluble phase partitioning were discussed and we identified the significant influences of effluent discharge. Results from this study can further provide information on metal toxicity prediction, environmental impact assessment and pollution mitigation.

## 2. Material and methods

### 2.1. Study sites and sampling

The Jiulong River, with a catchment area of 14,741 km<sup>2</sup>, discharges to the coastal sea of Xiamen City through the Jiulong River estuary. The estuary is a typical subtropical macro-tide system (Yan et al., 2012), with an annual net flow rate of 14 billion m<sup>3</sup>. This study focused on two sites of north branch of Jiulong River Estuary (Fig. 1), close to newly established industrial zones that host factories (Wang et al., 2011). Industrial effluents, characterized by high suspended particulate matter and organic matter concentrations, were often stored in holding ponds and were irregularly discharged into the estuary during low tide (Wang et al., 2014). Site 1 (S1) was directly receiving the visible intermittent effluent with dramatic change of environmental conditions, whereas Site 2 (S2) was

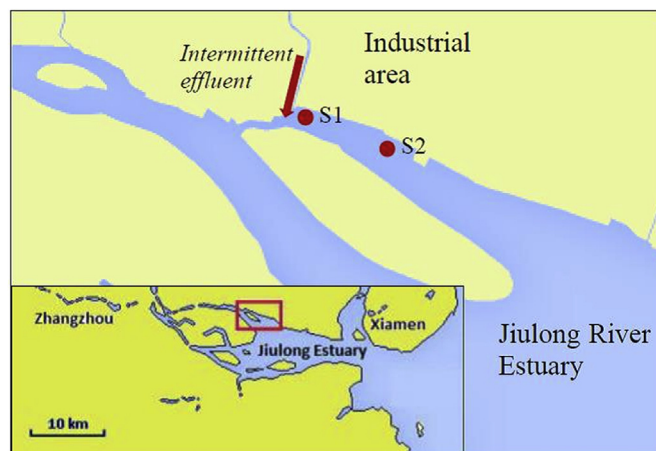


Fig. 1. Locations of the intermittent effluent and two sampling sites in the Jiulong River Estuary.

located at about 700 m downstream of S1 and much less influenced by the effluent discharge, although it had been potentially affected by continuous drainage water release.

Based on Before-After-Control-Impact design, two sampling campaigns were carried out during 30 Nov. 2014–2 Dec. 2014 and 27 Jan. 2015–29 Jan. 2015 respectively. The intermittent effluent discharge, which lasted for several hours, occurred on second day of 1st and on all three days of 2nd sampling campaign. On each day, there were 5 or 6 time points (with 1–2 h interval) for sampling at both the two sites, covering most time of a tidal cycle. At each site, both surface and bottom water samples were collected. At each time of sampling, two independent replicates were made.

Clean techniques were strictly employed during the whole sampling process to minimize contamination (Allen, 2000), and nitrile gloves were worn at all times. The plastic wares were previously acid-washed with 5% HNO<sub>3</sub>. Chemicals were of trace metal grade. Surface waters were collected by hand-dipping beneath the surface and a pre-cleaned perspex water sampler which was horizontal and adjusted to be precise on the sampling depth, helped collect estuarine waters from 50 cm above the bottom, and acid-washed 0.5 L low-density polyethylene bottles were used. On-board filtration was conducted with in-line syringe cellulose nitrate filters (0.2 μm, Sartorius) immediately after sampling to obtain total dissolved samples. All samples and filtrates were transported to the laboratory in ice-filled insulated containers and then stored at 4 °C in the dark before analysis.

In addition, physico-chemical parameters including salinity, pH, dissolved oxygen and temperature were measured *in situ* together with surface and bottom water sampling at each time point and were recorded with a calibrated multi-probe sensor (YSI Pro Plus).

### 2.2. Pretreatment of dissolved and particulate samples

Conventional water filtration did not completely separate soluble and colloidal forms of metals (Simpson et al., 2014). In this study, we chose centrifugal ultrafilter (Pall Corporation) with a molecular weight cutoff (MWCO) of 3 kDa; this approach has been well established for size fraction of colloids (Wang and Guo, 2001; Guo and Santschi, 2007; Chen et al., 2015). High molecular weight colloids were retained by the modified polyethersulfone membrane and the permeate represented soluble fractions. To check the apparent MWCO and the retention characteristics, two standard macromolecules with known molecular weights: 1.33 kDa vitamin B<sub>12</sub> and 4 kDa fluorescein tagged dextran were used. The vitamin

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