



## Development of novel assisting agents for the electrokinetic remediation of heavy metal-contaminated kaolin



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### ABSTRACT

A novel assisting agent (citric acid + calcium chloride, CA + CaCl<sub>2</sub>), combined the advantages of two different compositions, has been investigated for the electrokinetic (EK) remediation of multi-metals (Cd, Cu, Ni, Pb, Zn) contaminated kaolin simultaneously. The result shows that CA and CaCl<sub>2</sub> concentration significant affect the removal efficiency of heavy metals. The optimum concentration is obtained at the 0.2 mol/L CA mixed with 0.05 mol/L CaCl<sub>2</sub> in this study, and the removal efficiencies of Cd, Cu, Ni, Pb, and Zn were 98.19%, 95.24%, 98.95%, 86.21%, and 99.01%, respectively, with the consuming of 124.8 kWh/m<sup>3</sup> electric energy. Compared to traditional CA and HCl treatment, the CA + CaCl<sub>2</sub> poses the advantages of cost-efficient, eco-friendly, high removal efficiency for multi-heavy metals, may promote the progress toward commercial application.

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## 1. Introduction

Heavy metals contamination of soils is becoming a global problem. Most of heavy metals in soil are originated from a range of anthropogenic activities including mining operations, agricultural application, and refinery industries [1]. Especially, most of anthropogenic activities produced a serious soil contaminated with multi-heavy metals, such as cadmium (Cd), copper (Cu), nickel (Ni), lead (Pb) and zinc (Zn), which is frequently found as complex mixtures. Contamination of soils by heavy metals, which are toxic and carcinogenic to humans, animals, and plants, would cause serious environmental risk and potential health threat [2]. Various remediation techniques have been applied to remove heavy metals from soil such as soil washing [3], phytoremediation [4], and electrokinetic (EK) remediation [5]. Among of them, EK remediation is widely used [6] for extracting heavy metals from fine-grained soils [7] and seem to be a promising remediation process for simultaneous removal multi-heavy metals in soil [2,6,8].

During the EK remediation process, the movement and removal of heavy metals are dominant influenced by the electromigration and electro-osmotic flow (EOF) under the driving force of electric field [1,9]. Due to the fact that EK remediation can only removal

mobile heavy metals from soil [10], enhancement techniques are developed to solubilize heavy metals in soil and to keep them in anions or cations state for improving removal efficiency. Different enhancement techniques such as controlling the soil pH and adding chemical reagents (like chelating/complexing agents) have been reported in previous studies [10]. In general, soil pH is an important parameter that affects heavy metals existing form in soil [11]. The soil pH can be controlled by adding acidic solutions into the electrode compartments [12] or using of ion exchange membranes [13]. Furthermore, recent studies have proved that assisting reagents can effectively keep heavy metals in the mobile state and improve their removal efficiencies [14,15]. Kim et al. [7] reported that hydrochloric acid (HCl) and citric acid (CA) are effective in extracting heavy metals (Pb, Zn, Cu and Ni) from dredged marine sediment. CA had been used as an assisting agent because it is relatively inexpensive and environmentally benign [15]. However, many research revealed that CA was hard to simultaneous remove multi-heavy metals efficiently. Peng et al. [16] performed EK treatment for the removal of heavy metals from sludge and found that the relatively high removal efficiency of Cr and Zn were achieved in CA treatment, however, the removal efficiency of Cu and Pb were relatively low. Hence, it is important to find the assisting agents that can simultaneous remove multi-heavy metals with high efficiency. Soil washing research proved that acid composite with calcium chloride (CaCl<sub>2</sub>) can enhance the removal efficiently of heavy metals [17,18]. Zhou et al. [12]

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managed to remediate Cu and Zn by carrying out EK experiments with lactic acid and CaCl<sub>2</sub> solution. The use of combined CA and CaCl<sub>2</sub> (CA+CaCl<sub>2</sub>) as an assisting agent may be an effective alternative to simultaneously remove multi-heavy metals during EK process. In addition, to reduce the expense and environmentally hazards, it is desirable to reduce the quantity of assisting reagents used for soil remediation [19].

CA + CaCl<sub>2</sub> was used as an assisting agents for the simultaneous removal of Cd, Cu, Ni, Pb and Zn from contaminated kaolin and CA and HCl treatments were conducted for comparison. The influence of assisting agent concentration on the heavy metals migration and removal efficiency were investigated. We expected to find an environmentally benign and cost-effective assisting agent which can simultaneously remove multi-heavy metals efficiently.

## 2. Material and methods

### 2.1. Chemicals

All the chemicals used in the experiments were analytical grade. HCl was purchased from Beijing Chemical Works (China). All other chemicals were obtained from Sinopharm Chemical Reagent Co., Ltd (China). Milli-Q water was used in all the experiments.

### 2.2. Simulated heavy metal-contaminated kaolin

Commercial kaolin obtained from the Sinopharm Chemical Reagent Co., Ltd (China) was used as a model of soil in this experiment. The chemical properties and the mineralogical characterization of the kaolinite clay soil were summarized in Table 1. Sample of kaolin was artificially spiked with nitrates of Cd (II), Cu(II), Ni(II), Pb(II) and Zn(II). Nitrate salts of these heavy metals were dissolved in Milli-Q water. The aqueous solution pH was 4.26. And the heavy metals concentrations (mg/L) in aqueous solution were 100 for Cd, and 500 for Cu, Ni, Pb and Zn, respectively. Six hundred grams air-dried kaolin and 400 mL prepared solution were mixed using a mechanical stirrer for 10 min. A total of 410 g of the kaolin was loaded into the test cell using a tamper. The initial pH and zeta potential of contaminated kaolin was 4.32 and -27.6 mV. The initial heavy metal concentrations in dry kaolin were: 41.60 mg/kg for Cd, 293.7 mg/kg for Cu, 233.1 mg/kg for Ni, 280.9 mg/kg for Pb, and 252.2 mg/kg for Zn.

**Table 1**  
The chemical properties and the mineralogical characterization of the kaolinite clay soil.

Characteristics	Value
Particle size analysis (%)	
Sand (%)	2.02
Silt (%)	51.17
Clay (%)	46.81
Organic matter (%)	0.02
CEC (cmol/kg)	2.65
pH	6.28
Electrical conductivity (μS/cm)	266
Zeta potential (mV)	-29.4
Ratio of mineral (%)	
SiO <sub>2</sub>	52.55
Al <sub>2</sub> O <sub>3</sub>	42.16
TiO <sub>2</sub>	2.57
P <sub>2</sub> O <sub>5</sub>	0.98
Fe <sub>2</sub> O <sub>3</sub>	0.92
CaO	0.36
K <sub>2</sub> O	0.14

The extraction experiment of the contaminated kaolin has been performed in a 1 L glass beaker. Under magnetic stirring, ten grams of air dried contaminated kaolin was vigorously mixed with 1 L of deionized water and 0.05 mol/L CaCl<sub>2</sub> solution, respectively. The solution pH was adjusted using HNO<sub>3</sub> or NaOH to about 5. The heavy metals concentration in the aqueous phase was measured after 3 h [20].

### 2.3. EK experiments

The EK cell was constructed using polymethyl methacrylate which was divided into three compartments: a soil cell and two electrode chamber (Fig. 1). The soil cell (15 cm × 3.8 cm × 6 cm) was containing simulated metal-contaminated kaolin where connected to the electrode compartments in both ends, each cell was connected using perforated polymethyl methacrylate and a filter paper. The electrolytes were circulated from electrolyte reservoir into the corresponding electrode chamber using peristaltic pump at the rate of 10 mL/min and the electrolyte level in the electrode chamber was maintained equally.

All the experiments were conducted at room temperature, without pH control. A steady potential gradient of 1 V/cm was applied for 72 h. Graphite electrodes (7 cm × 3.5 cm × 1 cm) were used for the anode and cathode, respectively. The electrolytes of CA, HCl, and CA + CaCl<sub>2</sub> were used individually and deionized water was employed as a control set. Each electrolyte was prepared with different concentrations and used as anolyte and catholyte throughout the experiment. Each electrolyte reservoir was filled with 600 mL of catholyte or anolyte. A summary of all the run test conditions applied is listed in Table 2. During the experimental period, electric current was monitored by using a logger (MW100, Yokogawa Electric Corporation, Japan). EOF was calculated according to the volume changes of the two chambers. When EK experiment was terminated, the soil sample was sectioned into five equal parts and labeled as S1 to S5 from anode to cathode.

### 2.4. Analytical method

Soil pH and electrical conductivity (EC) were measured using a pH meter (S220 seven compact, Mettler-Toledo International Inc., Switzerland) and an EC meter (DDSJ-308A, Shanghai INESA Scientific Instrument Co., Ltd., China), respectively, by preparing slurries with soil to water ratio of 1:2.5 [21]. The zeta potential values of the original commercial kaolin, contaminated kaolin and the different electrolytes treated kaolin were determined with Zetasizer Nano ZSP (Malvern Instruments, Worcestershire, UK). Heavy metal extraction procedure was as follow: 3.0 g soil of each sliced section was added in 50 mL centrifugation tube containing 27.0 mL of 1 M HCl, shaken at 200 rpm for 6 h, and centrifuged at 4000 rpm for 15 min [21]. The concentrations of heavy metals ions in the supernatant were filtered through a 0.22 μm filters and analyzed by an Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES, ICP-5000, Focused Photonics, Inc., China).

### 2.5. Power consumption

The following equation was used for electrical energy consumption analysis [22].

$$E = \frac{1}{V_s} \int V I dt$$

Where  $E$  is the electrical energy per unit volume (kWh/m<sup>3</sup>),  $I$  is the applied current (mA),  $V$  is the voltage (V),  $t$  is the duration (d),  $V_s$  is the soil volume (m<sup>3</sup>).

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