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Efficacy of EDTA Enhanced Electrokinetic Technique on Metal Spiked Soil

Nuruddin Mohammad ¹[0000-0002-2504-5075], Arif Ali Baig Moghal ²[0000-0001-8623-7102] and Romana Mariyam Rasheed ³[0000-0002-6504-3517]

¹ Research Scholar, Department of Civil Engineering, National Institute of Technology, Warangal, India. Email: mn22cer1r08@student.nitw.ac.in

² Associate Professor, Department of Civil Engineering, National Institute of Technology, Warangal, India. Email: baig@nitw.ac.in; reach2arif@gmail.com

³ Assistant Professor, Department of Civil Engineering, TKM College of Engineering, Kollam; Research Scholar, Department of Civil Engineering, National Institute of Technology, Warangal, India.
Email: romanamrasheed@tkmce.ac.in; rm712020@student.nitw.ac.in

Abstract. There are many remediation techniques in the current scenario which facilitate the removal of contaminants from soil and groundwater. However, techniques such as bioremediation, soil vapor extraction, soil flushing, phytoremediation, vitrification, and others have proven incapable of transporting organic and inorganic contaminants in low permeable soils. Electrokinetic treatment (EKT) is one technique that can overcome this limitation. This paper illustrates EKT studies conducted to observe the effectiveness of EDTA solution (purging solution) in different concentrations for removing heavy metals from high plastic clayey soil. The EKT was conducted on soil spiked with copper and lead ions at 500 & 1000 mg/kg concentrations. Using two perforated iron electrodes, an electric gradient of 1 V/cm was applied to the soil specimens. The EDTA as a purging solution was used at the catholyte at varying molar concentrations of 0.05, 0.1 & 1M. EDTA as a purging solution increased the Cu²⁺ and Pb²⁺ removal efficiency by EKT in high plastic clayey soil. It was observed that 1M EDTA solution enhanced the Cu²⁺ removal efficiency by 41% and 48% from the 1000 mg/kg and 500 mg/kg loaded soils. Whereas, higher removal efficiency of 46% and 36% was observed for Pb²⁺ contaminated soil for load ratios of 1000 mg/kg and 500 mg/kg at 0.1M EDTA. This study proves the effectiveness of EDTA as a purging solution in decontaminating the high plastic clayey soil spiked with Cu²⁺ and Pb²⁺ using EKT.

Keywords: Decontamination, Electrokinetic, Electromigration, Electroosmosis, Heavy metals

1 Introduction

The soils contaminated with heavy metals can cause a severe threat to the environment and also to the living organisms. Due to urbanization and industrialization, the heavy metals in the soils are increasing [1, 2]. At many polluted sites, heavy metals, especially cadmium, chromium, arsenic, nickel, lead, zinc, and mercury, pose a significant public health problem [3, 4]. Electrokinetic (EKT) remediation has emerged as a potential method of removing heavy metals and a variety of organic contaminants from fine-grained soils [5, 6, 7] and has exhibited better performance compared to bioremediation and calcite precipitation [8, 9]. The setup consists of a minimum of two electrodes penetrated the soil through which the low DC voltage gradient is passed. The electrode can act as an anode, and the cathode depends on the electric flow direction. As the electric current is passed through the soil, the contaminants will transport depend on the different mechanisms such as electroosmosis, electromigration, and electrophoresis [10, 11, 12]. The primary mechanism involved in heavy metal removal is electromigration. In electromigration, the positive ions (cations) will migrate towards the negatively charged electrode (cathode). Similarly, the negative ions (anions) will migrate towards the positively charged electrode (anions) [13, 14, 15]. During EKT, the water at the anode zone generates the H^+ ions, which reduce the pH value at the anode. Similarly, water at the cathode zone produces OH^- ions, which cause an increase in pH at the cathode. The reduced pH helps in the desorption of heavy metal ions from the soil particle surface [15]. The heavy metals will precipitate at high pH and become difficult to remove using electric current.

The researchers followed different ways to improve the electrokinetic removal efficiency. One is using chelating agents, which can solubilize heavy metal contaminants at different pH conditions [16]. Chemical agents such as acetic acid (CH_3COOH), citric acid ($C_6H_8O_7$) and nitric acid (HNO_3), etc., are used to control the pH at the electrode zone. Many studies have shown that EDTA is an effective chelating agent and can reduce the pH enabling the desorption of heavy metals [17]. Even though these chemical substances are very effective in contaminant removal, they can cause damage to the environment. Plant growth can be affected due to the acidic nature of these chemicals. So, there is a need to control the use of acid for soil remediation. The main intention of this work was to study the effect of using different concentrations of EDTA solutions as catholyte in decontaminating copper and lead from the artificially spiked high plastic clayey soil.

2 Materials and Methods

2.1 Materials

The soil used in the present study was collected from a site in Batpalli cheruvu, located in the Telangana State of India. The basic soil properties were determined as per IS: 2720-1985 [18, 19, 20, 21] and are mentioned in Table 1.

Table 1. Basic properties of soil

S. No.	Properties	Value
	Atterberg limits [18]	
1	Liquid limit	53.63%
2	Plastic limit	19.54%
3	Plasticity index	33.09%
4	Specific gravity [19]	2.68
	Water content-dry density relation [20]	
5	Maximum dry density	1.72 g/cc
6	Optimum moisture content	19.70%
7	Permeability [21]	10^{-8} cm/s
8	Soil classification (USCS)	CH

2.2 Chemicals used

Copper nitrate ($\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$) of 99.5% purity and lead nitrate ($\text{Pb}(\text{NO}_3)_2$) of 99% purity (analytical grade quality) supplied by SRL Chemical, Mumbai, India were used for spiking the high plastic clayey soil. Ethylenediaminetetraacetic Acid (EDTA) was used as a chelating agent in the cathode reservoir to remove the metal contaminants effectively. The EDTA was prepared in different concentrations, and relative removal efficiencies were calculated using Equation 1.

$$\text{Removal efficiency (\%)} = \frac{\text{Initial contaminant concentration} - \text{Final contaminant concentration}}{\text{Initial contaminant concentration}} \quad (1)$$

2.3 Electrokinetic Setup

The electrokinetic setup was fabricated in the geotechnical engineering laboratory at the National Institute of Technology, Warangal. The electrokinetic cell body with a rectangular shape (18.3 cm length, 10.7 cm breadth, and 11.5 cm height) and top end open was fabricated using glass material as shown in Fig. 1. Regular iron plates with perforations to allow water to pass through were used as electrodes (Fig. 2).

A DC-Regulated power supply -PHY 8230D was used to control the voltage gradient, which can regulate the voltage up to 30 V. However, the length of the tank should be less than 30 cm to maintain the voltage gradient of 1 V/cm. Thus, the length of 18.3 cm was fixed while fabricating the setup. Likewise, the width and height of the setup were fixed according to the dimensions of the iron plate electrodes.



Fig. 1. Fabricated electrokinetic cell

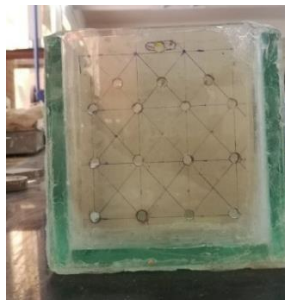


Fig. 2. Perforated iron sheet electrode attached to the electrokinetic cell

2.4 Soil Spiking Procedure

The soil was artificially spiked by dissolving the nitrate salts of copper (Cu^{2+}) and lead (Pb^{2+}) in water for respective cases. 3.8017 g of $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ was dissolved in 1000 ml of tap water to get 1000 ppm of copper stock solution. In similar lines, 1.598 g of lead nitrate ($\text{Pb}(\text{NO}_3)_2$) was dissolved in 1000 ml of tap water to get 1000 ppm of lead stock solution. Using these stock solutions, the soil was spiked with appropriate dosages to get 500 mg/kg and 1000 mg/kg targeted loads.

Generally, the contaminants discharging from the industries will dissolve in ground-water and adsorb on the soil particles. Here the chemicals were dissolved in tap water to replicate the same field condition.

2.5 Sample Preparation

Relying on the dry density curve of soil and the volume of the electrokinetic cell (2252 cc), the dry weight of high plastic clayey soil (passing through a 1.18 mm IS sieve) was calculated. This soil was spiked with Cu^{2+} and Pb^{2+} and filter papers were placed between the soil specimen and iron plate prior to placing the soil inside the EKT cell. The spiked soil was placed in the middle compartment of the EKT cell and compacted in three layers to achieve the targeted dry density (1.72 g/cc).

The respective solution was poured into the corresponding electrode reservoirs and ensuring soil specimen saturation. A low direct current (DC) was passed through the

electrodes to the soil and a voltage gradient of 1 V/cm was applied throughout the soil (Fig. 3). The test was terminated after a certain period of 20 days. The residual metal concentration in the untreated and treated soil was determined by subdividing the specimen into four parts. The concentrations of metal ions in samples procured from each subdivision were determined using Inductive Coupled Plasma Atomic Emission Spectrometry (ICP-AES) as per U.S. EPA 3050B.

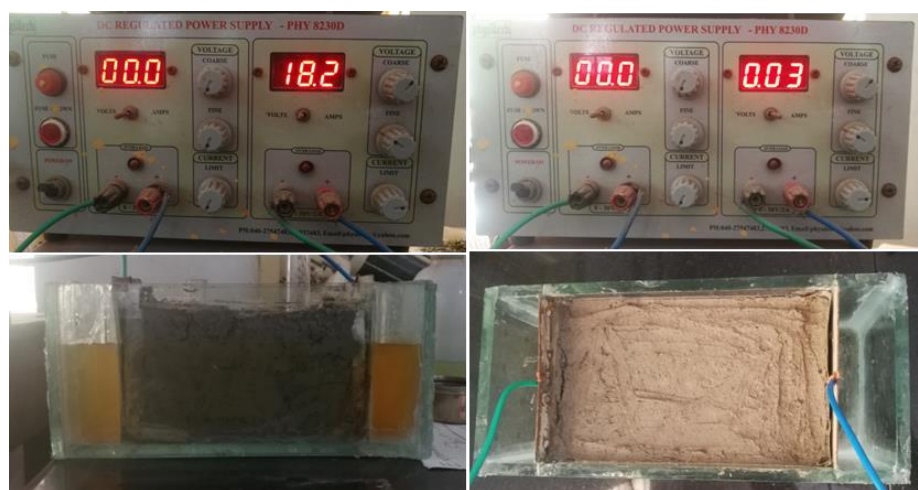


Fig. 3. Electrokinetic setup with soil connected to DC power supply unit

3 Results and Discussions

The soil samples were spiked with Cu^{2+} and Pb^{2+} at two different load ratios of 500 mg/kg and 1000 mg/kg. The EKT test was conducted using two different purging solutions i.e., tap water, and EDTA to study their effect on the Cu^{2+} and Pb^{2+} removal efficiencies. The soil in the middle compartment was subdivided into four parts and named sections 1, 2, 3, and 4. The samples were procured from each section to determine the residual metal concentrations after the completion of the test using ICP-AES.

3.1 Treatment with tap water

The soil samples spiked with Cu^{2+} and Pb^{2+} (at two different load concentrations of 1000 mg/kg and 500 mg/kg) were purged with tap water to study its effect in decontaminating the soils after a period of 20 days.

Samples spiked with 500 mg/kg and 1000 mg/kg heavy metals. The initial concentrations of Cu^{2+} and Pb^{2+} in soil samples spiked with a load concentration of 500 mg/kg were obtained as 454 mg/kg and 485 mg/kg, respectively. As shown in Fig. 4, the contaminant removal was relatively higher from section 1. Whereas, the precipitates of

Cu²⁺ and Pb²⁺ metal ions were higher in sections 2 & 3. The migration of Cu²⁺ and Pb²⁺ metal ions to the cathode was found to be very low.

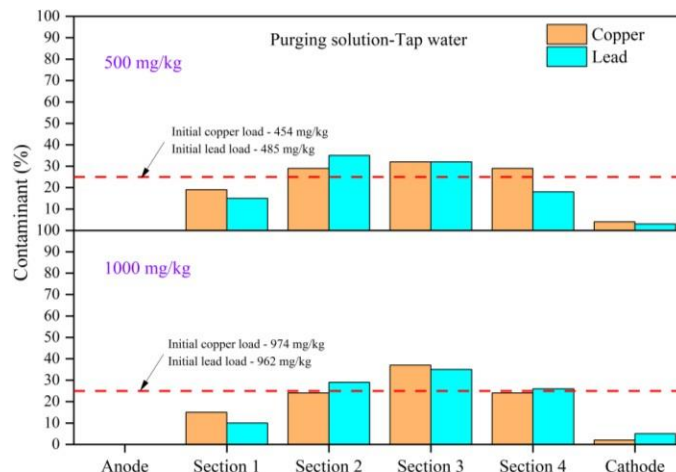


Fig. 4. Contaminant distribution of 1000 mg/kg and 500 mg/kg loaded soils after EKT (tap water in electrode reservoirs)

The initial concentrations of Cu²⁺ and Pb²⁺ in soil samples spiked with a load concentration of 1000 mg/kg were obtained as 974 mg/kg and 962 mg/kg, respectively (Fig. 4). The percentage of Cu²⁺ and Pb²⁺ that migrated towards the cathode from section 1 was more compared to the other sections. This trend was similar to the one obtained for 500 mg/kg loaded soils. Whereas, the residual contaminant concentrations were relatively higher in section 3. The lesser residual contaminant concentrations were observed at the cathode reservoir owing to high pH (12.9) which resulted in precipitation of metal species (Fig. 4). This indicates the need to control the pH at the cathode.

3.2 Treated with 0.05 M EDTA Purging Solution

The soil samples spiked with Cu²⁺ and Pb²⁺ (at two different load concentrations of 1000 mg/kg and 500 mg/kg) were purged with 0.05M EDTA to study its effect in decontaminating the soils after a period of 20 days.

Samples spiked with 500 mg/kg and 1000 mg/kg heavy metals. The initial concentration of Cu²⁺ and Pb²⁺ in soils spiked with a load concentration of 500 mg/kg load was obtained as 449 mg/kg and 471 mg/kg, respectively as indicated in Fig. 5. The treatment with 0.5M EDTA caused higher removal of Cu²⁺ and Pb²⁺ from section 1, whereas the lowest removal rates were observed in section 4. The amount of Cu²⁺ and Pb²⁺ that migrated to the cathode was higher compared to the trend observed for tap water. The results indicated that 25% of Cu²⁺ migrated towards the cathode whereas, only 18% of Pb²⁺ was collected in the cathode reservoir.

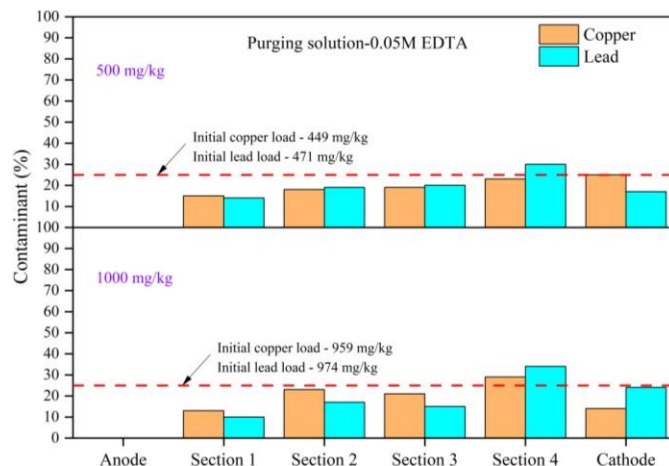


Fig. 5. Contaminant distribution of 1000 mg/kg and 500 mg/kg loaded soils after EKT (0.05 M EDTA as catholyte)

The initial concentration of Cu^{2+} and Pb^{2+} present in the 1000 mg/kg loaded soil samples was 959 mg/kg and 974 mg/kg respectively (Fig. 5). Similar to the trend observed for EKT on 500 mg/kg loaded soil, the residual concentrations were found to be the lowest in section 1 and highest in section 4. Due to the low pH (11.7) at the cathode, the contaminant concentrations were higher at the cathode. From the studies conducted using 0.05M EDTA solution, the 500 mg/kg loaded soil exhibited better Cu^{2+} removal than Pb^{2+} whereas, 1000 mg/kg loaded soil exhibited better Pb^{2+} removal than Cu^{2+} . It is worth noting that the 0.05 M EDTA enhanced EKT exhibited higher removal efficiency compared to tap water.

3.3 Treated with 0.1 M EDTA Purging Solution

The soil samples spiked with Cu^{2+} and Pb^{2+} (at two different load concentrations of 1000 mg/kg and 500 mg/kg) were purged with 0.1M EDTA to study its effect on decontaminating the soils after an interaction period of 20 days.

Samples spiked with 500 mg/kg and 1000 mg/kg heavy metals. The initial concentration of Cu^{2+} and Pb^{2+} present in the 500 mg/kg loaded soil samples was 468 mg/kg and 480 mg/kg respectively. When 0.1 M EDTA solution was used as the purging solution for 500 mg/kg loaded soil, the pH (9.1) at the cathode reduced significantly compared to the soil treated with 0.05 M EDTA solution. The pH reduction at the cathode caused the precipitated ions to dissolve in the pore fluid and migrate towards the cathode. The results indicated the presence of lesser residual contaminants in section 3 (Fig. 6). The removal rates of Cu^{2+} and Pb^{2+} in the cathode reservoir were found to be 40% and 30%, which was higher than treatment with 0.05M EDTA.

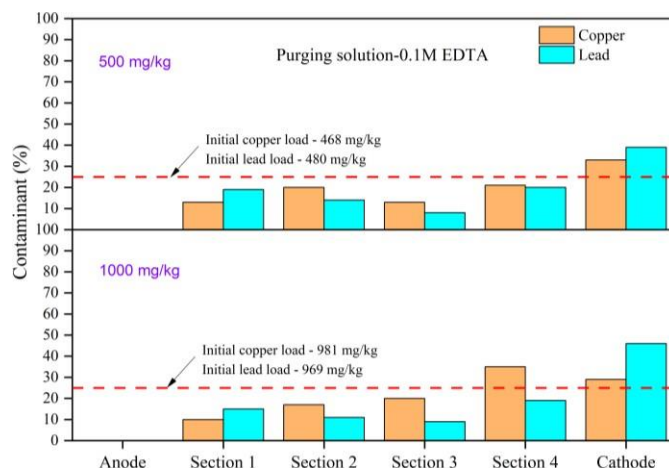


Fig. 6. Contaminant distribution of 1000 mg/kg and 500 mg/kg loaded soils after EKT (0.1 M EDTA as catholyte)

Fig. 6 indicates the initial concentrations of Cu^{2+} and Pb^{2+} in the 1000 mg/kg loaded soils as 981 mg/kg and 969 mg/kg. The lowest residual concentrations for Cu^{2+} and Pb^{2+} were observed in section 1 and section 3 respectively. The removal rates were found to be 48% and 29% for Cu^{2+} and Pb^{2+} in the cathode reservoir. Compared to the 0.05 M EDTA enhanced treatment, the soil treated with 0.1 M EDTA solution exhibited better removal of Cu^{2+} and Pb^{2+} from the soil.

3.4 Treated with 1 M EDTA Purging Solution

The soil samples spiked with Cu^{2+} and Pb^{2+} (at two different load concentrations of 1000 mg/kg and 500 mg/kg) were purged with 1M EDTA to study its effect on decontaminating the soils after a period of 20 days.

Samples spiked with 500 mg/kg and 1000 mg/kg heavy metals. The soil samples contaminated with 500 mg/kg of Cu^{2+} and Pb^{2+} separately, contained initial concentrations of 443 mg/kg and 465 mg/kg respectively, as shown in Fig. 7. The residual concentrations of Cu^{2+} and Pb^{2+} were found to be the least in section 1. The Pb^{2+} contaminant concentration of 38% was observed in section 3 whereas, about 48% of Cu^{2+} metal ions were collected in the cathode reservoir.

The initial concentration of Cu^{2+} and Pb^{2+} in soil samples spiked with 1000 mg/kg was found to be 975 mg/kg and 954 mg/kg using the ICP-AES test (see Fig. 7). The removal rates for Cu^{2+} and Pb^{2+} at the cathode were obtained as 42% and 19%. The pH was observed as 8.6 which is less compared to the pH of the soil treated with 0.1 M EDTA solution. The reduction in the pH at the cathode facilitated more copper removal. But the lead removal was less compared to the 0.1 M EDTA enhanced treatment. Hence the lead removal was ineffective using 0.1 M EDTA solution. Among all the sections,

the lesser contaminant residuals were accumulated in section 1. Examination of soil samples from sections 2 & 3 indicated that the lead concentration remained the same even after the enhanced treatment.

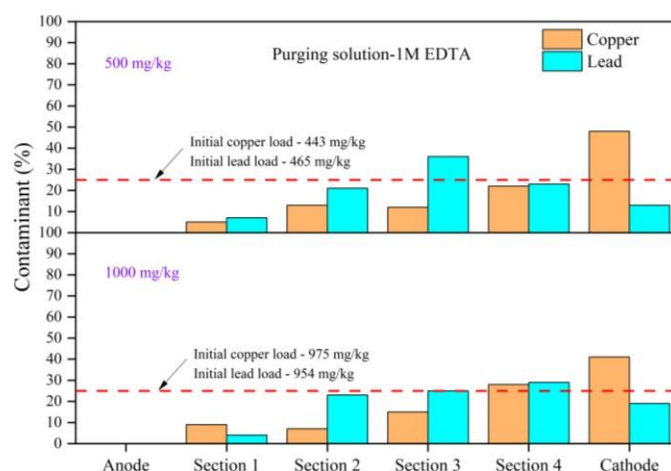


Fig. 7. Contaminant distribution of 1000 mg/kg and 500 mg/kg loaded soils after EKT (1 M EDTA as catholyte)

The Cu^{2+} removal rates with tap water, 0.05 M, 0.1 M, and 1 M EDTA were obtained as 2%, 14%, 29%, and 41% respectively when the soil was spiked with 1000 mg/kg. Similarly, removal rates of 5%, 25%, 33%, and 48% were observed with tap water, 0.05 M, 0.1 M, and 1 M EDTA respectively, when the soil was spiked with 500 mg/kg. It was thus observed that the Cu^{2+} removal rates increased with an increase in EDTA concentration for soils spiked with heavy metal concentrations of 500 mg/kg and 1000 mg/kg.

The Pb^{2+} removal rates with tap water, 0.05 M, 0.1 M, and 1 M EDTA were obtained as 4%, 24%, 46%, and 19% respectively when the soil was spiked with 1000 mg/kg. Similarly, removal rates of 3%, 17%, 39%, and 13% were observed with tap water, 0.05 M, 0.1 M, and 1 M EDTA respectively, when the soil was spiked with 500 mg/kg. Thus, the removal rates of Pb^{2+} increased with an increase in EDTA concentration up to 0.1M, beyond which the removal rate decreased for soils spiked with heavy metal concentrations of 500 mg/kg and 1000 mg/kg.

4 Conclusions

The feasibility of EKT in removing Cu^{2+} and Pb^{2+} from a high plastic clayey soil at different load ratios (500 mg/kg and 1000 mg/kg) with tap water and EDTA as purging solutions has been studied. The following conclusions are drawn:

- The removal efficiencies for Cu^{2+} and Pb^{2+} from soil samples purged with tap water and prepared at two different load ratios of 500 mg/kg and 1000 mg/kg were very low. With an increase in heavy metal load concentration, the removal rates of Cu^{2+} decreased by 50% and increased by 33% for Pb^{2+} .
- When soil samples were purged with 0.05M EDTA solution, the removal efficiencies for Cu^{2+} and Pb^{2+} were higher for 1000 mg/kg compared to 500 mg/kg loaded soils. With an increase in EDTA concentration, the 1000 mg/kg loaded soils exhibited higher removal efficiencies for Cu^{2+} and Pb^{2+} compared to 500 mg/kg loaded soils at 1M EDTA and a similar trend was observed with 0.05M EDTA.
- Compared to tap water, the removal efficiencies for Cu^{2+} and Pb^{2+} increased by 400% for 500 mg/kg and 1000 mg/kg loaded soils, respectively when purged with 0.05M EDTA. However, the relative improvement for Pb^{2+} removal in soil sample spiked with 500 mg/kg was only about 25%.
- At 0.1M EDTA, the removal efficiencies of Cu^{2+} and Pb^{2+} improved by more than 1950% and 375% for 1000 mg/kg loaded soil compared to tap water. Whereas, the removal rates were higher by 1100% and 333% for Cu^{2+} and Pb^{2+} for 500 mg/kg loaded soils.
- With increase in concentration of EDTA solution (1 M), the removal rates of Cu^{2+} were higher than Pb^{2+} owing to the comparatively lower value of pH (8.6) observed at the cathode. However, the Pb^{2+} removal rates were higher than Cu^{2+} at 0.1 M EDTA at a pH of 9.1.

Thus, it can be concluded that the remediation of soil contaminated with heavy metals such as Cu^{2+} and Pb^{2+} at load concentrations of 500 mg/kg and 1000 mg/kg can be effectively treated with EDTA enhanced technique compared to unenhanced technique. The EDTA solution as a purging agent increases the efficiency of EKT.

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