



Introducing hydrate aluminum into porous thermally-treated calcium-rich attapulgite to enhance its phosphorus sorption capacity for sediment internal loading management

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HIGHLIGHTS

- Al@TCAP was prepared by incorporating Al into heated porous Ca-rich attapulgite.
- The maximum P sorption capacity of Al@TCAP was nearly two times that of TCAP.
- P in overlying water and sediment mobile P can be effectively reduced by Al@TCAP.
- P bound by Al@TCAP was mainly formed by inter-sphere Al-P complexes.

ARTICLE INFO

Keywords:

Solid-phase P sorbent
Aluminum
Thermally-treated calcium-rich attapulgite
Sediment internal loading
Management

ABSTRACT

Solid-phase P sorbents are increasingly used in shallow eutrophic lakes for sediment internal loading management due to their strong ability to resist frequent resuspension. In this study, hydrate aluminum was incorporated into porous thermally-treated calcium-rich attapulgite (TCAP) to enhance its P sorption capacity for sediment internal P loading management. Batch studies indicated that 2 mol/L of aluminum modified TCAP (Al@TCAP) has the optimal P sorption performance and was well characterized. The maximum P sorption capacity of Al@TCAP with particle sizes 1–2 mm increased to 8.79 mg P/g at a pH of 7, which was nearly 2 times that of TCAP. P sorption on Al@TCAP performs well when the solution pH is in the range of 4–10, but efficiency decreases sharply when the solution pH exceeds 11. The co-existing ions including SO_4^{2-} , Cl^- , NO_3^- , and HCO_3^- that are frequently found in eutrophic lake waters have little effect on P sorption of Al@TCAP. Laboratory experiments showed that adding Al@TCAP does not cause pH changes in overlying water. An average P reduction of 59.7 to 72.5% in overlying water was recorded during the experiment's 63 days of aerobic and anaerobic incubation. Mobile P in surface sediment (0–2 cm) was significantly reduced and was accompanied by an evident Al-P increase and a moderate Ca-P increase in sediment when compared to the control treatment. This was further confirmed by ^{31}P solid-state NMR and XPS spectroscopy analysis, which indicated that P bound by Al@TCAP was mainly through formation of the Al-P complex. All results indicate that Al@TCAP can be used in eutrophic lake for sediment internal P loading management.

1. Introduction

Phosphorus (P) is the key nutrient in the induction of lake eutrophication, which can consequently lead to excessive algal blooms and subsequent water quality deterioration. It has been proven that successful phosphorus control is the key to lake eutrophication restoration [1]. However, the effective control of external P has not

guaranteed acceptable remediation of eutrophic lakes. This is because P can continuously be released into overlying water from the lake bed sediment, a process called internal P loading [2].

Enhancement of the P binding capacity in lakebed sediment using geoenvironmental material has proven an efficient method for restoring eutrophic lakes [3]. To date, many materials have been used to restore eutrophic lakes, including La-bearing materials such as lanthanum-

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modified bentonite (LMB) [4], aluminum compounds ($\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ and $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$) [5,6] and calcium compounds ($\text{Ca}(\text{OH})_2$ and calcite) [7]. Among these, LMB and alum have been extensively used in many lakes for eutrophication control [8]. Aluminum has been successfully used for 30 to 40 years for eutrophic lake restoration. However, study show that lake water with sustained (> 12 h) high pH levels ($\text{pH} > 9.5$) due to photosynthesis may increase the risk of releasing P from Al hydroxides in sediments [9]. Normally, the algal bloom can greatly enhance the pH value of overlying water in eutrophic lakes, which consequently affects the efficiency of aluminum [10]. But pH is 1–2 units lower near the sediment surface compared with overlying water. A Ca compound has been proposed for use in hard water lakes, which can form stable Ca-P complexes in high pH value water [11–13]. Therefore, if Al can incorporate with Ca-bearing materials, the resulting sorbents would be very effective in lake internal P remediation even during periods of extensive algal blooming.

Another problem associated with using aluminum in shallow lakes is its tendency to resuspend into a water column due to wind disturbance [14]. Thus, excessive wind will cause the release of Al from the inactivated sediment and will reduce its efficiency in binding sediment mobile P (the sum of labile-P, iron bound P and organic fraction of P). In comparison, solid phase P inactivation has a high weight itself, which means it can deposit quickly on surface sediment after application into overlying lake water. Solid phase P also has the ability to resist frequent sediment resuspension [15]. For example, Egemose et al. indicated that the consolidation of surface sediment was enhanced and resisted sediment resuspension after the application of LMB [16]. Consequently, if aluminum can be incorporated into a solid phase clay matrix, the resulting product will have an efficient P binding capacity. At the same time, the aluminum and clay matrix has the ability to resist frequent sediment resuspension in shallow eutrophic lakes. However, microorganism, aquatic plants and aquatic animals might also can change the micro-environment such as pH, redox potential and dissolved oxygen across sediment-water interfaces, which can consequently affect the sediment P binding capacity of P inactivation agents such as Al@TCAP.

Recent findings indicated that the thermally-treated calcium-rich attapulgite (TCAP) with a particle sizes of 1–2 mm can adsorb a certain amount of P (~ 5 mg/g), which can be used as a substrate in the constructed wetlands for rapid P removal [17]. TCAP has good mechanic strength, high density, and it remains a solid phase in water. Besides, the powder form of CAP can be granulated into large particle sizes from 0.5 to 8 mm (or larger) if required and can then be calcined and activated in high temperature. However, the alkaline nature of TCAP makes it unsuitable for sediment internal P remediation because high dosages can be toxic to benthic biota in lakes [18]. The low-cost and porous nature of TCAP makes it easy to be further modified by impregnating reactive elements such as aluminum, iron or lanthanum [19]. The aim of this study is to (1) introduce hydrate aluminum into the porous structure of TCAP to prepare a Ca-Al combined solid phase P sorbent, (2) to assess the P removal performance of the prepared Al@TCAP from its aqueous solution, (3) to assess the sediment P immobilization capacity using Al@TCAP during anaerobic and aerobic conditions under varied dosages, and (4) to elucidate the P binding mechanism by Al@TCAP using the ^{31}P solid-state NMR spectroscopy technique.

2. Materials and methods

2.1. Solid phase Ca-Al combined P inactivation preparation

Raw calcium-rich attapulgite (CAP) was collected from the town of Qiuji, Xuyi County, Jiangsu Province. The collected mineral was manually ground and sieved into a particle size of 1–2 mm, which was then calcined at 700°C for two hours to enhance its P sorption capacity following the method proposed in previous study [17].

The prepared thermally-treated calcium-rich attapulgite (TCAP)

(10 g, 1–2 mm) was added to 25 mL of $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ at different concentrations. The mixture was then placed on a shaker at a constant temperature of 160 rpm for 4 h before left to stand for 16 h. The Al-impregnated TCAP (Al@TCAP) was then harvested and dried at 90°C for 24 h after which, it was washed with deionized water several times until there was no Cl^- detection; finally, it was once again oven dried at 60°C to a constant weight. The remaining products were kept for further experiment.

2.2. Sorption experiments

2.2.1. Pre-screening P sorption

To identify the optimum adsorbent among the Al-modified TCAPs, a pre-screening sorption experiment was carried out. 0.50 g of TCAP (washed and unwashed with deionized water in order to investigate the loss of calcium from TCAP during modification) and the modified TCAPs were added to 25 mL of a phosphorus concentration solution (100 mg/L) with a pH of 7.0. After equilibration for 24 h in the temperature-controlled shaker (25°C), solutions were centrifuged, filtered and measured. The optimal Al-modified TCAP (Al@TCAP) was selected and set aside for further study.

2.2.2. Kinetics of P removal

A standard phosphate solution (1000 mg/L) was prepared by dissolving KH_2PO_4 in deionized water. The experimental P solution was prepared by diluting the standard solution to a known working concentration. The kinetics of phosphorus removal by Al@TCAP was investigated using synthetic wastewater. In brief, 0.5 g of Al@TCAP was added into a series of centrifuge tubes containing 25 mL of 5, 50, and 200 mg P/L synthetic solution. Duplicate centrifuge tubes were retrieved at time intervals of 0.5, 1, 2, 4, 6, 8, 16, 24 and 32 h for phosphorus and pH analysis.

2.2.3. Isotherm experiments

The maximum P sorption capacity of the Al@TCAP was measured in the batch sorption experiment. In brief, 0.5 g of Al@TCAP was added into a series of 50-mL centrifuge tubes containing 25 mL of various P concentrations (0.5, 1, 5, 10, 20, 50, 80, 100, 200 and 400 mg/L). The initial pH of the solution was adjusted to 7 with 0.5 mol/L HCl or NaOH. After maintaining equilibrium for 24 h in the temperature-controlled shaker (25°C , 160 rpm), solutions were centrifuged, filtered and measured for pH and P concentrations. To investigate the effect of pH and co-existing anions (sulfate (SO_4^{2-}), chloride (Cl^-), bicarbonate (HCO_3^-), and nitrate (NO_3^-)) on P removal of Al@TCAP, a series of batch studies were carried out. To obtain the optimal pH values, 1 M NaOH or 0.5 M HCl were used to adjust the pH of the tested P solution. All experiments were carried out in triplicate.

2.3. Sediment P inactivation experiments

A total of 16 sediment cores were sampled from Zhushan Bay of Lake Taihu using a core sampler ($\text{Ø}80$ mm \times L 500 mm) and transported into a laboratory within 4 h. Each sediment core was filled with 15 cm of lake water overlying the sediment-water interface. The sediment inactivated experiment was carried out by evenly placing Al@TCAP on the surface sediment. The dose of Al@TCAP applied was based on the theoretical maximum sorption capacity ratio of Al@TCAP to the amount of sediment mobile P (0–5 cm) proposed in previous research. The maximum of P sorption capacity was obtained from the batch study, which is around 9.0 mg P/g. However, a high dose of Al@TCAP was also investigated in this study in order to examine the dose effects on sediment internal P remediation. Four treatments were performed as follows: (1) Control, (2) Al@TCAP: Mobile P = 110:1, (3) Al@TCAP: Mobile P = 220:1, and (4) Al@TCAP: Mobile P = 330:1. All cores were incubated in darkness in a circulating water bath at 20°C (similar to the temperature of lake surface water during sampling).

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