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Bidirectional Pulsed Electric Field Enhancing Citric Acid Washing for Pb-Contaminated Soil Remediation

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Abstract: Reducing leaching-agent consumption while simultaneously enhancing heavy-metal removal efficiency remains a major challenge for soil-washing technologies. In this study, a treatment assisted by a bidirectional pulsed electric field (BPEF) was integrated with citric acid washing to efficiently remediate Pb-contaminated soil from an abandoned lead smelter site. The washing performance under different electrical parameters was systematically evaluated, and the mechanism underlying electro-enhanced removal was preliminarily investigated. The results demonstrated that the BPEF significantly improved Pb removal compared with citric acid washing alone. The Pb removal rate reached 72.7% after 6 h of treatment under the conditions of a pulse frequency of 0.08 Hz, a voltage of 24 V, and a duty cycle of 75%, representing a 22.4% increase over citric acid treatment alone, while reducing citric acid consumption by 50%. Mechanistic analyses indicated that BPEF altered soil pH, increased soil temperature, and enhanced ion electromigration, thereby inducing electro-osmotic and electroshock effects. These coupled processes facilitate the transformation of Pb into a more acid-extractable fraction, increase its activity, and strengthen its interaction with the functional groups of citric acid, ultimately promoting the washing efficiency. Overall, this technique effectively overcomes the limitations associated with conventional direct-current electro-assisted washing, such as electrode polarization and focusing effects, thus providing a promising approach for developing rapid, efficient, and more engineering-feasible technologies for heavy-metal-contaminated soil remediation.

Key words: bidirectional pulsed electric field (BPEF); chemical washing; citric acid; heavy metal lead (Pb); chemical speciation

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0 Introduction

Heavy metal contamination of soils has become a pervasive global environmental issue. Recent estimates indicate that 14%-17% of the world's arable land is contaminated by heavy metals, with 0.9-1.4 billion people living in regions where metal concentrations exceed agricultural and health safety thresholds. High-risk areas are

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primarily located in South Asia, the Middle East, southern part of China, and parts of Africa[1], which poses serious threats to both human health and ecological security[2-3]. Accordingly, the development of rapid and efficient remediation technologies for heavy metal-contaminated soils is of urgent importance. Conventional soil remediation approaches include solidification/stabilization, phytoremediation, chemical washing, electrokinetic remediation, and their integrated or coupled methods[4]. Among these, chemical washing has been widely recognized for its rapid action and high removal efficiency[5]. However, chemical washing is often limited by the high consumption of washing agents and the potential secondary pollution caused by residual chemicals. Consequently, various enhancement techniques—such as ultrasound, microwave treatment, and chemical oxidation/reduction—have been explored to improve removal performance[6]. Previous studies have demonstrated that electric field-assisted washing can substantially increase remediation efficiency while reducing chemical reagent consumption[6-7]. Nevertheless, most existing investigations have focused on direct current (DC) electric field-assisted washing, which is prone to issues such as electrode polarization, corrosion, and focusing effects[8]. These effects may also clog soil micropores, decreasing soil conductivity and, in turn, reducing removal efficiency[9]. To address these limitations, strategies such as introducing organic acids into the catholyte to alleviate focusing effects[10], or the periodic reversal of electrode polarity to eliminate polarization have been proposed. Building upon these advances, the researchers of the present study employ a bidirectional pulsed electric field system that integrates pulsed current with periodic electrode polarity reversal. This design effectively mitigates extreme pH variations near the anode and cathode, while the transient high-energy pulses promote the desorption of heavy metals adsorbed onto soil particles, thereby improving overall leaching efficiency.

1 Materials and Methods

1.1 Experimental Materials

The Pb-contaminated soil was collected from the surface layer (0-20 cm) of an abandoned lead smelting site in Chongqing, China. After sampling, visible debris was removed, and the soil was air-dried, ground, and sieved through a 10-mesh (< 2 mm) screen. The processed soil was thoroughly mixed to ensure a homogeneous distribution of heavy metals. The fundamental physicochemical properties of the test soil are presented in Table 1.

Table 1 Main physicochemical properties of the test soil

Physicochemical properties	Value
pH	6.4
Moisture content/%	7.1
Organic matter/(g·kg^{-1})	3.6
Cation exchange capacity/(cmol·kg^{-1})	13.0
Redox potential/mV	287.3
Total Pb/(mg·kg^{-1})	1 318.8

1.2 Experimental Setup

The schematic of the electric field-assisted washing device is shown in Fig. 1. The reactor had internal dimensions of 9 cm (length) × 5 cm (width) × 6 cm (height). Graphite plates were used as electrodes, with an interelectrode distance of 8 cm between the anode and cathode. The electric field was supplied by an MHS-5200P$^+$ pulse signal generator (Minghe Electronics Corp.). The waveform and parameter settings of the bidirectional pulsed electric field are illustrated in Fig. 2. The pulse period (t) equals the sum of the positive pulse duration (t_a) and negative pulse duration (t_c), while its reciprocal corresponds to the pulse frequency. The duty cycle (r) is defined as the ratio of t_a to the total pulse period (t).

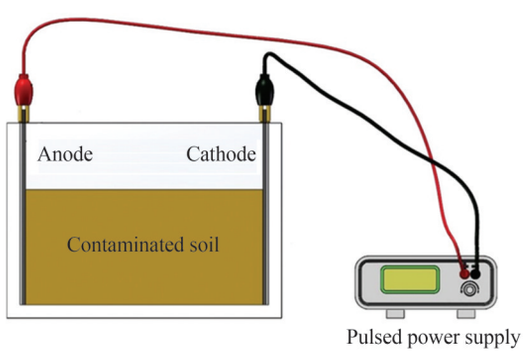


Fig. 1 Schematic drawing of the experimental setup

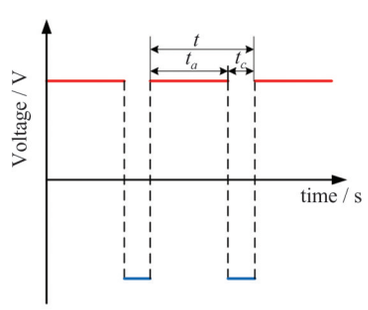


Fig. 2 Waveform of pulsed power supply (75% duty cycle)

1.3 Citric Acid Washing under Bidirectional Pulsed Electric Field

The contaminated soil was mixed with a citric acid solution at a solid-to-liquid ratio of 1∶5. The mixture was thoroughly stirred and then placed in the washing reactor connected to the pulsed power supply. The total washing time for each treatment group was 10 h. After washing, the mixture was centrifuged at 3 000 r/min, and the supernatant was collected for Pb concentration analysis. The centrifuged precipitate was washed with deionized water, oscillated for 15 min, and centrifuged again. Then, the solids were taken out and air-dried naturally for subsequent Pb speciation analysis. Operating parameters, such as citric acid concentration, pulse time, pulse voltage, and pulse frequency, were considered. An $L_9(3^4)$ orthogonal experimental design with four factors and three levels for each factor was adopted. The citric acid concentration was set at three levels (i=1,2,3): 0.05, 0.1, and 0.2 mol/L; the pulse frequency was set at 0.04, 0.08, and 0.16 Hz; the pulse time was set at 2, 4, and 6 h; and the pulse voltage was set at 8, 16, and 24 V. Based on preliminary tests, the duty cycle was fixed at 75% for all treatments. A control group (Treatment 10#) subjected to citric acid washing without an electric field was also included. Each experiment was performed in duplicate, and the mean values were recorded.

1.4 Analysis

Soil pH was measured using the standard potentiometric method at a soil-to-water mass ratio of 1∶2.5. Moisture content was determined gravimetrically. Organic carbon content was analyzed by combustion oxidation coupled with non-dispersive infrared absorption, and the obtained result was multiplied by the organic matter conversion factor of 1.724 to determine the soil organic matter content. The cation exchange capacity (CEC) was determined using the ammonium acetate exchange method. The oxidation-reduction potential (ORP) was measured following the "Soil Oxidation-Reduction Potential Determination-Potentiometric Method" (F-HZ-DZ-TR-0028). Soil mineral composition was identified by X-ray diffraction (XRD; X'Pert-Pro MPD, Netherlands). Total Pb content was determined after microwave-assisted aqua regia digestion, and Pb concentrations were quantified using atomic absorption spectrophotometry (AA-6300C, Shimadzu, Japan) after appropriate dilution. The chemical speciation of Pb in soil was analyzed using the BCR three-step sequential extraction procedure[11], and the Pb concentrations in the extracts were measured by inductively coupled plasma optical emission spectrometry (ICP-OES).

2 Results and Discussion

2.1 Efficiency of Pb Removal with Citric Acid Washing under Bidirectional Pulsed Electric Field

The residual Pb content and removal rate in the soil after bidirectional pulsed electric field-assisted citric acid washing are presented in Fig. 3 and Table 2, where the citric acid concentration for Treatment 10# (control) was 0.2 mol/L. As can be seen, compared with the control group (with the exception of Treatment 1#), the application of an electric field improved the leaching efficiency of citric acid. The highest removal rate of 75.8% was achieved under Treatment 7# with a pulse frequency of 0.04 Hz, a voltage of 16 V, a duty cycle of 75%, and an electric field application time of 6 h. For electric field-applied Treatments 3# and 5#, which used citric acid concentrations of 0.05 mol/L and 0.1 mol/L, respectively, the Pb removal rates were 70.4% and 72.7%. These rates are 18.5% and 22.4% higher than that of Treatment 10# (control, 59.4%), while the corresponding citric acid dosage was reduced by 75% and 50%, respectively.

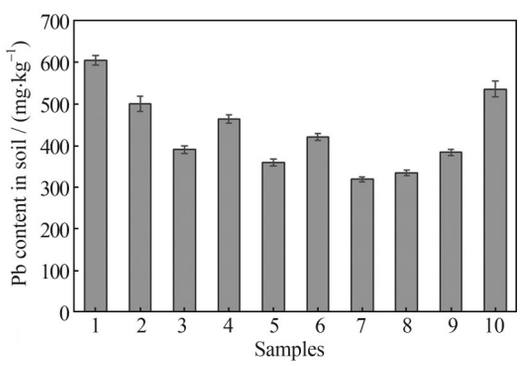


Fig. 3 Residual Pb in soils after washing under different parameters

Through intuitive analysis (Table 3), the order of influence of the parameters for bidirectional pulsed electric field-assisted citric acid washing of Pb was determined as follows: Citric acid concentration > Pulse voltage > Pulse time > Pulse frequency. The Pb removal efficiency was positively correlated with citric acid concentration, pulse voltage, and pulse time. The leaching of heavy metals from soil by citric acid relies on two effects. First, citric acid lowers soil pH, thereby increasing

Table 2 Original data of Pb removal rate under different parameters

Treatment	Citric acid concentration/(mol·L^{-1})	Pulse frequency/Hz	Pulse time/h	Pulse voltage/V	Pb removal rate/%
1#	0.05	0.04	2	8	54.20±0.91
2#	0.05	0.08	4	16	62.10±1.36
3#	0.05	0.16	6	24	70.40±0.68
4#	0.1	0.04	4	16	64.80±0.76
5#	0.1	0.08	6	24	72.70±0.61
6#	0.1	0.16	2	8	68.10±0.68
7#	0.2	0.04	6	16	75.80±0.45
8#	0.2	0.08	2	24	74.60±0.53
9#	0.2	0.16	4	8	70.90±0.53

the desorption and dissolution of heavy metal ions[12]. Second, the carboxyl and hydroxyl functional groups of citric acid form water-soluble metal complexes with these ions[13], thus enhancing their activity and mobility. A higher citric acid concentration provides more H^+ and organic ligands in the soil, significantly improving Pb leaching efficiency. Regarding the electric field parameters, pulse voltage had the greatest impact on the leaching effect. This is because the voltage gradient directly determines the migration rate of heavy metal ions. Increased voltage enhances the electric field force, accelerating ion migration, and also increases the electro-osmotic flow volume, thereby promoting contact between the washing solution and heavy metals. Furthermore, increased voltage intensifies redox reactions, disrupting heavy metal-soil bonds and promoting the conversion of bound heavy metals in the soil into soluble forms.

Table 3 Results of range analysis for orthogonal experiment

%

Factor	k_1	k_2	k_3	Range
Citric acid concentration	61.6	70.5	73.8	12.2
Pulse frequency	66.3	69.8	69.8	3.5
Pulse time	66.3	67.3	72.3	6.0
Pulse voltage	65.1	68.9	71.9	6.8

k_i, mean value of average Pb removal rates at the i-th level of each factor; Range, the difference between the maximum and minimum k_i values for each factor.

2.2 Mechanism Analysis of Bidirectional Pulsed Electric Field Combined Washing

Heavy metal speciation is a major factor affecting the removal of heavy metals. The acid-extractable fraction of Pb has higher mobility, and its content directly affects chemical extraction efficiency. Speciation analysis was conducted on the soil before washing, and on the soils from Treatment 7# (bidirectional pulsed electric field + citric acid) and Treatment 10# (control) after washing (Fig. 4). As can be seen, Pb in the initial soil was primarily in the Fe-Mn oxide-bound and acid-extractable fractions. After citric acid washing alone, the content of acid-extractable and Fe-Mn oxide-bound Pb fractions decreased significantly, while the content of organic-bound and residual fractions changed slightly before and after washing. By comparing the concentrations of different Pb fractions in each leaching treatment group (Fig. 4) with those in the control group, the corresponding removal efficiencies were calculated. The results indicate that the combined application of a bidirectional pulsed electric field and citric acid leaching enhanced the removal of all Pb fractions to varying degrees. Notably, the Fe-Mn oxide-bound fraction showed the most significant improvement, followed by the residual fraction; and their removal efficiencies increased from 61.37% and 4.04% to 87.52% and 16.80%, respectively. This indicates a synergistic effect between the electric field and citric acid, which even transforms and removes the most stable Pb fraction (residual). The pH range between the electrode plates after washing was 4.60-8.12 (Fig. 5), which is significantly narrower than the pH range (3.5-12.7) under a DC electric field[14], and the graphite electrodes remained largely intact after energization.

As shown in Table 1, and combined with the risk screening value of 400 mg/kg for the first-class land use specified in the "Soil Environmental Quality Risk Control Standard for Soil Contamination of Development Land (Trial)" (GB 36600–2018)[15], the Pb content in the test soil seriously exceeded the regulatory limit, indicating that the soil is moderately to highly Pb-contaminated. XRD results (Fig. 6) showed that the main mineral components of the soil were quartz, mica,

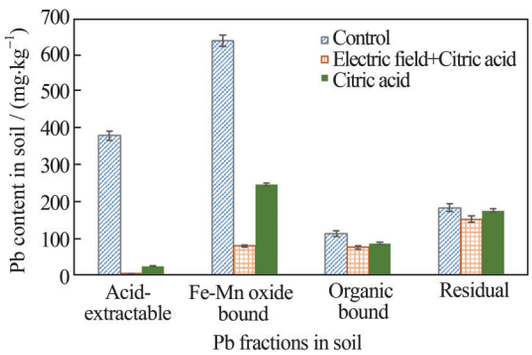


Fig. 4 Pb fractions in soils before and after treatment

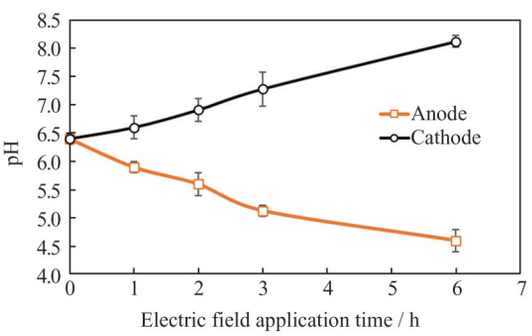


Fig. 5 Changes in soil pH at the anode and cathode during electric field treatment

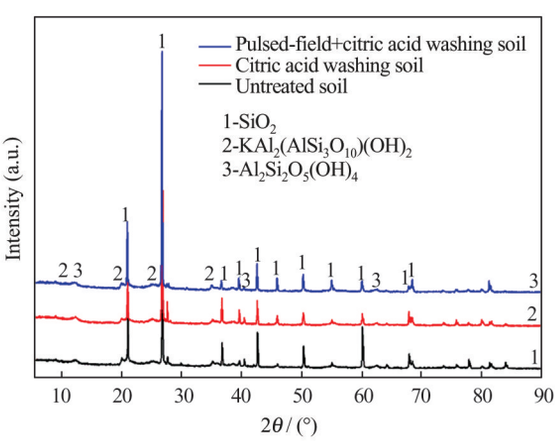


Fig. 6 XRD patterns of soil samples before and after treatment

and kaolinite. The mineral composition of the soil changed very little before and after remediation by citric acid alone or electric field-assisted remediation, with no obvious new phase peaks generated, indicating that application of electric field-assisted washing did not alter the original mineral structure of the soil. The total Pb content in the soil after bidirectional pulsed electric field-enhanced washing decreased significantly. The total Pb contents in soils from Treatments 3#, 5#, and 7# -9# were all below the risk screening value for the first category of land use. Furthermore, the distribution of Pb speciation in the soil followed the order: Residual > Fe-Mn oxide bound > Organic bound > Acid-extractable (Fig. 4), indicating low bioavailability of the residual Pb, thereby enhancing the environmental safety of the remediated soil. The Pb removal efficiency achieved by bidirectional pulsed electric field-assisted citric acid leaching was compared with that reported in previous studies, as shown in Table 4. Despite the higher initial Pb concentration, the Pb removal rate obtained in this study was comparable to that of earlier reports; however, due to the use of a 75% duty cycle, the actual electric field application time was shorter, thereby reducing overall energy consumption.

The reaction mechanism of citric acid washing for Pb-contaminated soil involves two aspects: (i) lowering the soil pH to increase the solubility of Pb-bearing compounds; and (ii) the complexation reaction between its –OH and –COOH groups and Pb^{2+} [21]. Therefore, the acid-extractable Pb is the primary target for citric acid. After applying the bidirectional pulsed electric field, not only was Pb promoted to transform into more easily extractable forms[22], but the electric field environment also increased the mobility of charged particles. Since Pb^{2+} and the active groups of citric acid carry opposite charges, their relative motion in the electric field environment increases the contact probability between Pb^{2+} and the active groups, promoting the formation of more soluble metal complexes, thus improving the overall utilization rate of citric acid and the washing efficiency. In addition, it was also observed in the experiments that the soil temperature near both electrode plates was nearly 20 ℃ higher than the room temperature. Therefore, the increase in soil temperature also accelerated the desorption and migration of Pb. Moreover, the shock effect of the pulsed electric field can weaken the binding force between Pb and the soil surface, promoting Pb desorption[23], thereby increasing the number of mobile Pb particles.

3 Conclusion

(1) The introduction of a bidirectional pulsed electric field effectively enhanced the washing performance of citric acid for Pb removal from soil. When the citric acid concentration was 0.1 mol/L, and a bidirectional pulsed electric field with a frequency of 0.08 Hz, a voltage of 24 V, a duty cycle of 75%, and an energization

Table 4 Comparison between this study and previous studies

Pb content/ ($mg \cdot kg^{-1}$)	Voltage/V	Reaction time	Citric acid concentration/($mol \cdot L^{-1}$)	Pb removal rate/%	Reference
1 000	7.5	24 h	0.051	82.93	[16]
1 000	30	24 h	0.051	96.98	
1 000	20	65 d	0.5	9.80	[17]
940.83	30	20 d	0.1	56.85	[18]
508	30	3 d	0.2	≈35.00	[19]
965.80	16.5	10 d	0.20	86.20	[20]
965.80	16.5	10 d	0.05	71.65	
1 318.80	16	6 h	0.20	75.80	This work
1 318.80	24	6 h	0.05	70.40	

time of 6 h was applied, the Pb removal rate reached 72.7%, which was 22.4% higher than that achieved with citric acid alone. The influence of washing parameters on Pb removal followed the order: Citric acid concentration > Pulse voltage > Pulse time > Pulse frequency.

(2) When the bidirectional pulsed electric field was applied to assist citric acid washing of Pb-contaminated soil, it affected soil pH and simultaneously increased the soil temperature and electromigration activity. Under the effects of electro-osmosis and electroshock, the bidirectional pulsed electric field increased the content of acid-extractable Pb in soil, promoted the mobility of Pb and the contact probability between Pb and the active functional groups of citric acid, thereby enhancing the leaching removal efficiency.

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双向脉冲电场强化柠檬酸淋洗修复Pb污染土壤的研究

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摘要：降低淋洗剂消耗的同时显著提升重金属去除效率一直是重金属淋洗技术面临的关键挑战。为此，本研究采用双向脉冲电场辅助柠檬酸淋洗，对某废弃铅冶炼厂的铅污染土壤开展实验室规模的修复实验，评估了不同电场参数下Pb的淋洗效果并探讨其强化机理。结果表明，相较于单一柠檬酸淋洗，双向脉冲电场不同程度地提高了柠檬酸对Pb的去除效果。在脉冲频率0.08 Hz、电压24 V、占空比75%的条件下，通电处理6 h后Pb的去除率可提升至72.7%，较单一柠檬酸淋洗去除率提高22.4%，且柠檬酸消耗量减少50%。机制分析表明，双向脉冲电场通过影响土壤pH、提高土壤温度和增强离子电迁移，产生电渗和电冲击效应，促使Pb向酸可提取态转化，提高Pb的活性和与柠檬酸活性基团的反应几率，从而显著强化淋洗过程。研究结果表明，该技术能有效克服传统直流电场中电极极化与聚焦效应等问题，为发展快速、高效且更具工程可行性的土壤重金属修复方法提供技术支撑。

关键词：双向脉冲电场；化学淋洗；柠檬酸；重金属铅（Pb）；化学形态

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