



Research article

Chemical and biological assessment of Cd-polluted sediment for land use: The effect of stabilization using chitosan-coated zeolite

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ARTICLE INFO

Article history:

Received 13 November 2017

Received in revised form

16 January 2018

Accepted 28 January 2018

Available online 22 February 2018

Keywords:

Sediment

Zeolite

Recycle

Heavy metal

Bio concentration factor

ABSTRACT

Disposal of dredged sediment contaminated with heavy metals on site or in landfills inevitably causes leaching of metals that generate new environmental problems. In this study, we investigated the effectiveness of stabilizing heavy metal Cd in sediment taken from Dongting Lake, China, using a chitosan-coated zeolite, and assessed the feasibility of reusing the stabilized sediment in river bank soil based on chemical and biological analyses. Results showed that the addition of chitosan-coated zeolite significantly reduced acid-exchangeable Cd by 8% in the dredged sediment and 7% in a sediment–soil mixture. Cadmium leachability was greatly reduced in the amended sediment or sediment–soil mixture. Toxicity bioassay using *Eisenia fetida* showed the mortality rate of worms reduced by 16% in sediment–soil mixture with a Cd concentration of 550 mg/kg and by 17% under a Cd concentration of 250 mg/kg, both with the addition of modified zeolite. Moreover, assimilation of Cd in the earthworms was decreased by a maximum of 36 mg/kg in the sediment–soil mixture with zeolite amendment. These results indicate that the reuse of Cd-contaminated sediment following chitosan-coated zeolite modification is a feasible option for treating the dredged sediment, and could thus benefit both aquatic and terrestrial systems.

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1. Introduction

Periodical dredging of river or lake sediment to allow for greater drainage capacity has been commonly employed worldwide. However, these dredging activities produce vast amounts of sediment waste and disposal of the dredged material has become an issue of concern. Much of the dredged sediment is sent to landfills or other disposal sites and has been reported to cause secondary contamination, especially of heavy metals, in the terrestrial part of alluvial systems (De Witte et al., 2016; Vandecasteele et al., 2002).

Previous work has demonstrated that dredged sediments could be applied to soil for agricultural purposes (Chen et al., 2002; Ruiz Diaz et al., 2010). It is assumed that sediment is derived from high-quality topsoils in the watershed and therefore it should have desirable soil characteristics such as efficient storage of plant-available water, good level of cation exchange capacity, and high fertility (Darmody and Marlin, 2002). However, elevated

concentrations of heavy metals from the dredged sediment may become a hindrance for direct application to soil due to potential bioaccumulation (Chen et al., 2002). An example of this problem is Dongting Lake located in mid-China where severe Cd contamination in sediment has been reported (Ding and Li, 2011; Li et al., 2013; Yi et al., 2017). This accumulation of Cd in the sediment is due to historical industrial activities conducted in the upper water stream over a long period (Yao et al., 2006). Therefore, it is necessary to produce a strategy that could convert the dredged sediments into a form that is appropriate for reuse in the terrestrial system. Yu et al. (2009) suggested and found that the heavy-metal-polluted sediment could be treated with *in-situ* stabilization treatment and *ex-situ* composting to allow for a safe use as a fertilizer for river bank plants. However, little further effort has so far been made in producing such a strategy.

It is well established that the solidification/stabilization treatment method, either *in-situ* or *ex-situ*, is successful in the remediation of polluted soils and sediments (Huang et al., 2016; Jacobs and Förstner, 1999; Khan et al., 2015; Muller and Pluquet, 1998; Tomasevic et al., 2013; Wen et al., 2018). This approach involves the addition of chemicals or cement to encapsulate

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contaminated sediments or convert them into less toxic and mobile forms. Among the various amendments, zeolite stands out as an excellent stabilizer because of its low cost and selectively high adsorption for heavy metals (Ashmawy et al., 2012; Janoš et al., 2010; Lin et al., 1998; Penilla et al., 2006). In view of its large specific surface area and selective adsorption through ion exchange, zeolite is capable of adsorbing various metals in a decreasing order of $\text{Ag}^+ > \text{Pb}^{2+} > \text{Cd}^{2+} > \text{Zn}^{2+} \geq \text{Cu}^{2+} > \text{Ni}^{2+} > \text{Hg}^{2+}$ (Mondale et al., 1995). Considerable research efforts have been devoted to the modification of zeolites, which enable them to possess characteristics conducive to metal adsorption (Ates and Akgül, 2016; Bowman, 2003; Kim et al., 2013; Peng et al., 2013). In particular, zeolite has been modified with a coating of chitosan, a metal binding chelant, to successfully remove organic or inorganic contaminants from aqueous solutions (Peng et al., 2013, 2015; Wan Ngah et al., 2013). Notably, most stabilization work using zeolite or modified zeolite material has been confined in a single system, i.e. soil, sludge or sediment. If the zeolite can be applied in the dredged sediment to alleviate the potential toxicity of heavy metals, it is possible that the sediment can be returned to the land, taking advantage of its high nutrient content while keeping its hazard to a low level.

As stabilization treatment aims to reduce the bioavailable forms rather than the total amount of metals in either soils or sediments, it is therefore necessary to provide an assessment of the bioavailable metals via chemical and biological methods. In particular, BCR sequential extraction proposed by the Community Bureau of Reference (now European Community Standards Measurements and Testing Programme) and the toxicity characteristic leaching procedure (TCLP) produced by the US Environmental Protection Agency (EPA) are two frequently applied testing methods for chemically evaluating the speciation and potential toxicity of heavy metals (Delgado et al., 2011; Fathollahzadeh et al., 2013; Ferraro et al., 2015; Kartal et al., 2006; Mittermüller et al., 2016; Pan et al., 2012). Toxicologists are more inclined to apply a toxicity bioassay to explain any ecological change raised by the heavy metals. In view of this, soil animals are often used as bioindicators in standard bioaccumulation tests and ecotoxicological studies. In particular, the earthworm *Eisenia fetida* is an ideal model animal for assessing the toxicity of pollutants in soil because of its prevalence in soil environment and sensitivity towards contaminants (Lee et al., 2009a, 2009b; Lemtiri et al., 2016; Nirola et al., 2016; Udovic and Lestan, 2007; Zhang et al., 2017). Mixing the stabilized sediment and soil may improve both physical and chemical conditions of the soil while diminishing the potential bioaccumulation of heavy metals in soil organisms. It is therefore worthwhile to analyze the chemical speciation alongside bioavailability of Cd during remediation and reuse of the Dongting Lake sediment. The purposes of this study were to evaluate the effectiveness of chitosan-modified sediment on Cd stabilization in the sediment and a sediment–soil mixture, and to understand the response of earthworms to Cd toxicity in soil amended with zeolite-modified sediment.

2. Materials and methods

2.1. Zeolite modification

The natural zeolite (particle size 0.5–1.0 mm) used in this experiment came from Gongyi, Zhengzhou (Henan Province, China). The zeolite was immersed in HNO_3 solution (1 mol/L) for 4 h and then washed with deionized water. Then zeolite and chitosan in a mass ratio of 1:1 were put in 5% acetic acid and mixed well on magnetic stirrer at 100 rpm for 3 h. The hydrogel of chitosan-zeolite complex was then cautiously added to NaOH solution (0.5 mol/L)

dropwise, and mixed on a stirrer at 100 rpm for 3 h. The precipitate was then triple washed with deionized water and left to air-dry (Wan Ngah et al., 2013). The natural and modified zeolite were then tested for pH and electrical conductivity (EC) (soil/water = 1:5 (w/v)). Both zeolites were subjected to total metal digest for possible concentrations of Cu, Pb, Zn, Cd, Cr and Ni. Concentrations of metals in solutions were determined by inductively coupled plasma-optical emission spectrometry (ICP-OES) (PE5300D, PerkinElmer, USA). The modified zeolite was subjected to X-ray diffraction analysis (XRD) (2555, Rigaku, Japan) to assess any structural change in crystallinity, and scanning electron microscopy (SEM) (JSM-6700F, JEOL, Japan) to assess morphological changes on the surface. An N_2 adsorption test was also conducted to obtain the specific area and pore size of the zeolite pre- and post-modification.

2.2. Sediment and soil sample preparation

Sediment samples were collected from Dongting Lake (113°07'13"N, 29°24'38"E), Hunan, China, in Spring 2016. Soil was also taken from an adjacent open land area (113°06'53"N, 29°25'20"E). The upper 20 cm depths of the sediment or soil were randomly collected, homogenized, and stored in prepared brown glass bottles prewashed with diluted nitric acid (10% v/v). All the collected samples were then air-dried, crushed, and passed through a 2-mm sieve. The sediment and soil subsamples were then tested for pH, EC, organic matter, water content and pseudo-total metal concentration of Cu, Pb, Zn and Cd.

To assess the effect of zeolite on stabilizing Cd in either the sediment or mixture, the natural or modified zeolite was added to a subset of the sediment or soil samples. In short, for the sediment only series, 10 g of natural or modified zeolite was mixed with each 100 g of sediment (d.w.). For the sediment–soil mixture, 25 g of the contaminated sediment was firstly mixed thoroughly with 75 g of the soil. Subsequently, 10 g of the natural or modified zeolite was added and mixed well with the soil–sediment mixture. The treatment setup was shown in Table 1. The sediment or sediment–soil samples were kept at 30% moisture in a dark and cool place for an incubation period of 3 months.

2.3. BCR sequential extraction

The BCR sequential extraction procedure was applied to all sediment + zeolite mixtures and sediment + soil + zeolite mixtures by adopting methods in Fathollahzadeh et al. (2013). In short, Cd in the sediment was divided into four geochemical fractions: acid-exchangeable fraction (F1), reducible fraction (F2), oxidizable fraction (F3) and residual fraction (F4). The pseudo-total concentration of Cd was determined by treating 0.2 g of sample with $\text{HNO}_3/\text{HClO}_4/\text{HF}$ acid mixture digestion method at 120 °C until the digested solution was clear (Sun et al., 2001). All reagents used to perform the extraction were of analytical grade. Prior to the

Table 1
Treatment setup of the study.

	Sediment (d.w.)	Soil (d.w.)	Zeolite (d.w.)
Sediment (S)	100 g	–	–
Sediment + Zeolite (S+Z)	100 g	–	10 g
Sediment + Chitosan-modified zeolite (S+CZ)	100 g	–	10 g
Soil + Sediment (S+S)	25 g	75 g	–
Soil + Sediment + Zeolite (S+S+Z)	25 g	75 g	10 g
Soil + Sediment + Chitosan-modified zeolite (S+S+CZ)	25 g	75 g	10 g

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