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Laboratory-Scale Electro-remediation of Toxic Metals from Soil Collected from Kala Shah Kaku Industrial Zone, Punjab, Pakistan

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Abstract

Unplanned industrial waste disposal, alongside wastewater-irrigated agriculture, has caused severe soil pollution with toxic metals in Pakistan's industrial zones. This study evaluates the laboratory-scale technical viability and efficacy of a locally fabricated Electrokinetic Remediation (EKR) apparatus for cleanup of chromium (Cr) and zinc (Zn) from silty clay soil collected from the Kala Shah Kaku industrial/agricultural zone in Punjab, Pakistan. The test soil was artificially contaminated with Cr and Zn and placed in an electrolytic cell packed between anolyte and catholyte compartments. Electrochemical washing was performed using sulphuric acid (0.05 M and 0.5 M H_2SO_4) as the electrolyte under a constant DC voltage of 15V for 83.25 hours (~3.5 days). Soil characterization and metal analyses before and after treatment were conducted using pH and Electrical Conductivity (EC) probes, X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), and Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES). The initial soil, anolyte, and catholyte pH levels were 6.61, 1.25, and 1.08, respectively. During remediation, pH values fluctuated before stabilizing, while EC values decreased in both electrolyte compartments. Mineralogical analysis identified quartz (71%), albite (8%), muscovite (13%), and chamosite (6%) as the major mineral constituents of the soil. SEM imaging showed that EKR disrupted soil aggregates, transitioning the structure from compacted particles to a more porous, open network. EKR achieved a high removal efficiency of 58.09% for Cr, 1.86% for Zn, and 2.25% for Fe. The findings confirm EKR as a viable, low-cost technique for remediating Cr from silty clay soils, though optimizing the electrolyte composition remains necessary for broader multi-metal extraction. Some recommendations for potential, cost-effective, and environmentally friendly EKR of toxic/heavy metal-contaminated soils are also discussed for future studies.

Keywords: Electrokinetic remediation; electrokinetic washing; electromigration; waste soils; soil pollution; toxic metals

1. Introduction

The indiscriminate disposal of untreated industrial effluents and solid wastes onto land and into watercourses has been exerting substantial pressure on the soil environment, exceeding its intrinsic buffering and attenuation potential and promoting the persistent accumulation of heavy metals and anthropogenic organic contaminants within the soil ecosystem. This practice is resulting in the deposition of diverse hazardous substances in the soil matrix that adversely affect soil health and the living biota associated with it (Kaur et al., 2010; Hu et al., 2013). Unlike natural lithogenic weathering, which introduces elements gradually, the chemical, metallurgical, textile, paint, tanning, and agriculture-related industries serve as localized point sources that heavily burden the environment with persistent anthropogenic contaminants (Oladimeji et al., 2024). In the rapidly industrializing landscape of Kala Shah Kaku, the most pervasive elements in the industrial and domestic sewage are those released by the leather tanning industry, synthetic fiber production industry, metal plating and polishing industry, galvanizing and steel industry, battery industry, chemical manufacturing complexes, steel re-rolling mills, and agriculture industry-related chemicals, dye and wood preservation industry, etc., which are identified as Cr and Zn, which may impact biotic life and human health. Chromium (Cr) is mainly added in to

the environment from multiple sources. For example, Cr is used for leather tanning, leather processing, paints, stainless steel, and pigment industries. The leather tanning industry uses basic chromium sulfate for chrome tanning of leather to make leather soft and durable. Ferrochrome is used in stainless steel and metallurgy-related industries to make stainless steel. The paints and pigments industries, like Diamond Paints, Berger, and Nippon, use Cr based chemicals such as Chrome Yellow and Chrome Green pigments and as anti-corrosion primers. Cr (VI) is used for electroplating and surface treatment such as hard chrome plating on auto parts and sanitary fittings. Likewise, Zn is also added to the environment from multiple sources. For example, the galvanizing and steel industry uses Zn-based chemicals such as zinc metal, ZnO, and $ZnSO_4$ for anti-rust coating for steel sheets, pipes, and G.I. sheets; in fertilizer as a plant nutrient; in wheat/rice to fight crop deficiency; and in anodes of power sources/batteries for anti-corrosion. Similarly, sodium dichromate is used for dyes, wood preservatives, and in catalysts. Zn deficiency is common in Pakistani soils (Rehman et al., 2020). Therefore, in the fertilizer and agriculture industry, such as Engro, Fauji Fertilizer, and Fatima Fertilizer, $ZnSO_4$ is used as a micronutrient fertilizer. In the Pakistani rubber and tire Industry (such as General Tire, Panther Tyres, Service Industries), ZnO is used for vulcanization. In textile and dyeing, $ZnCl_2$ is used as a mordant and catalyst. In the pharma and cosmetics industry, ZnO and $ZnSO_4$ are used in creams, sunscreens, supplements, and syrups. The paints industry (such as Nippon Paint, Diamond Paints, Berger) uses ZnO and Zinc phosphate as anti-corrosive primers. The improper disposal of solid wastes, domestic sewage, and effluents by these chemical industries onto the ground and into watercourses has led to an alarming and hazardous accumulation of toxic/heavy metals like Cr and Zn. These contaminants are widely transported through environmental systems, causing substantial degradation of aquatic, ecological, hydrogeological, biotic, and human health-related systems. Their continuous exposure to soil, aquatic, and groundwater environments is therefore becoming a serious threat to the living biota and is a primary cause of various waterborne illnesses. It has been reported that in these areas, more than 70% of the population is facing stomach and liver diseases; dysentery is 60%; while stomach diseases are 85% (Daud et al., 2017; Waseem et al., 2014).

In view of the above, the polluted soil and groundwater environment in the industrial zone of Kala Shah Kaku (KSK) calls for urgent remedial measures. Traditional soil cleanup methods, such as water or solvent flushing and bioremediation, are increasingly seen as costly and inefficient for treating on-site domestic and industrial pollution (Ferraro et al., 2016; Sharma et al., 2018; Michael-Igolima et al., 2022). For the past 60 years, electrokinetic remediation has been widely studied, updated, and applied to clean up contaminated waste soils, especially clayey soils containing inorganic toxic metal ions (Acar and Alshawabkeh, 1993; Weng and Yuan, 2001; Virkutyte, 2002; Srivastava, 2007, Hashmi et al., 2017; Sikander et al., 2021, Khalid et al., 2007; 2008; 2025; Song et al., 2020; Jameel et al., 2024), organic contaminants (Reddy and Parupudi, 1997; Vengris et al., 2001), and radionuclides such as thorium and radium (Kim et al., 2003; Sivapullaiah et al., 2015). Researchers have used different electrolyte recipes such as HCl (Probstein and Hicks, 1993); Sulphuric Acid (Alshawabkeh and Acar, 1996; Virkutyte et al., 2002); Nitric Acid (Bahemmat and Farahbakhsh, 2015); Citric Acid (Cameselle et al., 2021; Khalid et al., 2025) for electrokinetic remediation depending upon the nature of target pollutants.

For effective electrochemical decontamination, pollutants must either exist as soluble ions or molecules in the soil-water system, or be transitioned into the aqueous phase using appropriate chelating agents to ensure their mobility across the electric field (Iannelli et al., 2015). The EKR technique involves three electrokinetic processes, including: (i) electromigration, (ii) electro-osmosis, and (iii) electrophoresis (Oyanader-Rivera, 2004). The movement of charged ions and ion complexes toward oppositely charged electrodes under the influence of an electric field is termed electromigration (Cauwenberghe, 1997). Electromigration,

also called ionic migration, constitutes the transport of charged ions and ionic complexes toward the oppositely charged electrodes (anode or cathode). As the primary driver for the removal of heavy metal cations and inorganic anions, the rate of electromigration is governed by the ionic mobility and the local potential gradient within the soil matrix. Electro-osmosis refers to the bulk movement of pore fluid relative to a stationary, charged soil surface under the influence of an electric field. This mechanism is particularly critical in fine-grained soils, such as clays, resulting in a stable flow velocity that is independent of pore size, thereby overcoming the limitations of hydraulic conductivity. Electrophoresis, on the other hand, involves the translocation of charged colloidal particles, including organic matter, microorganisms, and fine clay clusters, across the electric field. While typically contributing less to the total mass transport than electro-migration in compacted soils, it serves as a pivotal mechanism for the mobilization of contaminants associated with suspended particulate matter.

There is no scientific data available on the decontamination of soil and/or groundwater environment of KSK industrial zone. Therefore, this paper describes preliminary results of the laboratory-scale electrokinetic remediation (EKR) of aqueous-phase toxic/heavy metal species such as Cr, Fe, Ni, Zn, Mn, Co, and Pb from silty clay soils collected near agricultural fields adjacent to industrial sites in Kala Shah Kaku (Punjab Province, Pakistan) using low DC voltage. The key objectives of the present investigations are as follows:

(i) To assess the performance and feasibility of a custom-built laboratory EKR system in removing aqueous Cr, Zn, and other metals from Kala Shah Kaku silty clay soils affected by municipal, industrial, and agricultural waste;
(ii) To evaluate potential operational failures during EKR experiments (e.g., cell leaks, power interruptions, electrolyte build-up, soil cracking, and mineral precipitation) and assess their impact on overall efficiency;
(iii) To quantify EKR efficiencies of Cr and Zn along with other naturally present toxic/heavy metals in silty clay soils of Kala Shah Kaku industrial/agricultural zone.

2. Materials and methods

2.1. Soil Sampling and Preparation for EKR Experimentation

Coarse silty clay soil was collected from the site adjacent to the Daik Nullah (31.724583° N, 74.268917° E) in the Kala Shah Kaku (KSK) Industrial Estate, a prominent industrial corridor along the Grand Trunk (G.T.) Road, Punjab Province, Pakistan (Fig. 1). The KSK sampling zone is characterized by a high density of diverse manufacturing units, including chemical plants, tanneries, and small-scale textile mills, along with major industries like Ravi Chemical Complex, Kohi Noor Oil Mills, and Itehad Chemical Industry, which contribute significantly to the local economy and industrial development. As a result, a diverse range of anthropogenic contaminants is generated, posing a substantial threat to the ecological stability and environmental sustainability of the local soil and groundwater systems (Anwar and Mazhar, 2024).



Fig. 1. Influx of domestic and industrial effluents in Daik Nullah adjacent to the sampling site in Kala Shah Kaku Industrial Estate.

A representative bulk sample (about 80 to 100 kg) of Kala Shah Kaku (KSK) soil was collected from a non-cultivated location between Daik Nullah and nearby agricultural fields. The collected soil was air-dried for a period of one week and manually cleaned of roots, shoots, shells, and pebbles. It was then ground, well mixed, and sieved using a mesh (size 180 µm) to attain a fine-grained soil. The so prepared soil fractions were packed in polyethylene bags and stored for subsequent laboratory physico-chemical, mineralogical phase analysis, SEM analysis, and EKR experimentation with special reference to toxic elements. The prepared KSK soil was spiked with Cr (IV) and Zn by mixing 1.5 kg of soil with a solution of 2.5461 g of potassium dichromate and 1.0007 g of zinc acetate dihydrate in one liter of deionized water. Therefore, spiked soil had Cr and Zn concentrations of 534.0 mg/l and 244.5 mg/l, respectively. Emphasis was mainly given to the analysis and EKR of Cr and Zn in the presence of other allied elements in KSK soil such as Cr, Fe, Ni, Mn, Co, Pb, and Sr.

2.2. Soil Analysis

The prepared soil samples were subjected to physico-chemical, mineralogical phase analysis, and morphological analysis before and after the EKR process. The pH and EC of soil and electrolytes were determined using a pH meter (Model: 3070 ELE and Model: PP-201 K) and an electrical conductivity meter (Model: WTW–LF 95), respectively. The morphological properties of the KSK test soil before and after the EKR experiment were determined using a scanning electron microscope (SEM, Model: Leo 440I). The Mineralogical Phase analysis was performed using X-ray Diffractometry (XRD, DMAX IIIC of M/S Rigaku Corporation, Japan). Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES; iCAP 6500 Thermo Fisher) was used to measure metal concentrations (Cr, Fe, Ni, Zn, Mn, Co, Pb, and Sr) in the soil before and after the EKR process, as well as in anolyte and catholyte aliquots collected during treatment.

2.3. Electrokinetic Remediation Apparatus

Figure 2 highlights the locally designed and fabricated experimental EKR set-up used in the present investigations. The detailed schematics and design of the EKR apparatus are given elsewhere (Khalid et al., 2007; 2008; 2025).

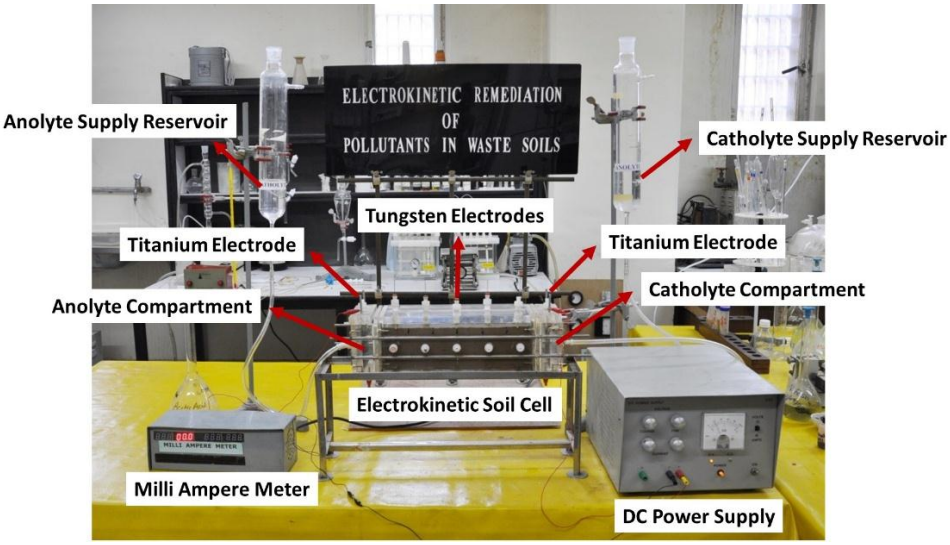

Fig. 2. Locally designed and fabricated experimental EKR apparatus.

2.4. The EKR Procedure

Well homogenized Cr and Zn-spiked moist KSK test soil (weight =1.5 kg) was loaded into the electrokinetic soil cell. The soil cell was gently shaken and vibrated to remove the trapped air within the soil particles and to settle the soil particles. Sulphuric acid (H_2SO_4) was used to prepare the electrolyte for the EKR experiment. About 350 mL of 0.05 M H_2SO_4 was used as electrolyte for the anolyte compartment, while 350 mL of 0.5 M H_2SO_4 was used as electrolyte for the catholyte compartment (Reddy and Chinthamreddy, 2003). The same concentrations of H_2SO_4-based electrolytes

were used to fill the respective electrolyte storage reservoirs attached to the anolyte and catholyte compartments. To ensure ionic transport occurred solely through the electrokinetic mechanism, a zero hydraulic head was maintained across the soil cell. This was achieved by aligning the liquid levels in the anolyte and catholyte compartments with the soil bed. When necessary, the acidic fluid level in the anolyte compartment was replenished using a 0.05 M H_2SO_4 solution from the storage reservoir. The electrokinetic remediation (EKR) process was initiated by applying a constant 15 V DC voltage (equivalent to 1 V cm-1) across the soil compartment for 83.25 hours. To simulate the natural hydrogeological conditions of the contaminated site, no pH control was applied during the test. Throughout the experiment, the pH and electrical conductivity (EC) of both electrolytes were monitored at regular intervals, alongside the system current and voltages across the active and dummy electrodes (D1–D5) (Khalid et al., 2025).

3. Results and discussion

3.1. Characterization of KSK Test Soil

Table 1 summarizes the predominant mineralogical phases of the KSK test soil used in the EKR experiment. The mineralogical phase analysis shows that quartz (71%) and albite (8%) constitute the main sandy or silty fractions, whereas muscovite (13%) and chamosite (6%) constitute the dominant clay minerals in the KSK soil. However, buffering minerals like calcite are not identified in XRD analysis. As such, the KSK soil may be termed coarse silty clay soil.

Table 2 summarizes the fundamental physicochemical properties of the KSK test soil used in the EKR experiment.

Table 1: Mineralogical phases analysis of experimental KSK soil

Mineralogical Phases Analysis	**Values (**% of bulk soil)
Quartz (SiO_2)	71%
Albite ($NaAlSi_3O_8$)	8%
Chamosite ($(Fe,Al,Mg,Mn)_6(Si,Al)_4O_{10}(OH)_8$)	6%
Muscovite ($KAl_2(AlSi_3O_{10})(OH)_2$)	13%

The analysis reveals that the KSK soil is slightly acidic (pH = 6.61), which might be due to the impact of flooding/recharge of acidic sewage from the nearby industries located around the soil sampling site. The lower EC value of 54.5 µS/cm of the KSK test soil may be attributed to the insolubility of metallic precipitates within the soil matrix or the significant leaching of soluble salts due to regional drainage patterns. Similarly, the bulk density of the test soil is 1.41 g/cm^3, while its specific gravity is 2.11, which is quite low and is likely attributable to the high organic content or the presence of lightweight industrial by-products within the soil matrix at the KSK site. Similarly, the moisture content of the test soil is 7.52%. The KSK test soil seems to have porosity values (49.4%) on the higher side of soil porosity, thereby reflecting the presence of large inter-granular spaces in the coarse silty clay soil, which are also quite visible in the SEM images of the KSK test soil (please refer to section 3.2). Notably, the acidic buffering capacity of the KSK test soil is also low (0.08 mM/g), and this low buffering potential of the test soil indicates an exhaustion of carbonate content and a low cation exchange capacity (CEC) within the soil's mineral framework, which may accelerate the propagation of the acid front during the EKR treatment.

Table 2: Physicochemical characteristics of experimental KSK soil.

Physicochemical Parameters	**Values**
pH	6.61
Moisture Content	7.52 (%)
Bulk Density	1.41 (g/cm^3)
Porosity	49.4 (%)
Specific Gravity	2.11
Organic Content	0.99 (%)
Acidic Buffering	0.08 (mM/g)
Electrical Conductivity	54.5 (µS/cm)

Table 3 shows the heavy/toxic metal concentrations in the experimental KSK soil as determined by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-EOS). Results reveal that Na (271 µg/g), K (4107 µg/g), and Mg (6469 µg/g) are in abundance in the KSK test soil, in addition to

significantly higher contents of Fe (22136 µg/g) and Mn (358 µg/g). The contents of trace metal elements Pb, Ni, Cr, Zn, Li, Co and Cu are 15 µg/g, 34 µg/g, 56 µg/g, 63 µg/g, 22 µg/g, 11 µg/g and 15 µg/g, respectively, and concentrations of these heavy metals in soil are in the order of Mn> Zn > Cr > Ni > Li >Pb, Cu > Co.

3.2. SEM Analysis

Figure 3 shows the SEM images of KSK soil taken at low, medium, and high magnification scales of 1000, 3000, and 5000 before and after the EKR process. It may be observed from SEM images of KSK test soil taken before the EKR process that the soil particles are well connected, with larger grain size, and reveal compacted aggregates with visible signatures of heavy metal deposits in the shape of bright, irregular clusters on the surface of the clay minerals. However, after the EKR process, the SEM micrographs display a cleaned mineral surface and an increase in micro-fissures within the compacted soil. This shows that the electro-remediation has changed the clay morphology, as the soil particles are segregated and the soil porosity seems to have increased in size.

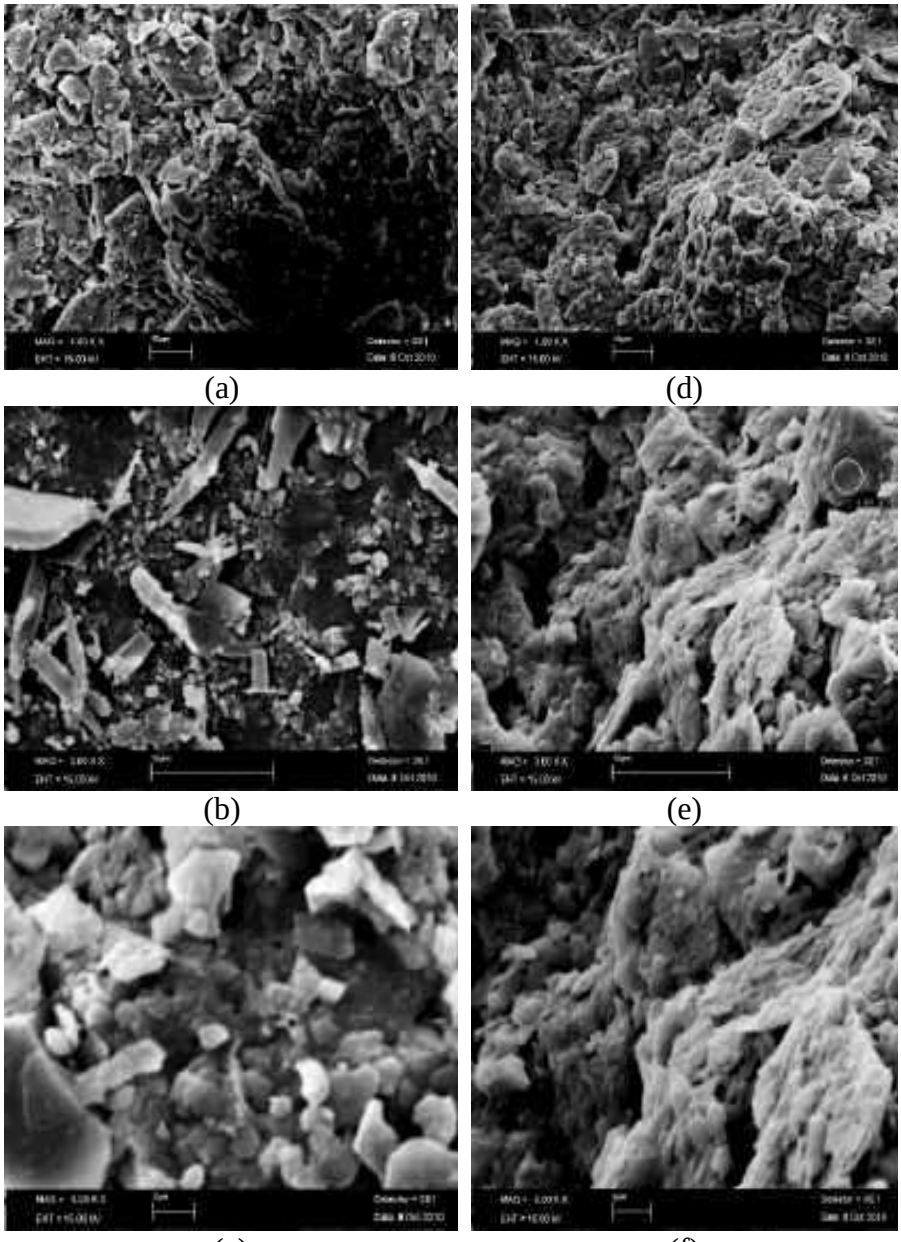

Fig. 3. SEM Micrographs of KSK Test Soil at Low magnification (scale 1000), Medium magnification (scale 3000), and High magnification (scale 5000) before (a, b, c) and after (d, e, f) EKR remediation.

Table 3: Content of metal elements in field collected/spiked KSK soil.

Metal Elements	Field Collected / Spiked KSK Soil Concentrations (µg/g)
Na	271
K	4107
Mn	358
Fe	22136
Cr	56 / (534.0)
Zn	63 / (244.5)
Ni	34
Mg	6469
Li	22
Pb	15
Cu	15
Co	11

The removal of the characteristic bright spots on mineral surfaces provides visual evidence of the solubilization and electromigration of metal ions, signifying efficient remediation of the soil solids. This may be attributed to the electrochemical washing of the KSK soil grains using a relatively strong acid solution (0.05 M H_2SO_4 in the anolyte and 0.5 M H_2SO_4 in the catholyte; pH ~1.25).

This acidic environment promoted clay mineral dissociation, leading to the partial breakdown of soil particles, precipitation of mineral phases at low pH, and subsequent alteration of the soil's morphology. This observation aligns with the findings of other researchers who noted that the EKR process effectively alters the morphology of contaminated medium by mobilizing metal ions away from the solid matrix (Yan et al., 2018; Hadi and Awadh, 2023; Khalid et al., 2025).

3.3. Variations in pH and EC of Electrolytes

Figures 4 and 5 illustrate the temporal variations in the pH and electrical conductivity of the anolyte (0.05 M H_2SO_4) and the catholyte (0.5 M H_2SO_4) during the course of the electrochemical washing process of the KSK test soil. The initial pH values were 1.25 and 1.08, and the initial EC values were 18.3 and 157 mS/cm, in the anolyte and catholyte compartments, respectively. It was observed that over the course of EKR, some precipitates started appearing in both the anolyte and catholyte compartments. As such, the pH value of the anolyte increased slightly by 0.61 units (i.e., from pH 1.25 to pH 1.86) (Fig. 4), while the EC value of the anolyte decreased from 18.3 mS/cm to 13.6 mS/cm (Fig. 5). The increase in pH and decrease in EC of the anolyte may be attributed to the precipitation of mineral salts in the anolyte compartment.

In contrast to the pH behaviour of the anolyte, it may be observed from Fig. 4 that the pH value of the catholyte dropped by 0.37 units (i.e., from pH 1.08 to pH 0.71) over the course of the EKR process. However, Figure 5 shows that, like the anolyte, the EC value of the catholyte also decreased from 157 mS/cm to 86.5 mS/cm over the course of the EKR process, which may be attributed to the precipitation of mineral salts in the catholyte compartment.

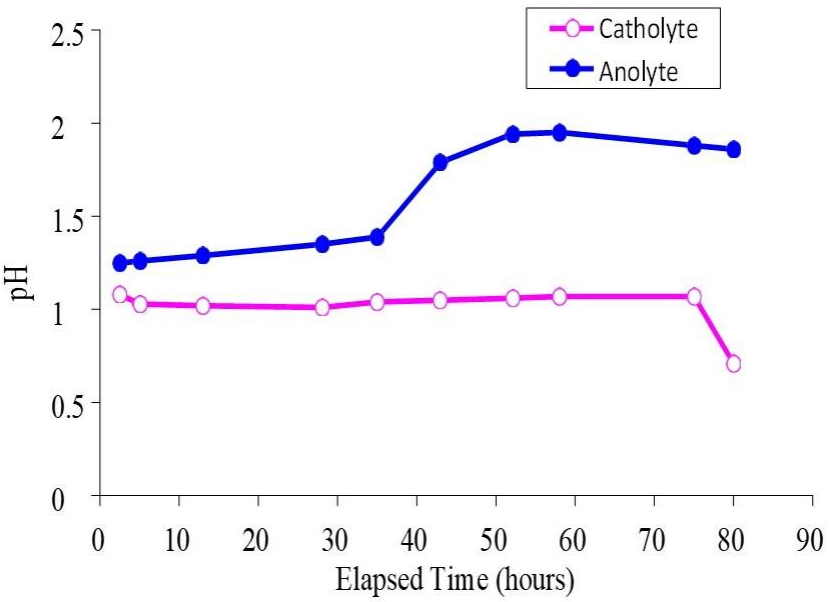

Fig. 4. Temporal variation of pH in anolyte and catholyte during EKR process.

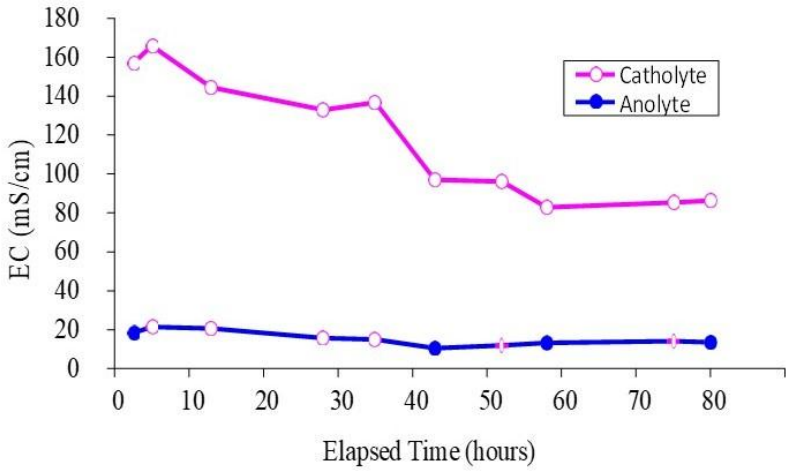

Fig. 5. Temporal variation of EC in anolyte and catholyte during EKR process.

3.4. Contaminant Transport across the Electrokinetic Soil Compartment

Figures 6a and 6b highlight the distinct electromigration patterns of various metal species over time in terms of dynamic transport and temporal accumulation of heavy metals (specifically Cr, Zn, Sr, Fe, Mn, and Ni) from the electrolytic soil compartment to the anolyte and/or catholyte compartments under the influence of the applied electric field. These profiles reveal that Cr has been significantly transported towards the anolyte compartment; however, a small amount of Cr and other metals have also been transported towards the catholyte compartment. A preliminary indication of successful transport of different ionic species of metals from the soil to the electrolyte compartments was assessed on the basis of the temporal colour changes in both the anolyte and catholyte compartments during the course of EKR of the metal species from the soil cell. Notably, the electrolytes did not show any visible change in colour during the initial 2-3 hours of the EKR process. However, a yellow colour started appearing in the anolyte compartment about 3 hours after the start of the EKR experiment. Later, at 20 hours, a slight greenish-yellow colour started appearing in the anolyte compartment. This chromatic evolution in electrolyte compartments is primarily attributed to the solubilization and oxidation of metal species, specifically the potential formation of chromate ions $(Cr_2O_4)^{2-}$ and the release of metallic complexes (like Fe^{3+}) from the soil matrix as the anode-induced acid front began to develop (Han et al., 2021). The indigo colour appeared in the catholyte compartment, which is mainly attributed to the transport of Co in the catholyte compartment.

The energy-dispersive spectrometry (EDS) analysis of KSK test soil was performed before and after the EKR process to evaluate the geochemical impact of the acidic electrolytes (0.05 M H_2SO_4 in anolyte and 0.5 M H_2SO_4 in catholyte; pH ~1.25) on the soil particles contained in the electrokinetic soil cell. It was hypothesized that the induced localized acidic environment would facilitate the hydronium-mediated dissolution of native silicate minerals. This geochemical transformation was expected to manifest as a measurable reduction of structural constituent elements, specifically aluminum (Al), silicon (Si), potassium (K), magnesium (Mg), and iron (Fe) from the solid phase, thereby validating the occurrence of mineral weathering during the electrokinetic process.

Table 4 shows the EDS-based concentrations of Al, K, Si, Fe (Atomic %) in the test soil particles before and after EKR of KSK soil. The results reveal the presence of significant contents of Al, K, Si, and Fe (Atomic %) in the soil, which is attributed to the presence of clay minerals such as Montmorillonite/ Chamosite and Muscovite in the KSK soil, as these elements are the main constituents of aluminosilicate minerals. However, due to electrochemical washing of the KSK soil with an acidic electrolyte (H2SO4), the contents of Al, K, and Fe in the soil were found to decrease (Al decreased by 11.61–16.23 atomic %, K decreased by 5.88–9.56 atomic %, and Fe decreased by 0.59–8.18 atomic %). No

significant change is observed for the element Ca in the soil particles. This clearly signifies potential breakdown of the soil particles under the action of acidic electrolyte, thereby resulting in leaching/washing of elements such as Al, K, and Fe from the soil medium. A marked increase in Si content (14.85–36.33 atomic%) was observed following electrokinetic treatment. The higher percentage of Si is perhaps due to the development of a Si-rich secondary coating or the formation of an amorphous silica residue following the leaching of Al, K, and Fe.

Table 4: EDS-based elemental concentrations of KSK soil before and after the EKR process.

	Before Electro-remediation		**After Electro-remediation**			
Elements	Particle – 1		Particle - 1		Particle – 2	
	Element %	Atomic %	Element %	Atomic %	Element %	Atomic %
Mg Kα	----	----	----	----	1.02	1.22
Al Kα	18.13	21.83	8.34	10.22	5.22	5.60
Si Kα	44.73	52.08	56.85	66.93	85.80	88.41
K Kα	14.03	11.72	6.90	5.84	2.92	2.16
Ca Kα	2.66	2.17	3.06	2.52	----	----
Fe Kα	18.44	10.79	17.23	10.20	5.04	2.61
Ni Kα	----	----	7.63	4.30	----	----
Total	100.00	100.00	100.00	100.00	100.00	100.00

Elements (Kα line) using Energy Dispersive Spectrometry (EDS)

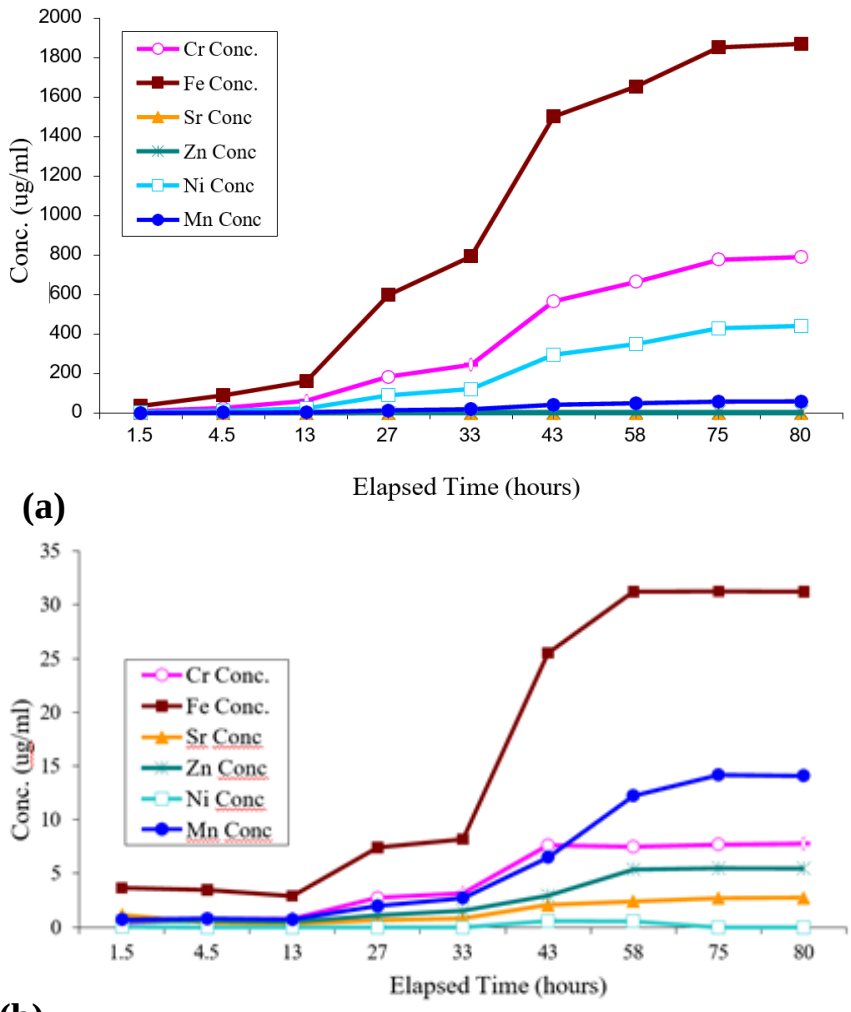

Fig. 6 Temporal changes in the content of metal species in anolyte (a) and catholyte (b) during EKR of KSK test soil.

Table 5: Mass balance calculations at the termination of the EKR experiment.

Elements ↓	Metals present in 1.5 kg experimental soil before EKR experiment			Metals remediated from soil and transported to electrolyte compartments			Metal element remained in 1.5 kg soil in the electrokinetic soil cell after EKR
	Initial mass of metal element in 1.5 kg test soil (A)	Mass of Contaminant metal used to spike soil (B)	Total mass of metal element in soil before EKR experiment (C=A+B)	Mass of metal element detected in catholyte (D)	Mass of metal element detected in anolyte (E)	Total mass of metal element in electrolyte (F=D+E)	Contents measured through numerical difference (H=C-F)
Units →	mg	mg	mg	mg	mg	mg	mg
Manganese (Mn)	537.0	NIL	537.0	6.07	29.03	35.10	501.9
Iron (Fe)	33204.0	NIL	33204.0	14.90	733.27	748.17	32455.83
Chromium (Cr)	84.0	450	534.0	3.73	306.50	310.23	223.77
Zinc (Zn)	94.5	150	244.5	2.37	2.17	4.54	239.96
Lead (Pb)	22.5	NIL	22.5	0.00	0.00	0.00	22.5
Cobalt (Co)	16.5	NIL	16.5	0.42	2.27	2.69	13.81

3.5. EKR efficiency

The effectiveness of the EKR process for removal of toxic/heavy metals was validated through a comprehensive mass balance analysis based on the concentration of the metals in the test soil and the concentration of the metals transported to and/or deposited in the anolyte and catholyte compartments. This quantitative evaluation accounted for the residual heavy metal concentrations remaining within the soil medium relative to the mass fractions electrochemically transported to, and subsequently collected in, the anolyte and catholyte compartments. Table 5 shows the mass balance calculations for the remediated metals in relation to the initial concentration of these metals in the test soil. Using the mass balance data shown in Table 5, the electrokinetic remediation (or removal) efficiencies for Mn, Fe, Cr, Zn, Pb, and Co were calculated by dividing the total mass of the element in the anolyte and catholyte compartments by the initial total mass of the same element in the KSK test soil as follows:

(a) % removal efficiency for Mn = (Total conc. of Mn in anolyte and catholyte / initial conc. of Mn in test soil) * 100= (35.10 / 537) x 100 = 6.54%

(b) % removal efficiency for Fe = (Total conc. of Fe in anolyte and catholyte / initial conc. of Fe in test soil) * 100= (748.17 / 33204) x 100 = 2.25%

(c) % removal efficiency for Cr = (Total conc. of Cr in anolyte and catholyte / initial conc. of Cr in test soil) * 100= (310.23 / 534) x 100 = 58.09%

(d) % removal efficiency for Zn = (Total conc. of Zn in anolyte and catholyte / initial conc. of Zn in test soil) * 100= (4.54 / 244.5) x 100 = 1.86%

(e) % removal efficiency for Pb = (Total conc. of Pb in anolyte and catholyte / initial conc. of Pb in test soil) * 100= (0 / 22.5) x 100 = 0.0%

(f) % removal efficiency for Co = (Total conc. of *Co* in anolyte and catholyte / initial conc. of *Co* in test soil) * 100= (2.69 / 16.5) x 100 = 16.30%

The mass balance calculations for EKR experiments conducted on the spiked KSK silty clay soil for a period of 83.5 hours have revealed that the removal efficiency of Cr was by far the highest, with a remediation efficiency of 58.09%, while Co was remediated by 16.30% and Mn by 6.54%. Similar remediation efficiencies for Cr have been recorded by various authors (Lu et al., 2012; Fu et al., 2017). Although the EKR process incorporates the combined influence of several electrokinetic mechanisms (such as

electromigration and electroosmosis) that contribute to the transport of heavy metal ions toward the electrodes, Fe and Zn were remediated very little, with efficiencies of 2.25% and 1.86%, respectively, while no Pb was found to be remediated from the electrolytic soil cell during the present short-term EKR experiment. The present EKR experimentation demonstrates that under the experimental conditions of EKR test soil (silty clay) and electrolyte selection and composition (use of 0.05 M H_2SO_4 as anolyte and 0.5 M H_2SO_4 as catholyte) and applied electric potential gradient of 15V DC, the EK remediation has been moderately effective in mobilizing and extracting the ionic species of Cr and Co from the test soil. Further, a removal efficiency of 58.09% for Cr suggests that the applied electric potential gradient was sufficient to enhance chromium movement from the contaminated KSK test soil matrix. Although complete or optimum extraction of chromium (and other elements) could not be achieved in the present studies, the residual contamination highlights a multi-factored resistance within the contaminated soil matrix towards mobilization and extraction of ionic species of Cr and other elements present in the KSK test soil towards electrolyte compartments. Through physical examination of the post-remediated soil contained in the electrokinetic soil cell, it was partially observed that the limitations to poor remediation efficiencies of some of the elements were governed by distinct mechanisms, such as the presence of localized precipitation zones near the anolyte or catholyte boundary resulting from OH^- generation and subsequent increase in pH that promotes chemical processes such as precipitation of metal hydroxides, and secondly due to the thermodynamic stability and low mobility of specific oxidation states or complexes of the ionic species. Wada and Umegaki (2001) have also quoted that the effective stabilization of the localized pH environment may suppress adverse phenomena like hydroxide precipitation, thereby maintaining the target contaminants in a highly mobile, soluble state conducive to electroosmotic and electromigratory extraction. It is also speculated that the combined effect of structural heterogeneity, hydraulic characteristics, and physicochemical and hydrochemical properties of sediments in KSK test soil impacted variations in tortuosity and porosity of the porous medium in the electrokinetic soil cell, which significantly constrained the overall transport kinetics of the aqueous phase contaminant metal species during the remediation period (Han et al., 2021; Virkutyte et al., 2002).

4. Conclusions

A preliminary short-term bench-scale EKR experiment has been successfully conducted for electroremediation of Cr and associated metals such as Mn, Fe, Ni, Pb, and Zn from a silty clay soil sample collected besides industrial cum agricultural zone of Kala Shah Kaku town (Punjab Province, Pakistan) using a locally fabricated apparatus. The soil sampling area is mainly facing threats from industrial cum domestic wastes (mainly sewage and solid wastes) from a variety of small- to large-scale industries, including the chemical industry, leather tanning and paint industry, and agro-pharma industry. The electrokinetic remediation was actuated by the use of H_2SO_4 (0.05 M H_2SO_4 as anolyte and 0.5 M H_2SO_4 as catholyte) and an applied electric potential gradient of 15V DC across the electrolytic soil cell containing KSK test soil. Mass balance calculations were used to establish the electroremediation efficiency for Cr, Zn, and associated metals (Mn, Fe, Ni, and Pb) by comparing their concentrations in the anolyte and catholyte compartments, as well as in the KSK test soil before and after treatment. The following conclusions have been drawn from the present baseline laboratory-scale EKR experimentation:

- It appeared that during the 83.25 hours of electrochemical washing of KSK soil with H_2SO_4 (in anolyte and 0.5 M in catholyte) in the presence of 15V DC across the soil Cell, the electro-remediation efficiency for Cr was well around ~ 58.09%, while those for co-contaminants were as low as 16.30% (for Co), 6.54% (for Mn), 2.25% (for Fe) and 1.86% (for Zn), and 0% (for Pb).
- This chromatic evolution in electrolyte compartments is primarily attributed to the solubilization and oxidation of metal species, specifically the potential formation of chromate ions $(Cr_2O_4)^{2-}$ and the release of metallic complexes (like Fe^{3+}) from the soil matrix as the anode-induced acid front

begins to develop (Han et al., 2021). The indigo colour appeared in the catholyte compartment, which is mainly attributed to the transport of Co in the catholyte compartment.

- The lower EKR efficiency of ~ 58.09% for Cr in KSK soil may be attributed to the abrupt increase in pH of the anolyte from pH~1.25 to pH~2 after about 45 hours of electromigration, which caused an alkaline soil environment in the vicinity of the anolyte compartment, causing precipitation of insoluble Cr species in the test soil. It may also be attributed to the presence of neutral complexes of Cr such as $Cr(OH)_3^0$ under the prevailing pH conditions in the soil within the electrolytic soil cell, precipitation of Cr salts, and/or synergistic effects of co-existing metallic species of Pb, Co, and Fe on removal of Cr from the test waste soil.
- The lower EKR efficiency of various target metal species in the present investigation is attributed to precipitation of mineral phases/salts within the anolyte and catholyte compartments, as is observed through a decrease in EC of electrolytes in both the electrolyte compartments.
- Chromium was primarily detected in the anolyte compartment, with only trace amounts found in the catholyte compartment. This indicates that chromium was originally present in the test soil as both Cr(VI) and Cr(III), depending on the cell's redox potential throughout the experiment.
- The EKR process effectively alters the morphology of the contaminated medium by dissociating clay minerals, leading to the partial breakdown of soil particles, and inducing mineral phase precipitation at low soil pH. Additionally, it mobilizes metal ions away from the solid matrix and toward the electrolyte compartments.
- Practical skills gained through the present studies may be utilized for laboratory and field-scale applications of the EKR technology to clean up waste-affected soils in the industrial and agricultural zone(s) in Pakistan and elsewhere.

5. Recommendations for future work

The electromigration of subsoil metal species towards anolyte and catholyte compartments is a quite complex phenomenon, and it mainly depends on a number of factors/parameters such as soil redox conditions, presence of co-contaminants, optimization of electrowashing fluids, electrode materials, and experimental geometry. Some typical recommendations for potential, cost-effective, and environmentally friendly EKR experimentation for removal of toxic/heavy metals from contaminated soil and subsoil water environment are as follows:

- The experimentalist must focus on enhancing contaminant removal efficiency while maintaining process sustainability and reducing potential adverse effects with respect to the parent soil and water environment (i.e., the hydrogeological system).
- The experimentalist must optimize fundamental operational parameters to enhance the performance of EKR process, specifically, w.r.t. regulation of pH profiles most critically alongside electrode boundaries.) is vital to mitigating the polarization effects and geochemical barriers that typically delay the migration and transport of ionic species towards electrolyte compartments.
- The efficiency of the electrokinetic remediation process may be enhanced by selecting a suitable geometry of the electrokinetic soil cell, such as square, rectangular, hexagonal geometry, etc., depending upon the spatial configurations of the contaminated soil zone or environment, as well as by selecting suitable electrode materials which are highly conducting and resistant to corrosion in the acidic electrolyte environment contained in the electrolyte compartments or wells.
- Calibration of the applied electrical potential is quite critical for enhancement of the efficiency of the EKR process. The experimentalist must carefully balance the application of an optimal voltage gradient. The applied voltage must be high enough to provide the driving force necessary to accelerate ionic electromigration and electroosmotic flow. However, the applied voltage needs to be kept within limits to avoid excessive ohmic heating and secondary water electrolysis reactions, which retard the efficiency for electrokinetic removal of toxic/heavy

elements from soil and water environment. Further, the applied voltage must be adjusted so as to maintain the economic viability of the EKR process through minimized energy expenditure (Yeung, 1994).

- The efficiency of the EKR process may be enhanced by accelerating the contaminant transport kinetics through deployment of optimized electrolyte conditioning solutions, alongside the integration of advanced enhancement configurations, such as the positioning of permeable reactive barriers (PRBs) or the introduction of targeted chemical conditioning agents such as reducing agents for Cr(VI). These auxiliary agents work synergistically to change the contaminant's oxidation state, suppress premature geochemical immobilization, and maintain a sustained electromigration flux across the treated sediment profile (Shen et al., 2007).
- The optimal EKR strategies must adopt a holistic approach that balances high-efficiency heavy metal extraction with economic and environmental sustainability. This may be achieved by maximizing target contaminant recovery while simultaneously minimizing the net energy consumption, suppressing the risk of secondary environmental pollution, and mitigating adverse alterations to the fundamental physicochemical and structural properties of the treated soil environment.

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Authors contributions

Maria Khalid performed field sampling, physicochemical analysis of soil, laboratory-scale electro-remediation experiment and data collection, write-up of manuscript including compilation of illustrations, tables, figures and submission of the proofread manuscript to the journal. Sajid Rasheed Ahmad was involved in proofreading and technical review of the manuscript before its submission to the journal. Riffat Mahmood Qureshi proposed the main concepts for electro-remediation-related experimental work, provided relevant literature, and facilitated arrangements for laboratory ICP-OES analysis of electrolyte samples, as well as XRD and SEM analysis of soil samples.

Conflict of interest

The authors declare no conflict of interest.

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