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Effect of soil washing with only chelators or combining with ferric chloride on soil heavy metal removal and phytoavailability: Field experiments

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HIGHLIGHTS

- On-situ soil washing with mixture of chelators (MC) combining with $FeCl_3$ exhibited efficient metals removal rates from soils.
- The plants growth in co-cropping was inhibited on the washed soil.
- Soil washing did not worsen groundwater contamination during the study period.
- Soil amendment with lime and organic fertilizer after soil washing improved crop growth.

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ABSTRACT

In a field experiment on multi-metal contaminated soil, we investigated the efficiency of Cd, Pb, Zn, and Cu removal by only mixture of chelators (MC) or combining with $FeCl_3$. After washing treatment, a co-cropping system was performed for heavy metals to be extracted by *Sedum alfredii* and to produce safe food from *Zea mays*. We analyzed the concentration of heavy metals in groundwater to evaluate the leashing risk of soil washing with $FeCl_3$ and MC. Results showed that addition of $FeCl_3$ was favorable to the removal of heavy metals in the topsoil. Metal leaching occurred mainly in rain season during the first co-cropping. The removal rates of Cd, Zn, Pb, and Cu in topsoil were 28%, 53%, 41%, and 21% with washing by $FeCl_3$+MC after first harvest. The application of $FeCl_3$ reduced the yield of *S. alfredii* and increased the metals concentration of *Z. mays* in first harvest. However, after amending soil, the metals concentration of *Z. mays* in $FeCl_3$+MC treatment were similar to that only washing by MC. The grains and shoots of *Z. mays* were safe for use in feed production. Soil washing did not worsen groundwater contamination during the study period. But the concentration of Cd in groundwater was higher than the limit value of Standard concentrations for Groundwater IV. This study suggests that soil washing using $FeCl_3$ and MC for the remediation of multi-metal contaminated soil is potential feasibility. However, the subsequent measure to improve the washed soil environment for planting crop is considered.

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1. Introduction

Heavy metal contamination in soil is a major environmental problem worldwide that is caused by the development of mining industry and its activities, waste water irrigation, and sewage sludge application (Luo et al., 2012; Zhang et al., 2013; Jelusic and Lestan, 2014a). This problem is particularly common in China (Li et al., 2014). According to the bulletin of the national soil pollution reported by Ministry of Environmental Protection and Ministry of Land and Resources of the People's Republic of China, 2014, the total over-standard rate was 16.1% in national soil, and the over-standard rates of Cd, Zn, Pb, and Cu were 7.0%, 0.9%, 1.5%, and 2.1%, respectively. Excessive accumulation of heavy metals in agricultural soils has led to elevated heavy metal uptake by crops. This phenomenon affects the yield and quality of the crops; heavy metals entering the food chain may potentially be hazardous to

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human beings (Kuo et al., 2006; Wu et al., 2007; Murakami et al., 2009; Alvarenga et al., 2014). The need for agricultural production is increasing because of population explosion. Therefore, studies on contaminated agricultural soil are urgently needed to remediate the soil and to improve food safety.

The remediation technologies for metal-contaminated soil include chemical, physical, or biological techniques. Chemical techniques, such as soil washing with chelating agents, are potentially useful for remediating contaminated soils (Peters, 1999; Lestan et al., 2008). Many different chelators have been tested for soil washing. In the literature, ethylenediaminetetraacetic acid (EDTA) is the most frequently cited chelating agent for potentially toxic trace metals extracted from soils because EDTA has strong chelating ability for different heavy metals (Sun et al., 2011; Lestan et al., 2008; Finzgar et al., 2014). However, after EDTA-enhanced soil washing, a significant part of metal-EDTA complexes are retained in the soil by the adsorption with the mineral surface such as soil iron oxides (Nowack and Sigg, 1996; Nowack, 2002; Zhang et al., 2010). Guo et al. (2013) found that residual EDTA that remained in the soil was 0.812 mmol kg^{-1} when soil was washed by EDTA at 10 mmol kg^{-1} and the addition of lime significantly increased the release of metal-EDTA complexes into the soil solution among EDTA-washed acid soil. Therefore, the removal rates of Cd, Zn, Pb, and Cu after soil washing with mixture of chelators (MC) at pH value 7 were 1.56-, 1.08-, 2.87-, and 1.75-fold higher, respectively, compared to the initial MC at pH 2.75 (Guo et al., 2011a).

Other washing chemicals include salt and high-concentration chloride solution, such as calcium chloride and iron (III) chloride (Makino et al., 2007). Makino et al. (2006) reported that $FeCl_3$ was selected as soil-washing chemical for Cd-contaminated paddy soils because of its Cd-extraction efficiency, cost effectiveness, and relatively low environmental impact. However, people pay more attention to the leaching risks of heavy metals in soil washing technology (Wu et al., 2004; Luo et al., 2006). Therefore, permeable barriers to reduce the leaching risk were tested in pot experiments (Kos and Lestan, 2003). Wei et al. (2011) reported no adverse impact to leached water with three MC applications (total application amount, 15 mmol MC kg^{-1} soil) and three crops in an acid ferralsol. The leached Cd and Pb accumulated in the subsoil (40–60 cm) for MC treated soil were indicated compared to the initial subsoil concentrations. The enhancement of deep layer fixation of heavy metals was proposed with addition of lime and Fe^{3+} to subsoil (Wei et al., 2010; Wei et al., 2011). Results suggested that the leaching risk of heavy metals is minimal in the acid ferralsol regions such as South China.

The aim of soil remediation with soil washing is to reduce the total and bioavailable concentrations of heavy metals in soil and to use such remediated soil as a plant substrate under field conditions. However, previous studies reported that washed soil was not directly suitable for plants (Jelusic and Lestan, 2014a; Jelusic et al., 2014b; Hu et al., 2014). Chinese cabbage planted in the remediated soil managed to absorb high concentrations of Pb, Cd, and Zn in their upper parts because of the residual EDTA-metal complexes left in the soil after remediation (Jelusic et al., 2013). EDTA washing decreased metal concentration in soil and plants, but the yield of white clover on washed soil was significantly lower than that on original soil (Zupanc et al., 2014). Our previous research also reported that the concentrations of Cd, Zn, and Cu in grains of *Zea mays* grown in MC-washed soil were significantly higher than those in grains of control plants (Guo et al., 2013). Therefore, the recommended treatment scheme is as follows: washing with appropriate washing solutions; repeated phytoextraction with hyperaccumulator; and addition of inorganic and organic amendments, followed by planting of a low metal-accumulating crop (Guo et al., 2013).

Co-cropping system is useful in the remediation of metal-contaminated soil (Jiang et al., 2010). A co-cropping system of *Thlaspi caerulescens* (hyperaccumulator) with *Thlaspi arvense* increased Zn uptake of the hyperaccumulator and increased the growth and reduced Zn uptake of the non-hyperaccumulator (Whiting et al., 2001). Recently, a co-cropping system (growing a metal hyperaccumulator plant alongside a low metal accumulating crop) was introduced to simultaneously phytoextract heavy metals from contaminated soil while growing an agricultural crop. The results showed that this co-cropping system effectively increased the hyperaccumulator's uptake of Zn from the soil, whereas the harvested agricultural crop met the Chinese standard for animal feeds (Wu et al., 2007). Continued agricultural production of safe animal feeds from contaminated soils is necessary considering the limited area of arable land in China and the time required for phytoremediation of metal-contaminated soils.

The objectives of this research are as follows: (i) to investigate the efficiency of Cd, Pb, Zn, and Cu removal through washing with only MC or combining with $FeCl_3$ on a multi-metal contaminated soil through the field experiment; (ii) to estimate the effects of a co-cropping system on the growth of *Sedum alfredii* and *Z. mays*, the extraction and accumulation of heavy metals through *S. alfredii*, and the production of safe feeds through *Z. Mays* in washed field site; and (iii) to evaluate the leaching risk of soil washing with only MC or combining with $FeCl_3$.

2. Materials and methods

2.1. Field description

The field plot experiments were conducted on a paddy soil located in Shangba Village of Wengyuan County, Shaoguan City, Guangdong Province, Southern China (24°31′ N; 113°43′ E). This site is located downstream of the Dabaoshan mine area, which is a large polymetallic deposit with limonite body, copper-sulfur deposit, and lead-zinc body. The nonferrous metal smelting companies in the area were built in the 1960s. Therefore, the site has a long history of surface irrigation with the mining wastewaters. The soil is contaminated with Cd, Pb, Zn, and Cu.

Wengyuan County has a subtropical monsoon climate with an average annual rainfall of 1694 mm and a mean annual temperature of 20.6 °C. Table 1 shows the metal concentrations and the selected soil properties of the soil in this study.

Table 1
Selected properties of the soil used in this study.

Parameter	Value
pH	4.32
Organic Matter ($g \cdot kg^{-1}$)	16.6
Sand/silt/clay(%)	15/62/23
Cation Exchange Capacity ($cmol \cdot kg^{-1}$)	6.56
Total N ($g \cdot kg^{-1}$)	1.42
Total P ($g \cdot kg^{-1}$)	0.66
Total K ($g \cdot kg^{-1}$)	17.5
Total Cd ($mg \cdot kg^{-1}$)	0.488
Total Zn ($mg \cdot kg^{-1}$)	329.3
Total Pb ($mg \cdot kg^{-1}$)	537.5
Total Cu ($mg \cdot kg^{-1}$)	482.9
NH_4NO_3–Cd($mg \cdot kg^{-1}$)	0.114
NH_4NO_3–Zn($mg \cdot kg^{-1}$)	12.83
NH_4NO_3–Pb($mg \cdot kg^{-1}$)	13.70
NH_4NO_3–Cu($mg \cdot kg^{-1}$)	4.12

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