Research and application of in-situ control technology for sediment rehabilitation in eutrophic water bodies
Bo Liu, Xuegong Liu, Jie Yang, David E. J. Garman, Kai Zhang and Honggui Zhang

ABSTRACT

Phosphorus (P) is often the limiting nutrient for algal growth, and P in sediments can be released under suitable conditions. To control P release, in-situ control technology with lanthanum (La) modified bentonite clay (Phoslock®) was proposed and its effectiveness was tested and evaluated both in laboratory and field trials. The results of static and dynamic simulation experiments under different environmental conditions showed that with the application rate of Phoslock® at 0.5 kg/m², the orthophosphate (PO₄-P) concentration of the overlying water decreased to a low level (<0.02 mg/L) within 10 days. Even under anaerobic and high pH (pH = 9.0) conditions, the phosphate release suppression efficiency reached 98.3%, and the P-release rate was −8.20 mg/m² d (negative value indicates P adsorption by Phoslock®). The monitoring data of the field sediments rehabilitation project were consistent with the results achieved in laboratory experiments, thus showing that the application of Phoslock® could inhibit the internal P release effectively.

Key words | eutrophication, in-situ control technology, lanthanum-modified clay, Phoslock®, phosphorus, sediment

INTRODUCTION

Nowadays, many water bodies in China and elsewhere in the world are eutrophic and experiencing an increased number of harmful cyanobacteria blooms (Hart et al. 2005). Because of the prevalence of toxic algal blooms, high priority is given to the control of phosphorus (P) inputs to water bodies, but algal blooms may continue to occur long after P inputs have ceased due to remobilization of P-bound to sediments (Gardolinski et al. 2004). P has been recognized as the most critical nutrient limiting lake productivity, as the trophic status of the water body is usually dependent on the P-concentration in water. In addition, the trophic development of the ecosystem is also influenced by P in sediments, which usually act as a sink of P. However, the concentration of P in sediments is often 1,000-fold higher than that in the water column, and sediments may become a potential P source under certain environmental conditions, mainly controlled by redox status and pH values inducing P release from the inorganic P pool by desorption processes or from the organic pool by mineralization (Jensen & Andersen 1992; Kaiserli et al. 2002; Zhou et al. 2005).

In recent years, to control P release from sediments, the application of ‘capping’ materials to contaminated sediments has been chosen as a low-cost alternative to conventional off-site technologies, such as dredging (Jacobs & Forstner 2003). The concept of capping sediments in-situ involves the placement of a cover over the sediment to seal it off and minimize release of contaminants to the water column. The cover material may simply provide a physical barrier over the sediment (e.g. sand), or may provide an active barrier, which is capable of actively demobilizing contaminants in the pore water by adsorption or precipitation processes (Hart et al. 2003; Gibbs et al. 2011). Thus Phoslock® was invented and developed for P-removal in the water column and P-inactivation in sediments (Groves 2010; Gibbs et al. 2011).

Sorption and desorption of P in sediments has been well investigated, because of the importance in regulating P concentration in the water column (Wang et al. 2009; Haghseresht et al. 2009). In contrast, the monitoring and analysis involved in the transformation of different P-forms in sediments after in-situ treatment remain poorly understood and such studies have seldom been carried out. Moreover, the
majority of sediment P-release studies have concentrated on the influence of single factors, that could not represent the real conditions in nature; consequently, studies on combined effects of multiple factors have been largely ignored.

The purpose of this study was to investigate the different P-forms present in sediments, analyse their possible contributions to the P-loadings of the system, analyse the transformation of P-forms and potential toxicity effect on the aquatic ecosystem after in-situ treatment of Phoslock®, and finally evaluate the effectiveness in both laboratory and field trials.

**MATERIALS AND METHODS**

**Study site**

The ALA artificial river, a relatively closed water body, with a length of 13 km, width of 20 m and depth of 2.5 m, is located in the Airport Logistics Area of Tianjin, China. Parts of the river have suffered from a high input of nutrients, mainly runoff from industrial–residential–commercial areas and flow from untreated sewage. As a result, cyanobacteria blooms have occurred yearly and the sediments have been substantially contaminated, especially with P. Both its cross-sectional dimension and the resulting data (Table 1) are representative of eutrophic artificial water bodies of North China. A water environment treatment project was carried out in this river in 2010, with a sewage interception project pursued firstly. Considering the cost and the environmental effects of dredging and disposal of the material that be removed, an in-situ control technology, Phoslock®, was selected to combat the high internal P-loading in a part of the river, with a dimension of 400 m (L) × 20 m (W) × 2.5 m (D).

**In-situ treatment materials**

Phoslock®, a kind of bentonite clay (~95%) modified with the rare earth element lanthanum (La, ~5%), is a new and effective P-inactivation product which was developed by the Commonwealth Scientific and Industrial Research Organization (CSIRO), Australia. La toxicity and the availability of its free form were dramatically reduced by incorporating the La ions into the structure of a high exchange capacity mineral (bentonite). La is adsorbed onto sites within the bentonite and becomes the active compound that removes phosphate (Equation (1)).

\[
\text{La}^{3+} + \text{PO}_4^{3-} = \text{LaPO}_4
\]

As a general rule, Phoslock® is applied at the rate of 100:1, i.e. 100 g Phoslock® is required to remove 1 g SRP (Groves 2010). But, dosage for each individual water body may be different, for historical physicochemical conditions and aquatic ecosystem of water bodies vary a lot.

**Sampling and chemical analysis**

Laboratory experiments were undertaken over a 1 yr period: from September 2009 to August 2010. Sediment samples were collected in August 2009 in the study site.

Sediment samples were collected from the top 10 cm layer using a cylinder sampling device. The samples were taken to the laboratory in air-sealed bags. To exclude the effects of biological activity on P release, sterilization was conducted under air-dried, homogenized conditions, after which the samples were passed through a 150 μm sieve.

For P determination, samples were digested by a combination of concentrated H₂SO₄ and HClO₄ acids, and concentrations were determined as per the procedure described for water. Water samples were collected 0.5 m below the water surface.

La concentrations were determined by the trichloroarsonazo spectrophotometric method (Sun et al. 1990).

All samples of the experiments were analysed in triplicate, and the data presented in the figures were averages, meanwhile, standard deviation for n = 3 was calculated (and shown in subsequent figures).

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Water and sediment analysis data of the study site</th>
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<tr>
<td><strong>Time</strong></td>
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<tr>
<td></td>
<td>SRP a (mg/L)</td>
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<tr>
<td>The year of 2009</td>
<td>0.865–2.090</td>
</tr>
<tr>
<td>Before application</td>
<td>1.024</td>
</tr>
</tbody>
</table>

*SRP, soluble reactive phosphorus; TDP, total dissolved phosphorus; TP, total phosphorus.*
**P-fractionation**

The long-term contribution of sediment bound P in promoting eutrophication can be more effectively evaluated on the basis of different P-fractions instead of total phosphorus (TP), for the concentration of TP in sediments cannot predict the potential ecological danger. P-fraction in sediments, a kind of sequential extraction method (Figure 1), was based on Psenner fractionation modified by Hupfer (Hupfer et al. 1995). The sediments were extracted with a series of extractants (NH₄Cl, bicarbonate–dithionite (BD), NaOH and HCl), resulting in the following P-fractions: NH₄Cl-P, redox-sensitive P (BD-P), NaOH-P, HCl-P and Res-P (occluded P). The extracts were centrifuged, and the supernatants were filtrated through 0.45 μm filter membrane. Both soluble reactive phosphorus (SRP) and total dissolved phosphorus (TDP) in each sample were determined by the molybdenum blue method (GB11893-89), and concentrations of non-reactive P (NRP) were calculated as the difference between TDP and SRP (Hupfer et al. 1998).

Of the P-forms in sediments, Bio-available P (BAP) has been defined as the sum of immediately available P and the P that can be transformed into an available form by naturally occurring processes (Boström et al. 1988). The BAP has been estimated by the sum of NH₄Cl-P, BD-P and NaOH-P (Stone & English 1995). Among them, NH₄Cl-P is the dominant form of P in the interstitial water, which is loosely adsorbed to the sediment surface, and is, immediately available P (Hupfer et al. 1995), while BD-P represents the redox-sensitive P fraction, mainly including P bound to Fe-hydroxides and Mn-compounds (Kleeberg & Gruenberg 2005), which is considered as a potentially mobile pool of P (Rydin 2000). The NaOH-P fraction is the P bound to metal (hydr)oxides, mainly of Al, which is exchangeable with OH⁻, anion of organic ligands and inorganic P compounds soluble in bases (Stone & English 1995; Rydin 2000).

**Static simulation experiment design**

The sieved sediments were mixed thoroughly and 15 L sediment–water mixture was measured and put into each tester for the release experiment. The overlying water was obtained through the microporous filtering film and then slowly flowed into the testers. Overlying water was adjusted to different temperatures (5°C, 15°C, 20°C, 30°C) and pH (pH at 6.0, 7.0, 9.0 and 11.0) with NaOH and HCl. Additionally, by adding oxygen and nitrogen, dissolved oxygen (DO) was controlled at 7–8 mg/L (aerobic) and 0–1 mg/L (anaerobic). Parts of the experiments were carried out with the treatment of Phoslock® (details of groups description see Table 2, and three replicates were pursued per experiment), and La concentrations were measured.

**Dynamic simulation experiment design**

Five grams of sieved sediment were measured and put into each centrifuge tube (100 ml, with cover), and then Phoslock® was added in. Finally the 50 mL phosphate

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**Figure 1** | Sequential extraction scheme of P in sediments.
solutions were injected with the concentration at 1.0 and 0.05 mg/L, concurrently, DO was controlled at 7–8 mg/L (aerobic) and 0–1 mg/L (anaerobic) by adding oxygen and nitrogen respectively, and the overlying water was adjusted to a nominated pH (pH at 7.0 and 9.0) with NaOH and HCl (details of scheme design see Table 3). All tubes were put into the isothermal oscillator (25 ± 1°C, 200 rpm), and were removed at different time intervals. Afterwards, the extracts were centrifuged, and the supernatants filtered through 0.45 μm filter membrane and SRP was analysed. The residual sediments were air-dried and sequential extraction of P was carried out.

Field trial

Based on the results of a series of laboratory experiments, the pilot project was pursued in April 2010; with the dose rate of Phoslock® at 0.5 kg m⁻², La concentrations were monitored with time, and samples were taken at different time intervals to analyse the P concentrations in water column and P-forms in sediments.

Statistics

Release rates of P

The rates of P release from sediments were calculated according to Equation (2),

\[ r = V(C_n - C_0) + \sum_{j=1}^{n} U(C_{j-1} - C_a), \quad R = r/tA \]  (2)

where \( r \) (mg) is the release quantity; \( V \) (L) is the volume of overlying water; \( n \) is the analysed time; \( C_n \) (mg/L) is the PO₄-P concentration measured every time; \( C_0 \) (mg/L) is the original PO₄-P concentration; \( C_a \) (mg/L) is the PO₄-P concentration after water refill; \( U \) (L) represents the sample volume every time; \( R \) (mg/m² d) is the release rate; \( t \) (d) is the release time; \( A \) (m²) is the water–sediment interface area.

Restraint ratio of P-release

A description of the P suppression efficiency of Phoslock® treatment was based on Equation (3), with the difference of equilibrium PO₄-P concentration between the experiments of Control Check (CK) and Phoslock® treatment

\[ \eta = \frac{C_c - C_m}{C_c} \times 100\% \]  (3)

where \( \eta \) is the restraint ratio, \( C_c \) (mg/L) is the PO₄-P concentration of CK; \( C_m \) (mg/L) is the PO₄-P concentration with Phoslock®.

P-inactivation rate of sediments

P-inactivation rate (transformation rate from BAP to non-bioavailable P) by in-situ treatment was calculated according to Equation (4),

\[ r_{pi} = \frac{C_b - C_a}{C_b} \times 100\% \]  (4)

where \( r_{pi} \) is the inactivation rate, \( C_b \) (mg/kg) is the concentration of BAP in the original sediments of the study site; \( C_a \) (mg/kg) is the concentration of BAP in the treated sediments.

Leaching rate of La

Leaching rate of La from Phoslock® to water body was calculated by Equation (5),

\[ r_{La} = \frac{m_1}{M \times n} \times 100\% \]  (5)

where \( r_{La} \) is the leaching rate of La, \( m_1 \) (g) is the quantity of La in water; \( M \) (g) is the quantity of Phoslock® added into the water body; \( n \) is La content of Phoslock®, which here is 5%.

RESULTS AND DISCUSSION

P-form analysis of the study site sediments

Release of BAP from sediments represented a significant internal P source for many water bodies. Through sequential extraction experiments, different P-forms and the relative proportions of the sediments in the study site were analysed (Figure 2), and the data indicated that the rank order of the P-fractions was NaOH-P > HCl-P > Res-P > BD-P > NH₄Cl-P. Of the TP in the sediments, the proportion of BAP (52.2%) was the largest. Potentially, the BAP can contribute substantially to the local primary algal production when the fraction reaches the water column during its growing season.
To conclude, the sediments of the study site are at a high risk of P release under certain environmental conditions.

**Effect of external factors on the efficiency of Phoslock®**

Lots of environmental factors might affect P release from sediments. A series of static simulation experiments were undertaken under different pHs, DO and initial PO₄-P concentrations of the overlying water before and after Phoslock® treatment (Table 2 and Figure 3(a)). The results were as follows.

**Effect of DO**

Anaerobic conditions stimulated BD-P release (Kleeberg & Gruenberg 2005). The PO₄-P concentration of the overlying water increased gradually with time in the untreated experiments. Although PO₄-P was released from untreated sediments under aerobic conditions, the SRP concentrations and the P-release rates were much smaller than those under anaerobic conditions. Under the same pH (CK2 & CK2’, CK1 & CK1’), the P-release rates in anaerobic conditions were 3.00 (pH = 7.0) and 1.92 (pH = 9.0) times higher than those in aerobic situations, respectively.

**Effect of pH**

Alkaline conditions promoted NaOH-P release greatly (Rydin 2000). When pH was increased from 7.0 to 9.0, even under aerobic conditions (CK2’, CK1’), the P-release
rate of the latter was 5.85 times higher than that of the former, which suggested that aeration could not inhibit P release under high pH conditions, not to mention the high pH coupling with anaerobic conditions (under pH = 9.0 and anaerobic conditions, the P-release rate arrived at 10.67 mg/m² d).

When cyanobacteria blooms occurred, both DO and pH of the water bodies changed greatly. Algal bloom and the
subsequent decay of algal matter led to oxygen depletion in the water, which caused release of phosphates that were previously bound to oxidized sediment. On the other hand, pH value could rise to 9.0 when algae proliferate and photosynthesize. BAP was apt to be released under such conditions (anaerobic and high pH). The P-release rate under aerobic and neutral conditions (CK2) was only 8.9% of that under anaerobic and alkaline circumstances (CK1). Moreover, the P-release rate under anaerobic conditions was less than that under high pH conditions, consistent with the results of the sediment P-fraction (NaOH-P > BD-P) of the study site.

**Effect of initial PO₄-P concentration of the overlying water**

When the PO₄-P concentration was relatively high (1.0 mg/L in the experiments), PO₄-P in the water column was adsorbed by sediments under aeration conditions (CK3), the average P-release rate reached -4.38 mg/m² d, and the PO₄-P concentration decreased by 70% in 40 days. On the contrary, P in the sediments was released to the water column when the PO₄-P concentration of the overlying water was as low as 0.05 mg/L (CK2), though the P-release rate was comparatively low (0.95 mg/m² d). When the condition was turned into high pH (pH = 9.0) and anaerobic, the P-release rate of low initial PO₄-P (CK1) was 1.8 times higher than that of high initial PO₄-P (CK3).

**Application rate of Phoslock®**

Considering the SRP of the overlying water and P quantity released from sediments at equilibrium, based on the general rule of Phoslock® application, the usage (for the water column and top 10 cm sediments) should be no less than 0.35 kg/m². While results of static experiments (Figure 3(b)) showed that the restraint ratio of P release increased sharply when the application rate of Phoslock® rose from 0.35 to 0.5 kg/m². Although P suppression efficiency depended on the amount of treatment material used, it changed little when Phoslock® dosage was above 0.5 kg/m². Under the experimental conditions of treatment 1, 2, and 3, the restraint ratio of P release reached 97.7, 96.5, 98.3% and 98.1, 96.9, 99.0%, with the application rate at 0.5 and 0.8 kg/m², respectively. Thus we preliminarily proposed the application rate of Phoslock® at 0.5 kg/m².

Compared with the experiments before/after Phoslock® treatment (CK1/Treatment1, CK2/Treatment2, CK3/Treatment3), the PO₄-P concentration of the overlying water decreased to a low level (≤0.02 mg/L) within 10 days with the treatment of Phoslock® at 0.5 kg/m² (Figure 3(a)), and the P-release rates reduced to -0.35, -0.50 and -8.20 mg/m² d respectively. With regard to the group of initial PO₄-P = 1.0 mg/L, the PO₄-P concentration decreased sharply during the first two days (with the P-release rate at -120.88 mg/m² d). It was indicated that Phoslock® adsorbed PO₄-P in the water column, and its effectiveness was quickly shown within two days.

**Transformation of P-forms with Phoslock® treatment**

Based on the results of the static experiments, and considering the high BAP-loading in sediments, the dynamic simulation experiments were pursued with the application of Phoslock® at 0.5 and 0.8 kg/m² respectively.

Results of the dynamic simulation experiments (Table 3 and Figure 4) indicated that, with the application of Phoslock® under varying conditions, the content of TP in sediments fluctuated little with time, while the content or ratio of different P-forms changed significantly (BAP decreased while HCl-P increased). With regard to the 1 yr period of *in-situ* treatments, compared with CK (original sediments), the P-inactivation rate of Group A was 58.4%, in which, the quantity of NH₄Cl-P, BD-P and NaOH-P decreased by 73.0, 64.0 and 56.1% respectively, while the proportion of non-bioavailable P (or HCl-P) increased by 1.2 times. When the condition was turned into anaerobic (Group B), where it was favourable for BD-P to be desorbed, there was no PO₄-P concentration increase in the water column, and correspondingly the quantity of BD-P decreased by 57.3% which was a little lower than that of Group A. And NaOH-P was apt to be released from untreated sediments when pH was increased to 9.0 (Group C); contrarily, the P-inactivation rate rose to 60.7% with the application of Phoslock® at 0.8 kg/m². And then, when the initial PO₄-P of the overlying water was decreased from 1.0 to 0.05 mg/L (Group D), where P was liable to be released, the P-inactivation rate remained at a better-than-expected level (57.6%) with the treatment of Phoslock® at
On this basis, if the application rate of Phoslock® was cut down to 0.5 kg/m² (Group E), although the P-inactivation rate dropped to 48.0% (the quantity of NH₄Cl-P, BD-P and NaOH-P decreased by 54.3, 34.5 and 53.0% respectively, and the proportion of non-bioavailable P doubled), nearly half of BAP was transformed into non-bioavailable P, which also helped to reduce the internal P release to a large extent. Thus the effectiveness of Phoslock® on P immobilization was demonstrated.

The results of laboratory experiments proved that the application of Phoslock® for sediment rehabilitation was effective and efficient. Even under anaerobic and high pH conditions, PO₄-P could not be released, but BAP could be transformed into non-reactive fractions to different degrees. The field monitoring data (Figure 4) indicated that, the PO₄-P removal rate in the water column was 96.5% in the first three months, meanwhile, with successive applications every three months, P-inactivation rate of the sediments reached 12.7, 19.5 and 31.0% at 3, 6, and 12 months respectively, consistent with the results achieved in laboratory experiments. There was no cyanobacteria bloom observed throughout the year.

Thus, the effectiveness of Phoslock® on in-situ sediment remediation can be concluded as follows.

Original sediments (labelled as CK), whose sorption capacities were dominated by Fe(OH)₃, released P during the development of hypolimnetic anoxia, which that resulted in the reductive dissolution of Fe(III) solid phases and the release of Fe²⁺ into solution or reprecipitation as Fe(II) sulfide. An increase in the pH of a water body above pH 8 might result in the release of P from the aluminium bound phosphate (Lewandowski et al. 2005). The active ingredient in Phoslock®, La, possessed a strong affinity for orthophosphate, forming the mineral rhabdophane with an extreme low solubility product $K_{sp} = 10^{-24.7}$ to $10^{-25.7}$ mol² L⁻² (Johannesson & Lyons 1994), which could prevent P release during anoxia and high pH value conditions by adsorbing the P liberated from Fe(OH)₃ and Al(OH)₃ respectively. Although this binding was not influenced by redox state and a wide range of pH values, pH influenced its adsorption capacity. The efficiency of Phoslock® dropped when pH > 9, which mainly resulted from hydroxyl ions competing with phosphate for La binding sites.

<table>
<thead>
<tr>
<th>Group number</th>
<th>DO (mg/L)</th>
<th>pH</th>
<th>Application rate of Phoslock® (kg/m²)</th>
<th>PO₄-P of the overlying water (mg/L)</th>
<th>Treated time (month)</th>
<th>P-inactivation rate (%)</th>
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</thead>
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<tr>
<td>CK</td>
<td>3.5</td>
<td>7.9</td>
<td>0</td>
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<td>31.0</td>
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Laboratory experiments indicated that the leaching rates of La were all less than 0.1% in the water bodies when pH ranged from 5 to 11, with the dose rates of Phoslock® from 0.2 to 3 g/L. Concerning the pilot project, the application rate of Phoslock® was 0.5 kg/m² (water depth: 2.5 m, application rate equalled 0.2 g/L), it was measured that the concentration of La in the water body was less than 10 μg/L. Related research results showed that long-term exposure to low-dose La (5.56 mg/L) might not cause a negative/toxicity effect on the activity and physical- or neurodevelopment of biology (Yu 2006). Thus it could be regarded as environmentally secure under the proposed
The optimal pH range for the application of Phoslock® was between 6 and 9. As for the representative eutrophic shallow water body of North China, the recommended application rate was 0.5 kg/m².

2. Phoslock® effectively restrained P release from sediments and the proportion of BAP in sediments was reduced, a majority of which was transformed into non-reactive species, even under anaerobic conditions and pH = 9.0.

3. With the successive applications of Phoslock® every three months in the field project, the sediments P-inactivation rate achieved 31.0% over a 1 yr period. This technology significantly reduced internal P release.

**REFERENCES**


