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SEDIMENT ENVIRONMENT AND POLLUTION CONTROL 2022

Higher magnitude of sediment phosphorus release stimulated by ammonium than nitrate

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Abstract

Purpose Human activities have increased the amount of reactive inorganic nitrogen (RIN) in the biosphere. Recently, the effect of RIN on lake ecosystems (e.g., stress to aquatic organisms) has received increasing attention, while only a few studies have investigated the effect of RIN on phosphorus (P) dynamics, which is considered a key element in eutrophication. This study aimed to test the effect of high RIN on sediment P release.

Materials and methods We conducted a two-month RIN-addition experiment in nine 150 L tanks with three treatments: controls (without N addition), nitrate addition treatments ($+NO_3^-$), and ammonium-addition treatments ($+NH_4^+$). $NaNO_3$ and NH_4Cl were added at a concentration of 10 mg L^{-1}.

Results and discussion We found that the total phosphorus concentrations (TP) of the water were significantly higher in the $+NO_3^-$ treatments than in the controls but lower than the TP in $+NH_4^+$ treatments. Meanwhile, labile P at the sediment−water interface indicated minor and significant P release in the $+NO_3^-$ and $+NH_4^+$ treatments, but P uptake in the control. Stimulated alkaline phosphatase activity was likely the dominant mechanism underlying the N-induced sediment P release. The magnitude of P release was higher in the $+NH_4^+$ treatment than in the $+NO_3^-$ treatment, likely because nitrate enhances the oxidation of sediment, counteracting to some degree the P-releasing effect.

Conclusions The findings imply that high ammonium pollution may cause increased risks of eutrophication than the same amount of nitrate; this is important for decision-making to control internal P loading under the scenario of continuous N amplification.

Keywords Eutrophication · Nitrate pollution · Ammonium pollution · Alkaline phosphatase activity · Oxidation of sediment

1 Introduction

Continued amplification of nitrogen (N) cycles induced by intensified human activities is receiving increasing attention. The amount of reactive N from human activities has more than doubled in the past decades (Stevens 2019). Much of this reactive inorganic N (including ammonium [NH_4^+] and nitrate [NO_3^-]) will ultimately be transported into aquatic ecosystems through fertilizer run-off, regional deposition, and wastewater effluents (Xu et al. 2018; Liu et al. 2019). NO_3^- and NH_4^+ are the dominant forms of inorganic N in lakes, as well as the major N pollutants in wastewater discharge (Mallin and Mclver 2012; Zieliński et al. 2013; Kinner et al. 2013; NBSC 2014). High concentrations of NO_3^- and NH_4^+ may cause stress to aquatic organisms, and could change species composition and decrease overall biodiversity (Camargo and Alonso 2006; Stevens 2019). High inorganic nitrogen can also alter the release of sediment phosphorus (P), which is a key nutrient fueling eutrophication in freshwater systems (Schindler et al. 2008; Ma et al. 2018; Kumar et al. 2019).

The dynamic release of sediment P to the overlying water involves two processes: one is the diffusion of pore water P, and the other is the resupply of solid P to pore water P through the release of P from the binding sites of sediment solids (Ding et al. 2015). Such processes are typically affected by environmental factors such as biological activity, water temperature, light, pH, dissolved oxygen (DO), hydrostatic conditions, and N loading (Jiang et al. 2008; Chai et al. 2021; Ma et al. 2021;

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Ding et al. 2022; Zhuo et al. 2023). For instance, the amount of P released from sediment increased under low DO conditions (Jiang et al. 2008; Ding et al. 2022). Chai et al. (2021) revealed that high hydrostatic pressure can promote sediment P release through increasing alkaline phosphatase activity (APA) and P-related bacteria. In an experiment by Ma et al. (2018), NH_4^+ addition stimulated P release and enhanced APA due to an imbalanced N:P ratio was the dominant process stimulating the release. NO_3^- has a similar mechanism to NH_4^+ for promoting P release (Ma et al. 2021). In contrast, nutrient addition experiments revealed that NO_3^- may reduce P release by increasing the redox potential and improving the P binding capacity in the sediment (Ripl 1976; Søndergaard et al. 2000; Ma et al. 2021). NO_3^- has been used as an oxidant with positive effects for restoration interventions in some stratified lakes during periods of hypolimnetic anoxia (Foy 1986; Søndergaard et al. 2000). For example, addition of 24 g N m^{-2} of NO_3^- (equivalent to 1.5 mg N L^{-1} concentration) to Lake White Lough (area 7.4 ha, mean depth 6.2 m), N. Ireland, resulted in a delay and reduction of P release (Foy 1986). Likewise, a large-scale NO_3^- addition experiment conducted in the hypolimnion of eutrophic Lake Lyng (area 10 ha, mean depth 2.4 m), Denmark, showed that a dose of 8–10 g N m^{-2} y^{r-1} (equivalent to 1.2–2.2 mg N L^{-1} in concentration) decreased the concentration of dissolved inorganic phosphorus in the hypolimnion from 1.6–2.9 mg P L^{-1} in untreated years to 0.8–1.2 mg P L^{-1} in NO_3^- treatment years (Søndergaard et al. 2000).

Taken together, P release from sediment likely responds very differently to NO_3^- and NH_4^+ enrichment, and the mechanisms involved need to be further investigated as it is essential to manage water environments and ensure efficient decision-making on N pollution control. We therefore hypothesize that the magnitude of N-stimulated P release from sediment will be more pronounced at high NH_4^+ loading than at same dosage of NO_3^- loading. To test our hypotheses and the underlying mechanisms, we conducted a N enrichment experiment with NO_3^- and NH_4^+ concentrations at 10 mg N L^{-1}.

2 Materials and methods

2.1 Experimental system set-up

We conducted the experiment in nine white cylindrical polyethylene tanks (0.5 m in diameter, 0.7 m in height, 150 L in volume) placed outdoors in Wuhan, China. Well-mixed sediment (total nitrogen [TN], 4.8 ± 0.15 mg g^{-1} dw; total phosphorus [TP], 0.8 ± 0.02 mg g^{-1} dw; organic matter [OM], 64.0 ± 1.4 mg g^{-1} dw) and water (TN, 0.8 ± 0.06 mg L^{-1}; TP, 0.08 ± 0.01 mg L^{-1}) collected in Lake Bao'an (N 30°17′17″; E 114°43′45″) were added to the tanks (Fig. 1). The depths of sediment and water were kept approximately at 0.2 m and 0.5 m, respectively.

2.2 Experimental treatments

Three treatments with three replicates were established: Control with no N addition, 10 mg $NaNO_3$ L^{-1} (+NO_3^-), and 10 mg NH_4Cl L^{-1} (+NH_4^+); $NaNO_3$ and NH_4Cl are

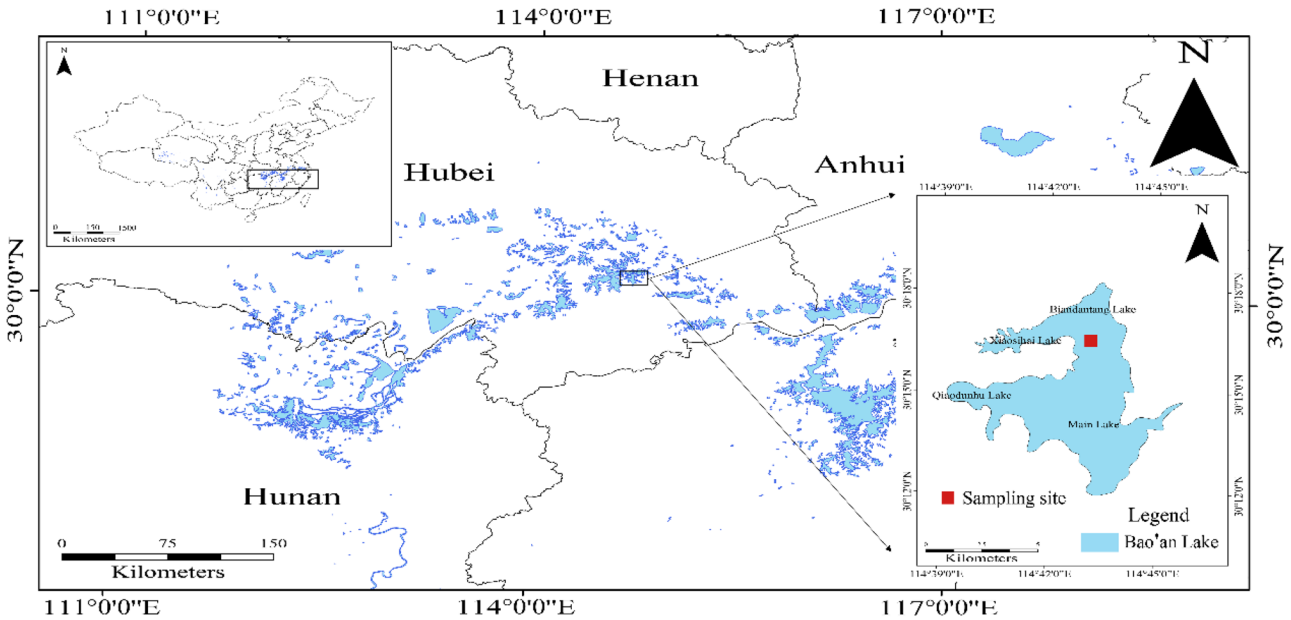

Fig. 1 Location of Lake Bao'an and the sampling site in this study

expressed as nitrogen. NO_3^- concentration of 10 mg L^{-1} represents the China standards for drinking water quality (SAC 2006) as well as the USA federal maximum level for drinking water (Camargo et al. 2005). NH_4^+ concentration of 10 mg N L^{-1} is within the range of Class B in the discharge standard of pollutants for municipal wastewater treatment plants in China (8–15 mg N L^{-1}) (GB-18918–2002). N concentration of 10 mg N L^{-1} is close to the N levels in most urban lakes in Wuhan (Li et al. 2016), although it is much higher than the actual N level in Lake Bao'an. $NaNO_3$ and NH_4Cl (> 99.5%) were added at a target concentration of 10 mg L^{-1} on March 27, April 11, and April 26, 2016. Total doses of 19.5 g $NaNO_3$ and 12.0 g NH_4Cl were applied in the + NO_3^- and + NH_4^+ treatments. The N fertilizer was dissolved with distilled water before injecting it evenly into the tanks at middle water depths by a 150-mL syringe.

2.3 Sampling and analysis

A survey covering four seasons was carried out from February 4 to December 17, 2016 providing the basic characteristics of Lake Bao'an: annual mean DO 9.8 mg L^{-1}, TN 0.8 mg L^{-1}, TP 0.08 mg L^{-1}, and phytoplankton chlorophyll *a* (Chl *a*) 36.7 μg L^{-1}. Water samples were taken three and 15 times before and after fertilization (on March 27); sampling depth was the middle of the water depth (approximately 25 cm above the sediment) and samples were collected with a 150-mL syringe. Well-mixed water (500 mL) was taken to the laboratory for chemical analyses including TN, TP, Chl *a*, and alkaline phosphatase activity in water (APA_{Water}). TN, TP, Chl *a*, and APA_{Water} were determined according to Ma et al. (2018). DO and pH were measured during water sampling with a YSI Pro-Plus (Yellow Spring Inc., USA) at the middle of the water depth. APA in sediment (APA_{Sed}) was monitored six times (approximately every 7 days) during the experiment, once before and five times after fertilization. A column glass tube (5-cm diameter, 20-cm length) was used to collect surface sediments (0–5 cm) for APA_{Sed} analyses. APA_{Sed} was determined according to Ma et al. (2018).

In situ measurement of labile P was performed using Zr-oxide diffusive gradients in thin films (DGT). Eighteen Zr-oxide DGT probes (Easysensor Co. Ltd, Nanjing, China) with standard DGT holders were inserted across the sediment−water interface of each tank on March 28 (pre-fertilization) and April 23 (post-fertilization). The probes were forced 10 cm into the sediment and kept 4 cm above the water surface. After 48 h, the probes were retrieved and

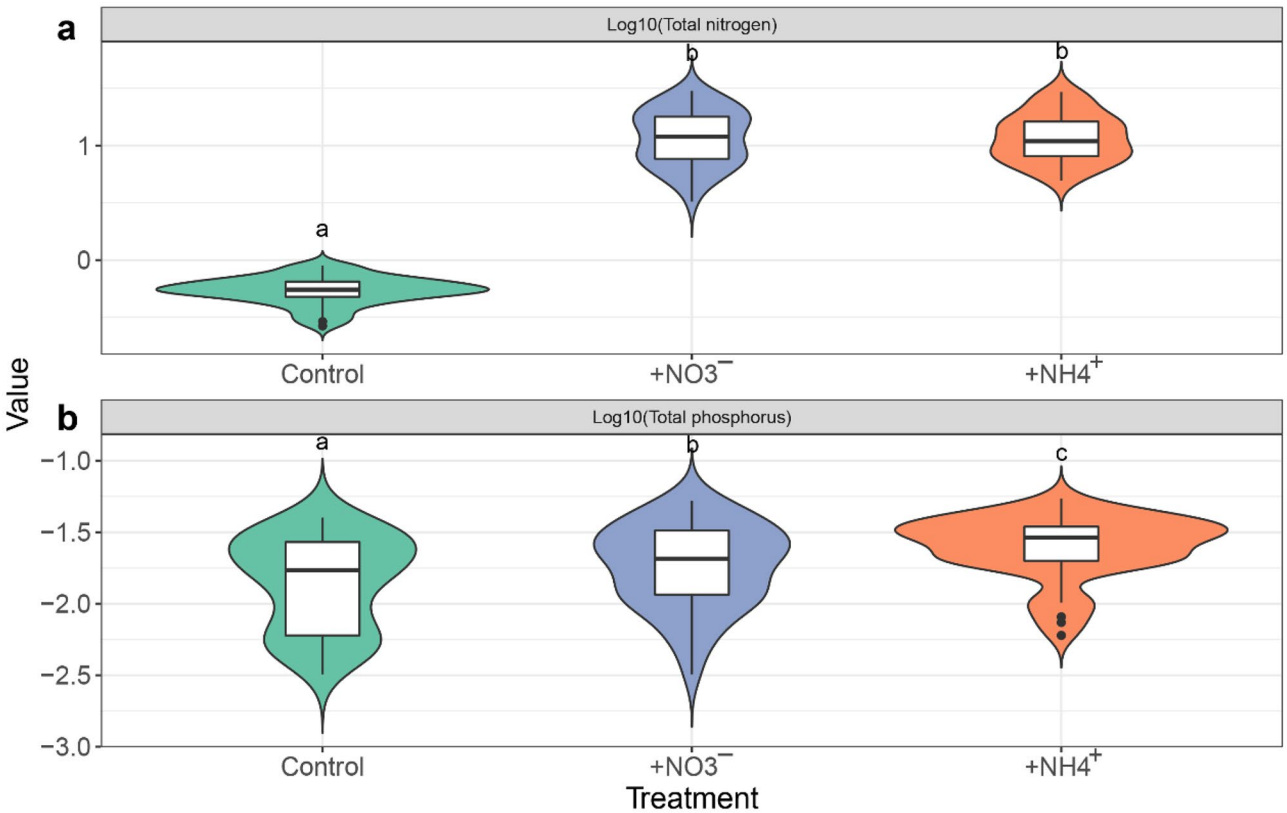

Fig. 2 Violin plots of **a** log10(total nitrogen) and **b** log10(total phosphorus) in water for the experimental treatments

brought to the laboratory for analyses. Further details on the data processing are given by Zhang and Davison (1999) and Ma et al. (2018). The oxide layer at the sediment−water interface was synchronously determined using FireStingGO2 (Pyro-Science, Germany).

2.4 Statistical analyses

To analyze the effects of N forms on P release, the experimental period was divided into two stages according to the time of fertilization, i.e., pre-fertilization (March 16–March 26) and post-fertilization (March 27–May 9). Excel 2010 and Origin 9.0 software were used for basic drawing. We used the percentages of increased DGT-labile P (PI) to determine the effect size of the N addition on the measured sediment P release because they equally weigh the negative and positive responses and facilitate statistical analysis (Ma et al. 2018). PI was calculated as the Δ values of DGT-labile P (the post-fertilization values minus the pre-fertilization values) divided by the pre-fertilization values of DGT-labile P. Positive values of PI represent promoted P release, whereas negative values indicate inhibited P release. To test the difference between treatments, the Friedman test (*F*-test) was made utilizing R. Wilcoxon–Nemenyi–McDonald–Thompson post-hoc tests (WNMT-test) were used if the Friedman test resulted in a significant *p* value (< 0.05) (Ma et al. 2018).

3 Results

In the pre-fertilization period (March 16–March 26), no statistical difference was found among the treatments for DO, TP, Chl *a*, and APA_{Sed} (all *p* values are presented in Table 1). However, TN was slightly higher and APA_{Water} was slightly lower in $+NO_3^-$ treatments than in the controls (Table 1), but the overall pre-fertilization data demonstrated comparable initial conditions for all the treatments. For the post-fertilization period (March 27–May 9), the average TN in the $+NO_3^-$ treatments was higher than in the controls, but similar to the $+NH_4^+$ treatments (Fig. 2a, Table 1). TP was significantly higher in $+NO_3^-$ treatments than in the controls, but lower than in the $+NH_4^+$ treatments (Fig. 2b, Table 1). TP was significantly higher in $+NH_4^+$ treatments than in the controls (Fig. 2b, Table 1).

As presented in Fig. 3, overlying water and sediment exhibited similar dynamic patterns for DGT-labile P. For the control,

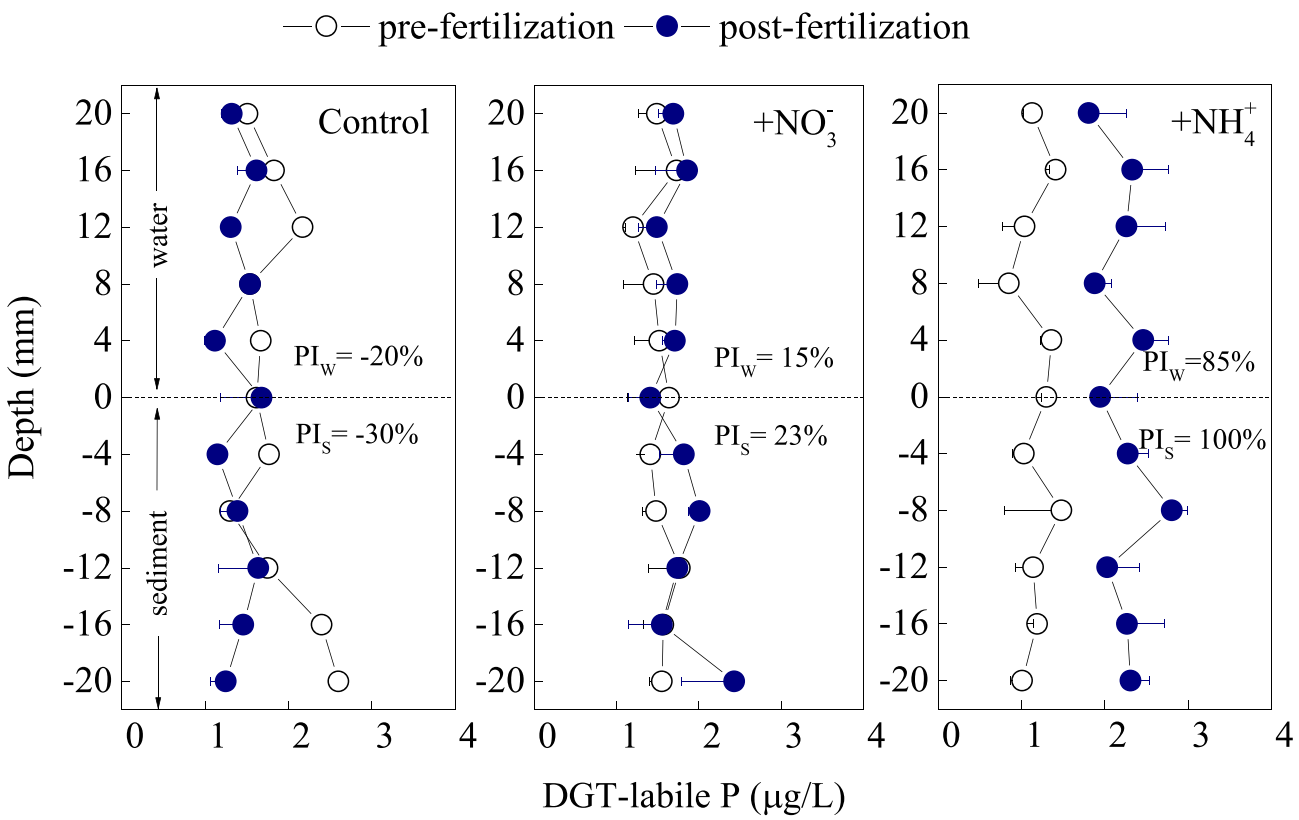

Fig. 3 The distribution of DGT-labile phosphorus (mean ± SE) in overlying water (0 to 20 mm) and sediment (–20 to 0 mm) profiles monitored in situ in pre-fertilization and post-fertilization for various treatments. The dashed lines indicate the sediment−water interface. The data above and below the dashed line represent the percentage of increased DGT-labile phosphorus (PI) in pre- compared with post-fertilization. Positive values suggest P release, while negative values suggest P uptake

the overall PIs averaged − 21% (ranging from − 39% through 0%) for overlying water and − 30% for sediment (ranging from − 52% to 7%), suggesting P uptake. In the $+NO_3^-$ treatment, the overall PIs had an average of 15% (ranging from 8 to 16%) for overlying water and 23% for sediment (from − 2% to 57%), suggesting P release. Positive and higher PIs were found in the $+NH_4^+$ treatment. The overall PI averaged 85% (60% to 121%) for overlying water and 100% for sediment (78% to 130%), suggesting significant P release.

During the entire post-fertilization period, DO in the $+NO_3^-$ treatment was higher than that in the control or $+NH_4^+$ treatment (Figs. 4a and 5a, Table 1). The thickness of the oxide layer at the sediment−water interface followed the same trend as DO, and was significantly higher in the $+NO_3^-$ treatment than in the control and $+NH_4^+$ treatment ($+NO_3^-$ > control > $+NH_4^+$) (Fig. 4b). Chl *a* in the $+NO_3^-$ treatment was not different from the control and was significantly lower than in the $+NH_4^+$ treatment (Fig. 5b, Table 1). Throughout the post-fertilization period, APA_{Water} in the $+NO_3^-$ treatment was not significantly different from the control, although it tended to be higher after nitrate addition; APA_{Water} was significantly higher in the $+NH_4^+$ treatment than in the control (Fig. 5c, Table 1). APA_{Sed} was higher in the $+NO_3^-$ than in the control, but not different from $+NH_4^+$ (Fig. 5d, Table 1).

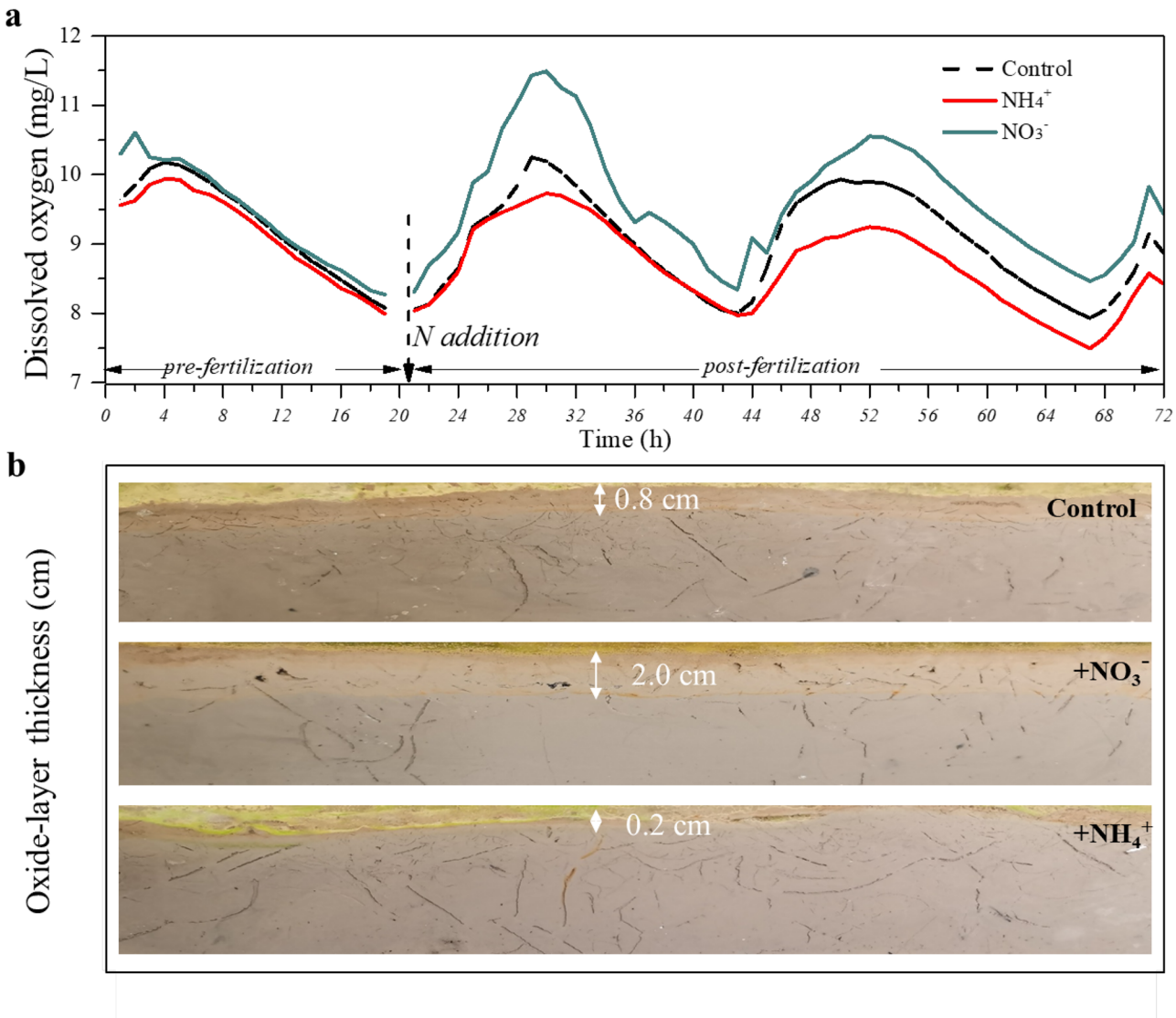

Fig. 4 **a** Changes in dissolved oxygen concentration in overlying water (continuous monitoring with a frequency of 20 min) and **b** the oxide layer thickness at the sediment–water interface for various treatments during the experiment. The black single arrow indicates the time of nitrogen addition; the white double arrows indicate the thickness of the sediment oxide layer

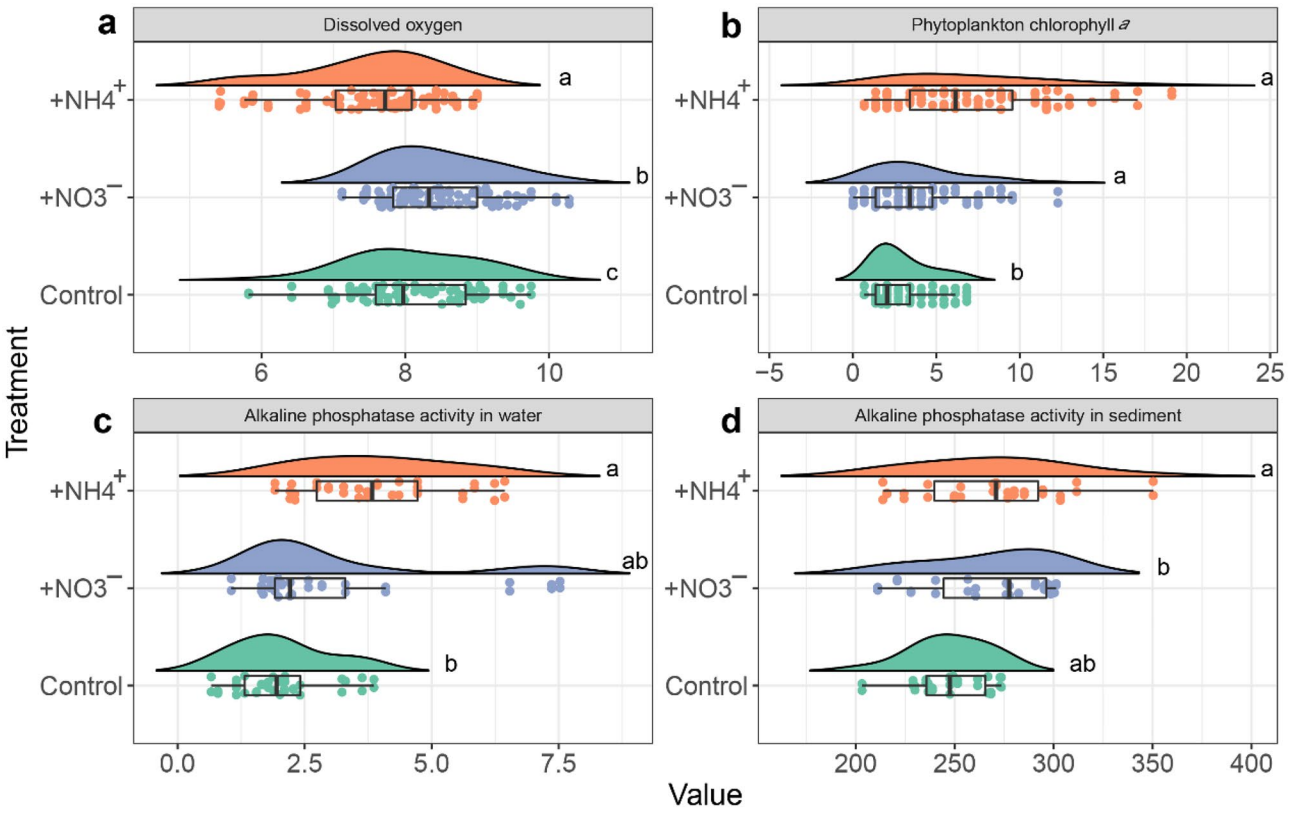

Fig. 5 Raincloud plots of **a** dissolved oxygen (mg L^{-1}), **b** phytoplankton chlorophyll a (μg L^{-1}), **c** phosphatase activity in water (μg P L^{-1} h^{-1}), and **d** phosphatase activity in sediment (μg P g^{-1} h^{-1}) among different treatments during the experiment

4 Discussion

Our 2-month tank experiment revealed that the increase of TP was most apparent in + NH_4^+ treatments, followed by + NO_3^- treatments, whereas it was almost unnoticeable in controls. Similarly, for DGT-labile P, noticeable increases in + NH_4^+ treatments and minor increases in the + NO_3^- treatments were observed in the sediment−water interface. The increases in TP and DGT-labile P suggest that high NO_3^- and NH_4^+ (10 mg N L^{-1}) promote sediment P release. However, the amount of P released was considerably higher in the + NH_4^+ treatment (1.56 times that of the control) than in the + NO_3^- treatment (1.25 times that of the control), suggesting that NO_3^- and NH_4^+ may involve different mechanisms altering sediment P dynamics.

The increase in water and sediment APA following NH_4^+ addition may be the key factor promoting enhanced P release, resulting in the observed higher TP. The involvement of APA in enhanced P release by breakdown of organic P has been extensively studied in terrestrial, marine, and freshwater ecosystems, and is one of the most important mechanisms used by organisms to adapt to high N:P stress (Zhou et al. 2011; Bogé et al. 2012; Ma et al. 2018; Li et al. 2021). Organic P is often known as an important P component in water environments (Feng et al. 2023), and its enrichment can facilitate P release through enzymatic hydrolysis and anaerobic desorption (Ma et al. 2023). In addition, the decrease in DO accompanying the loss of the oxide layer at the sediment−water interface, perhaps caused by the oxygen-consuming process of NH_4^+ decomposition, may also contribute to the observed P release. NO_3^- also promotes P release through the same mechanisms (APA production) as NH_4^+, but also is inhibitory by increasing DO and oxidizing the top sediment (2.5 times that of the control), contrary to the observations in the + NH_4^+ treatment. The oxidative effect of NO_3^- may partly counteract the amount of P released, resulting in a lower release of P in the + NO_3^- than in the + NH_4^+ treatment. Notably, the growth of algae (indicated by Chl *a*) stimulated by NO_3^- was significantly lower than that of NH_4^+ at the same N dose. This was likely because NO_3^- is less available to phytoplankton because the cells must first convert NO_3^- to NH_4^+ using the enzyme nitrate reductase (U.S. EPA 1999; Ou et al. 2014). The higher P release, together with the higher algae growth in + NH_4^+ than that of + NO_3^-, suggested that high NH_4^+ pollution poses a higher eutrophication risk than NO_3^- pollution.

Usually, NO_3^- addition acts as an electron acceptor and can inhibit sediment P release (Andersen 1982; Wauer et al. 2005), which differs from the observations

in this study. The different finding may reflect the dose-dependent effects of NO_3^- as well as oxygen-dependent conditions in the studied lakes. The dose of 10 mg N L^{-1} used here is much higher than the dose reported to inhibit sediment P release. For example, a NO_3^- concentration of approximately 2 mg N L^{-1} was found to completely inhibit sediment P release in an enclosure experiment (diameter: 10 m; depth: 8 m) conducted in Lake Dagow, Germany (0.3 km^2 in area, N 53°10′, E 13°02′) (Wauer et al. 2005). A field investigation of 31 lakes showed that P release was significantly reduced in deep and shallow lakes where the concentration of NO_3^- exceeded approximately 1 mg N L^{-1} and 0.5 mg N L^{-1}, respectively (Andersen 1982). Low doses of NO_3^- applied in previous case studies were predicted to prevent phytoplankton growth and APA production, and to effectively reduce the release of sediment P (Søndergaard et al. 2000). Moreover, NO_3^- in previous studies was added into the hypolimnion where effects such as stimulating algae and increasing APA could not occur (Foy 1986; Søndergaard et al. 2000; Cubas et al. 2014). In contrast, NO_3^- in our study was added into overlying water, and all tanks maintained relatively high oxygen conditions (7.8–9.9 mg O_2 L^{-1}) typical of shallow polymictic lakes, making redox-sensitive retention less pronounced.

To summarize, both NH_4^+ and NO_3^- might promote sediment P release through increasing APA, but the magnitude of P release was more pronounced for NH_4^+ than NO_3^- when applied in similar concentrations. This is likely because the oxidative effect of NO_3^- partly counteracts the amount of P released. Our findings suggest that high NH_4^+ pollution poses a higher eutrophication risk than NO_3^-, which is of relevance when determining N reduction actions needed for mitigating eutrophication. Although we tried to make the artificial and natural lake systems as close as possible, there was no mixing in our experiment and therefore likely a stronger oxygen gradient near the bottom than in natural shallow lakes. Consequently, large-scale field experiments are needed to fully clarify the coupling relationship between N and P.

5 Conclusions

NH_4^+ and NO_3^- addition can promote sediment phosphorus release, which was demonstrated through the increase in TP and DGT-labile P in both the N addition treatments. However, the magnitude of P release from sediment was more pronounced at high NH_4^+ loading than in high NO_3^- loading. NO_3^- may not only have a promotion mechanism through producing alkaline phosphatase (as for NH_4^+) but also has a retention mechanism as a result of increasing the thickness of the anaerobic layer at the sediment−water interface (as opposed to NH_4^+). Inhibiting effects likely offset the slightly stimulating effects, resulting in a weaker promotion by NO_3^- addition on the net sediment P release. Mesocosm experimental ecosystems typically lack critical whole ecosystem features (e.g., hydrological complexity and wide-ranging organisms), which may make it difficult to extrapolate and compare mesocosm-findings with those in natural lakes. Further studies with broader nitrogen loadings and at a larger scale under natural conditions will increase our understanding of the role of N loading on sediment phosphorus release in shallow lakes.

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Data availability Data will be made available on request.

Declarations

Competing interests The authors declare no competing interests.

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