



Research Paper

Rhamnolipid stabilized nano-chlorapatite: Synthesis and enhancement effect on Pb- and Cd-immobilization in polluted sediment



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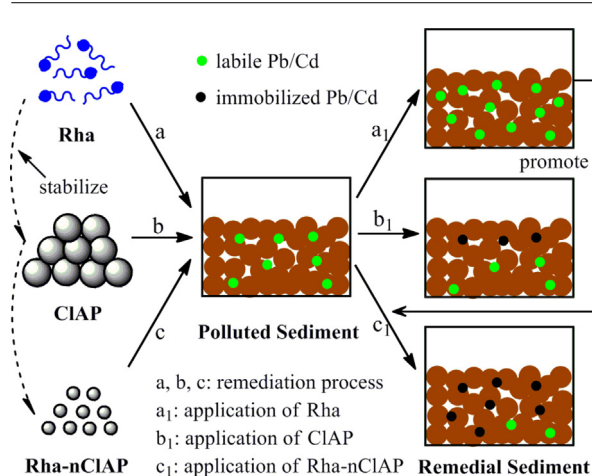
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HIGHLIGHTS

- Rha-nCIAP was synthesized with the combination of Rha and CIAP.
- Rha-nCIAP could effectively immobilize Pb and Cd and reduce the TCLP-leachable Pb and Cd.
- Rha could enhance the Pb and Cd immobilization by promoting the metal liberation from sediment.
- The organic matter and available phosphorus indicated the microorganism may involve.

GRAPHICAL ABSTRACT



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ABSTRACT

Phosphate (P) compounds are usually used as chemical amendment for *in situ* remediation of heavy metal polluted sediment. However, the low deliverability, weak utilization and potential risk of eutrophication inhibit the application of most P materials. Therefore, rhamnolipid (Rha), a kind of anionic biosurfactant which has algicidal activity, was employed in this study to synthesize a new kind of nano-chlorapatite (nCIAP) for Pb and Cd immobilization. Characterization results showed that the Rha stabilized nCIAP (Rha-nCIAP) was uniformly distributed in suspensions within about 5 nm. Experimental data demonstrated that the combination of Rha and nCIAP could greatly enhance the Pb- and Cd-immobilization efficiencies, promoting their transformation from labile fractions to stable fractions through precipitation or adsorption processes, especially when the Rha approached to its critical micelle concentration. And Rha-nCIAP could also decrease both the TCLP-leachable Pb and Cd with maximum reduction efficiencies of 98.12% and 96.24%, respectively, which also presented concentration dependence

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of Rha. Changes of available phosphorus implied the dissolution of nCIAP during the treatment and the detection of organic matter demonstrated that the microorganisms may involve in the remediation.

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1. Introduction

Lead (Pb) and cadmium (Cd) are widespread contaminants in sediments and soils [1–3]. They usually came into the environment through metal smelting, waste discarding, rubbish incineration and the application of agrochemicals [3–6]. Sediment polluted with Pb and Cd is of great concern to the government and researchers since they could accumulate in human body through the uptake by plants and other organisms [7–11]. Pb and Cd can not be degraded through microbial or chemical processes and they could persist in sediment for a long time, posing a potential toxicity to organisms [12–18]. It is necessary to find an effective method to deal with the polluted sediment since heavy metals in sediment are the intermediate sources for the occurrence of heavy metal pollution in aquatic ecosystem [19,20]. Current technologies for heavy metal remediation in sediment are divided into *in situ* and *ex situ* methods. Generally, the *ex situ* method aims to remove the polluted sediments for the further treatment or dispose it while the *in situ* method figures on preventing the toxic metals from accessing to the aquatic ecosystem by capping an inert material or transferring the metals into a stable fraction with the using of various amendments [21,22].

Phosphate (P) amendments, including soluble phosphate, insoluble phosphate and other synthesized P-based materials, are of great importance for heavy metal remediation since P-materials could cooperate well with many heavy metals such as Cd(II), Pb(II), Cu(II), and Zn(II) and convert these metals from unstable fraction to stable fraction, reducing their toxicity and bioavailability [23–27]. Phosphates of Pb [such as $Pb_5(PO_4)_3F$, $K_{sp} 10^{-71.6}$; $Pb_5(PO_4)_3Cl$, $K_{sp} 10^{-84.4}$; $Pb_5(PO_4)_3Br$, $K_{sp} 10^{-78.1}$; $Pb_5(PO_4)_3(OH)$, $K_{sp} 10^{-76.8}$] and Cd [such as $Cd_5(PO_4)_3(OH)$, $K_{sp} 10^{-42.49}$; $Cd_5(PO_4)_3Cl$, $K_{sp} 10^{-49.66}$] were much less soluble than other Pb and Cd compounds [28,29]. According to BCR (European Community Bureau of Reference) sequential extraction method, these metal-phosphate compounds are often recognized as the residual fraction (F-res) [other fractions are: acid soluble/exchangeable fraction (F-exc); reducible fraction (F-red) and oxidizable fraction (F-oxi), the stability of them are in order of: F-exc < F-red < F-oxi < F-res] which is insoluble and almost unreactive under most pH and oxidative conditions [30–32]. Therefore, more and more researchers pay their attention on the P-based materials for *in situ* remediation and results showed that P was effective to immobilize Pb and Cd in soils and eliminate their bioavailability [1,33–37]. Previous studies showed that the immobilization efficiency of P mostly depends on the amount of available phosphorus in contaminated sediments and soils since the soluble P performed much better than the insoluble P during the remediation [25,38,39]. However, the most intractable problem associated with P application appears to be the water eutrophication induced by the leaching of excess P, especially in case of soluble P [28]. In order to enhance the immobilization efficiency and decrease the eutrophication risk of P-materials, researchers tried to modify the insoluble P-material (in which the P is much less bioavailable to the algae than soluble P) with different substrates (such as carboxymethyl cellulose, sodium dodecyl sulfate and biochar) to get nano-sized, well-dispersed and easily-delivered P particles in recent years [1,2,35,40].

Rhamnolipid (Rha), a kind of environmental friendly anionic biosurfactant which produced by *Pseudomonas aeruginosa* and has algicidal activity on the harmful algal bloom species was employed

in this study to synthesize a new kind of stabilized nano-chlorapatite (nCIAP) for Pb and Cd remediation in polluted sediment [41]. In light of the problems associated with P application, this paper hypothesizes that the combination of rhamnolipid and chlorapatite would enhance the (Pb and Cd) immobilization efficiency of chlorapatite and at the same time decrease the eutrophication risk induced by P. To investigate the Pb and Cd immobilization efficiency of synthesized nano-chlorapatite and understand the immobilization mechanism involved, the BCR sequential extraction method, toxicity characteristic leaching procedure (TCLP), available phosphorus and organic matter are measured during the remediation.

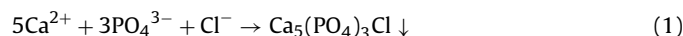
2. Materials and methods

2.1. Materials

All the chemicals used in this study were of analytical or higher grade. Rhamnolipid (Rha, the mixture of Rha-Rha-C₁₀-C₁₀ and Rha-C₁₀-C₁₀) was purchased from Huzhou Zijin Biological Technology company. The ultrapure water (18.25 MΩ cm, 25 °C) was prepared by an Ultra-pure Water System and all the equipments were washed extensively with the ultrapure water. The experiment was conducted in triplicate and the results were expressed as mean ± standard deviation.

2.2. Preparation and characterization of Rha-nCIAP

Rhamnolipid stabilized chlorapatite (Rha-nCIAP) was synthesized according to the steps proposed by Liu and Zhao [1] with some modifications: a 30 (50 and 60) mg/L Rha solution, a 26.8 mM Ca²⁺ and a 16.0 mM PO₄³⁻ solution were prepared by dissolving Rha, CaCl₂·2H₂O and Na₃PO₄·2H₂O into the ultrapure water accurately, respectively. The pH of Rha solution was adjusted to 8.40 by using an appropriate amount of 50 mM NaOH solution. Then 25 mL of 26.8 mM Ca²⁺ was dropped slowly (5–8 drops per minute) into 50 mL 30 (50 and 60) mg/L Rha solution by the acid burette under the constantly mixing (magnetic stirrer with a Teflon-coated stir bar) at 1000 r/min. After constantly stirring for 12 h, another 25 mL of 16.0 mM PO₄³⁻ solution was added to the mixture under constantly mixing with 1500 r/min at 5–8 drops per minute. To give a completely blending of Rha and CIAP, the mixture was continued stirred for 12 h at 1500 r/min after the addition of PO₄³⁻. A control experiment that used the ultrapure water (pH = 8.40) instead of Rha solution was prepared with other conditions unchanged. Fig. 1 is the laboratory flowchart about the synthesis of Rha-nCIAP. Concentrations of Rha were chosen on the base of its critical micelle concentration (CMC, about 25 mg/L) which exerts great impact on the properties of Rha. The molar ratio of Ca²⁺ to PO₄³⁻ was 1.67 in respect to the chemical formula of CIAP [Ca₅(PO₄)₃Cl] according to Liu and Zhao [1]:



To investigate the particle distribution of synthesized Rha-nCIAP and observe its cross section structure, a drop of Rha-nCIAP suspension was placed on a carbon-coated copper grid and kept at 28 °C for 24 h for transmission electron microscopy (TEM, JEM-3010, JEOL, Japan) analysis. Then the Rha-nCIAP suspension was freeze-dried by a freeze-dryer (LGJ-10B, Beijing sihuan scientific

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