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## DISTRIBUTION OF ADDED PHOSPHORUS IN SEDIMENT UNDER INTERMITTENT RESUSPENSION CONDITIONS

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### Abstract

The incorporation of phosphorus (P) into sediments and the changes in its bioavailability under intermittent P input and sediment resuspension conditions were investigated via laboratory experiments. The sediments were disturbed daily for 10 min and then allowed to settle for 1430 min after each disturbance to simulate the intermittent sediment resuspension conditions. The removal of soluble reactive P from the overlying water under sediment resuspension was greater than that under static conditions. Sequential fractionation indicated that most of the incorporated P (71%) was accounted for in the Fe/Al-P fraction under sediment resuspension conditions, whereas its value was up to 99.6% under static conditions. Bioavailable P (BAP) could be accurately estimated using the algal available P (AAP) and loosely associated P (NH<sub>4</sub>Cl-P). The contribution of the added P to BAP was 47.3% under sediment resuspension conditions, whereas the contribution was up to 76.4% under static conditions, which can be attributed to the formation of AAP. Less than 47% of the incorporated P into the sediments was accounted for in AAP under sediment resuspension conditions, whereas over 70% was accounted for under static conditions. The results suggest that intermittent resuspension can inhibit the incorporation of added P into AAP, which can be attributed to the binding of added P to crystalline iron oxides, aluminum oxides, and Ca.

*Key words:* adsorption, phosphorus, pollution, precipitation, remediation, sediments

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### 1. Introduction

Phosphorus (P) is a eutrophication-limiting nutrient (Lai and Lam, 2008; Rydin et al., 2011; McMahon and Read, 2013). Although many point sources of P discharges have been successfully controlled or reduced, nonpoint sources, such as those coming from agricultural catchments (Huisman et al., 2013; Wallbrink et al., 2003; Zhang et al., 2013), pose great dangers of increasing loads in adjacent water bodies (Reddy et al., 1996).

Increased phosphorus concentrations are associated with nuisance cyanobacteria blooms and excessive periphyton accumulation (Ellison and Brett, 2006). Moreover, phosphorus imported to a water body tends to accumulate in the sediments. Sedimentary P can be recycled to the overlying water under certain conditions after external inputs have

been eliminated, creating long-term water quality problems (Ellison and Brett, 2006; Li and Friedrich, 2006; Sun et al., 2009).

Dissolved P is expected to be adsorbed onto suspended sediments (De Vicente et al., 2010; Spears et al., 2007; Sun et al., 2009; Wan et al., 2010) because sediment resuspension may result in the expansion of fine-grained matter with high adsorption capacity in water (Nøges and Kisand, 1999). Moreover, free or sediment-attached microorganisms could also assimilate dissolved P through microbial immobilization (House et al., 1995). Thus, most of the P are incorporated into the different P fractions (i.e., Fe/Al-bound P and Ca-bound P) of sediments during sedimentation-deposition or sorption-precipitation (Nguyen, 2000; Pettersson, 2001). Li and Huang (2010) found that sediment resuspension results in a more efficient

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removal of dissolved inorganic P from the overlying water and in the decrease of sedimentary P bioavailability.

However, they did not investigate the removal of P from overlying water and the distribution of added P in the sediments under sediment resuspension and external P input conditions (Li and Huang, 2010). Evaluating the concentrations of bioavailable P (BAP) stored in the sediment is important for quantifying the release of P from the sediments (Ellison and Brett, 2006). In general, BAP can be estimated using the sum of mobile P fractions, such as loose P and Fe/Al-bound P.

However, Golterman (2001) and Selig (2003) found that not all Fe/Al-bound P contribute to the sedimentary P bioavailability, and only those P associated with amorphous Fe compounds (non-occluded Fe/Al-bound P) can be released. Moreover, Li and Huang (2010) found that sediment resuspension could reduce the concentrations of sedimentary P associated with amorphous Fe compounds, resulting in the decrease in BAP. However, information on the contributions of external P input to sedimentary P bioavailability under sediment resuspension conditions is limited.

Several studies (Nguyen et al., 1997; Nguyen, 2000) have investigated the partitioning of recently added P between inorganic and organic components of sewage-impacted wetland sediments. However, their results on the wetlands cannot be simply applied to the standing, heavy eutrophic canal.

Canal sediments are notoriously variable and are generally finer and less organic than sediments found in wetlands. Therefore, canal sediments are not expected to distribute P in the same way as wetland sediments do. In addition, previous studies lack data for the comparison of added P distribution in the sediments under sediment resuspension and static conditions. The comparison is an important knowledge gap because quantifying the sedimentary P transformation between mobile P fractions and inert P fractions is important to properly understand sedimentary P distribution and to foretell the potential of P release of sediments from the overlying water. The current study aims to investigate the influences of intermittent sediment resuspension on the amount of added P incorporated into different P fractions of sediments via a laboratory-scale experiment. The P fractionation schemes of Wang (2013) were used to trace the partitioning of input P into different P fractions. Moreover, changes in the BAP due to the redistribution of sedimentary P were investigated to understand the contributions of added P to sedimentary P bioavailability in terms of the effects of intermittent sediment resuspension and to compare them with the effects of static conditions.

## 2. Materials and methods

### 2.1. Site description

A campus canal with a relatively steady flow was used as the source of water and sediment

samples. Its length and average width were about 3150 and 18 m, respectively. Its maximum and mean depths were 3.30 and 1.26 m, respectively. The relatively small (1.95 km<sup>2</sup>) drainage area mainly ran through residential (~70%) and campus (~20%) areas. Although the canal under study did not receive any major point source nutrient inputs, some minor point sources (e.g., those coming from restaurants and bathhouses) and nonpoint sources pose great dangers of increasing loads in the canal. Appreciable concentrations of soluble reactive P (SRP) were observed in the water. The canal has had serious blue-green algae blooms every summer and autumn since 2004.

### 2.2. Experimental methods

Nine intact sediment cores (10 cm diameter) from the campus canal were sampled to a depth of 5 cm. The top 1 cm layers of all the sediment cores were extruded on site. Surface sediments were stored in capped glass flasks and then transported to the laboratory where the resuspension experiments were conducted immediately.

### 2.3. Resuspension experiment

Sediments [200 g wet weight (WW)] with depths of about 1.2 cm were placed into a Perspex container with a diameter of 154 mm and height of 300 mm. Canal water (2.5 L) with 0.42 mg L<sup>-1</sup> SRP was carefully placed into the container while preventing sediment disturbance. The depth of water was about 12.5 cm.

Two sets of containers (designated E0 and E1) were used in the current study, and each set was performed in triplicate. The sediments in E1 were disturbed using a blade with length of 5 cm at 60 rpm for 10 min daily for 39 days. The sediments were kept completely suspended during disturbance and then allowed to settle for 1430 min after each disturbance to simulate the intermittent sediment resuspension conditions. E0 was used as a control experiment to simulate static conditions. Sediments [200 g WW] and canal water (2.5 L) with 0.42 mg L<sup>-1</sup> SRP were placed into the container with the same size as E1. The sediments and water were not disturbed. Water samples (25 mL) were taken from each container for SRP measurement. At the same time, the same volume of canal water with 1 mL KH<sub>2</sub>PO<sub>4</sub> solution was added to each container, initially at two-day intervals for 39 days. The KH<sub>2</sub>PO<sub>4</sub> concentration was 1.25 mg P mL<sup>-1</sup> at days 3, 6, 9, 12, 15, 18, 21, and 24, and it was 2.5 mg P mL<sup>-1</sup> at days 27, 30, 33, and 36.

The experiment aimed to keep the P concentration of the overlying water in both sets at about 0.5 mg P L<sup>-1</sup> (SRP) from day 3 to day 24 and 1.0 mg P L<sup>-1</sup> (SRP) from day 27 to day 36 to simulate the external P input into the canal. Based on the calculations, a total of 20 mg P (SRP) was added into the water column over the 39-day period. After the

39-day resuspension experiments, the sediments were taken from each container, dried, ground, and then sieved using a standard 100-mesh sieve for chemical analyses.

#### 2.4. Phosphorus fractionation

Phosphorus fractionations were conducted according to Wang (2013). P was separated into the following: (a)  $\text{NH}_4\text{Cl-P}$  (loosely associated P) removed with 1 M  $\text{NH}_4\text{Cl}$  for 2.2 h; (b)  $\text{Fe/Al-P}$  (P precipitated with metal ions, such as Fe or Al) removed with 0.1 M  $\text{NaOH}$  for 16 h; (c)  $\text{HCl-P}$  (P precipitated with inorganic salts, such as Ca, removed with 0.1 M  $\text{HCl}$  for 16 h; and (d)  $\text{Res-P}$ , according to Jiménez-Cárceles and Álvarez-Rogel (2008). The sediments and extracted solutions were separated using a laboratory centrifuge (4,000 rpm for 20 min). The solid phase was used for the next extraction procedure, and the solution was filtered through a 0.45  $\mu\text{m}$  polyamide filter for subsequent SRP analysis.

The non-occluded  $\text{Fe/Al-P}$  could be assessed using algal available P (AAP) concentrations. The analytical procedure for measuring AAP was as follows: 0.50 g dry suspended particles were placed into bottles. Then, the samples were shaken in 200 mL of 0.1 M  $\text{NaOH}$  for 4 h using a reciprocating shaker at 220 rpm and 25 °C. The P extracts were centrifuged (10,000 rpm for 10 min) and filtered through a 0.45  $\mu\text{m}$  polyamide filter. Finally, the SRP concentration was analyzed. In the current study,  $\text{NH}_4\text{Cl-P}$  and AAP constituted BAP in the sediments.

#### 2.5. Analytical methods

SRP in overlying water and inorganic P (SRP) in all extracts were determined by the methylene blue method using DR 5000 ultraviolet spectrophotometer (Loveland, USA). Sediment pH (equilibrated with distilled water under disturbance conditions at a sediment-water ratio of 1:2.5 for 30 min) was measured using a pH glass electrode.

The sediments were dried at 105 °C overnight to determine the sediment water content. Organic matter was determined as a percentage of dry weight (DW) lost in ignition (LOI) after combustion at 550 °C for 4 h. Amorphous Fe (Ox-Fe) and amorphous Al (Ox-Al) in the sediments were extracted in the dark using acid ammonium oxalate (Loeppert and Inskeep, 1996). The concentrations were determined using an inductively coupled plasma optical emission spectroscopy (ICP-OES, Perkin Elmer, Optima 5300 DV, Maryland, USA).

The sample grain size distribution was analyzed using a laser grain size analyzer (ZetaPALS, New York, USA). The dried samples were placed into beakers and 50 mL distilled water was added to each beaker. Then, they were shaken for 5 min by hand prior to analyses. The percentages of grain size groups, including clay (0.02  $\mu\text{m}$  to 4  $\mu\text{m}$ ), silt (4  $\mu\text{m}$  to 63  $\mu\text{m}$ ), and sand fractions (63  $\mu\text{m}$  to 500  $\mu\text{m}$ ) were determined.

#### 2.6. Statistical analyses

All statistical analyses of the data obtained were conducted using SPSS software. Variations in SRP between treatments (static and sediment resuspension conditions) were tested for significance using the analysis of variance (ANOVA) procedure. Similarly, variations in added P in different sediment fractions, BAP, and AAP at the end of the experiment were tested for significance using ANOVA.

### 3. Results

#### 3.1. General sediment characteristics

The sediments in the canal had water content and bulk density of 76.8% and 0.85  $\text{g cm}^{-3}$ , respectively. The LOI and pH value of the sediments were 12.19% (DW) and 7.15, respectively. The concentration of Ox-Fe (13.7  $\text{mg g}^{-1}$ ) was higher than that of Ox-Al (11.2  $\text{mg g}^{-1}$ ) in the sediments.

The grain size distribution in the sediments was classified into clay, silt, and sand fractions. The grain size distribution in the sediments showed that the sediments mainly consisted of silt (83.10%). Table 1 shows the results of the sequential extraction. The sum of all sedimentary P fractions (Tot-P) had values of up to 1194.3  $\text{mg P kg}^{-1}$  (DW). Based on Table 2,  $\text{Fe/Al-P}$  was the dominant fraction in the sediments, comprising 56.52% of Tot-P, followed by  $\text{HCl-P}$  (35.02%),  $\text{NH}_4\text{Cl-P}$  (6.70%), and  $\text{Res-P}$  (1.76%).

#### 3.2. Variation of SRP in overlying water

The SRP in the overlying water under intermittent sediment resuspension conditions was lower than that under static conditions ( $P \leq 0.0001$ ) (Fig. 1). The SRP concentrations in the sediment resuspension treatment varied with time, but maintained a low level (about 0.02  $\text{mg P L}^{-1}$ ). However, the SRP concentrations in the static treatment gradually increased with the addition of P.

**Table 1.** Phosphorus fractions in the canal sediments ( $\text{mg P kg}^{-1}$ ). All the concentrations of P fractions in the sediments were expressed based on the DW of the sediments at 105 °C (unit:  $\text{mg P kg}^{-1}$  DW). The values reported are the mean  $\pm$  SD

<i>NH<sub>4</sub>Cl-P</i>	<i>Fe/Al-P</i>	<i>HCl-P</i>	<i>Res-P</i>	<i>Tot-P</i>
80.0 $\pm$ 18.0	675.0 $\pm$ 24.0	418.3 $\pm$ 38.2	21.0 $\pm$ 3.0	1194.3 $\pm$ 36.9

3.3. Incorporation of P into different P fractions

After the experiments finished, the Tot-P increased from 1194.3 mg P kg<sup>-1</sup> DW (initial state) to 1509 and 1373.3 mg P kg<sup>-1</sup> DW under the sediment resuspension and static conditions, respectively. The increase in Tot-P of the sediments subjected to resuspension was greater than that under static conditions (P ≤ 0.0001). Fig. 2 shows the changes in the individual P pool.

The amount of added P incorporated into the Fe/Al-P fraction was greater than that of the other fractions in the sediments under resuspension and static conditions (P ≤ 0.0001). The increase in HCl-P fraction was greater under sediment resuspension condition than that under static condition (P ≤ 0.05). The P in NH<sub>4</sub>Cl-P increased by 12.5% under static conditions, compared with initial state, but the P in NH<sub>4</sub>Cl-P under sediment resuspension conditions was relatively unchanged from their initial states after the 39-day period. In addition, the P in Res-P was relatively unchanged from their initial states after the 39-day period.

3.4. Incorporation of P into bioavailable P

The P forms in NH<sub>4</sub>Cl-P and non-occluded

Fe/Al-P (AAP) are available to algae. Therefore, the amount of BAP can be estimated using the sum of these fractions. Figs. 3 and 4 show the changes in AAP and BAP in these sediments, respectively. The addition of P into the water resulted in the increase in AAP from 305.5 mg P kg<sup>-1</sup> DW (initial state) to 432.3 and 452.9 mg P kg<sup>-1</sup> DW under the static and sediment resuspension conditions, respectively (P ≤ 0.0001).

In addition, about 46.8% of the added P incorporated into the sediments with resuspension was accounted for in AAP, whereas the concentration increased to 70.8% under static conditions, suggesting that sediments with resuspension may have a lower P release potential than sediments under static conditions after P input. Correspondingly, the concentrations in BAP in the sediments increased from 385.5 mg P kg<sup>-1</sup> DW (initial state) to 522.3 and 534.6 mg P kg<sup>-1</sup> DW for the static and sediment resuspension conditions, respectively (P ≤ 0.001).

The increase in BAP was assumed to be attributed to the added P. Thus, the contribution of added P to BAP was 47.3% under sediment resuspension, but it was up to 76.4% under static conditions.

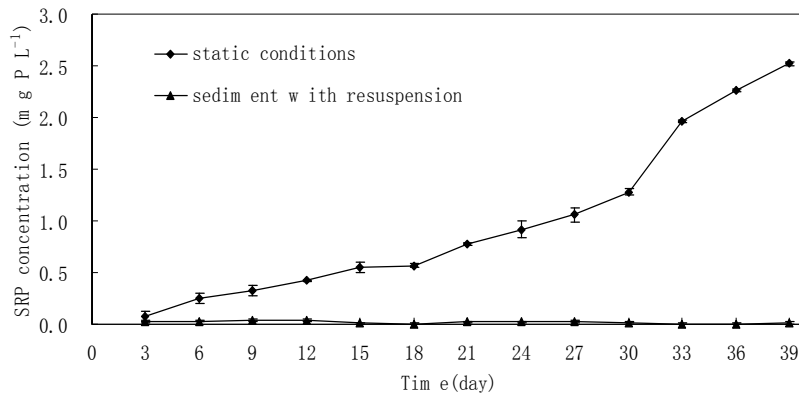


Fig. 1. Change in SRP in the overlying water with each P addition under sediment resuspension and static conditions. The values reported are the mean ± SD

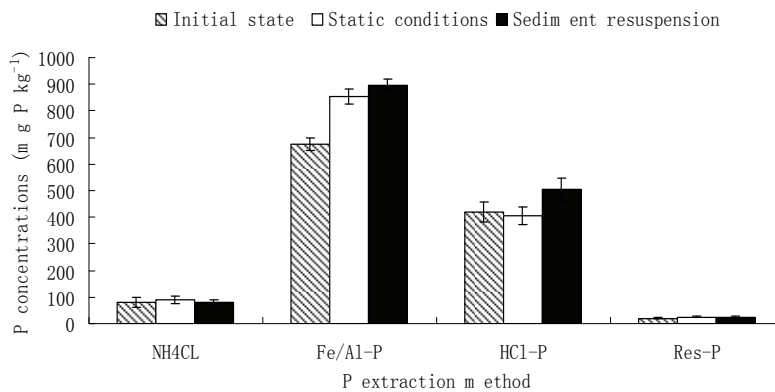
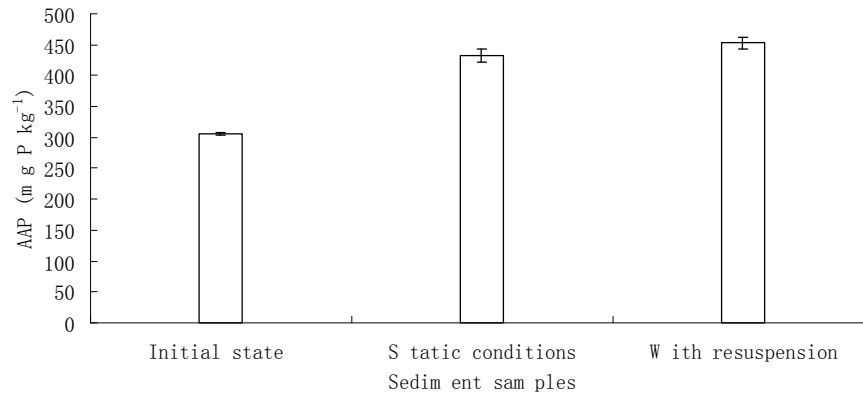
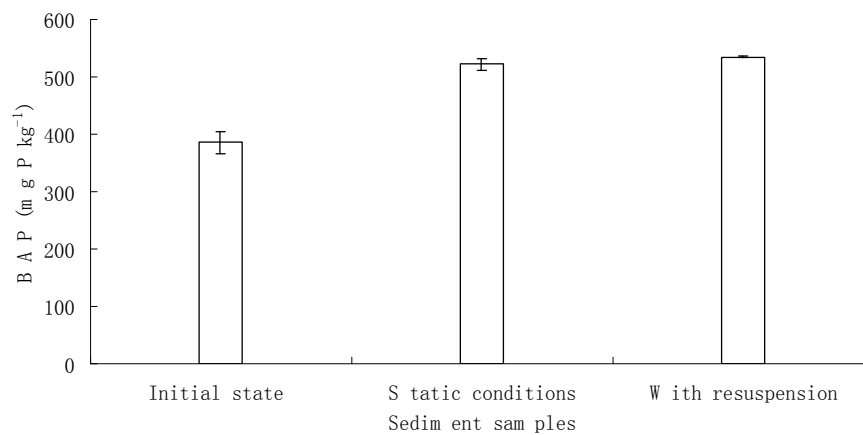


Fig. 2. Phosphorus concentrations in sediment under resuspension and static conditions after P input (the values reported are the mean ± SD)



**Fig. 3.** Concentrations of AAP in sediment under resuspension and static conditions after P input (the values reported are the mean ± SD)



**Fig. 4.** Concentrations of BAP in sediment under resuspension and static conditions after P addition (the values reported are the mean ± SD)

#### 4. Discussion

##### 4.1. Removal of SRP from the overlying water

The removal of SRP from the overlying water under sediment resuspension conditions was greater compared with that under static conditions because of the higher distribution of the phosphate reactants (e.g., amorphous Al, Fe, and organic matter) and sorbents (e.g., clay minerals and CaCO<sub>3</sub> particulates) in the overlying water caused by sediment resuspension (Eqemose et al., 2010; Huser and Pilgrim, 2014; Johannesson et al., 2011; Seo et al., 2005).

In general, amorphous Fe is very important in P absorption because of its relatively large specific surface area. However, the amorphous Fe decreased from 13.7 mg kg<sup>-1</sup> DW (initial state) to 11.2 mg kg<sup>-1</sup> DW under sediment resuspension, probably due to the amorphous iron oxides which became crystalline iron oxides via oxidation under sediment resuspension conditions (Kraal et al., 2009). Correspondingly, the non-occluded Fe/Al-P fraction (mainly existing as P bound via amorphous Fe oxides) was converted into occluded Fe/Al-P (Takeda and Fukushima, 2004). In addition, the presence of added P in the occluded Fe/Al-P may

also be attributed to the chemisorption of P by aluminum oxides (Nguyen, 2000; Wilson et al., 2010) because the P absorptive capacity of aluminum oxides is known to be constant under both aerobic and anaerobic conditions (Lijklema, 1993). Moreover, the significance of other P-binding elements, such as Ca, Mg, and Mn (Seo et al., 2005), and micro-organisms through microbial immobilization cannot be excluded as possible mechanisms that can contribute to the loss of SRP from the overlying water (Kisand and Nöges, 2003).

Dissolved oxygen (DO) penetration is another key factor that affects SRP removal through oxidized particles. The results in the present research show that the concentration of DO in the overlying water under sediment resuspension conditions increased gradually and then remained relatively constant (about 5.5 mg L<sup>-1</sup>) after 9 days. The supply of DO was in favor of the oxidation of Fe and Mn (Qin et al., 2004) and of the formation of ferric hydroxide/phosphate with associated high-energy sorption sites (House and Denison, 2000). Sediment resuspension can promote the interaction between SRP and oxidized particles, including metal oxides or hydroxides. Therefore, P removal from the overlying water is expected to be greater under sediment resuspension than under static conditions.

Kelderman et al. (2007) estimated that the sediments accumulated 65% of all imported P in the shallow Lake Taihu (China P. R.). In the current study, the accumulation value was obviously lower than that (99.85%) under sediment resuspension conditions. Lake Taihu is frequently influenced by wind and wind-generated waves, resulting in the resuspension of bottom sediments (Qin et al., 2004). Compared with the current study, the removal efficiency in Lake Taihu was lower because of the lower concentrations of suspended solids and because the shear stress caused by wind and wind-generated waves was lower.

#### 4.2. Distribution of incorporated P in P fractions

About 71% of added P incorporated into the sedimentary P fractions was accounted for in the Fe/Al-P fraction after sediment resuspension. The results suggest that the chemisorption of P by iron and aluminum oxides plays the most important role in controlling P incorporation (Nguyen, 2000; Palmer-Felgate et al., 2011). However, not all Fe/Al-P can contribute to P release. Saavedra and Delgado (2005) stated that amorphous iron oxides have much lower P retention capacities than crystalline iron oxides. Thus, the transformation of Fe between amorphous and crystalline oxides is important for P retention in sediments. DO penetration due to resuspension favors the transformation (Li et al., 2011), suggesting that sediment resuspension can reduce the mobility of Fe/Al-P, although most P are incorporated into this fraction.

HCl-P is refractory (Rydin, 2000) and contributes to a permanent burial of P in the sediments (Kaiserli et al., 2002). The formation of HCl-P accounted for more than 27% of the incorporated P under sediment resuspension conditions, reflecting that co-precipitation may be another P removal mechanism. Sediment resuspension could increase the contact probability between P and Ca in the overlying water. As a result, co-precipitation can be promoted. Moreover, calcium phosphate minerals contained in the sediments increased with the addition of P (House et al., 1995). On the contrary, HCl-P decreased slightly under static conditions, although in a smaller scale (13.3 mg P kg<sup>-1</sup> DW, 3.2%), suggesting that sediment resuspension plays an important role in promoting co-precipitation. Although this result cannot provide information on P transformation in sedimentary P fractions, it shows that sediment resuspension facilitates the formation of refractory P fractions compared with static conditions.

Rydin (2000) reported that the transformation of P from mobile fractions to inert fractions could occur with aging. In the current study, P incorporated into HCl-P of the sediments with resuspension included either added P or released P from mobile P of the sediments, i.e., the non-occluded Fe/Al-P fraction (weakly sorbed) (Nguyen, 2000). Although the relative importance of added P and released P

was not investigated, the proportions of Fe/Al-P and HCl-P to the total incorporated P of the sediments with and without resuspension suggest that the transformation of P from mobile fractions to inert fractions cannot be excluded as a possible mechanism for providing long-term P retention. Moreover, sediment resuspension may accelerate the transformation of mobile P fractions into inert P fractions.

#### 4.3. Contributions of additional P to bioavailable P

The results show that the added P may contribute to the bioavailability of sedimentary P, which is mainly attributed to the incorporation of P into the AAP. The concentrations of AAP in the sediments with and without resuspension increased by 48.3% and 41.5%, respectively, compared with the initial state.

On the other hand, the concentrations of occluded Fe/Al-P in the sediments with and without resuspension increased by 18.0% and 13.9%, respectively, suggesting that sediment resuspension facilitates the incorporation of added P into occluded-Fe/Al-P. The distinction between the sediments with and without resuspension is mainly attributed to the oxidation of amorphous iron oxides into crystalline iron oxides and the formation of aluminum oxides influenced by sediment resuspension (Nguyen, 2000; Takeda and Fukushima, 2004).

Sediments with resuspension are therefore expected to have a lower P release potential than sediments under static conditions after P input, i.e., sediment resuspension might favor the immobilization of input P (Qin et al., 2004; Li and Huang, 2010). Therefore, further research on the mechanism of the transformation of P into bioavailable forms and the transformation of mobile P fractions into inert P fractions caused by intermittent sediment resuspension is necessary.

### 5. Conclusions

Compared with static conditions, intermittent sediment resuspension could promote P removal from the overlying water. The suspended sediments also recorded higher Tot-P. Sequential fractionation indicated that most of the incorporated P (71%) into the sediments was accounted for in the Fe/Al-P fraction, followed by HCl-P (27%) under sediment resuspension conditions.

On the other hand, all of the incorporated P were almost found in the Fe/Al-P fraction under static conditions. Although the Fe/Al-P fraction was a predominant P fraction (about 60% of Tot-P in both sediments under resuspension and static conditions) in the sediments, the non-occluded Fe/Al-P (AAP) were considered the main Fe/Al-P fractions available for P release from sediments to the overlying water under anoxic environments. Therefore, BAP can be accurately estimated using the sum of NH<sub>4</sub>Cl-P and AAP.

The concentrations of BAP increased from their initial states under sediment resuspension and static conditions. The increase was attributed to the added P incorporated into the sedimentary P fractions. However, the contribution of added P to BAP under sediment resuspension was less than that under static conditions mainly because the lower fraction of added P to AAP accounted for 80% in BAP under sediment resuspension conditions.

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