



Mechanisms of the photochemical release of phosphate from resuspended sediments under solar irradiation



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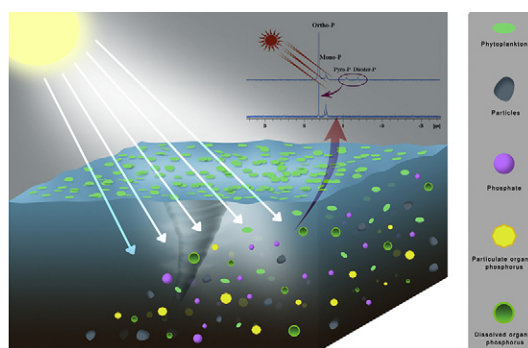
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HIGHLIGHTS

- Dissolved P release increased when resuspended sediments were exposed to simulated solar irradiation.
- The fraction and composition of organic phosphorus has been changed after solar irradiation.
- OH plays an important role in PO_4^{3-} photo-released.
- Photochemical processes offer the potential to PO_4^{3-} released from sediment in the aquatic environment.

GRAPHICAL ABSTRACT



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ABSTRACT

In previous studies, resuspended sediments that were exposed to simulated solar irradiation could release dissolved phosphate (PO_4^{3-}). However, the mechanisms of phosphate release remain unclear. In this research, a battery of experiments was performed to reveal the mechanisms of the photochemical release of phosphate from resuspended sediments of a shallow eutrophic lake under solar irradiation. The results show that the PO_4^{3-} released in resuspended sediments was significantly higher than that in the dark control or in water alone after treatment with solar irradiation for 6 h. The results of sequential chemical extractions showed that the concentrations of labile organic, moderately labile organic and residual organic phosphorus decreased in the resuspended sediment after 6 h of solar irradiation; of these, moderately labile organic phosphorus was the greatest contributor to the release of dissolved phosphate in resuspended sediment. Orthophosphate, phosphate monoesters, phosphate diesters and pyrophosphate were detected with ³¹P NMR. It is worth mentioning that the diester-P and pyro-P species disappeared after 6 h of irradiation. In addition, enzyme activity and radical trapping experiments were applied to identify the roles of biomineralization and photochemical degradation during phosphate release from resuspended sediments under solar irradiation. The amount of PO_4^{3-} released in fresh sediment was greater than that in the autoclaved sediment, which should be attributed to the higher alkaline phosphatase activity in the fresh sediment. However, the PO_4^{3-} released from the photochemical degradation of organic phosphorus is the primary phosphate source during sediment resuspension under 6 h of solar irradiation. The phosphate photorelease was inhibited when methanol was added to the suspension and decreased

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significantly when the concentration of methanol was increased from 0.5 M to 2.0 M. All of these results suggest that photochemical processes may lead to PO_4^{3-} release from sediment in aquatic environments.

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1. Introduction

Phosphorus, an essential element for organisms in aquatic environments, has been recognized as the most significant nutrient that influences the trophic status of a lake (Conley et al., 2009; Meinikmann et al., 2015). Internal phosphorus in the sediment is a main component of phosphorus in the aquatic environment and a significant source of phosphorus (Qin et al., 2015; Zhu et al., 2015). Phosphorus stored in sediment can be released to the overlying water through physical, chemical and biological processes, such as the diffusion of surface sediment, mineralization or sediment resuspension (Gardolinski et al., 2004; Jensen et al., 2006; Li et al., 2011; Kim et al., 2015). Sediment resuspension is a frequent event in shallow lakes, resulting in the direct exchange of phosphorus with the water column due to its immediate contact with the overlying water; this mechanism could represent an important pathway of phosphorus cycling (Morin and Morse, 1999).

A recent study demonstrated that dissolved nutrients could be released when resuspended marine and estuarine sediments were irradiated by sunlight (Kieber et al., 2006; Mayer et al., 2009; Helms et al., 2014). In a suspension constituted with autoclaved sediment (<30 μm) and filtered sea water, more dissolved PO_4^{3-} was released under solar irradiation than in dark conditions. Because biological effects had been eliminated, the increase in PO_4^{3-} in the suspension was attributed to the photochemical decomposition of organic phosphorus from sediments (Southwell et al., 2010). These findings suggested that photocatalysis had a significant impact on organic phosphorus production into the surface waters, leading to the increase of the concentration of available phosphorus (Southwell et al., 2011). These earlier studies have found that photochemical processes play an important role in nutrient recycling in aquatic environments, but there is scant research of environmental controls and mechanisms of the phosphate released from resuspended sediments under solar irradiation.

The photochemical transformation of organic matter in the environment includes direct and indirect photolysis. Sometimes, organic phosphorus itself can absorb solar energy and direct photolysis (Nowack, 2003; Lesueur et al., 2005); this phenomenon is correlated with the structure and morphology of organic phosphorus. More commonly, as shown in previous work, organic phosphorus can decompose to release PO_4^{3-} by reactions with reactive oxygen species (ROS) (Senthilnathan and Philip, 2011; Li et al., 2015), such as hydroxyl radicals ($\bullet\text{OH}$) (Keen et al., 2014; Xie et al., 2015; He et al., 2015). Natural constituents of water, such as nitrate (NO_3^-) and iron ions (Fe^{3+}), can take part in the photodecomposition of organic matter in natural water (Kim and Zoh, 2013). For example, NO_3^- and Fe^{3+} can produce $\bullet\text{OH}$ in water when excited by solar light (Passananti et al., 2013; Shah et al., 2015; Boucheloukh et al., 2012); these radicals can mediate the photochemical decomposition of organic matter in the natural environment. As far as we know, the relation between the produced $\bullet\text{OH}$ and the PO_4^{3-} released from organic phosphorus photodegradation during sediment resuspension under solar irradiation has not been reported.

Here, we undertook a detailed study of PO_4^{3-} release from resuspended sediments of a shallow eutrophic lake, under simulated solar irradiation. The initial concentration of the resuspended sediment, the particle size of the sediment and the radiation spectrum were considered when the effects of environmental parameters on the photorelease of PO_4^{3-} were explored. Sequential chemical extractions and ^{31}P NMR were used to investigate variations in the phosphorus speciation during sediment resuspension under solar irradiation; these techniques are useful in understanding the major forms of phosphorus and the pathways of phosphorus photorelease. In addition, enzyme activity and

radical trapping were applied to identify the biomineralization and photochemical degradation of the phosphate released from resuspended sediments under solar irradiation. The results will be useful to understanding the phosphorus cycle in aquatic environment.

2. Experimental

2.1. Sample collection and processing

The water and sediment used in the experiment were collected from Lake Nanhu in Wuhan, China, on May 16, 2015, and the specific sampling location is shown in Fig. S1. Lake Nanhu is a shallow, hypertrophic lake with an average depth of 1.5–3 m that receives urban rainwater and is polluted by municipal wastewater. The surface sediment was collected from the lake using a Peterson dredge, put into clean plastic bags, and transported back to the lab; the overlying water was collected at the same site with high-density polyethylene bottles. The properties of the water and sediment in Lake Nanhu are summarized in Table 1. Then, in the lab, the five sediment samples were mixed, and a part of the mixed sediment was autoclaved at 115–120 °C for 30 min. The mixed lake water was stored at 4 °C after being filtered with a 0.2 μm polysulfone filter (Supor-200, PALL) to remove microbes and suspended particulate.

2.2. Experimental procedures and analytical methods

2.2.1. Experimental setup

In total, 2 g of autoclaved sediment was added to the filtered lake water, which was then transferred into a 1 L beaker; the sediment was completely resuspended using a stirrer. The suspension was allowed to settle for 5 min to remove the coarse sediments. The top 20 cm of the suspension was then decanted into high-density polyethylene bottles to be used in our experiments. The particle size of the sediment that was transferred in the suspension was <10 μm , and the particle size distribution is shown in Fig. S2. The photolysis experiments were carried out in a rotating photoreactor, which is an instrument that has been used widely in photochemical experiments (Xie et al., 2014). The suspension was placed into quartz sample tubes with magnetic stirrers. A 500 W xenon lamp was put in the center of a cold hydro pump that circulated cooling water, and the temperature of photoreactor was 35 ± 2 °C. Then, the quartz tubes were put into the photochemical reaction instrument. The average light intensity on the surface of the quartz tubes was approximately 130 ± 5 mW cm^{-2} according to a light intensity test instrument (Newport Corporation, Beijing, China), which was approximately the same light intensity on the lake surface water on a midsummer day. The samples (5 mL) were collected at scheduled times and filtered with a 0.45 μm microfiltration membrane. The concentration of dissolved phosphate was determined using the molybdenum blue method.

2.2.2. Effect of environmental parameters

To determine the effect of the resuspended sediment concentration on the amount of PO_4^{3-} released under solar irradiation, the suspension was prepared by adding 1, 2 and 3 g of autoclaved sediment to filtered lake water. The resuspended sediment in the suspension was quantitated with a 0.45 μm glass fiber membrane, and the concentration of the sediment remaining in the suspension was 33, 73 and 101 mg/L, respectively. To determine the effect of the sediment particle size on the phosphate released from the resuspended sediment, the suspension residue in the fraction of particles smaller than 10 μm was consecutively filtered with 20 μm sieves to remove the >20 μm sediment particles and obtain

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