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Lake sediment treatment with aluminum, iron, calcium and nitrate additives to reduce phosphorus release

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Abstract: Treatment of lake sediments with salts is a promising approach for preventing phosphorus release from sediments. Five 35-d treatments of undisturbed sediment cores in the East Lake, Wuhan, China were applied under anoxic conditions: nothing added (control), $Al_2(SO_4)_3$ added, $FeCl_3$ added, $CaCl_2$ added, and $NaNO_3$ added. To identify changes in the P binding sites in the sediment caused by the treatments, different P binding forms were extracted from the sediment before and after the treatments. We found that the mean P release rates for anoxic treatments with $Al_2(SO_4)_3$, $FeCl_3$, $CaCl_2$ and $NaNO_3$ were −0.6, 0.03, 0.6 and 2.6 mg/(m^2·d), respectively, while the P release rate with no additives was 7.3 mg/(m^2·d). In suboxic conditions, the concentration of total phosphorus ($TP_{average}$ 657 mg/kg) in sediment was much lower than that of untreated sediment ($TP_{average}$ 688 mg/kg) and treatments with salts (TP($Al_2(SO_4)_3$) 793 mg/kg, TP($FeCl_3$) 781 mg/kg, TP($NaNO_3$) 802 mg/kg, TP($CaCl_2$) 747 mg/kg). We also found that adding $CaCl_2$ prevented P release because of apatite formation and because P_{Ca} (Ca bound P) increased at the sediment surface. Addition of Fe^{3+} and NO_3^- to the sediment increased the amounts of $P_{Fe, Mn}$ (Redox-sensitive P, mostly Fe and Mn compounds), since iron oxide has the ability to combine P. Addition of $Al_2(SO_4)_3$ increased the fraction of $P_{Al, Fe}$ (P bound to metal oxides (Al, Fe)) and decreased the P and Fe in the water above the anoxic sediment, showing the greater ability of Al in binding P. The results showed that $Al_2(SO_4)_3$, $FeCl_3$, $CaCl_2$ and $NaNO_3$ all had an effect in controlling phosphorus release. The effect was related to the forms of phosphorus existing in the sediment before treatment and the forms resulting after adding the four reagents. The combination of Al^{3+} or Fe^{3+} with NO_3^- promises to be a reasonable chemical treatment for increasing the P retention capacity of sediments in eutrophic lakes. If chemical treatment is combined with bioremediation, the aim of environmental repair may be achieved.

Key words: Sediment, Internal phosphorus, Aluminum, Iron, Calcium, Nitrate, P fraction
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INTRODUCTION

Lake sediments play an important role in phosphorus metabolism in lakes. Phosphorus has long been considered a key factor affecting the water quality of lakes (Li *et al*., 2006). The main sources of phosphorus in lakes include external loading (such as rainfall, runoff, soil leaching, industrial and municipal effluents) and internal loading. Internal loading will often determine the eutrophication status of the lake and the time lag for recovery after reduction of the external loading (Kaiserli *et al*., 2002; Gao *et al*., 2005). In eutrophic lakes, P-release from anaerobic sediments often makes up the major fraction of the total P-load (Wauer *et al*., 2005a).

Lake sediments can function as either a sink or source of diluted forms of phosphorus, depending on the prevailing physicochemical conditions of the sediment-water interface. The sediment depth interacting with the lake water is probably lake-specific and highly dependent on lake morphology, sediment characteristics and wind exposure. Most often, phosphorus in the upper 10 cm or so is considered to take part in the metabolism of the whole lake, but mobility of phosphorus from depths down to 20~25 cm has been found (Søndergaard *et al*., 2003). It should be emphasized that lake sediments can be highly variable in terms of their chemical composition. Parameters such as dry weight and the contents of organic matter, iron, aluminum, manganese, calcium, clay and other elements with the capacity to bind and release phosphorus may all influence sediment-water interactions (Wauer *et al.*, 2005b).

At present, lake sediment treatments include in-situ and off-site treatment technologies. In-situ treatment technologies have developed rapidly recently because of their technological and economic advantages. Treatments can also be divided into chemical, biological and stabilizing types. In-situ chemical treatment is a new technology in which chemical agents (such as aluminum sulfate and calcium nitrate) are injected into sediment to reduce phosphorus release into water bodies.

A significant amount of research has been conducted on the reduction of P in internal loads and the immobilisation of P. These studies have included: restoration techniques including hydrological and chemical manipulation aimed at reducing P in the inflows and in the lake itself, as well as case studies (Stuben *et al*., 1998); the impact of the release of sediment nutrients on water quality and restoration projects (Kim *et al*., 2003; Fytianos and Kotzakioti, 2005); and restoration methods disrupting P-release from sediments aimed at improving their P retention capacity (Søndergaard *et al*., 2000; Prepas *et al*., 2001). Ripl (1976) first developed a restoration technique involving injecting $Ca(NO_3)_2$ into the sediment. However, this method is not widely used because of its negative effects on the water environment, such as accelerating eutrophication.

In this study, we evaluated the impact of aluminum, iron, calcium and nitrate additives on phosphate release from anoxic sediment in the East Lake, Wuhan, China, during a period of more than one month using undisturbed sediment cores. The test results revealed the process of migration of phosphorous in the sediments and provided guidance for restoration of the lake.

MATERIALS AND METHODS

Study area

The East Lake (30°33′ E, 114°23′ E) is on the alluvial plain of the central basin of the Yangtze River, northeast of Wuhan City, China. Its mean depth is 3~4 m with a maximum of 4.5 m. The lake is composed of several basins: Guozheng Hu, Tanglin Hu, Hou Hu, Niuchao Hu and Shuiguo Hu. The catchment area is 97 km^2, including urban areas (26.8 km^2), mountain areas (36 km^2) and arable land (34.2 km^2). Primary production by phytoplankton in the water column of the lake area has been rated as 6.3 mg $O_2/(m^2\cdot d)$ over the years (Jiang and Shen, 2006).

Municipal wastewater is still released into the East Lake through five main sewage outlets. About 60% of the phosphorus is retained in the lake, resulting in a sharp increase in the phosphorus concentration in the sediment (Xie and Xie, 2002). A large amount of sediment is suspended because of the wind-induced wave action in this shallow lake, resulting in natural eutrophication. The problem of eutrophication in the East Lake is now very serious. The percentage of eutrophicated water is now over 62%, while the concentrations of total nitrogen (TN) and total phosphorus (TP) in the most eutrophic parts of the lake are 7 and 0.4 mg/L, respectively (Yang *et al*., 2005).

Sampling

In June, 2007, undisturbed sediment cores (Φ5 cm) were taken from the connection between the Guozheng Hu and Shuiguo Hu basins using a home-made sediment corer. The sediments were black because they contained some plant debris, smelled bad and looked like slush. A total of 3 tubes of 35 cm depth and 15 tubes of 10 cm depth were collected for use in the experimental system. The samples of 35 cm depth were analyzed only to establish the original sediment composition. For each analysis test, the average value was adopted as the result after three parallel samples were analyzed. All salts were added by gently mixing the salts dissolved in deionized water into the top 2 cm of each core.

Dry weight was measured by drying sediment to a constant weight at 105 °C. Organic matter was measured by the loss in weight after oven-drying at 550 °C. The physico-chemical properties of the sediments are: pH=7.59, $W(H_2O)=18.0\%$, $W_{org}=3.8\%$, TP content $W_P=835$ mg/kg, NH_3-N content $W(NH_3\text{-}N)=229$ mg/kg, and TN content $W_N=$ 6200 mg/kg.

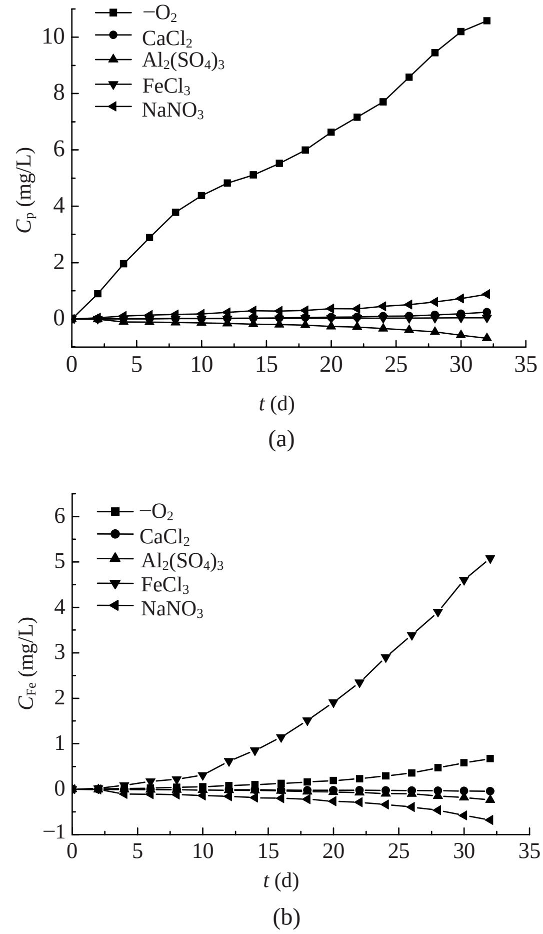
Test arrangements and analytical methods

Under suboxic conditions, five treatments were bubbled with N_2 during a 35-d experiment each using undisturbed sediment cores: nothing added (control-O_2), $Al_2(SO_4)_3$ added, $FeCl_3$ added, $CaCl_2$ added, and $NaNO_3$ added. In each batch of experiments, the volume ratio of water-sediment was 5:1 and samples were incubated in light at room temperature. The suboxic tanks were bubbled with N_2 for 2 h every day after which they were closed with a lid. The oxygen concentration was about 1 mg/L. 5 g of each chemical ($Al_2(SO_4)_3$, $FeCl_3$, $CaCl_2$, $NaNO_3$) were added as particles to the upper 5 cm of the sediments. Then the original lake water was gently added to the tank using a siphon. Water samples were replaced with lake water after they were taken for phosphorus flux C_P and Fe flux C_{Fe} analysis. After addition of the salts, efflux measurements were carried out daily. The efflux was calculated from the change in concentrations in the overlying water.

The sequential extraction scheme of TP fractional composition in the sediment is shown in Table 1. O_2 concentrations, temperature and O_2 saturation in the water were measured using a dissolved oxygen meter (Thermo Orion, USA). Concentrations of TN were determined using a BÜCHI 339 Kjeldahl analyzer unit (BÜCHI Analytical, Inc., USA), and TP was measured by colorimetry of a single reagent ascorbic acid after H_2SO_4+$HClO_4$ digestion. Concentrations of NH_3-N were analyzed on a Lachat Instruments flow injection analyzer (Lachat Instruments, a Hach Company Brand, USA). Fe concentration was measured on an atomic absorption spectrophotometer (Analyst 800, PerkinElmer, USA).

Table 1 Sequential phosphorus fractionation

Mark	Extractant/time	Expected P binding form
$P_{Fe,\ Mn}$	0.11 mol/L $Na_2S_2O_4$/$NaHCO_3$/ 0.5 h (40 °C)	Redox-sensitive P, mostly Fe and Mn compounds
$P_{Al,\ Fe}$	0.1 mol/L NaOH/16 h	P metal oxides (Al, Fe)
P_{Ca}	0.1 mol/L HCl/16 h	Ca- and Mg-bound P compounds
$P_{residual}$	Ignition at 550 °C/2 h	Organic P



(a)

(b)

Fig.1 Water effluxes in treated sediments. (a) Accumulated P-release; (b) Accumulated Fe-release. $-O_2$ means suboxic conditions

RESULTS AND DISCUSSION

Comparison with efflux

There was a significant difference in phosphorus fluxes C_p (mg/L) between the treated and untreated sediments. In sediment cores with added chemicals ($Al_2(SO_4)_3$, $FeCl_3$, $CaCl_2$ and $NaNO_3$), the accumulated P-release were generally low compared with those having no additives (Fig.1a). The flux of TP from the sediment in control cores was about five times higher than that of sediments treated with chemicals, and the rate of average TP effluxes was 7.3 mg/(m^2·d). The phosphorus release from sediment is determined by redox-sensitive iron dynamics (Wauer *et al.*, 2005). In anoxia, iron(III) was reduced to iron(II) and subsequently both iron and adsorbed phosphate returned into solution. In sediment treatment with $Al_2(SO_4)_3$, a low efflux of P was observed. However, this was not significantly different from the low efflux in the sediment treatments with added $FeCl_3$, $CaCl_2$ or $NaNO_3$. The mean accumulated P release rates for sediment treatments with $Al_2(SO_4)_3$, $FeCl_3$, $CaCl_2$ and $NaNO_3$ were −0.6, 0.03, 0.6 and 2.6 mg/(m^2·d), respectively.

The sediment treatment with $Al_2(SO_4)_3$ resulted in P uptake from the water phase. Our results indicate that $Al_2(SO_4)_3$ is more efficient in immobilizing P from both the sediment and water phase than $FeCl_3$, $CaCl_2$ or $NaNO_3$. The reason is that sol aluminum and a single- or multi-core of a hydroxyl polymer can be generated by hydrolytic polymerization after $Al_2(SO_4)_3$ is added to water. Sludge is formed through flocculation precipitation. Furthermore, with stronger absorption characteristics, the floccule of $Al_2(SO_4)_3$ is larger than that of $FeCl_3$. The hydrolysis of $Al_2(SO_4)_3$ may reduce the pH value and produce toxic $Al(OH)^{2+}$ and Al^{3+}. However, $Al_2(SO_4)_3$ has three advantages: it is not poisonous in the correct dosage, it is almost irreversibly bundled because of its high phosphorus absorption capacity, and it is more sensitive to oxidation-reduction potential compared with ferric chloride.

A lower P efflux was found in the NO_3^- treatment compared with that in sediment with no additives (Fig.1a). This is because NO_3^- penetrated deeper into the sediment than O_2 and therefore kept more Fe compounds oxidized (Wauer *et al*., 2005a). Under anoxic respiration, NO_3^- has the preferential right to be used as an electron acceptor than sulfate, which also controls the P binding capacity, because of a diminished formation of FeS. Through increasing the supply of nitrate, denitrification is stimulated, organic matter is decomposed, and the chemical energy content of the labile degradable organic matter is lowered. Furthermore, the reduced FeS is oxidized to iron oxide/hydroxide (FeOOH) and absorbs phosphate concentrated in the interstitial water (Zong and Zhang, 2006). Thus, only in the $NaNO_3$ treatment there was no black FeS layer found.

The low rate of phosphorus release in the sediment used by molysite is because there is a negative correlation between the TP concentration in the overlying water and the Fe/P ratio of the sediment. In other words, the greater the Fe/P ratio in sediments, the lower the TP concentration in the overlying water (Jensen *et al*., 1992). To keep phosphate largely within the sediment, the available iron has to reach a sufficient concentration.

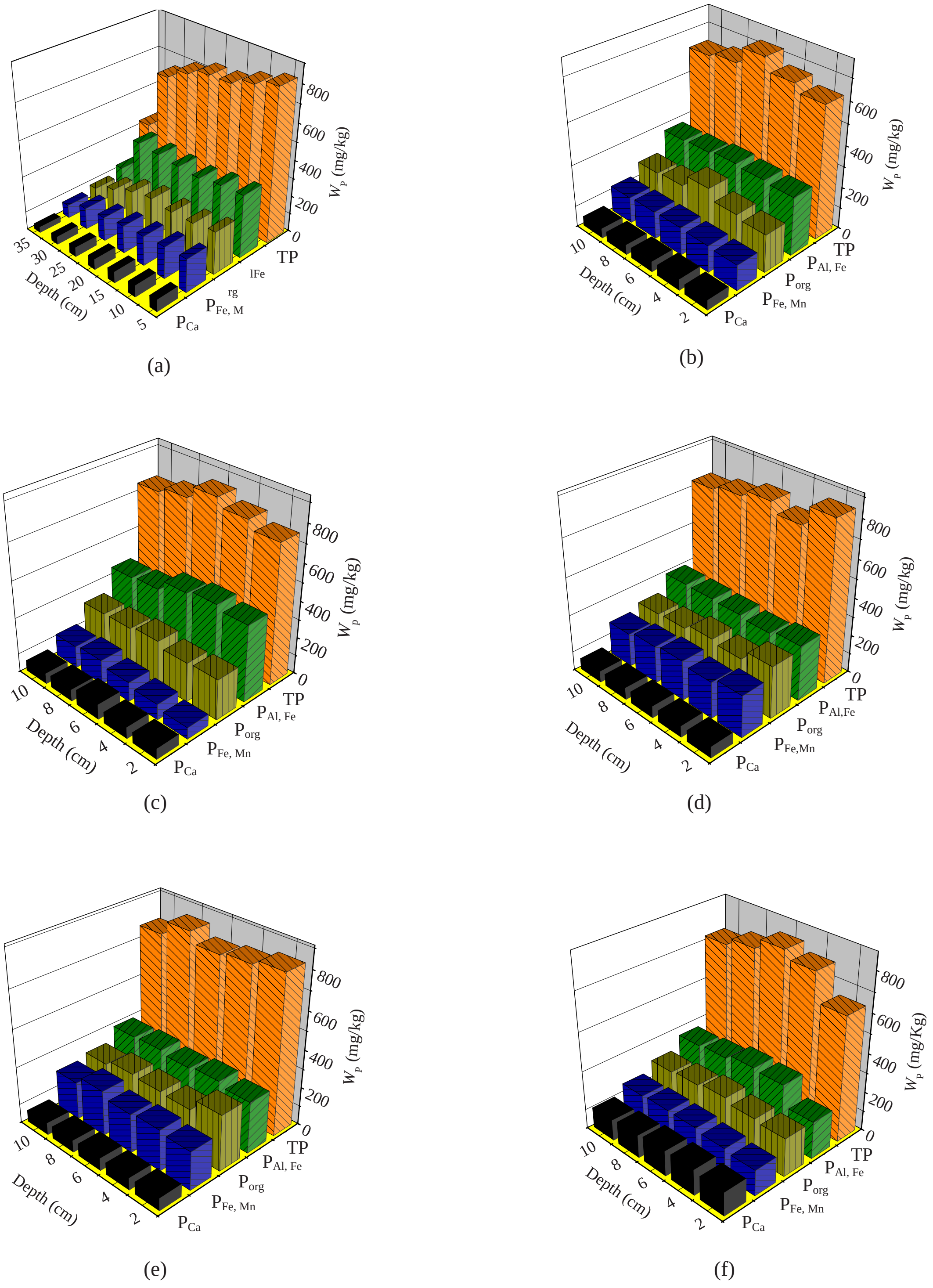
The effect of $CaCl_2$ added to sediment was similar to that of $FeCl_3$. Treatment with Ca^{2+} can prevent phosphorus release because of apatite formation. Thus, we found a low efflux of P in the sediment treatment with $CaCl_2$. Even if the water environment has been changed, phosphorus is not easily released from calcium phosphate.

In the sediment treatment without additives, Fe efflux C_{Fe} (mg/L) showed a moderate increase (Fig.1b) because anoxic conditions lead to a reduction of Fe^{3+} to Fe^{2+}, and Fe^{2+} is released into the water phase. Fe release under anoxic conditions with no additives was significantly higher than those with added $NaNO_3$, $CaCl_2$ or $Al_2(SO_4)_3$ (Fig.1b). This is because iron oxide is susceptible to oxidation-reduction potential (Jensen *et al*., 1992). Lehtoranta and Heiskanen (2003) found that almost all of the iron phosphate in sediments had active properties and could be released in a deoxidized environment, proving that iron phosphate was unstable. This was confirmed by our results. The Fe efflux correlated with the P efflux from the same treatment (Fig.1). The correlation suggests that Fe is the most important compound controlling anoxic P release from sediment.

CHANGES IN SEDIMENT COMPOSITION

Different forms of phosphorus in pretreatment sediment

Analysis of the three fresh sediment cores showed that TP concentration decreased slightly from 835 mg/kg at the sediment surface to 646 mg/kg at 25~30 cm depth (Fig.2a). At 30 cm below the surface of the sediment cores there was a significant drop of 324 mg/kg in TP concentration, which was caused by a change in the sediment composition from fine particulate mud to sand. The average P contents for the 0~35 cm depth sediments were: P_{Ca} 8%, $P_{Fe,\ Mn}$ 17%, P_{org} 28.7%, $P_{Al,\ Fe}$ 42.3%. The concentrations of $P_{Fe,\ Mn}$ and P_{org} declined with sediment depth, while P_{Ca} decreased slightly with depth. At 10~15 cm depth, the $P_{Al,\ Fe}$ concentration was at a minimum and below this depth, the $P_{Al,\ Fe}$ concentration increased with depth. At 30~35 cm depth, the $P_{Al,\ Fe}$ concentration dropped sharply.



(a) (b)

(c) (d)

(e) (f)

Fig.2 Concentrations of different forms of phosphorus before and after treatment of sediment. (a) Untreated sediment; (b) suboxic conditions; (c) $Al_2(SO_4)_3$; (d) $FeCl_3$; (e) $NaNO_3$; (f) $CaCl_2$

Changes in sediment composition

At the end of the incubation period, the characteristics of the sediment cores of the five different treatments were observed. Ochre deposits were found on the sediment surface in the $FeCl_3$ treatments. No black layer was found in the surface sediment of the $NaNO_3$ cores. Only 1 cm deep black deposits were discovered in calcium treatments. In the $Al_2(SO_4)_3$ cores, a black layer of 6 cm was observed. This layer penetrated much deeper into the sediment compared to the other treatments where this layer was present only in the top 1~2 cm. The vertical distribution of P compounds in the sediment at the end of the experiment is given in Figs.2b~2f. In suboxic conditions, the concentration of total phosphorus ($TP_{average}$ 657 mg/kg) in sediment was much lower than those of untreated sediment ($TP_{average}$ 688mg/kg) and treatments with salts ($TP(Al_2(SO_4)_3)$ 793 mg/kg, $TP(FeCl_3)$ 781 mg/kg, $TP(NaNO_3)$ 802 mg/kg, $TP(CaCl_2)$ 747 mg/kg). Therefore, a higher P efflux was found in the treatment without salts as shown in Fig.1a.

The black sediment layer in the $Al_2(SO_4)_3$ treatment resulted from precipitation of FeS. The added $Al_2(SO_4)_3$ contained a high amount of sulfate, which can be reduced to sulfide by bacterial respiration. Sulfide ions can precipitate with Fe^{2+} and form FeS. Fe^{2+} and sulfide may have diffused deeper into the sediment than the depth to which the salts were mixed, thus forming a black FeS precipitate throughout the whole depth of sediment (Zong and Zhang, 2006). We suggest the use of $AlCl_3$ for lake restoration purposes. In the $Al_2(SO_4)_3$ treatment, the P concentration in the $P_{Al, Fe}$ fraction was much higher in the upper 4 cm (441 mg/kg) than that in the other treatments (Fig.2c). This is because P was adsorbed onto Al-oxides more firmly than other compounds. The low P concentrations in the $P_{Fe, Mn}$ fraction can be explained by the fact that Al hydroxides are capable of adsorbing $Fe_3(PO_4)_2$, which is mobilized when Fe^{3+} is reduced under suboxic conditions. Thus, mobile P was immobilized to $P_{Al, Fe}$ with the addition of $Al_2(SO_4)_3$ under anoxic incubation. Fytianos and Kotzakioti (2005) suggested that Al-bound P will appear in the NaOH fraction. This was confirmed in our experiment.

The $P_{Fe, Mn}$ fraction (62 mg/kg) in the surface sediments was much lower in the $Al_2(SO_4)_3$ treatment than that in the other treatments except for the treatment without additives, which showed surface sediments act as redox media. By comparing the control treatment without O_2 with the treatment of adding $NaNO_3$, a clear effect of NO_3^- is observed by an increased concentration of $P_{Fe, Mn}$ (from 203 to 238 mg/kg) in the upper layer of sediment and a significantly decreased concentration of P_{org} (from 295 to 219 mg/kg), indicating that $NaNO_3$ played the role of an oxidant and prevented the reduction of Fe^{3+}. Nitrates also stimulated the denitrification, and promoted the degradation of organic sediment. Organic phosphorus was released to some degree into overlying water by $NaNO_3$ treatment. Thus, the TP in overlying water after $NaNO_3$ treatment was higher than that after aluminum, calcium and iron salt treatment (Fig.1a).

Addition of $FeCl_3$ increased the $P_{Fe, Mn}$ at the sediment surface. By the end of the experiment, the surface of the sediment in the $FeCl_3$ treatment was ochre red, indicating that Fe was still in its oxidized form, thus verifying the importance of oxidized Fe in retaining P in surface sediments. The Fe^{3+} treatments had the highest P concentration (222 mg/kg) in the top surface of the sediment in the $P_{Fe, Mn}$ fraction. This is because a higher amount of available Fe^{3+} can bind more P than the other treatments.

We found that the content of P_{Ca} was the highest (119 mg/kg) after $CaCl_2$ treatment. Adding $CaCl_2$ prevents the liberation of phosphorus because of the formation of minerals, especially apatite. As a result, the P_{Ca} increased at the sediment surface.

While the method of chemical treatment is efficient, it may cause ecological damage in the long term. Bioremediation is slow but causes no pollution to the original ecosystem. If chemical treatment is combined with bioremediation, the aim of environment repair may be achieved (Qin *et al.*, 2006).

CONCLUSION

We found that the addition of salts (ferrous and calcium, and especially aluminum and nitrate) into the sediment can inhibit the transfer of phosphorus in sediments. The composition of sediment in terms of its various forms of phosphorus was changed after chemical treatment. Only treatment with $Al_2(SO_4)_3$ resulted in negative growth of P in the water phase. Chemical treatment with salts, especially aluminum and nitrate resulted in a high capacity for inhibition of endogenous phosphorus release and increased sediment stability.

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