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## Enhancement of active thin-layer capping with natural zeolite to simultaneously inhibit nutrient and heavy metal release from sediments

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#### ABSTRACT

This study strove to enhance the efficiency of sediment capping using active thin-layer capping (ATC) with natural zeolite to simultaneously prevent the release of nutrients ( $PO_4^{3-}$  and  $NH_4^+$ ) and heavy metals ( $Pb^{2+}$ ,  $Cd^{2+}$ ,  $Mn^{2+}$ , and  $Zn^{2+}$ ) from polluted reservoir sediment. A series of lab-scale column experiments were performed in different conditions by varying capping thickness, ionic strength of Na<sup>+</sup> and Ca<sup>2+</sup>, and the redox environment in overlying water. Results showed that the release of nutrients and heavy metals could be prevented effectively using ATC with zeolite. There was no significant difference in the efficiency of nutrient (except  $PO_d^{3-}$ ) and heavy metals inhibition among three capping thicknesses. The greatest efficiency was obtained with 2 cm thickness, which the inhibition rates of  $NH_4^+$ ,  $PO_4^{3-}$ ,  $Cd^{2+}$ ,  $Pb^{2+}$ ,  $Mn^{2+}$ , and  $Zn^{2+}$ ,  $Pb^{2+}$ ,  $Mn^{2+}$ ,  $Mn^{2+}$ ,  $Nn^{2+}$ ,  $Nn^{2+$ were 91.9%, 57.7%, 35.7%, 85.7%, 65.6%, and 57.8%, respectively. And, the capping thickness of 1 cm was sufficient to simultaneously prevent the release of nutrients and heavy metals from sediment. The presence of high  $Ca^{2+}$  concentration in the overlying water promoted the release of nutrients and heavy metals. However, aerobic environment could enhance the efficiency of inhibition of nutrient and heavy metal (except  $Cd^{2+}$ ) release from sediment. Additionally, fluxes of  $NH_4^+$ ,  $PO_4^{3-}$ , and  $Pb^{2+}$  in aerobic conditions with capping were significantly lower than those in anaerobic conditions without capping in an in situ capping simulated experiment. Therefore, the efficiency of inhibiting the release of nutrients and heavy metals from sediments could be enhanced by ATC with natural zeolite through a series of measures, including increasing suitable capping thickness, creating an aerobic environment, and reducing the content of  $Ca^{2+}$  in overlying water.

### 1. Introduction

Wastewater from agriculture, industrial and recreational activities has been repeatedly discharged into water bodies, resulting in an increase in the amount of harmful substances in water and sediments (Miao et al., 2015; Salem et al., 2014). Nitrogen, phosphorus, heavy metals, and insoluble organic pollutants have all been deposited into sediments, and maintain a certain dynamic balance at the sedimentwater interface (Salem et al., 2014). The resuspension of nutrients, heavy metals, and other pollutants is affected by changing of environmental conditions (e.g. redox condition, pH, and hydrodynamic conditions), resulting in secondary pollution of water bodies (Cheng et al., 2015; Zheng et al., 2013). Nitrogen and phosphorus are the main limiting factors for phytoplankton growth and the main cause of eutrophication in water bodies (Domis et al., 2016; Drewry et al., 2009; Xu et al., 2010). Heavy metals are usually toxic to aquatic organisms, and can affect human health through the food chain (Achary et al., 2017).

Many sediment remediation techniques have been developed to prevent the release of contaminants into water. Current sediment remediation technologies include active capping (Knox et al., 2014), phytoremediation (Meagher, 2000), washing (Löser et al., 2007), dredging (Erftemeijer et al., 2012), electrochemical remediation (Virkutyte et al., 2002), and Phoslock (Spears et al., 2013) etc.. The active capping technology with chemically reactive materials can sequestrate and/or degrade contaminants from sediment and reduce their mobility, toxicity, and bioavailability (Knox et al., 2014; Zamparas et al., 2013). Capping has become a focus of current research and has

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developed rapidly from passive thick-layer capping to active thin-layer capping (Huang et al., 2011; Knox et al., 2014; Lin et al., 2011; Zhou et al., 2016).

Many materials can be used in active barrier systems, such as apatite (Knox et al., 2014), organ clay (Meric et al., 2012), zeolite (Baker et al., 2009; Lin et al., 2011), Fe-modified bentonite (Zamparas et al., 2013), calcite (Lin et al., 2011) etc.. Natural zeolite is a crystalline hydrated aluminosilicate of alkali and alkaline. It has a complex structure that consists of a framework formed by tetrahedral SiO<sub>4</sub> and AlO<sub>4</sub> that creates high cation exchange and adsorption capacities (Leyva-Ramos et al., 2008). Thus, natural zeolite is often used as an active thin-layer capping material to prevent the contaminations release from sediments. Huang et al. (2011) reported that 90–100% of total nitrogen (TN) and 89.6% of ammonium was inhibited from eutrophic lake sediment release to overlying water by zeolite active capping of 3 cm thickness in anoxic media, and the inhibition efficiency was significant better than ceramicite and light porous media capping respectively. Zhou et al. (2016) showed that TN reduction efficiency of overlying water reached a maximum (56.7%) at day 34 with bioactive thin-layer capping with biozeolite (thickness of about 2 mm). Lee and Park (2013) noted that long term release of heavy metals became negligible for a 20 mm reactive layer with a distribution coefficient of 10 g/L. However, studies on the efficiency of sediment capping with ATC using natural zeolite of different thicknesses to simultaneously inhibit nutrient and heavy metal release under anaerobic conditions are very limited. Jacobs and Förstner (1999) and Knox et al. (2014) showed that release of heavy metals was significantly inhibited under oxidized conditions. In addition, some studies founded that cations (Na<sup>+</sup>, Ca<sup>2+</sup>, Mg<sup>2+</sup> etc.) can affect on the ion exchange capacity between heavy metals and the ions of zeolite, thereby impact the adsorption of heavy metals on zeolite (Biškup and Subotić, 2005; Teutli-Sequeira et al., 2009). However, the effect of the ionic strength of  $Na^+$  and  $Ca^{2+}$ , and redox conditions in overlying water on simultaneously inhibiting the release of nutrients and heavy metals has rarely been studied.

The objectives of the present study were to enhance the efficiency of ATC using natural zeolite as a capping material to control the release of nutrients ( $NH_4^+$  and  $PO_4^{3-}$ ) and heavy metals ( $Pb^{2+}$ ,  $Cd^{2+}$ ,  $Mn^{2+}$ , and  $Zn^{2+}$ ) from sediments by changing the thickness of the capping material, the ionic strength of Na<sup>+</sup> and Ca<sup>2+</sup>, and redox conditions in the overlying water. Finally, in order to verify an ATC system with optimum conditions to prevent nutrient and heavy metal release from sediment, the efficiency of inhibition was evaluated by an in situ capping simulated experiment.

#### 2. Material and method

#### 2.1. Experimental material

Surface sediments (0–10 cm) were collected from a polluted reservoir in Guangdong Province, China. The sediments were freezedried, then preserved in polyethylene bags after sieving through a 100 meshsieve. The main characteristics of the sediment are as follows: TOC (total organic carbon) 1.9%, pH 6.0, TP (total phosphorus) 1103.1 mg/ kg, TN 1664.4 mg/kg, Cr 89.0 mg/kg, Cu 115. 9 mg/kg, Mn 1022.8 mg/ kg, Ni 72.6 mg/kg, Pb 74.3 mg/kg, Zn 281.7 mg/kg, and Cd 0.5 mg/kg.

Natural zeolite with a particle size of 7–8 mm was purchased in Guangdong Province, China. Its chemical composition is as follows: CaO (0.9%), SiO<sub>2</sub> (69.0%), Fe<sub>2</sub>O<sub>3</sub> (2.0%), Al<sub>2</sub>O<sub>3</sub> (10.3%), MgO (0.5%), MnO (0.06%), K<sub>2</sub>O (0.6%), Na<sub>2</sub>O (3.4%), P<sub>2</sub>O<sub>5</sub> < 0.01%. Its properties are as follows: specific surface area 14.2 m<sup>2</sup>/g, porosity 0.02 cm<sup>3</sup>/g, and aperture 7.4 nm.

#### 2.2. Adsorption experiment

The purposes of these experiments were to investigate the adsorption capacity of  $\rm NH_4^+$ ,  $\rm PO_4^{3-}$ ,  $\rm Pb^{2+}$ ,  $\rm Mn^{2+}$ ,  $\rm Zn^{2+}$ , and  $\rm Cd^{2+}$  on natural zeolite, and to discuss the mechanism of inhibiting release using ATC with natural zeolite. Standard solutions (pH 7.0) were prepared of  $\rm NH_4^+$ ,  $\rm PO_4^{3-}$  (5–100 mg/L),  $\rm Cd^{2+}$  (10–150 mg/L),  $\rm Pb^{2+}$ ,  $\rm Mn^{2+}$ , and  $\rm Zn^{2+}$  (30–300 mg/L) using stock solutions diluted with Milli-Q water. Standard solutions (40 mL) of different concentrations were added to a 250 mL Erlenmeyer flask with 2.5 g natural zeolite. After stirring for 24 h in a constant incubator (25 °C, 200 rpm), the aqueous samples were centrifuged at 4000 rpm for 15 min, and then supernatant was taken for  $\rm NH_4^+$ ,  $\rm PO_4^{3-}$ ,  $\rm Pb^{2+}$ ,  $\rm Mn^{2+}$ ,  $\rm Zn^{2+}$ , and  $\rm Cd^{2+}$  analysis. Each experiment was performed in triplicate.

#### 2.3. Column experiment

#### 2.3.1. Inhibition efficiency experiment

The aim of these experiments was to research the efficiency of ATC with natural zeolite in preventing nutrient and heavy metal release from sediments by changing experimental conditions. Black polypropylene columns were constructed in column experiments of 20 cm height and 20 cm inner diameter. The content of nutrients and heavy metals in previous studies is significantly higher than that found in sediment from our study reservoir (Antunes et al., 2013; Guhathakurta and Kaviraj, 2000; Özmena et al., 2004). In order to improve the applicability of our findings on inhibiting the release of contaminants from sediment, reservoir sediment was spiked with a mixture of heavy metals and nutrients. The final concentration of contaminants was:  $Pb^{2+}$  300 mg/kg,  $Cd^{2+}$  50 mg/kg,  $Mn^{2+}$  1000 mg/kg,  $Zn^{2+}$  500 mg/kg,  $PQ_4^{3-}$  500 mg/kg, and  $NH_4^+$  1000 mg/kg. Contaminated sediments were placed in the bottom of each column to a depth of 3 cm. A total of 30 columns were set up, divided into 3 experimental cells.

(i) *Thickness*: natural zeolite was added, with a thickness of 0 (control group), 0.5, 1.0, and 2.0 cm (experimental groups), on the top of contaminated sediments in columns. Then, 4 L Milli-Q water was added to each column, which was sealed with a lid to maintain an anaerobic environment.

(ii) Ionic strength of  $Na^+$  and  $Ca^{2+}$  in overlying water: different concentrations of CaCl<sub>2</sub> and NaCl (0 + 0, 0.01 + 0.01, 0.01 + 0.05, and 0.05 + 0.01 mol/L) were added to the overlying water. Then, natural zeolite with a capping thickness of 1.0 cm was added to columns for experimental groups, and 4 L Milli-Q water was added into columns for the control group. All columns were sealed with lids to maintain an anaerobic environment.

(iii) *Redox conditions*: Milli-Q water (dissolved oxygen (DO) < 1.0 mg/L) was added to all columns, some of which were sealed with lids as anaerobic control groups. The experimental groups were subjected to an aerobic environment with no lid and artificial aeration lasting for an hour every 6 h to maintain DO > 5.0 mg/L.

Experimental design is shown in Fig. A1. Each experiment was performed in triplicate.

#### 2.3.2. In situ capping simulated experiment

Bench-scale in situ capping simulated experiments with optimal combinations of capping thickness and redox conditions were set up to evaluate the efficiency of sediment capping using ATC with natural zeolite in inhibiting nutrient and heavy metal release from sediment. Reservoir sediments were filled in the bottom of each column to a depth of 3 cm, and reservoir water was added. The inhibition efficiency experiments showed that high concentrations of  $Ca^{2+}$  (0.05 mol/L) promoted the release of pollutants from sediment, while low

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