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Short Communication Investigation on the Electrokinetic Removal of Pb(II) from Contaminated Silt Soil

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Electrokinetic remediation (ER) is a new green remediation technology for heavy metal pollution, and the remediation effect is better for coarse grained soil, while the remediation effect for fine grained soil needs to be verified. In this paper, an ER experiment of Pb(II) contaminated silt soil was carried out by adopting different electrode materials and remediation times (t), and the variations in the current (I), potential (U), pH value, removal rate (R), and energy consumption (E) during the ER process were analyzed. The results showed that as t increased, I first increased, then decreased and subsequently increased, while U first increased and then decreased. The soil pH value close to the cathode was significantly higher than that near the anode, and it was significantly affected by t, whereas was less affected by the electrode material. During the experiments, the minimum R for Pb(II) was only 14.25%, whereas the maximum R reached 95.27%. For Pb(II) contaminated silt soil, the R was higher and the E was lower when t was 4 d and a ruthenium-iridium coated titanium mesh was used as the electrode. The research results provide a guideline for the ER engineering practice of Pb(II) contaminated soil.

Keywords: Electrokinetic remediation; lead; silt soil; removal efficiency; energy consumption

1. INTRODUCTION

With the rapid development of industrial technologies, the soil environment has deteriorated, leading to one of the main problems of global pollution. Among many forms of soil pollution, heavy metal (such as Pb) pollution is particularly prominent [1-3]. Pb pollution mainly comes from batteries, dyes, gasoline, minerals, and exhaust gas. When Pb enters the soil and groundwater, it is absorbed by

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affecting health [4-6]. Therefore, the remediation and treatment of Pb-contaminated soil are urgent tasks. Electrokinetic remediation (ER) is a green technology that has emerged in recent years. It has the advantages of a short remediation time, a high efficiency, easy operation, and no secondary pollution. This technology is particularly suitable for the treatment of pollution in low-permeability soils and has become a research hotspot in the field of soil remediation [7]. By conducting ER with the polarity exchange method, Cai et al. [8] found that the removal efficiency (R) of Pb reached 87.7%, which was significantly higher than that of the conventional method (61.8%), and the focusing effect could be avoided with the combined method. Ng et al. [9-10] concluded that the R of Pb was improved by combining ER and soil washing methods, and found that using citric acid as the wash solution led to a high R, a low soil solution ratio, a stable pH and electrode integrity, verifying the feasibility of the method and enabling in-situ recovery in the cathode chamber via electro-deposition. Li et al. [11] investigated the ER characteristics between a Pb contaminated natural soil and kaolinite, and discovered that the enrichment ratios in the kaolinite soil were between 2.11 and 4.45 when using KNO₃ or EDTA-2Na as the catholyte, and significantly superior to the enrichment ratios in the natural soil. Zhang et al. [12] noted that the R of Pb and Cd increased by 23.52% and 28.60%, respectively, when sodium chloride and citric acid were used as composite electrolytes, indicating that the use of composite electrolytes is an effective improved method to remove Pb and Cd from paddy soils. Putra and Tanaka [13] found that the Pb in contaminated soil could be transferred into the entrapping zone by electrokinetic processes and immobilized by adsorption sites on polymeric aluminum residuals in aluminum drinking water treatment residuals. Based on the in-situ ER test with a hexagonal electrode configuration, Jeon et al. [14] found that the R after 24 weeks for As, Cu, and Pb reached 44.4%, 40.3%, and 46.6%, respectively. Based on the ER of Pb contaminated soil, Li et al. [15] investigated the effect of catholytes and anolytes on soil conductivity, and found that H⁺ with a higher ionic conductivity result in the increased of soil conductivity, whereas Fe^{2+} with a lower ionic conductivity induced the decreased of soil conductivity. Altin and Degirmenci [16] pointed out that the R of Pb changed from 60% to 70% for a natural soil containing clinoptilolite, gypsum and calcite minerals, and varied from 50% to 95% for a kaolinite soil. Suzuki et al. [17] found that the remediation effect for a Cd and Pb contaminated clay soil could be improved by adding ethylenediaminedisuccinic acid (EDDS) under neutral pH conditions, which provided a guideline for the optimization of an ER system. Tahmasbian et al. [18] concluded that the combination of electrokinetic with a cow manure extract or a poultry manure extract could decrease the effects on soil microbes, suggesting that manures can be used as catholytes to alleviate the environmental risks of electrokinetic methods. Alcantara et al. [19] studied the ER of Pb contaminated clay or sandy soils, and revealed that the use of 1% Tween-80 and 0.1 M EDTA as a processing fluid was most effective remediating polluted soils. Based on the results of zeta potential and electrokinetic experiments, Murillo-Rivera et al. [20] indicated that ammonium acetate and acetic acid were the best electrolytic solutions to remove Pb from a sandy soil contaminated with PbCO₃.

Previous studies verifying the reliability of the ER method have mainly focused on coarse grained soil, whereas research on fine grained soil is less conducted. In addition, the corrosion of electrode materials increases the energy consumption (E) and decrease the R, leading to a bottleneck

and limiting the development of the methods. Therefore, a new electrode material that can improve the R and reduce the E is urgently needed, and the reliability needs to be verified in the meantime. In this paper, an ER experiment for a Pb(II) contaminated silt soil was conducted by adopted different electrode materials and remediation times, and the variations in the current (I), potential (U), pH value, R, and E during the ER process were analyzed. The research results provide a guideline for practical engineering of ER for Pb(II) contaminated soil.

2. EXPERIMENTAL METHOD

2.1 Materials

The silt soil used in the test was collected from treelawns without heavy metal contamination at Dalian University of Technology. After the removal of debris, the soil samples were passed through a 2-mm sieve before being dried for later use. Table 1 lists the basic physical parameters of the silt soil.

Table 1. Physical parameters of silt soil

$G_{\rm s}$	$w_{\rm L}(\%)$	w _P (%)	$I_{\rm P}$	
2.64	24.4	16.5	7.9	

2.2 Instruments



Figure 1. Test equipment

The ER test equipment is shown in Figure 1, and it mainly included a model box, a DC power supply, an *I* and *U* measurement system, an electrolyte circulation system, an overflow collection system, and a data acquisition system. The model box was made of plexiglass, with a size of 310 mm (length)×90 mm (width)×120 mm (height). The size of the sample chamber was 100 mm×70 mm×100

mm, the size of the electrode chamber was 70 mm×70 mm×100 mm, and the size of the electrode was 70 mm×100 mm. The electrode chamber and the sample chamber were separated by double-layer porous plexiglass plates. During the test, filter paper was placed between the two plexiglass plates to prevent soil particles from entering the electrode chamber. Small holes were situated at the bottom of the electrode chambers on both sides of the soil chamber so that the peristaltic pump could pump the circulating electrolyte into the electrode chamber. Overflow holes were opened on both sides of the electrode chamber, and their positions were flush with the soil after loading to ensure that the upper surface of the soil was not covered by the electrolyte during the test. The main instruments and equipment used in the test are shown in Table 2.

Table 2. Main instrument and equipment

Instrument	Manufacturer
GPS-2303CDC stabilized power	Good Will Instrument Co., Ltd
DHG-9011A drying oven	Shanghai Jinghong Laboratory Instrument Co., Ltd
CT15RT freezing high speed	Shanghai Tianmei Biochemical Instrument and Equipment
centrifuges	Engineering Co., Ltd
CP214 analytical balance	OHAUS Instrument (Shanghai) Co., Ltd
FIVEEASY PLUS28 pH meter	Mettler Toledo Instrument (Shanghai) Co., Ltd
AA60000 atomic absorption	Shanghai Tianmei Biochemical Instrument and Equipment
spectrometer	Engineering Co., Ltd
VC86E digital multimeter	Double King Industrial Holdings Co., Ltd
YZ15 peristaltic pump	Baoding Lead Fluid Technology Co., Ltd

2.3 Method

As shown in Table 3, six groups of ER tests were conducted for the Pb(II) contaminated silt soil. To ensure the uniformity of the sample, the sample density was controlled at 1.42 g/cm^3 . During the test, *U* was set to 30 V, 0.1 mol/L KNO₃ was used as the anolyte, 0.15 mol/L citric acid was used as the electrolyte and circulating in the cathode at a rate of 1 mL/min. In addition, different electrode materials and treatment times were adopted, and all the tests were repeated.

Table 3. Test schem

Test No.	Treatment time (d)	Electrode
T1	1	Ruthenium-iridium coated titanium mesh
T2	2	Ruthenium-iridium coated titanium mesh
T3	3	Ruthenium-iridium coated titanium mesh
T4	4	Ruthenium-iridium coated titanium mesh
T5	4	Rhodium-iridium coated titanium mesh
T6	4	Ruthenium-iridium coated titanium plate

2.3.1 Preparation of soil samples

To prepare the Pb(II) contaminated soil sample with a concentration of 1000 mg/kg, firstly, 2 kg of dried silt soil, and a sufficient amount of distilled water were measured, and 3.1969 g of Pb(NO₃)₂ was dissolved in a beaker. Subsequently, the Pb(NO₃)₂ solution was added to the dry soil, and the beaker and glass rod were washed with distilled water two to three times. Then, distilled water was added to the soil until the soil was in a flow-plastic state, and the soil sample was stirred with a stirrer for 1 h to evenly distribute the Pb(II) in the soil. Finally, the soil was dried and crushed. During the crushing process, water was sprayed by a watering can to prevent the generation of toxic dust, and the crushed soil was passed through a 2-mm sieve for later use. After the sample was loaded, the electrolyte was injected into the electrode chamber, followed by equilibration for 48 h.

2.3.2 Sample collection

After the test, four thin metal sheets were inserted into the soil sample chamber at an interval of 2 cm, and the soil was divided into five equal parts. A spoon was used to dig out the soil, the areas from the cathode to the anode were labeled as S1-S5 sequentially, and the soil was dried for testing.

2.3.3 Measurement of I and U

The I and U were measured by a digital multimeter, the U on the sample chamber was measured, and the I of the entire system was measured. During the test, a timed camera system was used to take the I and U readings, and then the data were collected.

2.3.4 Measurement of pH

During the pH measurement of the soil sample, 5.0 g of the dried soil after the test was placed in a 50-mL polypropylene centrifuge tube, and 25 mL of distilled water was added. After the mixture was shaken for 2 h to fully mix the soil and water, the sample was placed in a high-speed centrifuge and centrifuged at 5000 r/min for 10 min. The supernatant was collected in a 50-mL centrifuge tube, a pH probe was inserted into the solution, and the mixture was stirred gently until the reading stabilized.

2.3.5 Determination of Pb(II) C

In the test, a digestion procedure combining step-by-step extraction and a single extraction method was adopted. After multiple measurements, the extraction rate of HCl was between 90% and 95%, while the extraction rate of EDTA was between 75% and 80%. Therefore, HCl was used as the extractant for the Pb(II) contaminated silt soil. After a corresponding fold-dilution of the obtained solution, the *C* of Pb(II) was measured with an atomic absorption spectrophotometer.

The *R* of Pb(II) was calculated as follows:

$$R = \frac{\left(m_0 - m_e\right)}{m_0} \times 100\%$$
 (1)

where *R* is the removal rate, %; m_0 is the initial total mass of pollutants, mg; and m_e is the total mass of the remaining pollutants, mg.

2.3.6 Calculation of E

The *E* of ER was calculated as follows:

$$E_c = \frac{1}{M_c} \int UIdt \ (2)$$

where E_c is the electric consumption for removing a unit mass of pollutants, W·h/mg; M_c is the total amount of pollutants removed, $M_c=m_0-m_e$, mg; U is the potential, V; I is the current, A; and t is the treatment time, h.

3. RESULTS AND DISCUSSION

3.1 Effect of current

Figure 2 shows the relationship between *I* and *t* during the ER process. The *I* reached the peak at 10 h, started to decrease, and then reached a minimum at 20 h. As t further increased, I continuously increased and stabilized at 60 h (0.5 A). The increase in I could be attributed to the increase in the concentration of soluble ionics due to the electrolysis reactions [21]. The variation pattern was different from that in reference [8], where I first increased and then decreased. In the meantime, Ahmed et al. [22] concluded that I increased during the first 18 h, then decreased and abruptly, and subsequently increased. The increase in I at the beginning of the test was related to the electrolysis of water. The continuous production of H^+ and OH^- provided sufficient mobile ions in the system [23, 24]. H⁺ entered the soil to form an acidic environment, promoting desorption and increasing the mobility of Pb(II) [25]. The I was maintained at a high level throughout the test, indicating that the circulating fluid continuously provided sufficient ions. The *I* of T1~T4 was significantly lower than that of T5 and T6 at the beginning of the test, but the difference was small at the later stage (60 h). The results showed that when ruthenium-iridium coated titanium mesh was used as the electrode, the I at the beginning of the test was significantly lower than that obtained with the rhodium-iridium coated titanium mesh and the ruthenium-iridium coated titanium plate, whereas the effect of the electrode material on the I was small in the later stage of the test.



Figure 2. Curves of current versus treatment time during electrokinetic remediation with 30 V potential, 1000 mg/kg initial concentration, and 0.15 mol/L citric acid as the catholyte





Figure 3. Curves of potential versus treatment time during electrokinetic remediation with 30 V potential, 1000 mg/kg initial concentration, and 0.15 mol/L citric acid as the catholyte

Figure 3 shows the relationship between U and t during the ER process. For T1~T3, U increased with t at the beginning of the test (20 h) and then gradually decreased with t, which is consistent with the results of reference [22] and different from the tendency of uniform descent [26]. The high pH decreased the ionic electrical conductivity of the soil, thereby causing the U to drop [27]. For T4~T6, the U at the beginning of the test first decreased, then increased, and started to decrease again after the U reached the peak. During the test, U was set to a constant 30 V, but the measured value was significantly lower than the set value. In addition, a black precipitate was observed in the cathode chamber during the test. This precipitate was soluble in 1 M hydrochloric acid. The black

precipitate first appeared when the *I* plateaued (approximately 60 h into the test). Then, a large amount of black precipitate appeared, and the amount stopped increasing at approximately 80 h. At this time, the *I* started to increase again, and the *U* also began to fluctuate. The main reason for the above phenomenon was that at the beginning of the test, as the number of free ions in the soil decreased, the conductivity of the soil decreased, and the corresponding resistance increased [28, 29]. The soil resistance accounted for a large proportion of the overall system resistance, so the *U* gradually increased in the early stage. After the experiment had continued for a period of time, the H⁺ produced by water electrolysis led to an increase in the soil conductivity. The cations in the soil migrated to the cathode under the acidic conditions. The ions that migrated to the cathode side precipitated on the separator or in the separator gap. As a result, the separator resistance increased.

3.3 Effect of soil pH

Soil pH is an important factor affecting the removal efficiency of heavy metal ions during ER. Figure 4 shows the pH distribution in the soil region after the ER test. S1 is the region close to the cathode, and S5 is the region near the anode. To effectively control the catholyte pH and facilitate the removal of Pb(II), a peristaltic pump was used to continuously pump 0.15 mol/L citric acid solution to the cathode to ensure that the cathode pH was maintained at a low level. The pH distribution in the soil region was found to be highly dependent on the electrolyte agent [28]. The pH in S1 and S2 (3.3~6) was significantly higher than that in S3~S5, indicating that the soil pH on the cathode side was significantly higher than that on the anode side. This result is consistent with the pH distribution laws in Pb contaminated soil in references [16, 30], and the acidification of the sample near the anode during ER as the operating time increased [21, 23]. Under an electric field, water was electrolyzed at the cathode, producing OH⁻. OH⁻ was dispersed in the cathode chamber and diffused into the soil near the cathode, alkalinizing the soil. Similarly, H⁺ was produced by the electrolysis of water at the anode and acidified the surrounding soil. As the ER process proceeded, H⁺ and OH⁻ continuously migrated. Because H⁺ migrated faster than OH⁻, H⁺ and OH⁻ finally met at the cathode side of the soil chamber. Pb ions precipitated under the alkaline conditions, resulting in a lower Pb mobility. Citric acid, which was injected into the cathode during the test, is a weak tribasic acid with a relatively strong acidity. The ionization degree of citric acid increases as the pH increases. Additionally, citric acid can form a complex with Pb and increase the mobility of Pb ions. The soil pH of T1 was high, while the difference in the soil pH of T2~T6 was small, indicating that the soil pH decreased with t and the electrode material had little effect on the soil pH.



Figure 4. Curves of pH versus soil region after electrokinetic remediation with 30 V potential, 1000 mg/kg initial concentration, and 0.15 mol/L citric acid as the catholyte

3.4 Effect of removal efficiency



Figure 5. Curves of Pb(II) concentration versus soil region after electrokinetic remediation with 30 V potential, 1000 mg/kg initial concentration, and 0.15 mol/L citric acid as the catholyte

Figure 5 shows the Pb(II) *C* distribution in the soil region after the ER test. The Pb(II) *C* in S1~S3 was significantly higher than that in S4 and S5, which is consistent with the Pb(II) *C* distribution laws in Pb contaminated soil in references [16, 30]. The Pb(II) *C* near the cathode was significantly higher than that near the anode, indicating that Pb(II) migrated from the anode to the cathode during the ER process [31]. The Pb(II) *C* of T1~T3 was high, and the Pb(II) *C* of T4~T6 followed the order T6>T5>T4, indicating that both *t* and the electrode material had great effects on the Pb(II) *R*. Figure 6 shows the Pb(II) *R* under different treatments. The Pb(II) *R* of T1 was only 14.25%, while the Pb(II) *R* of T4, T5, and T6 reached 95.27%, 91.17%, and 86.19%, respectively, which was significantly higher than the results of references [32, 33]. The results indicated that when the ruthenium-iridium coated titanium mesh was used as the electrode and *t* was 4 d (T4), the test results were the best, and the Pb(II) removal was the highest. In addition, black substances with different

morphologies were found on the surface of the cathodes after remediation. The plate electrode was covered with the black substance. The surface layer was relatively loose and washed off with water, but another layer of black substance underneath was difficult to remove. This layer was removed by soaking the electrode in 1 M hydrochloric acid. On the mesh electrode, the black substance was flocculent and could be completely removed by washing with water. The different morphologies of the black precipitates on the electrode surfaces might be related to the differences in the *R*. At the end of the ER, a large amount of black substance appeared, and the large-scale migration of Pb(II) was completed. At this time, the surface of the plate electrode was covered with a black substance that was difficult to remove, resulting in a decrease in performance. On the other hand, the black flocculent precipitate on the mesh electrode did not significantly affect the performance of the electrode.



Figure 6. Histogram of Pb(II) removal efficiency after electrokinetic remediation with 30 V potential, 1000 mg/kg initial concentration, and 0.15 mol/L citric acid as the catholyte

3.5 Effect of energy consumption

Table 4. Pb(II) removal efficiency and energy consumption

Test No.	Removal efficiency	Energy consumption	$E_{\rm c}$ (W·h/mg)
	(%)	(kW·h)	
T1	84.56	0.22078	0.27
T2	89.55	0.47406	0.55
T3	90.42	0.83579	0.96
T4	95.27	1.22697	1.34
T5	91.17	1.33692	1.52
T6	86.19	1.37851	1.66

E is an important factor for the practical application of electrokinetic treatment [22]. Table 4 summarizes the Pb(II) *R* and *E* of different tests. A comparison of T1~T3 showed that the *R* and *E* of T3 were relatively high. This result indicated that the Pb(II) *R* and *E* gradually increased with *t*, which is consistent with references [12, 14], and were significantly higher than the Pb(II) *R* when citric acid was used as the electrolyte agent [28]. In addition, increasing *t* and more acidification of the sample are

proposed to improve the removal efficiency, which will result in unacceptable costs and impractical operation in practical engineering [31]. A comparison of T4~T6 showed that T4 had a higher R and a lower E. Therefore, under the same t, the ruthenium-iridium coated titanium mesh electrode had a better removal effect and a lower E. Overall, the ruthenium coated was more economical than the rhodium coated, and the material consumption of the mesh electrodes was significantly lower than that of the plate electrodes. Therefore, it is recommended to use ruthenium-iridium coated titanium mesh as the electrode for the ER of Pb(II) contaminated silt soil. In the meantime, a good remediation effect can be achieved when t is 4 d.

Table 5 presents a comparison of Pb(II) removal for approaching the ER method. It is worth noting that it is difficult to directly compare the results due to the effects of the electrolyte agent, electrode, t, and C_0 . Although the adopted U gradient varied from 0.4 to 4 V/cm, the Pb(II) R changed from 7.41 to 95.27%, especially the highest R reached in this study. Furthermore, the E obtained in this study was slightly higher than that of reference [29] and significantly lower than that of reference [12], indicating that using the ruthenium-iridium coated titanium mesh used as an electrode can lead to better results when conducting ER tests. In addition, the electrolyte agent is a crucial factor in the application of the ER method. From an engineering perspective, citric acid is a good electrolyte due to it can be easily obtained, inexpensive and effective for most heavy metals. However, the feasibility of the ER approach needs to be further verified by taking into account the effects of scaling up, the electrode arrangement, the heavy metal ions and the soil types [28].

Electrolyte	Electrode	Treatment	Initial	Removal	Energy	Reference
agent		time	concentration	efficiency	consumption	
		(h)	(mg/kg)	(%)		
Distilled water	Graphite	192	1160	61.80		[8]
EDTA-2Na	Titanium	480	10000	7.41	131.7 kWh/m ³	[11]
Citric acid	Graphite	480	940.83	80.37	3.458 kWh	[12]
Citric acid	Graphite	216	3668	51.31	481.37 kWh/m ³	[21]
Citric acid	Platinum	168	1656	42.00	148 kWh/m ³	[22]
Citric acid	Graphite	168	2194	31.50	224 kWh/m ³	[23]
EDTA/Na ₂ SO ₄	Steel	360	1000	54.00	2534 kWh/m ³	[24]
Citric acid	Graphite	120	639.11	34.0	2342.23 kWh/t	[25]
Distilled water	Graphite	120	103.50	18.49		[26]
$NH_4C_2H_3O_2$	Titanium	120	45.0	65.6	720 kWh/m ³	[27]
EDTA	Graphite	24	402.20	64.31	0.0036 kWh	[28]
Citric acid	Steel	150	1000	32.52	0.92 kWh	[29]
Distilled water	Graphite	216	103	22.00		[31]
HNO ₃	Graphite	504	100	17.81	1151.58 kWh/m ³	[32]
Na ₂ -EDTA	Steel	360	955	79.41	1814.40 kWh/m ³	[33]
Acetic acid	Graphite	220	853.25	63.15	289 kWh/t	[34]
HNO ₃	Graphite	480	2500	38.14		[35]
Citric acid	Titanium	96	1000	95.27	1.23 kWh	This study

Table 5. Comparison of Pb(II) removal by electrokinetic remediation

4. CONCLUSIONS

(1) As t increased, the I showed an overall trend of increasing-decreasing-increasing, while the U showed an overall trend of decreasing-increasing-decreasing.

(2) After ER, the soil pH on the cathode side was significantly higher than that on the anode side. The soil pH decreased with *t*, and the electrode materials had little effect on the soil pH.

(3) The *C* of Pb(II) in the soil near the cathode was significantly higher than that near the anode. Pb(II) migrated from the anode to the cathode during the ER process. The *R* of Pb(II) of T1 was only 14.25%, while that of T4 reached 95.27%.

(4) Based on the test results and the *E* analysis, it is recommended to use ruthenium-iridium coated titanium mesh as the electrode for the ER of Pb(II) contaminated silt soil. In addition, a good remediation effect can be achieved when t is 4 d.

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CONFLICT OF INTEREST

On behalf of all authors, the corresponding author states that there is no conflict of interest.

DATA AVAILABILITY

The data used to support the findings of this study are available from the corresponding author upon request.

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