

Enhanced Electrokinetic Remediation of Zinc Contaminated Soil by Changeover of Composite Electrolyte

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Abstract: The combination of electrolytes 0.1 M potassium chloride (KCl) and 0.1 M citric acid (CA) was investigated in a bench scale reactor for removal of zinc (Zn) from soil using electrokinetic remediation (EKR). The study was conducted over a period of 144 hours for different experimental set-ups. When KCl was used as a single electrolyte, 49.38 % removal of zinc was obtained with the changeover of electrolyte at every 24 hours, as compared to 12.5 %, when the reactor was operated as batch reactor without changeover. The use of composite electrolyte in cathode compartment and single electrolyte in anode compartment showed 79.06 % removal, whereas the use of composite electrolyte in both anode and cathode compartment gave 21.74 % removal.

Key words: Electrokinetic remediation, composite electrolyte, removal of zinc.

Introduction

In the past few years, the occurrence of heavy metal contaminated sites has surged as a consequence of discharges of high metal waste from fast growing industrial areas, mining activities, land application of fertilisers and pesticides, wastewater irrigation, spillage of petrochemicals and atmospheric deposition of coal combustion residues (Wuana and Okieimen, 2011). Heavy metals are hazardous to plants and organisms through bioaccumulation and biomagnification in the food chain. Once the soil suffers from heavy metal contamination, it is difficult but not impossible to be remediated (Su et al., 2014; Wuana and Okieimen, 2011).

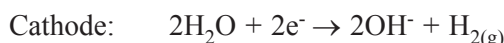
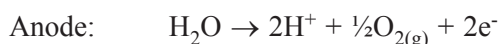
Zinc (Zn) is a heavy metal considered a transition metal, having an atomic number of 30. Naturally, Zn found in crustal rocks is about 70-100 mg kg⁻¹ but it is rising unnaturally due to anthropogenic

activities (Wuana and Okieimen, 2011). The amount of zinc present varies extensively from agricultural to contaminated soils. (Asati et al., 2016; Long et al., 2003). Zn can interrupt the activity of microorganisms and earthworms related to the decomposition process of organic matter; another impact is the contamination of ground water due to the leaching of heavy metals from contaminated soil. Therefore, efforts are required to remediate contaminated sites considering the most relevant technologies for contaminant species, such as soil replacement, soil isolation, soil washing, solidification/stabilisation, soil vitrification, phytoremediation, electrokinetic remediation (EKR), etc. (Khalid et al., 2017).

EKR is an in-situ type of process with reasonable demand for energy. No excavation and replacement of soil are needed for the treatment of soil and it is effective for low permeability soils too. The main

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aim of EKR is the migration of contaminants under the influence of the electric field. Three predominant phenomena responsible for contaminant migration are summarised as electromigration, the accumulation of ions or complexes of ions towards the opposite charge of electrodes, electroosmosis, the movement of soil pore fluid from anode to cathode, and electrophoresis, the transport of colloids with the bounded contaminants in the imposed electric field. In EKR, various eco-friendly chelating or complex agents are generally used as an electrolyte, e.g., sodium chloride (NaCl), potassium chloride (KCl), EDTA, citric acid (CA), etc. These agents serve as a conducting medium and enhance the removal efficiency of metals from the soil by sorption, precipitation, and dissolution reactions. The most important electron transfer reactions at the electrodes during the electrokinetic (EK) process due to electrolysis of water are as follow:



The H^+ ions produced decrease the pH near the anode and at the same time OH^- ions generated at the cathode increase the pH near the cathode. This will form the acid front and alkaline front at the anode-side and cathode-side of the soil, respectively. Soil is divided into two zones, acidic zone and alkaline zone, with intense pH change. In acidic conditions, metal desorbs from soil and solubilizes and in alkaline condition adsorbs to soil particles or makes precipitates with the hydroxide ion. These H^+ and OH^- ions are at some point in soil combined in water form, which is then transported towards cathode through electroosmosis. Oxygen and hydrogen gas formation occurs at anode and cathode respectively during the EK process. These electrode reactions are modified by the use of electrolytes. Depolarisation or modification of electrode reactions enhances the transport of metal ions by preventing premature precipitation as hydroxides. (Acar et al., 1995). When KCl and CA are used as electrolytes, K^+ , Cl^- , H^+ and citrate ions are formed (Zhang et al., 2018). While CA converted to citrate and in combination with metals will form soluble metal citrate complex. This helps to reduce metal concentration from soil (Zhang et al., 2018).

The efficiency of EKR for the removal of contaminants depends on specific conditions such as type of soil, type of contaminant, pH of the soil, organic content and temperature. There are some limitations to EKR, such as interference of the ionic impurities, corrosion, reduced efficiency of the EK process due to gas bubbles of H_2

and O_2 produced at the surface of the electrode during the treatment (Virukyte et al., 2002). Leaching of useful elements from soil or accumulation of elements from the electrolysis of electrolyte during EKR is also a constraint for use of soil after EKR.

Materials and Methods

Collection of Soil Sample

The soil used for experimental work was sampled from an industrial area, G.I.D.C. Ranoli, Vadodara, at Latitude: 22.40 °N and Longitude: 73.14 °E.

Colour : Brownish

Organic Content : 0.3 %

Specific gravity : 2.173

Zn concentration : 46 mg/kg

K concentration : 1057 mg/kg

Preparation of Soil Spiked with Zn

Synthetic Zn contaminated soil was prepared in the laboratory. Primarily, the soil was dried in a hot air oven at 150°C for 2 hours. The dried soil was then spiked with $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ solution of 500 mg/L Zn concentration (Li, 1996; Selvi and Rajshekhar, 2018). The soil and Zn solution was stirred vigorously once a day for five days. After five days, the excess solution was drained with a cloth filter. The spiked soil was then dried in a hot air oven at 150°C for 5 to 6 hours. The dried soil sample was acid digested and analysed for initial Zn concentration using atomic absorption spectrometry.

Experimental Set up

The experimental set up is demonstrated in Figure 1. Acrylic reactor with internal dimensions of 220 mm, 70 mm, and 80 mm (Zhang et al., 2018; Zhou et al., 2018) was divided into three compartments: the soil compartment (length 120 mm, width 70 mm, and depth 50 mm) and two electrolyte chambers (length 50 mm, width 70 mm, and height 50 mm each). Graphite sheets

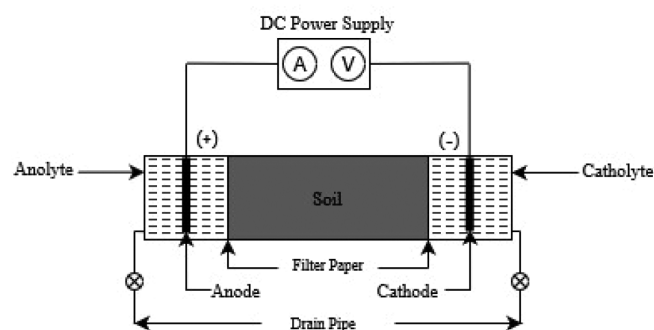


Figure 1: A schematic diagram of experimental set up.

having dimensions of 45 mm × 70 mm and thickness of 5 mm, used as working electrodes. The current was applied by a digital DC power supply unit. Soil and electrolyte chambers were separated using Whatman filter paper (Whatman-42) (Selvi and Rajshekhar, 2018; Zhou et al., 2018).

About 725 g of spiked soil mixed with 200 ml distilled water placed in the soil compartment. The voltage of 33 V was applied by the DC power supply unit. All experiments were carried out under a voltage gradient of 2.4 V/cm for the period of 144 hours (6 days) (Zhou et al., 2018). At the end of 24 hours, the electrolyte was replaced with a new batch of the same electrolyte.

Potassium chloride (KCl) 0.1 M solution was used as an electrolyte to provide conductance to enhance the EK process and also to have increased potassium concentration in soil to work as fertiliser (Zhou et al., 2018). KCl was used in combination with citric acid (CA). CA is an eco-friendly chelating agent, and generates a large amount of H⁺ ions on electrolysis, reduces the pH of the soil compartment and helps in the dissolution of Zn ions (Zhang et al., 2018). It has a higher electrical conductivity than CA but the ability of dissolution of Zn in KCl is weaker than CA. To balance these deficiencies, a mixture of 0.1 M KCl and 0.1 M CA was used as anolyte and catholyte. Experimental conditions are listed in Table 1.

To assess migration or accumulation of Zn in soil, the residual amount of Zn was analysed in the soil at five sections at every 3 cm distance from anode to cathode. The pH of the soil was also measured at each section. To evaluate the overall removal efficiency, at the end of 144th hour, the soil was removed from the reactor, mixed homogeneously and analysed for zinc concentration.

Observations taken for removal of Zn such as current intensity, pH, migration and extraction of Zn in the soil and the electrolyte are discussed in the following sections. The effect of KCl as an electrolyte on soil was assessed by determining potassium (K) and chloride (Cl) concentrations in the homogeneous mixture of soil at the end of the EKR process.

Power Consumption

The formula used for the determination of total power consumption during EKR is presented as below:

$$P = \frac{1}{Mc} \int_0^t \frac{V * I}{1000}$$

where, P = power consumption (kWh/g)

V = Voltage (V)

I = Average electric current of the soil (A)

Mc = Mass of contaminant removed (g)

Analytical Methods

Zn and K concentrations were determined using atomic absorption spectrophotometry (ECIL make atomic absorption spectrophotometer AAS4141), pH and chloride concentration was determined using digital pH meter and titrimetric method respectively (APHA, 2017).

Results and Discussion

Change in Current Intensity

Figure 2 shows variations in current intensity through the soil over a period of 144 hours for experimental conditions A, B, C and D as given in Table 1. For runs A, B, C and D current intensity through soil is observed at peaks of 333 mA, 430 mA, 487 mA and 648 mA, respectively, at about 2 hours from the start of the run. Run A has a continuous decreasing slope whereas run B, C and D show peaks due to the changeover of electrolyte every 24 hours. At the end of 144 hours, the values of current intensity in the runs A, B, C and D are 19 mA, 19 mA, 26 mA and 48 mA. Initial highest peaks are due to mobilisation of ionic contents of soil including electromigration of Zn under electric field. With further passage of time, current intensity decreases as most ions have migrated to the opposite electrodes and OH⁻ ions at the cathode region causing Zn to precipitate, which hindered further migration and increased the resistance. Moreover, electromigration of H⁺ and OH⁻ ions towards cathode and anode,

Table 1: Experimental conditions for EKR

<i>Runs</i>	<i>Anolyte</i>	<i>Catholyte</i>	<i>Initial amount of Zn in soil (mg)</i>	<i>Changeover of electrolyte</i>
A	0.1 M KCl	0.1M KCl	377	Without
B	0.1 M KCl	0.1M KCl	487	With
C	0.1M KCl+ 0.1M CA	0.1M KCl + 0.1 M CA	489	With
D	0.1 M KCl	0.1 M KCl + 0.1 M CA	425	With

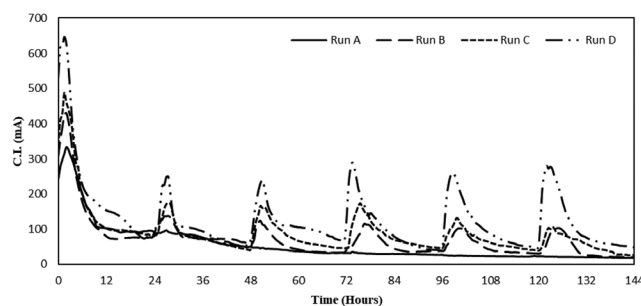


Figure 2: Current intensity across soil during EKR.

respectively, neutralise each other and form water. This decreases the conductance in the EKR system. Higher peaks at every 24 hours in run D, compared to run A, B and C demonstrates higher mobility of ions in run D, which further results in greater removal efficiency.

Changes in Soil pH

Figure 3 demonstrates changes in pH of the soil at five soil sections from anode front towards cathode front after the duration of 144 hours of EKR. The initial pH of soil ranges from 5.9 to 7.4. The soil pH varied across the soil owing to the reasons stated in the above section. For run A and run B, at the anode front, the pH of the soil was 2.3 and 2.9, which gradually increased to 9.9 and 10.7 at the cathode front, respectively. These data direct towards inadequate control of pH as we proceed towards the cathode region when KCl was used as a single electrolyte. In run C, the soil pH of 2.5 at anode front gradually increased to 3.9 at the cathode front and for run D soil pH varies from 2.5 to 3.8 from anode front to cathode front. For run C, the mixture of 0.1 M CA and 0.1 M KCl (composite electrolyte) was used as anolyte and catholyte as well, whereas in run D KCl was used as a single anolyte and composite electrolyte was used as catholyte. CA with KCl in the cathode compartment indicates better pH control in the cathode region and inhibited precipitation to have enhanced removal efficiency. However, no additional effect on

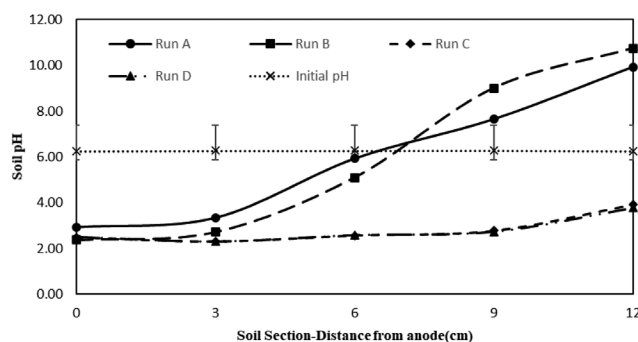


Figure 3: Effect on pH of soil.

soil pH, due to the use of composite electrolyte in both electrode compartments was observed.

Removal Efficiency of Zn from Soil

Figure 4 illustrates the initial and residual amount of Zn over the period of 144 hours at different soil sections. Initial Zn in soil varies from 377 mg to 489.1 mg.

The formation of H^+ ions at anode and their migration towards the cathode decreases the pH of the soil in the reactor and dissolves Zn ions from the soil. The presence of a higher concentration of OH^- ions near the cathode stalled the dissolution of Zn from contaminated soil and make an impermeable wall for the Zn to pass through. Zn dissolved from the soil was transferred to anode and cathode by electroosmosis and electromigration. The combined effect of these two phenomena leads to the removal of zinc from spiked soil. OH^- ions at the cathode front and H^+ ions at the anode front are essential parameters for electromigration and removal of Zn metal.

To assess percentage removal, at the end of each run, soil from the reactor was removed, homogeneously mixed and zinc concentration was determined.

In run A, B, C and D, the removal of 12.5%, 49.38%, 21.74% and 79.06 % were obtained, respectively. When electrolytes were refreshed every 24 hours in run B, higher decontamination of zinc from soil was observed as compared to run A, in which electrolytes were not refreshed. Employment of composite electrolyte as catholyte in Run D enhances the removal of zinc to 79.06 %.

Changes in the Amount of Zn in Electrolyte

Figure 5 demonstrates the trend for extraction of Zn in anolyte and catholyte for run B, C and D.

In run D, the amount of zinc obtained in catholyte is more than run C and hence run D exhibits 79.06 % removal of zinc from soil. In run C when composite

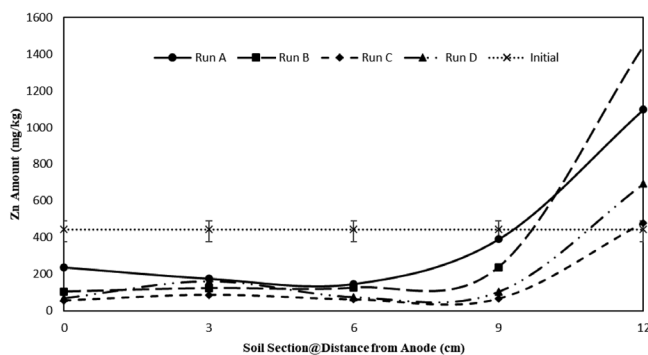


Figure 4: Electromigration of Zn in soil.

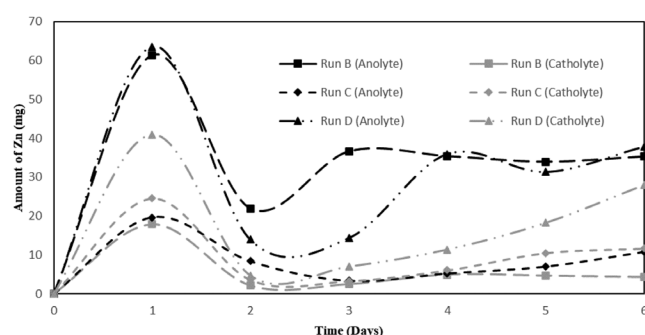


Figure 5: Extracted amount of Zn in electrolyte.

electrolyte was used in both anode and cathode compartments there is the possibility of unexpected reactions leading to lower conductivity in the soil compartment; thus, the zinc removal from soil was only 21.74 %. Mass balance for the amount of zinc in soil and electrolyte is analysed and shown in Table 2.

Effect on Potassium and Chloride Content of Soil

Potassium (K) and chloride (Cl) have a significant impact on the fertility of the soil. EKR basically affects metals, organic and inorganic contents of the soil. Potassium and chloride both are essential for crop growth (White and Broadley, 2001). Initial concentrations of K and Cl in soil were 1057 mg/kg and 154 mg/kg, respectively.

Results for the analyses of K and Cl contents in soil at the end of the EKR process for run B, run C and run D are displayed in Figure 6.

Run B had KCl as both anolyte and catholyte; therefore, it was seen that the dissociation of KCl increases the K and Cl concentration of soil by 5% and 83%. Run C in which CA was one of the components

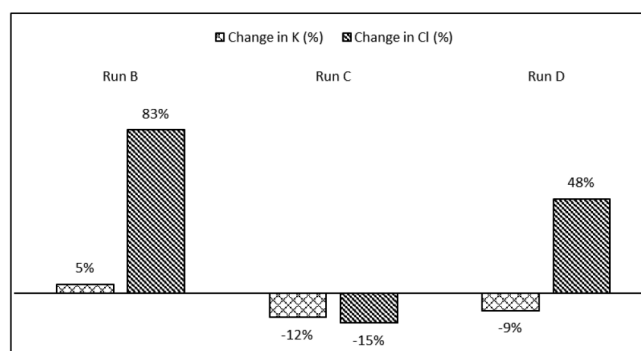


Figure 6: Change in K and Cl from soil after EKR.

of composite electrolytes, leaching of K was observed and the K and Cl contents decreased below initial value; while for run D, where CA was present only in catholyte, a 48 % increase in Cl content is observed.

Power Consumption

Table 3 illustrates total power consumption for runs B, C and D over a period of 144 hours. Table 4 shows power consumption reported in similar studies.

Comparison of power consumption with similar studies as shown in Table 4 indicates similar trends.

Conclusion

Among various combinations of electrolytes used in experiments, 0.1 M KCl as anolyte and composite electrolyte of 0.1 M KCl and 0.1 M CA as catholyte imparted maximum removal of 79% of Zn from the soil over a time period of 144 hours, with the power consumption of 1.68 kWh/g. With this combination of

Table 2: Mass balance for Zn

Run	Initial amount of Zn in soil (mg)	Residual amount of Zn in soil (mg)	Cumulative amount of Zn in electrolytes (mg)	Zn removal (%)	Mass balance accuracy (%)
A	377	329.8	48.34	12.52	102.0
B	487.2	246.6	261.0	49.38	108.0
C	489.13	382.77	114.0	21.74	107.0
D	424.7	88.94	307.2	79.1	92.0

Table 3: Power consumption

Run	Total power consumption (kWh)	Power consumption kWh/g
B	0.35	1.33
C	0.43	3.79
D	0.60	1.68

anolyte and catholyte, a decrease in the K content of the soil is a limitation. As K is a nutrient for plant growth, remediated soil will have this constraint.

Acknowledgements

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Table 4: Similar studies from literature

Details	Total power consumption (kWh)	Power Consumption (kWh/g)
Yingjie Zhang et al. (2017) Electrolyte-0.1 M NaCl + 0.1 M C.A. 30 V power supply for 20 days	3.458 kWh for removal of 80.37 % Pb and 90.86 % Cd	-
Yang et al. (2011), Electrolyte-Deionized water, 25 V and 35 V power supply for 70 hours, - respectively.	-	68.68 and 188.72 for removal of 25.4% and 31.7% Cd

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