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Enhanced electrokinetic removal of mercury from sandy soil using thioglycolic acid and lignocellulosic sawdust at acidic conditions

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ABSTRACT

Mercury is a resistant environmental contaminant that is difficult to remediate due to its low mobility in natural soils, especially under neutral and alkaline conditions. Therefore, in this study, the efficiency of electrokinetic remediation of mercury-contaminated sandy soil was investigated using different laboratory conditions within varying pH ranges (4, 7, and 10) and using both thioglycolic acid and sawdust. The sandy soil was leached with mercury nitrate at a concentration of 300 mg/kg, and treatment was carried out under a constant electric field of 1.5 V/cm. The results showed that the acidic environment and the presence of thioglycolic acid achieved a maximum removal efficiency of 92.4%. This provided a stable complexation of mercury, increasing its transport towards the cathode, thus demonstrating maximum improvement in mercury mobility through increased solubility and decreased mercury hydroxide ($Hg\ (OH)_2$) deposition. Meanwhile, sawdust retained mercury via ion exchange and hydrogen bonding. While the removal efficiency reached 86.8% under acidic conditions of pH 4 alone. The pH and electrical conductivity patterns facilitated the electrochemical gradients that stimulated metal transport. These results demonstrate the synergistic potential of green chelators and biodegradable adsorbents in electrokinetic remediation systems to provide an effective and environmentally friendly method for remediating mercury in sandy soils.

Keywords: electrokinetic remediation, mercury, thioglycolic acid, heavy metals, lignocellulosic adsorbents.

INTRODUCTION

Mercury (Hg) is one of the most toxic and persistent heavy metal found in various forms, including elemental, organic, and inorganic species like methylmercury, which is bioaccumulate and neurotoxic (Aljanabi et al., 2024; Rosestolato et al., 2015). Anthropogenic activities such as artisanal gold mining, and coal combustion, contribute significantly to Hg release into terrestrial ecosystems (U.S. Environmental Protection Agency, 2017). Once in the soil, Hg binds tightly to organic matter, sulfides, and clay minerals, making its removal a complex task (Al-Khyat et al., 2023; Hamdan, 2021). Traditional remediation techniques, including excavation and thermal desorption, are capital-intensive and often environmentally disruptive. In recent years, electrokinetic remediation (EKR) has emerged as a promising in-situ approach for removing heavy metals from low-permeability soils. This technique involves applying a low direct current (DC) electric field across electrodes inserted into contaminated soil. The electric field induces several transport mechanisms – electromigration, electroosmosis, and electrophoresis – allowing contaminants to migrate towards collection zones (Reddy and Cameselle, 2009). Electrolysis at the anode generates H^+ ions, while OH^- ions accumulate near the cathode, resulting in acidic and alkaline fronts, respectively. Hg cations (Hg^{2+}) tend to precipitate under high pH, reducing removal efficiency (Ge et al., 2022). necessitates the use of enhancing agents to maintain solubility and mobility. Chelating agents like EDTA and citric acid have been explored, but they pose environmental concerns due to low biodegradability (Aljanabi et al., 2024; Hamdan et al., 2024; Jama and Saeed, 2023). thioglycolic acid (TGA) have shown promise. TGA ($HSCH_2COOH$) contains both thiol and carboxyl functional groups, capable of forming stable complexes with Hg^{2+}, facilitating its mobility under electrokinetic fields. In parallel, sawdust as a bio-based adsorbent has gained attention due to its low cost and metal-binding properties (Li et al., 2023; Zaman et al., 2021). Lignocellulosic structures in sawdust contain hydroxyl and methoxy groups that contribute to cation exchange and complexation with heavy metals. Sawdust also functions well as a barrier in EKR cells to limit reverse osmosis and trap migrating ions (Meez et al., 2021). Despite individual advantages of TGA and sawdust, little research has examined their combined use in EKR systems for Hg under varying pH levels. This study addresses this gap by evaluating the synergistic effect of thioglycolic acid and sawdust on Hg remediation at pH 4, 7, and 10, under controlled EKR conditions.

MATERIALS AND METHODS

Soil sampling and preparation

Soil was collected from Baghdad, Iraq, fields that are sandy and have low organic matter. The soil was air-dried, milled, and sieved using a 2 mm sieve to standardize particle size distribution. Initial physicochemical properties were analyzed: pH 7.2, electrical conductivity 614 µS/cm, and organic matter 0.04%. The soil's texture was sandy according to USDA classification. Table 1. Outlines the properties of soil.

Mercury contamination procedure

Mercury nitrate [$Hg(NO_3)_2 \cdot H_2O$] was used to simulate soil contamination. A 2000 mg/L stock solution was prepared by dissolving 3.26 g of the compound (324.61 g/mol) in 1 L deionized water. The stock solution was diluted and mixed well with 5 kg of anhydrous soil to have a uniform concentration of 300 mg/kg Hg^{2+} across the sample, corresponding to a total of 1500 mg Hg^{2+}. The soil was then aged for 24 hours to reach equilibrium.

Thioglycolic acid

Thioglycolic acid ($HSCH_2COOH$), an organic disulfur containing two functional groups (–SH and –COOH), was chosen as the chemical enhancer. TGA is present in its undissociated state at pH 4 and 7. A volume of 100 mL of 0.1 M TGA was placed in the anode compartment for treated configurations.

Sawdust

Sawdust was collected from local carpentry workshops. It was washed with distilled water, dried at 60 °C, and sieved (size 0.5 mm). This biomass adsorbent was placed between the cathode and soil chambers in the electrokinetic cell as a reactive barrier. The lignocellulosic portion of sawdust provides multiple adsorption sites (hydroxyl, carbonyl) for the adsorption of Hg^{2+} (Hawal et al., 2021).

Electrokinetic cell

The electro-kinetic cell used in this research is shown in the form of a 3D sketch in Figure 1. The electro-kinetic cell is fabricated from glass. The internal dimensions of the cell are 60 cm in length, 15 cm in width, and 15 cm in height. The effective length of the soil specimen in this cell is 30 cm. In the electro-kinetic cell, 6 cm thick sawdust was placed as a barrier between the soil and the cathode compartment. The sawdust barrier has a height of 15 cm. The electrode compartments of size 12 × 15 × 15 cm had a valve, 10 cm from the bottom, which was utilized for flow control in the cell and electrode. The electrodes were made in the form of a cylinder using graphite material, 12 cm high and 5 cm diameter. The electrodes were drilled at the highest point to create a threaded shape for the purpose of conducting electricity. Perforated plastic plates were used to establish a barrier between the sawdust and the soil at one end, and between the sawdust and the cathode electrode at the other end. The plates are 15 × 15 cm in size and have holes of 7 mm diameter. The holes are 1 cm apart, as measured from the center of one hole to the center of the other. A filter paper (Whatman 40) is placed in front of each perforated plastic plate. A DC power supply (LDESTAR, LP 3005D) was utilized to provide the cell with a constant voltage via the electrode. The electric current flowing through the soil during the experiments was measured by a multi-meter.

Experimental work

The electrokinetic remediation cell was thoroughly washed with distilled water to remove any

Table 1. Physicochemical properties of the soil samples

Property	Value
Particle size distribution (ASTM D 422) Sand (%) Silt (%) Clay (%) Texture classification	 93.35 3.20 3.45 sand
Specific gravity	2.64
EC (μS/cm)	614
Organic matter content (%)	0.04
Initial pH	7.2
Soil porosity (n)	51.7

residual contaminants that might affect the accuracy of the experimental procedure. After cleaning, filter paper was carefully placed between the soil and the electrode chambers to prevent soil migration and ensure a consistent flow interface. Laboratory-prepared mercury-contaminated soil, prepared at a concentration of 300 mg/kg, was tamped into the central soil chamber using a soil shaper to ensure even contact and uniform distribution of the electric field. The system was then left undisturbed for 24 hours to stabilize. Throughout the test period, a constant solution level was maintained in the anode and cathode chambers to prevent hydraulic gradient fluctuations that might distort ion migration. The electrodes were installed in their respective chambers and connected to a DC power supply. A constant voltage of 1.5 V/cm^3 was applied to the system. Plain tap water was used as the base solution in all experiments. Four experiments were conducted to evaluate the effect of pH (4, 7, 10) and thioglycolic acid on mercury removal. For the first three experiments, tap water was adjusted with hydrochloric acid and sodium hydroxide (NaOH) to achieve the desired pH. In the fourth experiment, 100 ml of 0.1 M TGA was added to the aqueous medium. During operation, the pH, electrical conductivity, and current of the solution were continuously monitored, while the pH of the aqueous medium was continuously adjusted to maintain the desired pH under the specified experimental conditions by adding small amounts of HCl or NaOH. At the end of each experiment, the soil column was divided into five equal aliquots. Each aliquot was extracted, labeled, and stored for subsequent analysis of residual mercury content. Table 2 summarizes all environmental and operational parameters of the laboratory setup.

ANALYTICAL METHODOLOGY

To determine the mercury concentration in soil, the three-acid digestion method was used to treat soil samples prior to mercury analysis. The digestion solution consisted of 5% nitric acid (HNO_3), 3% hydrochloric acid (HCl), and 1% perchloric acid ($HClO_4$). This mixture is an excellent chelator for organic and inorganic compounds, allowing mercury ions to enter the solution. For each air-dried soil sample, 1 g was added to a digestion flask, and 5 ml of the acid mixture was added. The sample used was allowed to predigest overnight at room temperature. The next day, the flasks were placed semi-dry on a hot plate in a fume hood. After cooling, 20 ml of distilled water was added to the residue to dissolve it. Whatman filter paper was used to

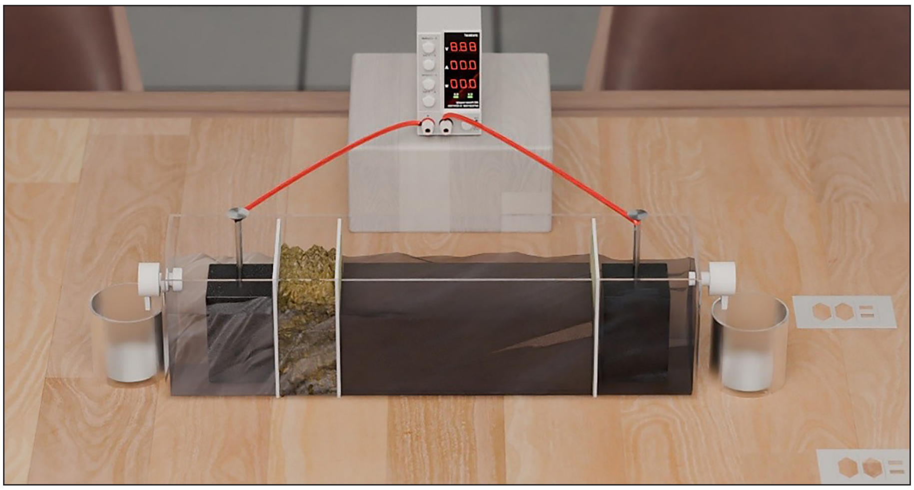

Figure 1. Three-dimensional schematic diagram of the EKR cell

Table 2. Summary of experimental and operating conditions

EX. No.	Initial Hg (mg/kg)	pH	Voltage (V/cm)	TGA	Sawdust	Objective of the experiment
EX-1	300	4	1.5	No	Yes	Baseline (Acidic)
EX-2	300	7	1.5	No	Yes	Baseline (Neutral)
EX-3	300	10	1.5	No	Yes	Baseline (Alkaline)
EX-4	300	4	1.5	Yes	Yes	Effect of TGA

filter the solution, and the filtrate was diluted to a total volume of 25 ml in volumetric flasks. Test solutions were analyzed using cold vapor atomic absorption spectrometry (CVAAS) according to U.S. EPA Method 7471B. The instrument was calibrated using mercury standards, and the resulting absorbance values were correlated to standard calibration charts and recorded in terms of concentrations. Thus, residual mercury was quantitatively measured in each soil fraction.

Influence of pH

In this study, pH was a critical parameter affecting the migration and removal efficiency of mercury ions from contaminated soil via electrokinetic remediation. Three control experiments (EX-1 to EX-3) were conducted without amendments to analyze the individual effect of pH. The soil was initially injected with 300 mg/kg of mercury (Hg^{2+}) and then exposed to pH values of 4, 7, and 10. The removal efficiency at EX-1 (Ph 4) was 86.78%, indicating that the acidic environment facilitates mercury dissolution and transport. Figure 2 shows a significant decrease in mercury concentrations near the anode and an accumulation of mercury toward the cathode, based on the efficiency of electrokinetic migration under acidic conditions. This is consistent with (Acar and Alshawabkeh, 1993a), who also indicated that H^+ ions enhance metal solubility by reducing complexes with soil particles. In EX-2 (pH 7), the soil conditions were neutral, and there was moderate mercury mobility. Electromigration continued, but complexes with soil oxides and organic matter in the micro-phases reduced transport efficiency. Precipitation of $Hg(OH)_2$ near the cathode may occur due to the high pH, resulting in a reduction in removal efficiency of 84.8%. (Yeung and Gu, 2011) also reported the same, noting that neutral pH conditions can slow contaminant transport due to competing adsorption mechanisms and low ionic strength. At EX-3 (pH 10), alkaline conditions showed strong inhibition of mercury migration. At higher pHs, mercury readily precipitated as insoluble hydroxides according to a reaction: this precipitation was primarily on the cathode side, where OH^- ions accumulated because of water electrolysis. Consequently, mercury became fixed in the soil matrix, particularly in the central and cathodic regions, with a removal efficiency of only 74.2%. This is known as the "concentration effect," where metal ions concentrate and precipitate near the cathode, preventing further migration (Sharma et al., 2020). Figure 3 shows a clear gradient in soil pH – from (6.2–3.9) near the anode to the (9.7–7.4) near the cathode, due to the differential mobility of H^+ and OH^- ions. The acid front moved more rapidly, creating more favorable conditions for migration at the anode side, while the alkaline front hindered metal movement near the cathode (Acar and Alshawabkeh, 1993b). Furthermore, Figure 4 shows the electrical conductivity distribution, which was highest in the middle portions (2.22–2.38 µS/cm) and decreased significantly towards the cathode (~1.31 µS/cm), indicating reduced ionic mobility due to precipitation. Based on this (Hamed and Bhadra, 1997), lower pH improves metal solubility and electric current due to increased ion availability, while higher pH promotes precipitation and reduces current flow. In general, pH critically controls the behavior of mercury in EKR systems through its influence on the dissolution and transport of Hg^{2+} under acidic conditions (pH 4) and its precipitation and migration inhibition under alkaline conditions (pH 10 in acidic conditions). Therefore, proper EKR performance requires strict pH control to maintain mercury in a mobile and extractable state throughout the treatment period.

Influence of thioglycolic acid

The use of thioglycolic acid in the chelating metal extraction process plays a pivotal role in overcoming the limitations imposed by metal precipitation, especially in alkaline regions.

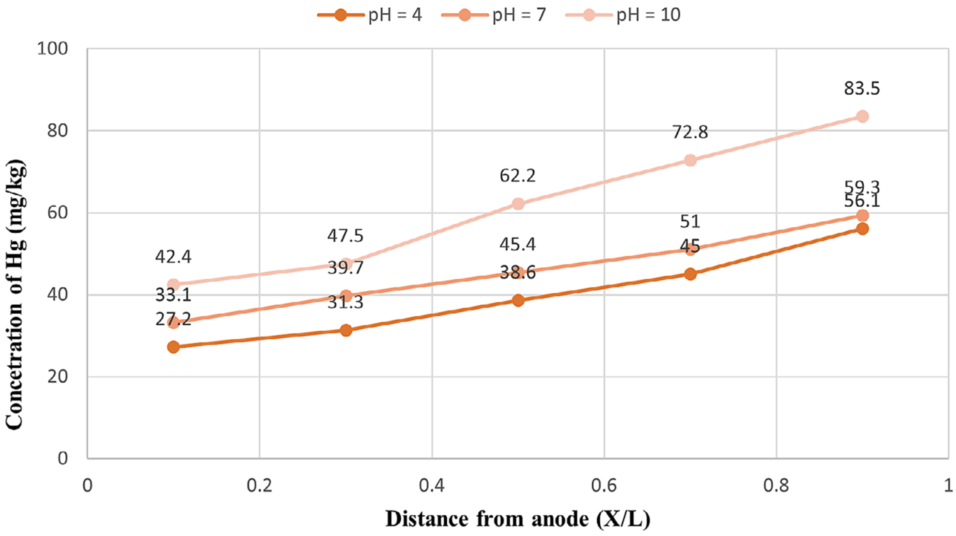

Figure 2. Residual mercury concentration (mg/kg) in soil under varying pH

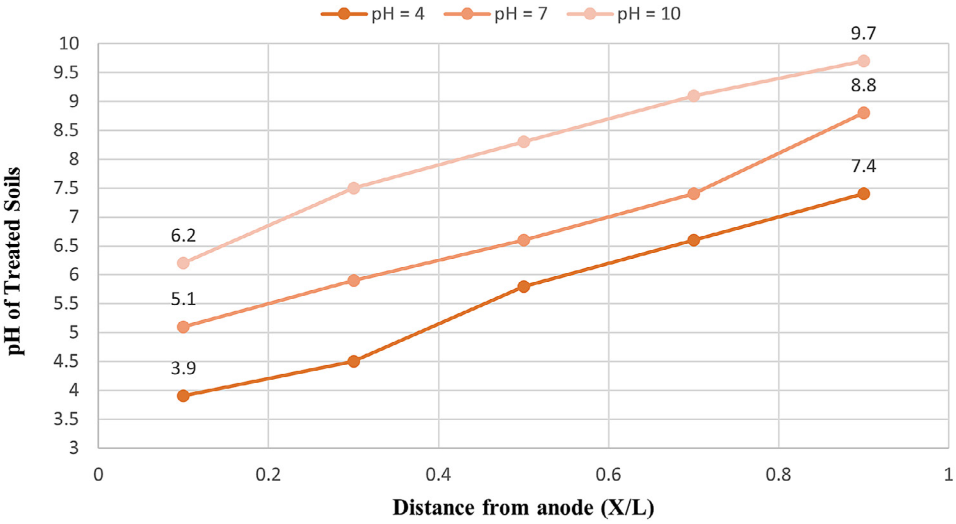

Figure 3. pH distribution in soil under varying initial pH conditions

During the EX-4 experiment, 100 mL of 0.1 M TGA was introduced into the anode tank. As shown in Figure 5, the mercury concentration pattern showed a more uniform decrease compared to the unoptimized setup. The results were remarkable, with mercury concentrations decreasing to 32.89 mg/kg near the cathode and 14.6 mg/kg near the anode. This indicates that the chelating effect of TGA effectively stabilized Hg^{2+} ions in solution, preventing their precipitation as hydroxides and facilitating their continuous transport through the soil column. TGA helped maintain mercury solubility across varying pH ranges, an important advantage given the sharp pH shifts typically observed in EKR systems. Figure 6 shows that the pH in TGA-treated cells was slightly more regulated due to the acid contribution, further enhancing the conditions for metal migration, ranging from 4 to 7.4 from the anode towards the cathode. Figure 7 reveals higher EC across the soil column in TGA-enhanced systems, It reached its highest point at 2.83 µS/cm, resulting from increased ionic species in the solution, confirming the improved mobility of metal-ligand complexes. In addition to improving metal solubility, TGA is biodegradable, cost-effective, and environmentally safer than traditional synthetic chelators such as EDTA. Previous studies (e.g., Ge et al., 2022) have also demonstrated the success of organic acids such as TGA in increasing the EKR efficiency of metals such as Cd and Pb. Thus, the

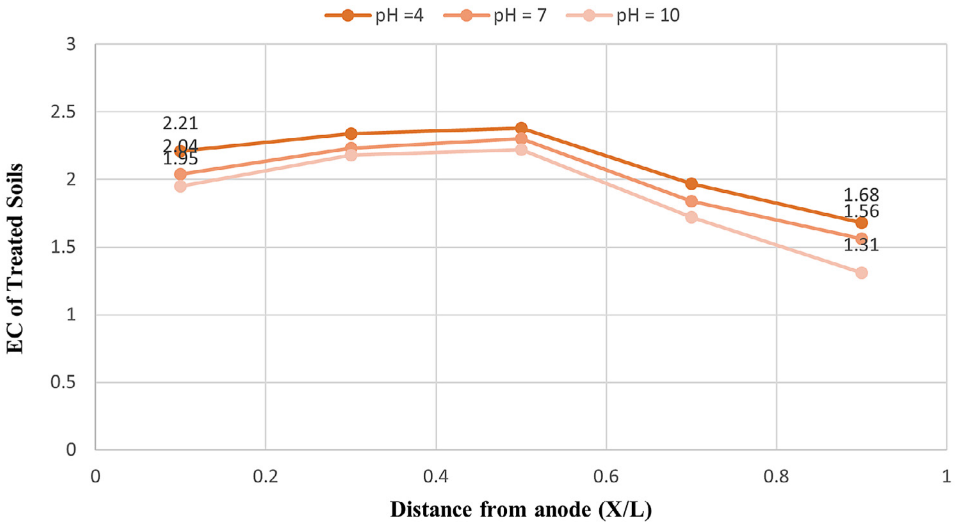

Figure 4. Electrical conductivity of soil under varying pH

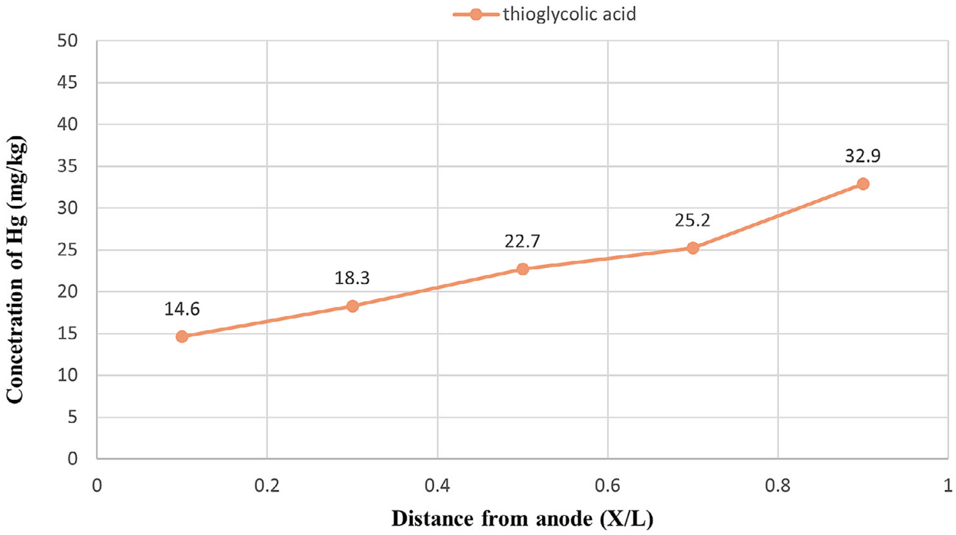

Figure 5. Mercury concentration (mg/kg) in soil after thioglycolic acid addition

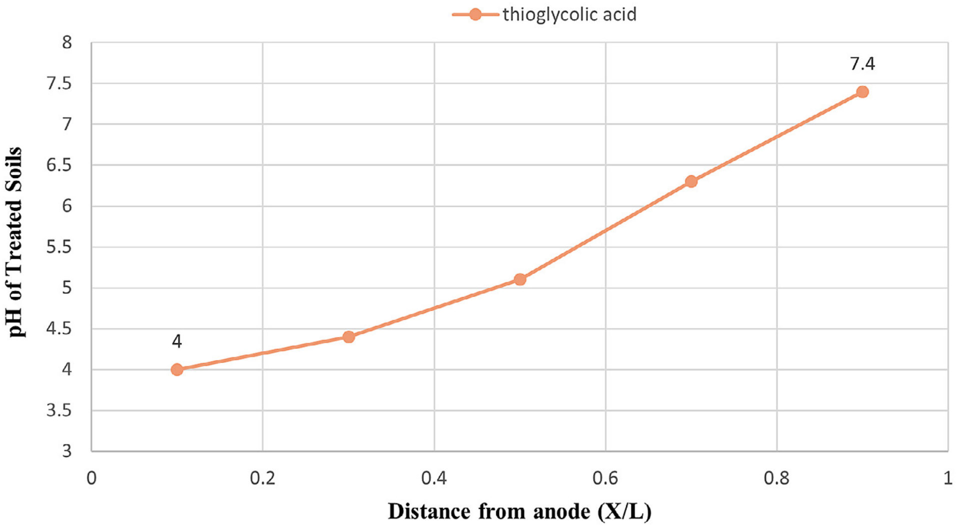

Figure 6. Profile of soil pH under thioglycolic acid enhanced conditions

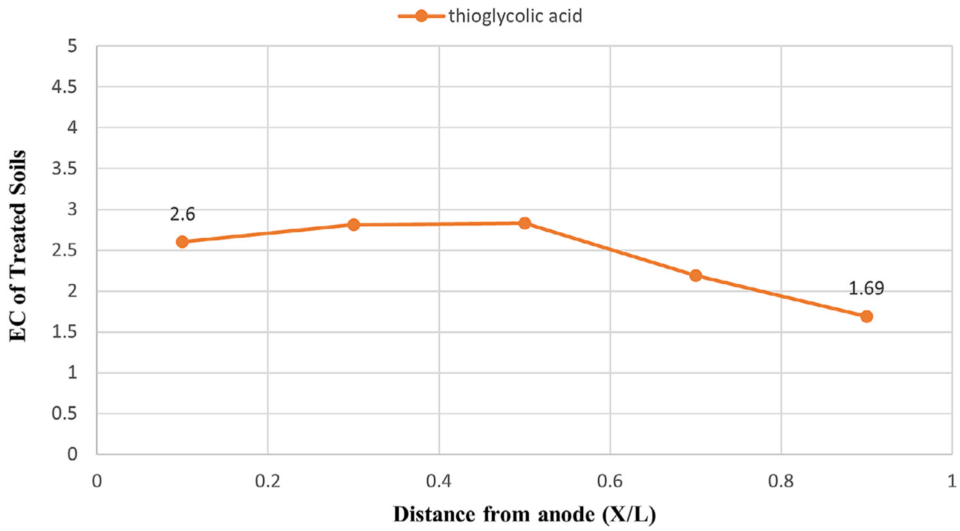

Figure 7. Profile of soil EC under thioglycolic acid enhanced conditions

Table 3. Final removal efficiency of mercury

Experiment	Description	Average concentrations	Removal efficiency (%)
EX-1	pH 4 – no enhancers	39.64	86.78
EX-2	pH 7 – no enhancers	45.7	84.8
EX-3	pH 10 – no enhancers	102.8	74.2
EX-4	pH 4 + TGA	22.74	92.4

use of TGA in this study significantly enhanced mercury removal efficiency, especially under acidic conditions.

Mercury removal efficiency and concentration reduction

Efficiency of mercury removal in EKR must be measured to allow assessment of system performance and comparison of treatment conditions. For the purposes of this research, removal efficiency (RE) was calculated from a standard mass-balance equation (1) comparing the pre-and post-treatment mercury concentration:

$$RE\ (\%) = \left(\frac{Ci}{Ci - Cf}\right) \times 100\% \qquad (1)$$

where: Ci – Initial mercury concentration (mg/kg) = 300 mg/kg; Cf – Average post-treatment mercury concentration (mg/kg).

This equation, widely applied in remediation studies (Reddy and Cameselle, 2009; Yeung, 2011), allows the comparison of different experimental conditions according to a standard basis. The following Table 3. Shows the last average concentrations of mercury and the corresponding removal efficiencies for all experiments:

It can be seen from the Table 3 and Figure 8. That the highest removal efficiency was in EX-4 (TGA + pH 4) at 92.4%, which reflects the high chelation property of TGA to keep Hg^{2+} soluble and mobile. While EX-1 with pH 4 performed well with only 86.78%, but lower than the improvised setups. The lowest efficiency was in EX-3 (pH 10), where mercury got precipitated out as insoluble hydroxides, limiting migration and removal. These results verify the importance of chemical amendment and pH management to improve electrokinetic remediation for mercury-polluted soil.

CONCLUSIONS

The experiments in this study confirmed the effective role of electrokinetic treatment in treating sandy soil contaminated with mercury, using thioglycolic acid as a chemical chelating agent and sawdust as a biologically derived and environmentally friendly adsorbent. The entire series of laboratory experiments was carefully conducted to

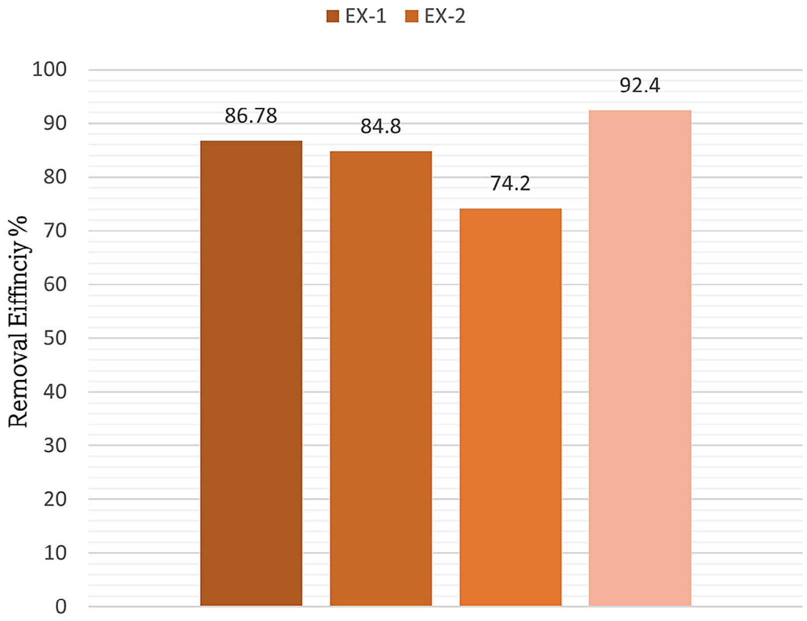

Figure 8. Mercury removal efficiency (%) under various remediation conditions

evaluate the effects of pH variation, TGA chelation, and sawdust adsorption on mercury ion migration and removal efficiency. The results showed that the acidic medium (pH 4) of the disinfection solution in EX-1 significantly enhanced the migration and removal of mercury ions to a removal efficiency of 86.78%, unlike EX-2 and EX-3, i.e., when neutral and basic conditions (pH 7 and pH 10) prevailed, the removal efficiency was 84.8% and 74.2% respectively. Meanwhile, in EX-4, when TGA was used as the chelating agent, the maximum removal efficiency was achieved, with TGA increasing the removal efficiency by approximately 6% which reach to 92.4% compared to unenhanced conditions.

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