



In situ remediation of metal contaminated lake sediment using naturally occurring, calcium-rich clay mineral-based low-cost amendment



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HIGHLIGHTS

- Natural calcium-rich clay minerals were firstly used as in situ sorbent amendments.
- NCAP and NCSP can effectively reduce mostly mobile fraction of metal in sediment.
- Metal uptake by benthic organism with different feeding behavior can be reduced.
- NCAP and NCSP have the potential to be as amendments for sediment remediation.

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ABSTRACT

In situ sorbent amendments have gained attention owing to their role in contaminated sediment remediation and their cost-effectiveness. In this study, the effectiveness of two naturally occurring, low-cost calcium-rich clay minerals, natural calcium-rich sepiolite (NCSP) and natural calcium-rich attapulgite (NCAP), were investigated as potential in situ sorbent amendments for remediation of Pb and Cd using a sediment water microcosm and 15-day bioaccumulation assays. The two amendments were mixed with sediment at rates of 0%, 2.5%, 5% and 10%. The results indicate that the two amendments can effectively reduce most of the mobile fraction of metal in sediment, which in turn reduces metal uptake by benthic organisms. Additionally, the remediation efficiency increased with application dosage. Overall, sediment treated with the highest rate of 10% NCAP and NCSP led to reductions of 56.2% and 34.2%, for Cd, and 81.5% and 77.4%, for Pb, in the acid-soluble fraction. Similarly, uptake of Pb and Cd by benthic organisms was reduced by 33.5–53.3% and 20.9–37.1%, respectively. These results highlight the potential for using these naturally occurring, calcium-rich clay minerals as in situ sorbent amendments for sediment remediation.

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1. Introduction

Sediment is the sink of heavy metals in aquatic ecology systems such as lakes, rivers and estuaries [1]. However, metals cannot be fixed in sediment forever, and they could be released into overlying water when environmental conditions change (e.g. pH, redox potential) [2]. Hence, it is essential to develop methods to manage these metal-polluted sediments.

To date, the strategy for in situ remediation of contaminated sediment has mainly included sediment dredging and in situ sorbent amendment [3,4]. Sediment dredging, which is based on the idea of pollutant removal, is expensive and requires large areas

of land for sediment disposal [5]. In addition, sediment dredging may cause the resuspension of contaminants into the water column thereby reducing effectiveness [6]. In contrast, amendment with in situ sorbents is a relatively low-cost and low-impact approach to remediation of sediments polluted with heavy metals or hydrophobic organic contaminants [3]. Sorbents used for in situ remediation have a strong capacity for binding pollutants in sediment; hence, the pollutants sorption capacity of the amended sediment can be greatly enhanced by their addition. This sorbent binding capability limits the mobility of contaminants in sediment [1,7], which in turn, results in reduced uptake by benthic organisms [8]. Alternatively, sorbents can be physically mixed with surface sediment or applied directly to the surface sediment as capping material [3].

To date, various amendments have been used in the field and laboratory for remediation of metal contaminated soil and

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sediment [1]. These include industrial waste (red mud, slag) [9], iron oxide or hydroxide [10], clay minerals (zeolites, sepiolite, apatite) [11], nano-materials (nano-hydroxyapatite particles, stabilized iron sulfide nanoparticles) [12,13] and activated carbon [4]. When compared with amendments used in soil, those used in sediment usually have high sorption capacity, low water solubility, and higher stability under reducing and oxidizing conditions [1]. The relatively low-cost and ecological safety of clay has received a great deal of attention in soil remediation [1]. However, few studies have been conducted to assess the capability of modified or natural clay minerals for metal-polluted sediment remediation. The large quantity of OH groups and exchangeable cations (Ca^{2+} , Mg^{2+} or Na^+) result in clay minerals being an effective amendment for remediation of metal-polluted sediment [1,14]. Some researchers have found that Ca^{2+} in apatite or hydroxyapatite can exchange with metals in sediment and, therefore, be effectively applied for the remediation of metal-polluted sediment [15,16]. However, these amendments could induce eutrophication when used in lakes or other aquatic ecosystems due to the release of phosphate from the amendment. Hence, additional studies should be conducted to identify low-cost metal amendments for lake sediment remediation.

Sepiolite ($\text{Mg}_4\text{Si}_6\text{O}_{15}(\text{OH})_2 \cdot 6\text{H}_2\text{O}$) and attapulgite ($(\text{Mg}, \text{Al})_2\text{Si}_4\text{O}_{10} \cdot 4\text{H}_2\text{O}$) are natural hydrated magnesium silicate minerals with structures consisting of parallel ribbons of in 2:1 layers. These materials have similar mineralogy characteristics and have been widely applied in environmental protection and soil and water remediation [14,17]. Sepiolite and attapulgite are abundant in China. When compared with sepiolite or attapulgite from other countries, those in China (partial attapulgite) are low grade [18,19]. This is mainly due to the high levels of calcite and dolomite contained in sepiolite and attapulgite [18,19]. Sepiolite and attapulgite, with high levels of calcium, have previously been used as amendments for Pb- or Cd-polluted soil remediation, at which time they were shown to effectively immobilize metals in contaminated soil and reduce metal accumulation in plants, including rice [14,20]. However, it is not clear if they are suitable for remediation of metal-polluted sediments. In this study, natural calcium-rich sepiolite (NCSP) and natural calcium-rich attapulgite (NCAP) were used as low-cost amendments for the remediation of Cd- and Pb-polluted sediment. The performance of these two amendments was evaluated through sequential extraction, toxicity characteristic leaching procedure (TCLP) and benthic organism bioaccumulation in the amended sediment.

2. Materials and methods

2.1. Materials

NCSP and NCAP were collected from Henan province, Nanyang city, Jiangsu province, and Xuyi city based on a previous study [17,21]. The minerals were manually ground to be able to pass through a 100 mesh sieve, then dried at 105 °C for 24 h to achieve a constant weight. Cd-polluted sediment was collected from Lake Changdanghu as previously described [22]. Briefly, surface sediment (0–20 cm) was collected using a Petersen sampler and then homogenized, after which subsamples were either freeze-dried or kept at 4 °C until analysis. Both Pb and Cd solutions were prepared using nitrate salts (>99.5% purity). All reagents used in this experiment were of analytical grade or higher.

2.2. Sorption kinetics and isotherm studies

For the sorption isotherms, 0.50 g of NCSP or NCAP were added to 50 mL polyethylene centrifuge tubes with 25 mL of various Pb

and Cd concentrations (1–400 mg/L) at pH 6.0. The tubes were then placed on a constant temperature shaker (25 °C) for 24 h at 160 rpm to ensure complete mixing. Next, the solutions were centrifuged and the supernatants were filtered through a 0.45 μm membrane. The resulting solutions were stored at 4 °C until further analysis. The Pb and Cd sorption kinetics were determined at 25 °C using initial Pb or Cd concentrations of 50 mg/L at pH 6.0. Each kinetics experiment involved a series of vials, and three of the vials were sacrificed to measure the residual aqueous metal concentrations at a predetermined time.

2.3. Sorption kinetic and isotherm models

To understand the controlling mechanisms of the adsorption kinetics and provide improved understanding of metal sorption processes on the two calcium-rich minerals, the adsorption kinetics data were further analyzed using the following two typical kinetic equations:

$$\text{the Pseudo-first-order equation : } \log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (1)$$

$$\text{and the Pseudo-second-order equation : } \frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (2)$$

where, q_t and q_e are the amounts adsorbed at time t and at equilibrium (mg/g), respectively, and t is the sorption time. k_1 and k_2 are the rate constant for the pseudo-first-order kinetics and pseudo-second-order kinetics, respectively, which can be determined from regression of the experimental data.

The adsorption data were analyzed using the Langmuir and Freundlich models. The non-linear form of the two models can be expressed as follows:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad (3)$$

$$q_e = K_F C_e^n \quad (4)$$

where, q_e is the amount of Pb or Cd adsorbed on NCAP or NCSP (mg/g), C_e is the equilibrium metal concentration in solution phase (mg/L), K_L is the equilibrium adsorption constant related to the affinity of binding sites (L/mg), q_m is the maximum amount of metal per unit weight of adsorbent for complete monolayer coverage, K_F is roughly an indicator of the adsorption capacity and n is the heterogeneity factor, which has a low value for most heterogeneous surfaces.

2.4. Spiked sediment preparation

Owing to the low Pb concentration in collected sediment (<30 mg/kg), additional Pb was added into the sediment slurry as Pb solution. Very high levels of Pb (1000 mg/L) were added into a known weight of sediment slurry, after which the solution/sediment slurry was mechanically mixed daily to ensure that the Pb solution was adsorbed by the sediment slurry. After two months of stabilization, sub-samples of the sediment were analyzed by ICP-MS. The final concentrations of Pb and Cd in the spiked sediment were 199 mg/kg and 18.7 mg/kg dry weight, respectively, and the concentrations exceeded the Effects Range-Medium (ERM) sediment quality criteria [23].

2.5. Sediment water microcosms

Laboratory sediment water microcosms were established to simultaneously assess the impact of amendments on metal fractionation and bioaccumulation. These microcosms were similar

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