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A three-year in-situ study on the persistence of a combined amendment (limestone + sepiolite) for remedying paddy soil polluted with heavy metals

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ABSTRACT

In order to study the persistence of a combined amendment (LS, limestone + sepiolite) for remedying paddy soil polluted with the heavy metals Pb and Cd, a three-year in-situ experiment was conducted in a paddy soil near a mining area in southern Hunan, China. LS was applied at rates of 0, 2, 4, and 8 g/kg (w/w); rice was subsequently planted for the three consecutive years of 2012 (first season), 2013 (second season), and 2014 (third season). Experimental results indicated that LS significantly increased soil pH values for all three seasons, and the enhancement ranked as follows: first season > second season > third season. Under the experimental conditions, the effect of LS on decreasing exchangeable concentrations of soil Pb and Cd was as follows: first season (97.6–99.8% for Pb and 88.3–98.9% for Cd) > second season (80.7–97.7% for Pb and 28.3–88.0% for Cd) > third season (32.6–97.7% for Pb and 8.3–71.4% for Cd); the effect of LS on reducing Pb concentrations in brown rice was: first season (73.5–81.2%) > third season (29.6–68.1%) > second season (0–9.7%), and that for reducing Cd concentrations in brown rice was third season (72.7–81.0%) > first season (56.1–66.8%) > second season (20.9–32.3%). For all three seasons, the effect of LS on reducing Cd content in brown rice was better than that for Pb. The highest translocation factors for Pb and Cd were from rice straw to husk, implying that the husk of rice plants was the main organ in which heavy metals accumulated. The effect of LS for decreasing soil exchangeable Cd content was relatively persistent, but that for Pb gradually decreased with time, implying that LS was more suitable for the long-term remediation of Cd-polluted soil than Pb-polluted soil.

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1. Introduction

With the rapid development of agriculture and industry, soil environmental pollution is increasing, especially with regard to heavy metal contamination. Many sources of contamination, such as metal manufacture, mining industries, residue disposal, and transportation, originate from human actions (Zhang et al., 2005). During the process of mining and the grinding and concentrating of ores, dust is emitted, wastewater is discharged, and mine tailings are created (Liu et al., 2005; Lei et al., 2010). Heavy metal contamination involves the greater than normal accumulation of heavy metals in soil, mainly from human activities, which result in a deterioration of the ecological environment. Heavy metals are different from other pollutants; they are non-degradable, non-biodegradable, and hard to translocate. They can accumulate in plants and animals, and can be extremely persistent in the environment (Ren et al., 2009; Arwidsson et al., 2010). Following their accumulation in the edible parts of crops, they can enter human bodies through the food chain and threaten human health (Basta et al., 2001; Madrid et al., 2006). For this reason, soil heavy metal pollution has led to public concern, and many methods have been adopted to deal with this problem. As a type of in-situ remediation technology, chemical fixation has developed rapidly in recent years due to its efficiency and low cost. Chemical remediation is a particularly effective method for the treatment of mild-to-moderate pollution of heavy metal contaminated soil (Mulligan et al., 2001; Diels et al., 2002; Madrid et al., 2006). For non-point source pollution caused by agricultural activities, chemical remediation also has obvious advantages, but existing methods of physical, chemical, and biological analysis provide only limited information. Methods to monitor the bioavailability of metallic elements in soil (Guo et al., 2005) and to ensure remediation efficiency over long periods will be key research

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priorities for the future remediation of heavy metal soil contamination.

At present, numerous additives have been incorporated into soils polluted with metal (loids) to immobilize pollutants (González et al., 2012). These additives include individual additions of lime, calcium carbonate, fly ash (Lombi et al., 2003; Friesl et al., 2006; Liu et al., 2007), metal oxide (Markus et al., 2004; Kumpiene et al., 2006), and combinations of amendments, including limestone and fly ash (Herwijnen et al., 2007), and phosphate rock and phosphoric acid (Cao et al., 2009). However, these studies have focused predominantly on pot experiments, with little field experimentation or tests of remediation efficiency over long periods of time.

In order to study the persistence of a combined amendment (LS, limestone+sepiolite) for remedying paddy soil polluted with the heavy metals Pb and Cd, an in-situ experiment was conducted. Limestone, hydroxyapatite, sepiolite, zeolite, diatomite, and bentonite were selected and tested for their ability to remediate heavy metal contaminated soil in experiments that took place within the laboratory. Single amendment and combined amendment (two amendments combined in the following ratios: 1:2, 1:1, and 2:1) were used in a preliminary experiment. LS (consisting of limestone ($CaCO_3$) and sepiolite, 2:1) was found to be more effective in decreasing heavy metal concentrations of exchangeable fractions in soil than a single amendment or other combined amendments (Zeng et al., 2012). Therefore, we made a one-off application of the latter combined amendment to an area of paddy soil and subsequently cultivated rice on it for three consecutive years. The main objectives of this study were to observe changes in the physicochemical properties of the tested soil, to understand the translocation and accumulation of Pb and Cd in the soil and rice plants, and to study the persistence of remediation effects on heavy metal contaminated soil under different LS applications.

2. Materials and methods

2.1. Experimental materials

Due to many years of exploitation, heavy metal contamination of paddy soil around the selected mining area (26°34.7′N, 112°35.6′E) in southern Hunan, China, had become increasingly serious. In April 2012, an in-situ chemical modification test using the combined amendment LS took place near the mining area to attempt to remedy the polluted soil. The mean annual temperature for the area ranged between 16.8 and 17.2 °C, and mean annual precipitation was about 1400 mm. Irrigation water was taken from a clean pool near the paddy soil. The basic physicochemical properties of the paddy soil before LS application are shown in Table 1. The information of rainfall and sunshine days on rice growing conditions during experiments for the years 2012, 2013, and 2014 are given in Table 2.

The combined amendment (LS) was mixed evenly at a ratio of limestone: sepiolite of 1:2. LS particle size was <0.2 mm. Limestone (calcium carbonate content $>98\%$) and sepiolite (sepiolite content $>80\%$) does not dissolve basically in water. These materials were both provided by Shang Jie Ore Powder Factory, Ningxiang County in Hunan Province, and the properties of the ingredients are shown in Table 1. The rice cultivar used was "Huang-Hua-Zhan" (conventional rice, Hunan Agricultural Abundant Seed Industry Co., Ltd). Fertilizer applications included plant ash, urea, and ammonium bicarbonate.

Table 1
Basic properties of the tested materials.

	pH	BS (%)	CEC (cmol/kg)	Pb (mg/kg)	Cd (mg/kg)	Cu (mg/kg)	Zn (mg/kg)
Paddy soil	5.39	3.66	19.93	257	3.03	43.9	349
National Standard II	–	–	–	250	0.30	50.0	200
Sepiolite	5.54	–	–	2.75	–	–	48.2
Limestone	9.13	–	–	1.63	–	–	7.76

Table 2
Information of rainfall and sunshine days on rice growing conditions for years 2012, 2013, and 2014.

	Climate condition	May (days)	June (days)	July (days)	August (days)
First season	Rainfall	25	24	14	16
	Sunshine	6	6	17	15
Second season	Rainfall	20	16	19	15
	Sunshine	11	14	12	16
Third season	Rainfall	23	19	20	21
	Sunshine	8	11	11	10

2.2. Experimental methods

LS was applied evenly to the selected paddy soil plots. Each plot area was 9 m^2 (3 m × 3 m). The amount of LS applied was set as 0, 2, 4, and 8 g/kg (w/w), and three replicates were conducted. The experimental fields were divided into 12 sample plots, all plots were randomly arranged, and soil moisture was maintained for 21 days until rice transplantation. Rice was planted in the paddy soils for three consecutive seasons. Accelerated germination and rice seedling culture took place in the middle of April 2012, 2013, and 2014, respectively. At the beginning of each May, the rice cultivar "Huang-Hua-Zhan" was transplanted, and three guard rows were set up around each soil plot. Planting density and management were set according to customary local agricultural practice. Plant ash application was 500 g/plot and mixed with wet soil for 15 days before rice cultivars. Urea application was 250 g/plot 30 days later than plant ash applying to paddy soil. Ammonium bicarbonate application was 220 g/plot 5 days later than urea applying to paddy soil. Water managements were used controlled irrigation according to the methods of local agricultural production. At harvest in the middle of August 2012, 2013, and 2014, three rice plants from each soil plot were collected randomly and were stored in polyethylene bags. Rhizosphere soil from each rice plant was separated from the roots by gently shaking off the soil attached to the roots. All samples were stored in polyethylene bags for transport. After plants were taken back to the laboratory, they were cleaned using tap water and distilled water, placed in a 105 °C drying oven for 30 min after airing, and then placed in a 70 °C drying oven until the weight of samples remained constant. Rice plants were then separated into three tissues parts (root, straw, and grain). Grain was hulled with a small huller (JLGJ4.5, China), husk and brown rice were collected, and straw was crushed using a micro mill (RT-02B, China). The crushed powder was passed through a 100-mesh sieve and kept in a sealed plastic bag for analysis. Rhizosphere soils were air-dried, crushed, passed through a 100-mesh sieve, and preserved for further analysis.

2.3. Sample analysis and testing methods

Soil pH was measured with an acidity meter (PHS-3C, ray magnetic), and the solid-to-liquid ratio was m (solid): v (liquid)= 1:2.5. Soil samples were acid-digested with aqua regia and

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