Effect of Soil Washing with Ferric Chloride on Cadmium Removal and Soil Structure

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Abstract: In China, arable soils contaminated with cadmium (Cd) threaten human health. Ferric chloride (FeCl₃) is a highly efficient agent that can remove Cd from contaminated soils. However, it is unknown whether FeCl₃ damages the soil structure and consequently affects crop growth. In this study, we investigated the impacts of Cd extraction by FeCl₃ on the structure of a paddy soil on the basis of comparisons of control (without washing agents) and hydrochloric acid (HCl) treatments. According to our results, the removal efficiency increased with the decrease in soil initial pH, as adjusted by FeCl₃. However, the low pH of 2.0 caused a partial loss of soil mineral components, with an Al release of 4.4% in the FeCl₃-treated soil versus 1.3% in the HCl-treated soil. In contrast, the amount of released Al was less than 0.2% in the control and in the FeCl₃ treatments with initial pH values of 3.0 and 4.0. The washing agents caused soil TOC loss of 27.1%, 17.5%, and 2.76% in the pH 2.0, 3.0, and 4.0 FeCl₃ treatments, compared with 15.5% in the initial pH 2.0 HCl treatment. The use of FeCl₃ represents an optimum tradeoff between removal efficiency and the loss of soil components to restore Cd-polluted soils by adjusting the initial pH to 3.0 with the addition of FeCl₃. Under this condition, the amount of Al loss was less than 0.2%, and the extraction efficiency reached 40.3%, compared to an efficiency of 39.7% with HCl at an initial pH of 2.0. In conclusion, FeCl₃ could effectively remove Cd from contaminated soil.

Keywords: cadmium pollution; ferric chloride; soil restoration; soil structure

1. Introduction

Due to rapid industrialization and urbanization, arable soils in China are increasingly subjected to cadmium (Cd) pollution [1]. On a national level, Cd is widely distributed and, in some areas, exceeds the threshold value outlined in the Chinese Environmental Quality Standard for Soils (GB 15618-1995) [2]. According to the Bulletin of the National Soil Pollution [3], the areas with excessive Cd levels account for 7.0% of the investigated area of 6.3 million square kilometers, with 0.5% being heavily polluted. When Cd enters the food chain, it can cause itai-itai disease in humans, with significant health impacts [4]. Compared to phyto remediation, soil dressing, or soil stabilization, soil washing is a remediation technology which is time- and cost-efficient and can permanently remove heavy metals from contaminated soil [5–7]. However, the key issue is the selection of a suitable reagent, which ideally is highly effective and cheap, without causing secondary pollution. Ethylenediaminetetraacetic acid (EDTA) is an efficient chelating reagent for extracting Cd from soils due to its strong chelating ability and the high solubility of metal–EDTA complexes in solution [8,9]. However, in a previous study, a significant
portion of metal–EDTA complexes were retained in the soil after washing [10], most likely because metal–EDTA complexes, such as Ni–EDTA, Co(II)–EDTA, and Cd–EDTA, have a quinquedentate configuration; therefore, the carboxylate group not coordinated to the metal center can be adsorbed onto the soil mineral surface [11]. Similarly, the low-molecular-weight organic acids (LMWOAs) such as citric, oxalic, and succinic acids, secreted by plant roots, can form soluble complexes with Cd, thus increasing the release of Cd from the soils into the soil solution. However, in a previous study, the Cd from Cd–LMWOA complexes was easily adsorbed onto the soil particles with the biodegradation of the LMWOAs. Renewal of LMWOAs is necessary to maintain the steady release of Cd from the soils [12].

Unlike organic chelating reagents, ferric chloride (FeCl$_3$) is a stable and highly efficient extracting agent; ferric iron can dissociate water molecules to release hydrogen ions, which can extract heavy metals adsorbed onto soil surfaces [13–15]. This difference is reflected in the pH of the extraction solution, indicating that the primary mechanism of Cd extraction by FeCl$_3$ is proton release coupled with hydroxide generation, as iron hydroxides are insoluble. Washing with FeCl$_3$ led to the formation of Cd–chloride complexes, enhancing Cd extraction from the soils [13]. Moreover, the formation of metal–chloride composites can also enhance the extraction efficiency. Above an extraction pH of 2.4, FeCl$_3$ could extract soil Cd more effectively than HCl [13]. Gao et al., using leaching experiments, reported that 20 mmol·L$^{-1}$ FeCl$_3$ removed 66.5% Cd from a contaminated paddy soil in Hunan Province, China [16]. Although FeCl$_3$ is a promising washing reagent, it is not clear whether it causes damage to the soil structure, an important factor for crop cultivation.

In this context, the objectives of this study were to reveal the impacts of Cd extraction by FeCl$_3$ on the soil mineral components and organic matter on the basis of comparisons with hydrochloric acid (HCl) and to confirm the feasibility of this method to restore Cd-contaminated arable soils.

2. Materials and Methods

2.1. Soil Properties

Soil samples were collected from the plow layers (0–15 cm) of a Cd-contaminated paddy field in Maoming City (21°44′ N, 110°46′ E), Guangdong Province, China. Prior to chemical analysis, the samples were air-dried and passed through a 2 mm sieve. Soil texture was as follows: sand 17.3% (0.05–0.60 mm), silt 73% (0.002–0.05 mm), and clay 9.7% (<0.002 mm), with a pH of 4.57 (1:5.0, CaCl$_2$), 22.5 g·kg$^{-1}$ of total C, and 4.3 cmol$_c$·kg$^{-1}$ of CEC. The total Cd concentration of the paddy soil was 2.10 mg·kg$^{-1}$, which was substantially higher than the background value (average 0.20 mg·kg$^{-1}$) in uncontaminated soils in China [2].

2.2. Batch Experiment

Batch-scale soil desorption experiments were performed to confirm the extraction of Cd from the Cd-contaminated paddy soil by washing agents. Briefly, 4.0 g of soil samples and 40 mL of 0.01 mol·L$^{-1}$ NaCl were placed in 50 mL centrifuge tubes with screw caps and spiked with FeCl$_3$ and HCl, keeping a soil-to-suspension ratio of 1:10 (g/mL) in each sample. The suspensions were shaken at a constant shaking speed of 150 rpm at room temperature (25 °C), under the influence of initial solution pH values (2.0, 3.0, and 4.0) on Cd extraction from the soils adjusted by different amounts of FeCl$_3$; the extraction kinetics (at 0.17, 0.33, 0.50, 0.67, 0.83, 1.0, 3.0, 6.0, 12, 24, 72, 144, and 288 h) were evaluated under the above conditions. The experiment with an initial solution pH value of 2.0, adjusted by HCl, and the control experiment without pH adjustment were conducted under the same conditions. Each sample was collected using a 2.5 mL syringe and then filtered through a 0.22 μm Millipore filter for Cd determination; we also analyzed dissolved organic carbon (DOC) and Fe (II). After the reaction, the residual soils were separated by static settling and supernatant removal for analyzing the speciation of Cd, soil mineral components, and total organic carbon (TOC). Each treatment was replicated three times.
2.3. Measurements

The measure method of soil particle size distribution was formulated from Miroslava’s research [17]. The air-dried soil was sifted through a sieve of 0.60 mm. To obtain a homogeneous soil sample of specific amount, the quartering method was used. From the prepared fine soil, 0.5 g was weighted and added to 10 mL of 1 mol·L⁻¹ sodium hexametaphosphate. Dispersing time was 24 h. Before the measurement, the samples were exposed to ultrasound for 10 min. The prepared sample was analyzed in a laser particle size analyzer (Mastersizer 2000, Malvern, UK).

The concentrations of total Cd and Al were determined via ICP-MS (Thermo iCAP RQ, Waltham, MA, USA) [18]. The dissolved Fe(II) concentration in the solution was determined using a UV/visible spectrophotometer (Agilent Cary 60, Santa Clara, CA, USA) at 510 nm [19].

The speciation of Cd was analyzed using the Tessier sequential extraction procedure [20]. For this, five defined fractions were sequentially extracted, i.e., exchangeable Cd (Exch), Cd bound to carbonates (Carb), Cd bound to iron and manganese (Fe–Mn) oxides, Cd bound to organic matter (Org), and residual Cd (Re).

The TOC and DOC were determined using a TOC analyzer (Multi N/C 3100, Jena, Germany) [21,22], while the X-ray diffraction (XRD) patterns of the soil samples were obtained using Rigaku Smartlab with a Cu Kα radioactive source (1.54059 Å). Continuous scans from 10° to 80° 2θ were collected at a scanning rate of 5°·min⁻¹ following Singh’s study [23].

2.4. Statistical Analysis

Means and standard deviations were calculated with Microsoft Excel; differences between treatments were examined using one-way ANOVA. The significance of differences between treatments was determined according to Duncan’s new multiple range test (p < 0.05), using the statistical software package SPSS. Graphs were generated using Origin.

3. Results and Discussion

3.1. Effects of Ferric Chloride on Cd Extraction Efficiency

To comprehend the extraction process for Cd by ferric chloride, a set of kinetic experiments was conducted at a reaction time from 0.17 to 288 h. According to the results, a two-step process occurred during the reacting period, with a rapid extraction of soil metals within the first 12 h, followed by a gradual release up to equilibrium at 144 h. In the control treatment, the proportion of Cd desorption to the total soil Cd was 1.73%, versus values of 48.0%, 40.3%, and 6.4% at an initial pH of 2.0, 3.0, and 4.0, adjusted by FeCl₃ (Figure 1). In contrast, HCl extracted Cd by 39.7% at an initial pH of 2.0, which was close to the value at an initial pH of 3.0 but lower than that at an initial pH of 2.0 of the FeCl₃ solutions. At the same initial pH, ferric chloride had a better extraction efficiency than HCl, most likely because ferric iron could continuously generate hydrogen ions through the equilibrium reaction of hydroxide formation. Yoo et al., using FeCl₃ and Fe(NO₃)₃ to remove Pb from shooting range soil, obtained similar results, with higher removal efficiencies than HCl and HNO₃ at an initial pH of 2.5 [24].

In the untreated soil, Cd bound to Fe–Mn oxides was the main fraction (37.9%), followed by exchangeable Cd (28.1%), residual Cd (16.2%), carbonate Cd (16.0%), and Cd bound to organic matter (1.80%; Figure 2). After washing with FeCl₃, the concentrations of exchangeable Cd and carbonate Cd significantly decreased in all three treatments. At the initial pH values of 2.0, 3.0, and 4.0, the concentration of exchangeable Cd decreased by 87.4%, 75.5%, and 21.8% versus 59.1% in the washed soil by HCl, and the concentration of carbonate Cd decreased by 68.8%, 47.7%, and 25.2% versus 64.4% in the HCl treatment. The efficient extraction of soil Cd by FeCl₃ is mainly attributed to the lowering of the solution pH through proton release from the chemical equilibrium of hydroxide formation [24]. Moreover, Fe³⁺, as a trivalent cation, might partly promote ion exchange with Cd²⁺ on the surface of soil particles compared to HCl [13]. Additionally, Cd bound to Fe–Mn oxides
was even reduced by 60.2% and 44.2% in the initial pH 2.0 and 3.0 FeCl₃ solutions versus 58.3% in the initial pH 2.0 HCl treatment. Udovic and Lestan reported that the removal of labile heavy-metal species from the soil would disturb the chemical equilibrium among different species, thus potentially re-establishing such an equilibrium and shifting heavy metals back to more available chemical forms [25]. Gao et al. reported that the easily reducible and oxidizable fractions of Cd were transformed into easily extractable fractions after the soil was leached by the composite reagents with citric acid and FeCl₃ [16]. Thus, Cd bound to Fe–Mn oxides might have undergone this transformation during the washing process and been removed by FeCl₃ or HCl in this study. Similarly, Yoo et al. reported that Pb was extracted into solution by EDTA as a result of a fractional change from the strongly bound fractions to soluble, exchangeable, and carbonated fractions [26]. In our study, the Cd bound to the organic matter fraction was also reduced by 42.1%, 36.8%, and 9.41% in the initial pH 2.0, 3.0, and 4.0 FeCl₃-treated soil versus 35.1% in HCl-treated soil. This may relate to the reduction of organic carbon in soils treated with FeCl₃ and HCl (Table 1).

![Figure 1. Kinetics for the Cd extracted from contaminated paddy soil by FeCl₃.](image)

![Figure 2. Sequential extraction of Cd from contaminated paddy soil with FeCl₃. Note: Exch, Carb, Fe–Mn, Org, and Re refer to exchangeable Cd, Cd bound to carbonates, Cd bound to iron and manganese oxides, Cd bound to organic matter, and residual Cd.](image)
### Table 1. TOC, DOC, and Fe(II) concentrations in different treatments.

| Treatments          | TOC (g·kg⁻¹)   | DOC (mg·L⁻¹) | Fe (II) (mg·L⁻¹) |
|---------------------|----------------|--------------|-----------------|
| Control             | 22.3 ± 0.24    | 6.63 ± 0.30  | 0.08 ± 0.00     |
| HCl, pH 2.0         | 19.0 ± 0.06    | 9.01 ± 0.19  | 0.37 ± 0.08     |
| FeCl₃, pH 2.0       | 16.4 ± 2.15    | 19.0 ± 1.62  | 28.9 ± 0.75     |
| FeCl₃, pH 3.0       | 18.5 ± 0.08    | 7.64 ± 0.08  | 0.12 ± 0.04     |
| FeCl₃, pH 4.0       | 21.9 ± 0.45    | 7.07 ± 0.14  | 0.08 ± 0.00     |

Lowercase letters within the same column compare different treatments at the equilibrium stage. Values represent means ± SE. Values followed by the same letter do not differ significantly according to Duncan’s test (p < 0.05).

#### 3.2. Changes in Soil Clay Minerals during Extraction

Washing with FeCl₃ or HCl could decrease the soil pH values (Table 2), potentially resulting in a negative disturbance of the soil structure [15,27,28]. However, due to the buffering capacity of soil, the equilibrium pH values were restored to 2.12, 3.94, and 4.88 in the initial pH 2.0, 3.0, and 4.0 FeCl₃ solutions versus 2.77 in the initial pH 2.0 HCl solutions. In the initial pH 2.0 FeCl₃ treatment, the soil solution pH had a slight resilience until equilibrium compared to that in the HCl treatment; however, both washed soils deviated far from the untreated soil in terms of the pH value. At a similar Cd extraction efficiency, the initial pH 3.0 FeCl₃ treatment restored the pH more closely to that of the untreated soil compared to the HCl treatment.

### Table 2. Solution pH values during the washing process.

| Treatments          | Initial pH | Final pH |
|---------------------|------------|----------|
| Control             | 5.06 ± 0.01| 5.25 ± 0.04|
| HCl, pH 2.0         | 2.00 ± 0.00| 2.77 ± 0.12|
| FeCl₃, pH 2.0       | 2.00 ± 0.00| 2.12 ± 0.05|
| FeCl₃, pH 3.0       | 3.00 ± 0.00| 3.94 ± 0.05|
| FeCl₃, pH 4.0       | 4.00 ± 0.00| 4.88 ± 0.06|

Values represent means ± SE.

The XRD was performed on HCl- and FeCl₃-treated soils to reveal the effects of these reagents on the soil mineral components. The results (Figure 3) indicate that the untreated soil was mainly composed of quartz (SiO₂) and kaolinite (Al₂O₃Si₂O₃·2H₂O); hematite (Fe₂O₃), goehite (FeO(OH)), and magnetite (Fe₃O₄) were probably also present. The FeCl₃-treated soils did not differ from the untreated soils in terms of mineral components, indicating that no minerals disappeared under the decrease in pH due to FeCl₃ addition. Moreover, Cutler et al. reported a new formation of fine-crystalline Fe phases with ferrihydrite-like morphology in FeCl₃-treated soil by transmission electron microscopy analysis; iron (oxy)hydroxides can be converted to more stable and crystalline iron oxides over time [24,29]. In contrast, the Fe₃O₄ peak did not appear in HCl-treated soil.

The results of the XRD can, however, not sufficiently prove that FeCl₃ treatment did not damage the soil components, as we performed no quantitative analysis. Kaolinite (Al₂O₃Si₂O₃·2H₂O) is the most abundant and basic clay mineral of the soil [15], and Al can indicate the extent of kaolinite dissolution into the aqueous phase in HCl- and FeCl₃-treated soils (Figure 4). The amount of released Al into solution as a proportion of total soil Al was less than 0.2% in the control and in the FeCl₃ treatments with initial pH values of 3.0 and 4.0. These results are in agreement with the findings of Makino et al., who indicated that washing with FeCl₃ is not likely to cause serious damage to the soil clay minerals [10]. The amount of Al in the initial pH 2.0 HCl and FeCl₃ solutions fell within the range of 1.0–5.0%, which was significantly greater than that in the control treatment due to the considerably lower pH. At the same initial pH value, the percentage of Al loss in FeCl₃-treated soil was significantly higher than that in HCl-treated soil (p < 0.05), most likely because FeCl₃-treated soil had a lower equilibrium pH value because of the higher amount of hydrogen ions from the chemical equilibrium of hydroxide formation.
Figure 3. X-ray diffraction patterns for differently treated soils.
nificantly correlated with the concentration of Fe(II) (correlation coefficient = 0.987; DOC and Fe(II) [30]. Similarly, in our study, the DOC in the solutions positively and sig-

mical iron reduction on ferrihydrite-adsorbed natural DOC led to the coupled release of iron oxides in soils, which are important sorbents for DOC. Pan et al. reported that microbial iron reduction on ferrihydrite-adsorbed natural DOC led to the coupled release of DOC and Fe(II) [30]. Similarly, in our study, the DOC in the solutions positively and significantly correlated with the concentration of Fe(II) (correlation coefficient = 0.987; p < 0.01). However, it is not clear whether reducing microorganisms played a role under the conditions of initial pH 2.0 in HCl- and FeCl₃-treated soils. In addition, the amount of DOC in solution increased with the decrease in equilibrium pH. Thus, a change in soil pH may also contribute to the DOC loss [31,32].

3.3. Changes in Total Organic Carbon during Extraction

Compared with the untreated soil, there were no significant losses of TOC in the control and initial pH 4.0 FeCl₃ treatments (Table 1). However, in the initial pH 2.0 HCl and in the pH 2.0 and 3.0 FeCl₃-treated soils, the content of TOC decreased significantly by 15.5%, 27.1%, and 17.5%, respectively.

The organic carbon in the treated soils was partly released into the solutions. In contrast to the TOC in the soil, the amount of DOC in the initial pH 2.0 FeCl₃ solution was significantly higher than that in the other treatments, followed by the HCl-treated solution (Table 1). The release of DOC into the solutions may be attributed to the dissolution of iron oxides in soils, which are important sorbents for DOC. Pan et al. reported that microbial iron reduction on ferrihydrite-adsorbed natural DOC led to the coupled release of DOC and Fe(II) [30]. Similarly, in our study, the DOC in the solutions positively and significantly correlated with the concentration of Fe(II) (correlation coefficient = 0.987; p < 0.01). However, it is not clear whether reducing microorganisms played a role under the conditions of initial pH 2.0 in HCl- and FeCl₃-treated soils. In addition, the amount of DOC in solution increased with the decrease in equilibrium pH. Thus, a change in soil pH may also contribute to the DOC loss [31,32].

3.4. Applications and Suggestions

Although most of the research of soil washing with ferric chloride on cadmium removal has been conducted in laboratory experiments, there have been some studies on slurry reactor soil and other practical equipment, such as a circulating washing system device and soil leaching machine. Shi conducted batch and slurry reactor soil washing experiments to explore the removal of Cd, Cr, Pb, and Zn using seven agents. Citric acid (CA) and ferric chloride (FeCl₃) exhibited an obvious synergistic effect on the removal of heavy metals [33]. This technology is already in use in some countries, such as Japan.

The side-effect of its application has raised concern. Liang studied a comparison extraction of Cd and Pb with EDTA, DTPA, citric acid, and FeCl₃, as well as their effects on soil fertility. Results indicated that slight changes in soil fertility, including pH, cation exchange capacity, organic matter, and soil extractable phosphorus, were observed [34]. Furthermore, if overcommitted, ferric chloride will become a new contaminant in the soil. Hence, in the process of application, the amount of FeCl₃ should be controlled. Zhan’s research focused on how to treat the wastewater after soil washing since water pollution is a severe threat to the water security of China. Ca(OH)₂ and two kinds of biochars (biochar and ZnCl₂-modified biochar) were tested to treat the waste FeCl₃ washing effluent [35].
All of these issues need further study. Furthermore, the mechanism of ferric chloride for the removal of Cd should be studied in the future to support the application of this technology.

4. Conclusions

According to our results, FeCl$_3$ could effectively remove Cd from contaminated soil. The removal efficiency increased with the decrease in soil initial pH, as adjusted by FeCl$_3$. However, the low pH of 2.0 caused partial loss of soil mineral components, with an Al release of 4.4% in the FeCl$_3$-treated soil versus 1.3% in the HCl-treated soil. This is an optimum tradeoff for restoring Cd-polluted soils by adjusting the initial pH to 3.0 with the addition of FeCl$_3$. Under this condition, the amount of Al loss was less than 0.2%, and the extraction efficiency reached 40.3%, compared to an efficiency of 39.7% with HCl at an initial pH of 2.0. The addition of different agents caused no variation in the soil mineral species. However, the washing agents caused a loss of soil TOC, which was 27.1% and 17.5% in the pH 2.0 and 3.0 FeCl$_3$ treatments, and 15.5% in the initial pH 2.0 HCl treatment.

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