



# An investigation of the effects of elevated phosphorus in water on the release of heavy metals in sediments at a high resolution



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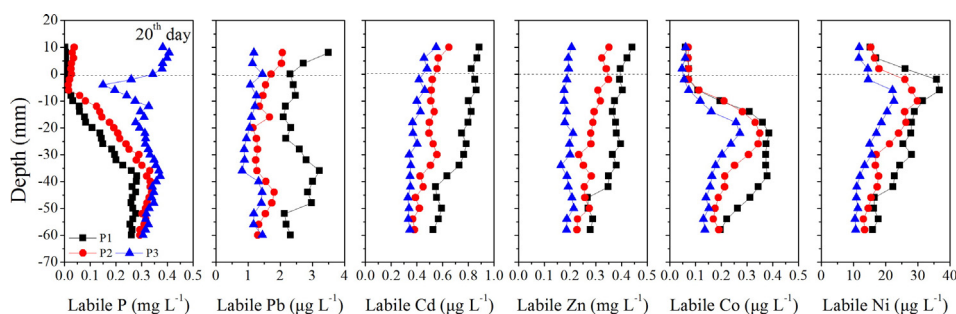
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## HIGHLIGHTS

- DGT and Peeper techniques were employed to investigate P effect on heavy metals at a millimeter scale.
- The precipitation of metal P reduced DGT-labile and dissolved metals during a 20-day incubation.
- The recovery of DGT-labile metals appeared on the 40th day during incubation due to solid resupply.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Excessive phosphorus (P) input plays an important role on the release of heavy metals in sediments under the eutrophic environment. In this study, a microcosm experiment with 40-day incubation using homogenized sediments was performed to investigate this aspect at a millimeter resolution. Diffusive gradient in thin films (DGT) and dialysis (Peeper) techniques were employed to simultaneously measure labile and dissolved P, Pb, Cd, Zn, Co and Ni in sediments at a millimeter scale, respectively. The results showed that an increase of water P from 0.02 mg L<sup>-1</sup> to 0.20 and 2.4 mg L<sup>-1</sup> generally led to intensified decrease of DGT-labile metals from the 10th to 20th days after the onset of incubation. The decrease in dissolved metals in pore water also appeared on the 20th day. The degree of decrease in the five metals was in the order of Pb > Cd > Zn > Co > Ni, which has a negative correlation with the solubility constants ( $K_{sp}$ ) of each metal-P precipitate. This indicated that the negative effect was caused by the precipitation of metal P. On the 40th day during incubation, the concentrations of DGT-labile metals had different increasing pattern compared to those on the 20th day. The extent of metal recovery was positively correlated with the change of desorption rate constant ( $k_{-1}$ ) and negatively correlated with the change in the characteristic time ( $T_c$ ) to reach equilibrium from DGT perturbation derived from DGT-induced fluxes in sediments (DIFS) modeling. This suggested that the recovery of metal lability from elevated water P was a result of the increased releases of metals from sediment solids.

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## 1. Introduction

Heavy metal pollution in the environment including the air, soil, lake and sea has been recognized as a serious problem for more than six

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decades (Agarwal, 2009; Gao and Chen, 2012; Harada, 1995). In particular, heavy metal pollution of the lakes has adverse impacts on the aquatic ecosystem, and the food chain through bioaccumulation in aquatic organisms (Buccolieri et al., 2006; Fan et al., 2002). Studies have evaluated the environmental health risks based on the contents of various heavy metals in the lake water and sediments (Gao and Chen, 2012; Karlsson et al., 2010; Tang et al., 2010). There are a great number of studies focused on the sources of heavy metal pollution (Gundersen and Steinnes, 2003; Peng et al., 2009; Salomons and Stigliani, 1995). The studies found that factors such as pH, Eh and organic matter have significant influences on heavy metal pollution (Bonnissel-Gissing et al., 1998; Gundersen and Steinnes, 2003; Kelderman and Osman, 2007; Peng et al., 2009). For example, when pH in water decreases, the fixation percentage of heavy metals adsorbed on particles may decrease from nearly 100% to almost zero (Gundersen and Steinnes, 2003). Consequently, the adsorption ability of the dissolved metals decreases, resulting in an increase in the mobility of heavy metals. Similarly, when Eh in sediments increases, the degradation rate of organic compounds increases correspondingly, accelerating the release of the adsorbed heavy metals (Calmano et al., 1993). For instance, with increasing the value of Eh in sediments, the organic sulfide bound Cd would decrease from 65% to 30% from a stable metal form to a more mobile form (Kelderman and Osman, 2007; Zoumis et al., 2001). In some lakes, organic matter bound heavy metals are the largest fraction in sediments. Additionally, the solubility of organic matters in sediments usually directly determines the mobility of heavy metals (Sekaly et al., 1999).

Besides these factors, Peng et al. (2009) reported that  $H^+$  strongly competed with dissolved metals for ligands such as  $PO_4^{3-}$ , indicating that the dissolved metals could be associated with  $PO_4^{3-}$ . The influence of the phosphorus (P) on heavy metal pollution in lakes remains poorly understood, though the influence in soil has been widely investigated (Bolan et al., 2003a; Fang et al., 2012; Maenpaa et al., 2002; Zwonitzer et al., 2003). According to Maenpaa et al. (2002), the high concentrations of phosphorus ( $KH_2PO_4$ ) significantly reduced the bioavailability of heavy metals such as Pb, Zn, and Cd because of the formation of metal-phosphate complexes in soil. Therefore, further investigation is necessary to understand whether the same mechanism is applicable to sediments. Additionally, toxic, food web altering, hypoxia generating cyanobacterial algal blooms (CyanoHABs) are multiplying world-wide in lakes because of nutrient enrichment, posing a serious threat to water resources (Paerl et al., 2011). One of the major reasons for water eutrophication and harmful algal blooms in lakes is the excessive P inputs from fertilizer use, municipal and industrial wastewater (Conley et al., 2009). Whether the excessive P inputs in water affect the lability of heavy metals in sediments and their releases to water have not been well studied.

The aim of this study was to understand the processes affecting the releases of heavy metals in the presence of high concentrations of phosphate in lake sediments. An incubation experiment was performed using homogenized sediments to investigate the effects of elevated water P on the mobility of five heavy metals, namely Pb, Cd, Zn, Co and Ni in sediments at a millimeter scale. Dialysis (Peeper) and diffusive gradient in thin films (DGT) techniques were employed to simultaneously measure dissolved P, Pb, Cd, Zn, Co and Ni in pore water and labile P, Pb, Cd, Zn, Co and Ni primarily released from sediment solids, respectively. The precipitation effect of P was assessed based on their changes in concentration, along with the kinetics of Pb, Cd, Zn, Co and Ni in sediment solids.

## 2. Materials and methods

### 2.1. Preparation of the DGT and peeper probes

The loaded DGT probes were provided by Easysensor Ltd. ([www.easysensor.net](http://www.easysensor.net)). Two types of binding gels were used in the assembly

of DGT devices. One was the zirconium oxide (Zr-oxide) gel used for DGT measurement of P. The other was the Chelex binding gel used for simultaneous measurements of Pb, Cd, Zn, Co and Ni. Both measurements had a vertical resolution of 4 mm. The Zr-oxide was prepared according to Ding et al. (2010a). The Chelex binding gel was modified from the ZrO-Chelex gel reported by Xu et al. (2013), in which the amounts of half-dried Zr-oxide powder and Chelex-100 used in casting the gel were changed to 0.2 g and 2.0 g, respectively (Wang et al., 2016). A new flat-type holder was used for assembling the DGT, which has removed the raised bottom edge of the existing one provided by DGT Research Ltd. and significantly reduced the sample disturbance during probe insertion into sediments (Ding et al., 2016a). In the assembly of the DGT probe, a binding gel was placed on the bottom of a flat DGT holder, which was sequentially covered by the agarose diffusive gel and the Durapore PVDF membrane (Millipore, 0.45  $\mu m$  pore size, 100  $\mu m$  thickness). The loaded DGT device was stored in 0.01 M NaCl prior to deployment in sediments.

The Peepers technique was developed by Ding et al. (2010b) for sampling pore waters in sediment at a 4 mm vertical spatial resolution. The Peepers is constructed of Perspex plates (20 cm) with 30 equally spaced 200- $\mu L$  chambers. The chambers were filled with deionized water and covered with a 0.45  $\mu m$  cellulose nitrate membrane. Assembled peepers were placed in water flushed with  $N_2$  overnight before usage. The exposure time of the sampler in the sediments was at least 48 h to ensure the equilibrium status was achieved between the concentrations of dissolved substances in the pore water and the chamber water.

### 2.2. Experimental setup

Sediments used in the microcosm experiment were collected from Meilang Bay of Lake Taihu (31°30'31.1"N, 120°10'31.0"E), which is the third largest freshwater lake in China. Previous studies reported that the surface sediment pH in the sampling site was 6.82, while the concentrations of TOC, TP, Pb, Cd, Zn, Co and Ni were 8.26  $mg\ g^{-1}$ , 810  $mg\ kg^{-1}$ , 44.7  $mg\ kg^{-1}$ , 0.78  $mg\ kg^{-1}$ , 135.60  $mg\ kg^{-1}$ , 19.9  $mg\ kg^{-1}$  and 65.20  $mg\ kg^{-1}$ , respectively (Chen et al., 2015; Ren et al., 2015; Yuan et al., 2011). Algal bloom occurs frequently in this bay due to eutrophication caused by sewage discharges since the 1980s. About 18 sediment cores (11 cm in diameter) were collected using a gravity corer (11 cm  $\times$  50 cm, Rigo Co., Japan) on the 26th of September 2015, along with overlying water in plastic buckets. The samples were transferred to the laboratory within 4 h. Each sediment core was sectioned at a 2 cm interval. The sediment layers at the same depth for all the cores were pooled together and thoroughly homogenized. Thereafter, macrofauna and large particles were removed from the homogenized sediments by passing through a 0.6 mm pore-size mesh sieve. The sediments were then put into 18 Perspex tubes according to their original depth order, and the final depth of the sediment cores used for incubation was 12 cm. Afterwards, each 6 sediment cores were put into a tank (totally 3 tanks), to which the overlying water filtered through a 0.45  $\mu m$  filter was added up to 45 cm depth. The three microcosms were incubated at 20 °C for two months. The water was pumped together with air for 10 min every hour to maintain the  $O_2$  saturation during the incubation.

At the end of the pre-incubation, the concentrations of total P in the three microcosms were conditioned at 0.02  $mg\ L^{-1}$  (P1), 0.20  $mg\ L^{-1}$  (P2) and 2.4  $mg\ L^{-1}$  (P3), respectively, by adding phosphate (in the form of  $Na_2HPO_4$ ) into water samples. The water pH was stabilized at the original value of 7.6 through diluted HCl and NaOH adjustment.

### 2.3. Sampling

One Peepers was inserted into a sediment core of each microcosm at the 10th, 20th and 40th days after the onset of incubation. After 24 h, the DGT probes were inserted into the same sediment cores. The probes

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