



## Effect of natural zeolite capping on phosphorus release from sediments

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### ABSTRACT

As an aluminosilicate mineral, natural zeolite can inhibit sediment nutrient release by forming an active coating layer on the sediment surface. Laboratory experiments demonstrated the inhibitory effects of zeolite on sediment phosphorus release. And the influence of temperature, disturbance, pH value, the thickness and particles size of the zeolite on phosphorus release was also investigated. Phosphorus release experiments were simulated in a cylinder 600 mm high, 60 mm inner diameter, 70 mm outer diameter over a period of 60 d. The results showed that at 30 d, the total phosphorus (TP) release intensity of the experimental group was 48% of the control group, and the inhibitory effect tended to be stable after 30 d. The effect of the zeolite to prevent P release is sequentially affected by the temperature, zeolite thickness, pH, and zeolite particle size, but is not affected by the disturbance. Moreover, the unit price of zeolite which inhibits the release of phosphorus in sediments is 2.78 yuan/m<sup>2</sup>, which is very economical. This indicates that the use of a small amount of zeolite can effectively suppress the release of phosphorus in a large amount of sediment.

*Keywords:* Phosphorus release; Zeolite; Temperature; Disturbance; pH value; Thickness and size of zeolite

### 1. Introduction

Eutrophication is a water pollution phenomenon caused by excessive nitrogen and phosphorus. If the external source load of water pollution is high, the sediment becomes a “sink” of contaminants, reducing the overlying water pollution. When the lake is undergoing remediation, the sediment can also act as “source” of the contaminants, contributing to the overlying water pollution [1,2]. Once the external source pollution has been effectively controlled, sediment contaminant release is an important process affecting water quality and eutrophication [3]. Sediments can release nitrogen, phosphorus and organic matter into water bodies, contributing to eutrophication and delaying water remediation [4,5]. Studies have shown that the control of external pollution sources may not be able to

improve the overlying of water pollution without endogenous control [6–10]. Therefore, it is very important to study the control of phosphorus release from sediments to control lake eutrophication.

In the process of sediment phosphorus release, phosphorus first enters the sediment pore water, thereby diffuses to the sediment-water interface, and then enters the overlying water, increasing the phosphorus load of overlying water [11]. The whole process is affected by DO (dissolved oxygen), pH, temperature, and disturbance etc [12,13]. Phosphorus releases is significant under anaerobic conditions, but limited under aerobic conditions [14–16]. The phosphate forms in sediment are as follows: aluminum phosphorus (Al-P), iron phosphorus (Fe-P), calcium phosphorus (Ca-P), organic phosphorus (OP), inorganic phosphorus (IP), and total phosphorus (TP), respectively. High pH promotes the release of HCl-P (mainly Ca-P), and low pH promotes the release of NaOH-P (Al-P, Fe-P) [17,18]. Microbial activity is

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enhanced at higher temperature, improving bioturbation and increasing oxygen consumption due to organic matter mineralization in sediments. The oxygen content of the interstitial water decreases, reducing the redox potential, and shifting from oxidizing to reducing conditions. The reduction of  $\text{Fe}^{3+}$  to  $\text{Fe}^{2+}$  will accelerate the release of iron-bound phosphorus in sediment [19]. Sediment suspension will be accelerated by perturbation, leading to the rapid release of phosphorus in pore water into the overlying water [20]. The influence of disturbance on sediment phosphorus release is only temporary. The disturbance effect would be no longer prominent when the phosphorus concentration of overlying water reached a certain level [21]. The high speed disturbance results in the internal phosphorus release being lower than the low speed. However, the intermediate-level disturbance has the highest phosphorus release rate [22].

Sediment pollution control technology is mainly divided into ectopic control technology and in-situ control technology [5]. The ectopic control technology mainly includes sediment dredging and treatment. The research on lake dredging is mostly aimed at the impact of dredging on ecosystems, benthic organisms and water quality, but dredging is still controversial for improving water quality and eliminating water eutrophication. The in-situ control technology of sediment pollution mainly includes sediment cover, in-situ treatment of sediment, chemical passivation and artificial aeration. The sediment cover is covered with one or more layers on the upper part of the contaminated sediment. It is a sediment pollution control technology that separates the sediment from the overlying water and prevents the release of sediment from the sediment. The chemical passivation technology mainly fixes nutrients (mainly phosphorus) in water and sediment by adding chemical reagents (aluminum salt, iron salt and lime, etc.), and forms a coating layer on the upper surface of the sediment. Sediment in situ treatment refers to the use of physicochemical or biological methods to reduce the volume of contaminated sediment in situ, reduce the amount of pollutants or reduce the solubility, toxicity or migration of pollutants, and reduce the release of pollutants. The in-situ control technology of sediment pollution can improve the dissolved oxygen level and redox potential of the sediment–water system, and can effectively control the release of nitrogen and phosphorus from the sediment [11–13].

The main principle of in situ capping technology is laying one or more clean layers on the contaminative sediment surface to inhibit the migration of contaminants from sediments to the overlying water [23]. Natural zeolite is a typical cation exchange agent, which has been shown to be effective for the removal of  $\text{NH}_4^+$ , heavy metals, and radioactive nuclear elements. Zeolite has a tetrahedral structure with

a large number of micropores and a pore diameter of less than 1 nm, allowing only molecules or ions smaller than the micropores to pass [12]. This porous structure forms a large electrostatic attraction such that it has good physical adsorption properties. And the larger the specific surface area, the better the adsorption performance. Zeolites are mainly composed of silicon oxide and aluminum oxide. The replacement of silicon by a portion of aluminum results in a negative charge excess, which results in ion exchange of the zeolite, and the ion exchange capacity is related to the ratio of silica to alumina [13,24]. And the adsorption capacity of zeolite for phosphorus is 0.3511 mg/g in water [24]. The advantage of zeolites is low cost and does not contribute to secondary pollution to the water environment. Other adsorbent materials have significant disadvantages. For example, flyash can cause a pH rise; Fe can cause water discoloration, and Al is biologically toxic. Thus, in order to find a good adsorbent material which has absorbing effect on both nitrogen and phosphorus, the inhibition of phosphorus release by zeolite from sediments is also worth studying (Fig. 1). There have been some studies about zeolites on the control of contaminants. Bona et al. studied the effect of aluminum-modified zeolite on sediment containment release in Venice Lagoon Lake [25]. They showed that phosphorus release can be completely inhibited by aluminum-modified zeolite under aerobic and anaerobic conditions. Under anaerobic conditions, As, Hg and  $\text{NH}_4^+$  release rates were decreased. Liu et al. also showed that phosphorus release can be effectively controlled by natural zeolite under anaerobic conditions. Temperature rise decreases phosphorus release and zeolite layer thickness has little influence on phosphorus release between 1–1.5 cm [3]. However, there is currently a lack of systematic research on the factors that influence the release of phosphorus by zeolites. Moreover, these influencing factors have an important influence on the phosphorus control effect of zeolites.

Based on this, the aims of this study are to investigate (1) the efficacy of zeolite on sediment phosphorus release, and (2) the influence of temperature, disturbance, pH value, zeolite cover layer thickness, zeolite particles size on sediment phosphorus control. In order to provide data reference for the sediment containment control of GonghuBay of Taihu Lake, China. The equation for the flux of P release from sediments into the overlying water were provided below.

The rate of phosphorus increase and rate of phosphorus inhibition and the release rate of phosphorus from the sediment are calculated as follows:

- Rate of phosphorus increase = (Final of phosphorus – initial phosphorus concentration) / Initial phosphorus concentration  $\times 100\%$

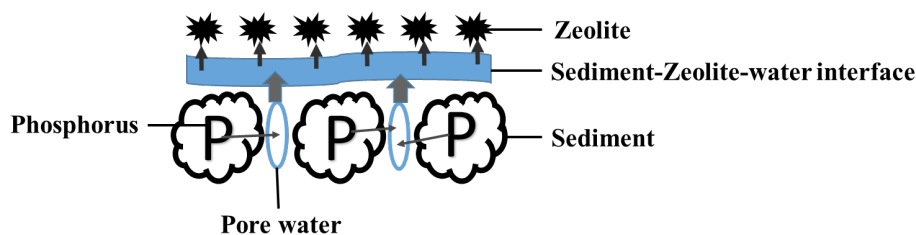


Fig. 1. The transfer of phosphorus in sediments.

- Rate of phosphorus inhibition = (phosphorus in the control group – phosphorus in the experimental group) / phosphorus in control group × 100%
- The release rate of phosphorus from the sediment = phosphorus concentration in water × the volume of water / the surface area of zeolite / days

## 2. Materials and methods

### 2.1. Materials

The chemical composition of natural zeolite is shown in Table 1. Natural zeolite was purchased from a company in Henan Province, China, with particle diameter of 1–5 mm and porosity of 46.4%.

Sediment samples were collected from GonghuBay of Wuxi in May 2014, which experiences serious phosphorus pollution during the summer. Surface sediment samples (0–10 cm depth) were collected by the Peterson dredge. The collected sediment samples were put in a plastic bag after being mixed and exhausted. Mixed sediment samples were frozen and dried, grinded and sieved (100 mesh) after taken back to the lab. TP concentrations in the sediments were all about 300 mg/kg.

Water was simulated using deionized water, sodium bicarbonate, and potassium chloride. The simulated lake water had an alkalinity of 40 mg/L (CaCO<sub>3</sub>), 0.01 mol/L KCl (to increase the ionic strength of the water), a pH value of 7.0, and DO content of 4.0 mg/L.

### 2.2. Experimental method

The schematic view of the experiment apparatus is shown in Fig. 2. Experimental apparatus and sampling time were set as follows: the sediment was mixed evenly in a large container. Eighteen cylinders were constructed, with a height of 600 mm, inner diameter of 60 mm, and outer diameter of 70 mm. Two kilograms of sediment was put in the cylinder, and allowed to settle for 3 d prior to adding the simulated lake water. The sediment layer was 200 mm high. The simulated lake water was added to the cylinder with siphon method, and a height ratio of soil to water was 3:2. The experimental period was 60 d. 10 ml overlying water was periodically sampled to determine TP concentration, with 10 mL simulated lake water added to replenished the column.

The experiments were divided into two parts. The first part explores the inhibitory effects of zeolite (thickness of

1 cm, particle size of 2–3 mm) on sediment phosphorus release. The second part investigate influence of the following factors on sediment phosphorus release control using orthogonal experiment design: pH, temperature, disturbance, zeolite cover thickness and particle size of the zeolite. The experiment factors designs of zeolite inhibiting phosphorus release from sediment by 5 kinds of influence factors are shown in Table 2. And the sediments were disturbed by a stirrer.

The dissolved oxygen meter (TOA Do meter Do-11P) was used to determine dissolved oxygen. pH meter (Yi Source F-20 pH-mv Meter, PHE-3C) was used to measure the pH value. Molybdenum-antimony-anti-spectrophotometry was used to determine TP and dissolved total phosphorus (DTP) concentration. SPSS 17.0 software was used to do statistical analysis.

## 3. Results and discussion

A comparison of rate of phosphorus inhibition using zeolite control is shown in Fig. 3. The rate of phosphorus inhibition of the experiment group was 92% of the control group on the first day, but reduced to 48% on 30th day, this indicated that the zeolite was effective in controlling phosphorus release, and that the phosphorus release was stabilized after 30 d. Sediment TP release is less at the beginning of the experiment, resulting in the phosphorus release of experimental and blank groups were similar 10 d before.

As shown in Table 2, in the 18 experiments, the 9<sup>th</sup> group has the lowest phosphorus release intensity of 1.212 mg/(m<sup>2</sup> · d). TP concentration in overlying water is 0.242 mg/L, belongs to surface water Standard III water quality.

The results of zeolite inhibiting phosphorus release from sediment are shown in Table 3. The results of variance analysis of zeolite inhibiting phosphorus release from sediment are shown in Table 4. As shown in Tables 3 and 4, among the factors influencing sediment phosphorus release, all factors had significant influence on sediment phosphorus release after the zeolite coverage, except for disturbance. The rea-

Table 1  
Chemical composition of natural zeolite in 1–5 mm diameter

Natural zeolite	Composition (%)
SiO <sub>2</sub>	66.83
Al <sub>2</sub> O <sub>3</sub>	13.73
Fe <sub>2</sub> O <sub>3</sub>	1.06
CaO	3.17
MgO	1.28
K <sub>2</sub> O	2.81

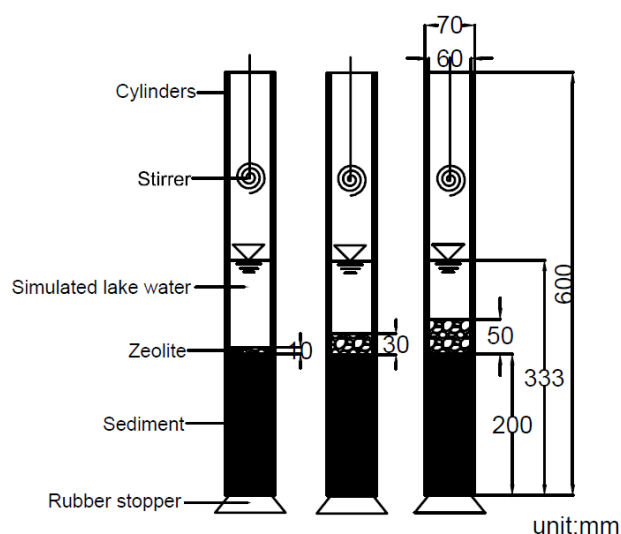


Fig. 2. Schematic view of the orthogonal experiment apparatus.

Table 2  
Experiment factors designs

Factors Experiment number	pH value	Temperature (°C)	Disturbance	Zeolite thickness (cm)	Zeolite particle size (mm)	Phosphorus release rate (mg/m <sup>2</sup> ·d)
1	1(5)	1(10)	2(N)	1(1)	1(<2)	1.92
2	1(5)	2(20)	–	3(5)	2(2–3)	2.74
3	1(5)	3(30)	1(Y)	2(3)	3(3–5)	3.38
4	2(6)	2(20)	2(N)	2(3)	2(2–3)	1.97
5	2(6)	3(30)	–	1(1)	3(3–5)	2.84
6	2(6)	1(10)	1(Y)	3(5)	1(<2)	1.53
7	3(7)	2(20)	1(Y)	1(1)	3(3–5)	1.70
8	3(7)	3(30)	2(N)	3(5)	1(<2)	1.88
9	3(7)	1(10)	–	2(1)	2(2–3)	1.21
10	4(8)	1(10)	–	3(5)	3(3–5)	1.64
11	4(8)	2(20)	1(Y)	1(1)	1(<2)	2.11
12	4(8)	3(30)	2(N)	2(3)	2(2–3)	2.49
13	5(9)	3(30)	1(Y)	3(5)	2(2–3)	1.89
14	5(9)	1(10)	2(N)	2(3)	3(3–5)	1.83
15	5(9)	2(20)	–	1(3)	1(<2)	2.39
16	–	3(30)	–	1(1)	1(<2)	2.60
17	–	1(10)	1(Y)	2(3)	2(2–3)	1.65
18	–	2(20)	2(N)	3(5)	3(3–5)	2.12

Note: The numbers outside the brackets are levels, and the numbers in brackets are parameter value. Where “1 (5)” in the first cell of the pH column indicates that the parameter at level 1 is pH 5.

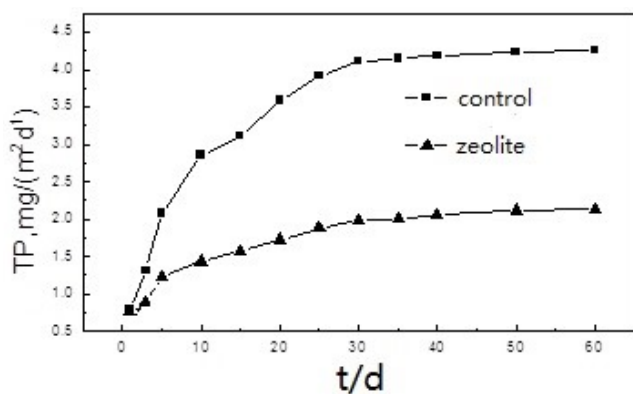


Fig. 3. Comparison of inhibiting effect on sediment TP release.

son is that zeolite is in direct contact with the overlying water, and zeolite is less likely to be suspended compared with uncovered sediments [26]. Furthermore, after covering with the zeolite, the phosphorus needs to diffuse through the zeolite cover layer, increasing the difficulty of sediment suspension [22,27]. Wu [17] showed that the effects of factors on phosphorus release control in descending order were: DO > temperature > pH value. Lin [28] did orthogonal experiments in the Shuanglong lake, showed that the control effect of factors on sediment phosphorus release in the descending order were as follows: DO concentration > temperature > pH value > disturbances. In this study, after zeolite was used to covering the sediment, the factors influencing sediment phosphorus release in descending order

Table 3  
Results of zeolite inhibiting phosphorus release from sediment

Factors	pH value	Temperature (°C)	Disturbance	Zeolite thickness (cm)	Zeolite particle size (mm)
$k_{1j}$	2.68	3.26	4.08	4.51	4.14
$k_{2j}$	2.10	4.32	4.07	5.30	3.98
$k_{3j}$	1.60	5.02	–	3.93	5.50
$k_{4j}$	2.08	–	–	–	–
$k_{5j}$	2.03	–	–	–	–
$R_j$	1.08	1.76	0.01	1.37	0.52

Note:  $k_{ij}$ ,  $R_j$  represent the mean and the range of the total test results for factor  $j$  and level  $i$ .

were: temperature > thickness > pH value > diameter > disturbance. The importance of temperature, pH and disturbance value for phosphorus release in our study was in good agreement with the results of Lin [28]. Lin [29] showed that the smaller the particle size of the zeolite or the layer thickness of the cover, the better the control effect of phosphorus. This is similar with our study in this paper. The main reason why thickness has greater influence is that there is no control effect by capping zeolite when the zeolite layer is thin [30]. On the other hand, while the zeolite layer was too thick, a lack of oxygen on sediment surface would take place, reducing the oxidation-reduction potential of the sediment surface, and increasing Fe/Al-P release in sediment [31]. This is similar with the results of Wu [17].

Table 4  
Results of variance analysis of zeolite inhibiting phosphorus release from sediment

Source of variance	Square of deviance	Degree of freedom	Mean square	F ratio	Significance
pH value	16.00	4	4.00	1.52*10 <sup>8</sup>	0.0000
Temperature	42.54	2	21.267	1.96*10 <sup>9</sup>	0.0000
Disturbance	0.00	1	0.00	0.07	0.7950
Zeolite thickness	25.34	2	12.67	1.140*10 <sup>10</sup>	0.0000
Zeolite particle size	0.43	2	0.21	46211.36	0.0000

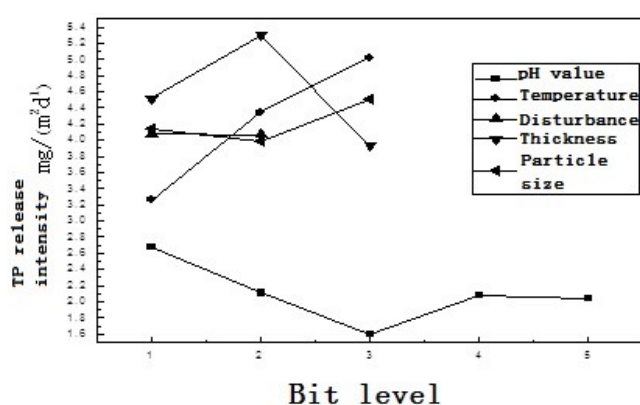


Fig. 4. Effect on zeolite inhibition of sediment phosphorus release by different factors affecting levels.

The effect on zeolite inhibition of sediment phosphorus release by various factors affecting levels is shown in Fig. 4. When the overlying water is acidic or alkaline, TP release intensity increases [32]. The neutral conditions of the overlying water resulted in the lowest TP release intensity. The influence of pH and temperature could be interpreted as follows. Release of Fe/Al-P from sediments increased under acidic conditions, while the release of Ca-P in sediments increased under alkaline conditions [33]. In addition, the adsorption of phosphorus by zeolite was affected by pH value, decreasing in acidic or alkaline conditions [34]. The main principle of zeolite adsorption of phosphorus is the physical adsorption of the porous structure [35]. Raising the overlying water temperature increased TP release intensity. In the case of temperature, high temperature could increase microbial activity in sediment, and accelerate the conversion of organic phosphorus to inorganic phosphorus, phosphorus release intensity increases [36]. Another explanation is that molecular diffusion is strengthened as temperature rises, leading to the decrease of zeolite adsorption, and TP release intensity increase [37]. The optimum thickness of zeolite is 3 cm. Excessive thick zeolite layers can block oxygen diffusion into the sediment and lowering the oxidation–reduction potential in surface sediments, which can increase TP release intensity. The smaller the particle size of the zeolite, the larger the adsorption surface, and the greater the TP adsorption intensity [38,39]. This result is consistent with Jiang [40].

The control effect of phosphorus release by zeolite-aeration and zeolite-Phoslock were superior to using zeolite alone. However, we have not been able to do more research on the factors affecting zeolite-aeration and zeolite-Phos-

lock. Hope we can supplement this research in the future, and do more researches on the combined effects of zeolite with other materials.

#### 4. Cost analysis

In the single cylinder simulation experiments, 15.43 g zeolite was added to 0.00286 m<sup>3</sup> of sediment (0.05 m thickness) for a surface of 0.27 kg/m<sup>2</sup>. The cost of the zeolite is 0.020 yuan/m<sup>2</sup>, while the zeolite is priced at 750 yuan/ton.

#### 5. Conclusions

Covering sediment with zeolite inhibited sediment phosphorus release. When the height ratio of sediment to zeolite is 20:1, the inhibition rate of zeolite on TP release is 52%, and the inhibitory effect could best able for a long time after 30 days, which indicated the use of a small amount of zeolite can effectively inhibit the release of phosphorus from a large number of sediments. Also, the unit price of zeolite that inhibits the release of phosphorus from sediments is 2.78 yuan/m<sup>2</sup>, which is very economical. And the efficacy of zeolite to retard P release is affected by pH value, temperature, particle size of zeolites, zeolite thickness, but not affected by disturbance. The order of factors affecting zeolite inhibition of phosphorus release from zeolite-covered sediment were as follows: temperature > zeolite thickness > pH value > zeolite particle size > disturbances.

Looking forward to the future, we can continue to study in depth the combination of zeolites and other technologies to control the release of phosphorus from sediments.

#### Additional information

Competing Interests: The authors declare that they have no competing interests.

Data availability statement: The authors agree that the data is available.

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