Evaluating the Effect of the Approaching Electrode on the Migration for Metal Ions through the Electro-Kinetic Technique for Contaminated Soil

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ABSTRACT

In this study, three experiments using the fixed anode technique and approached anode technique were carried out. The first experiment (EK-1) used distilled water as leaching solution (purging solution) at the anode and cathode chamber under voltage gradient was equal to 1.5 V/cm. In the second and third experiments, a solution enhanced with 1M acetic acid (AA) was used as catholic solution. The results indicate that residual concentration of Zn^{+2} decreases, beginning with an initial value of 850 mg/kg in all portions of soil sample EK-1, EK-2, in addition to EK-3. It seems that the residual concentration of EK-3 (approached anode technique) was lower than that of EK-1 and EK-2 in identical portions.

Keywords: Zinc, electro kinetic, remediation, Approached anodes.

INTRODUCTION

Heavy metal-contaminated soil has been regarded as one of humanity's most pressing ecological issues. Geologic origins and anthropogenic activities such as mining, smelting, fossil fuel consumption, industrial discharge, waste disposal, and agricultural irrigation largely contribute to soil metal enrichment. Humans are most concerned about cadmium_ (Cd), copper_ (Cu), lead_ (Pb), zinc_ (Zn), chromium_ (Cr), mercury_ (Hg), selenium_ (Se), and arsenic_ (As). Heavy metals, unlike organic pollutants, cannot be biodegraded or chemically destroyed. Therefore, metals can easily build in soil over time. Metals in the soil can be absorbed by plants, contaminating the food chain as a result. Excessive accumulation of heavy metals in soil endangers agricultural ecosystems and human health. Therefore, contaminated soil with heavy metals must be remedied, as cited by Wang et. al. (2021).

Electrochemical remediation, especially electro kinetic remediation, has emerged as a promising technology, and tremendous strides have been achieved in the cleanup of contaminated soils using this technique during the past two decades. In an ordinary electro kinetic remediation system, the heavy metals in the soil move to electrodes with opposite charges by electro osmosis, electro migration, and/or electrophoresis in the presence of a direct-current electric field. However, electro kinetic remediation is typically employed for the treatment of fine-grained soils with low permeability (poor hydraulic conductivity). In certain instances, the extraction effectiveness of metals via electrochemical remediation is quite limited. Combining it with other technologies, such as bioremediation and extraction of heavy metals from soil particles using leaching solution (chemical agents), often results in an improvement. However, these single or combined processes require considerable electric fields (1–1.5 V/cm) to facilitate the migration of heavy metal ions, resulting in the electrolytic breakdown of water and electrode wear. Undesirable side reactions will significantly increase energy consumption, and supplementary equipment is required to control

the pH of electrolytes. In electrochemical soil remediation methods, reducing the applied voltage can effectively reduce energy consumed and costs, as cited by Yang et. al. (2020).

Amal and Ban (2017) investigated the efficacy of saturating soil with various alternatives for purging: acetic acid (1 M AA), ethylenediaminetetraacetic (0.1 M EDTA), sodium dodecyl sulfate (SDS), and ammonium citrate (1 MAC).The study also studies the use of a catholyte solution coupled with zeolite as a permeable reactive barrier in the electro-kinetic (EK) process (PRB). Faisal and Rashid (2015) looked into six various tests that were carried out on silty clay soil spiked with lead in concentrations of 1500 mg/kg. All of these tests were done with a constant DC voltage gradient of 1 V/cm with duration time of 7 days. During the electro-kinetic remediation process, different purging solutions and addition configurations, such as injection wells, were tried out to improve the extraction of lead from the Iraqi silty clay soil. The results of the experiments showed that 18%, 37%, 42%, and 29% of the lead was removed with distilled water, 0.1 M acetic acid, 0.2 M EDTA, and 1 M ammonium citrate, respectively, as the purging solutions. But adding injection wells as another way to improve the tests used the same purging solutions that were talked about above which have key role in increasing the removal efficiency up to 59 %. Shen et al. (2007) showed the enhanced EK in remediating soil during cation exchange membrane (CEM), the H+ concentration and the redox potentials are very high near the anode. Speeding up Cd electro-migration is made when pH is low and redox potential is high, soils near the anode can be quickly remedied. The EK process can work with a fixed anode (FA). To speed up the electromigration effect, a new CEM-improved EK technique with approaching anodes (AAs) is used. In turn, they were moved from the anode to cathode. Hence, migration of high redox potentials and high H+ concentration to the cathode become quick. In consequence, remediating of soil can be done very quickly and approximately 44% of the energy and 40% of time can be saved. Wei et al. (2016) carried out EK experiment to compare the influence of fixed electrodes and approaching anodes (AAs) with and without using reducing agent, which is sodium bisulfite ($NaHSO_3$). When $NaHSO_3$ was added to the soil before EK, treatment decreased

Cr (VI) to Cr (III) by 90.3%. The adverse effect of contrasting migration of Cr (III) and Cr (VI) species, as indicated by EK tests (III). Thus, the practical application of this method was discarded when the reducing agent is utilized. Wan et al. (2019) investigated the influence of moving anode polygonal electrodes in restoring cadmium polluted soil which was prepared in the laboratory. A regular hexagonal electrode electric restoration experimental apparatus was set up for the study. The repair effect of fixed anode method and approaching anode method was compared and analyzed under the condition of constantly shortening the distance between anode and cathode and the electric field strength being 2 V/cm. The results showed similarity in the current variation trend between the fixed anode method and the approaching anode method. There is also similarity in the pH variation for both methods. In the approaching method, there is significant decrease in the pH value at the later stage of restoration. The removal rate of Cd (II) using a method with a fixed anode was 68%, whereas the removal rate using a method with an approaching anode was 95% at type A2 sampling points. %. The energy usage of the approaching anode method was reduced by 65%, as compared to the fixed anode method. The approaching anode method is more effective. The purpose of this research was to examine the potential of an electro-kinetic technique for the removal of zinc from contaminated soil. This was accomplished by employing acetic acid (AA) as leaching solution and approaching of the anode electrode.

EXPERIMENTAL SECTION

Soil Preparation and characterization

The tests in this paper naturally made use of Iraqi soil as a porous media. It was washed, dried, and sieved again to ensure a consistent quality. A breakdown of the physicochemical characteristics of used soil is shown in (Table 1). In order to obtain zinc-contaminated soil with a concentration of roughly 850 mg/kg, a soil sample was artificially contaminated with Zn $(NO_3)_2$, prior being packed into the reactor cell in an effort to achieve equilibrium, the contaminated soil was allowed to sit for a period of three days. Following the same technique as Faisal and Rashid (2015).

Table 1. Composition and properties of the native soil

| Property	Value
Particle size distribution Silt (%) Sand (%) Clay (%)	 36.4 39.9 16.2
pH	8.5
$CaCO_3$ (%)	11.432
Chloride ions, Cl^{-1} (mg/l)	1.859
Organic content (%)	3.8
EC (ms/cm)	2.2

Experimental setup

The schematic diagram of the electro-kinetic experimental setup used in this work is shown in Figure 1a. The dimension of rectangular chamber was 14 cm D, 10 cm W, 26 cm L. The cell used in this study consisted of two cylindrical anode chamber, one rectangular cathode chamber, and a power supply (seen Figure 1(b)).It is feasible to enlarge the base zone and decrease the base zone area, maximizing the heavy metals removal and improving process efficiency, according to the research reported by Peng et al. (2013). Therefore, two perforated plexiglass cylinders with a diameter of 3.5 centimeters, height of 12.5 centimeters and eight holes measuring 0.3 centimeters each were placed in the other end of the rectangular cell, which represented anode chambers. The position of the cathode chamber in cell was located at the opposite direction of the position of the two anode chambers in the rectangular cell and had the following dimensions: 7 cm L, 10 cm W and 14 cm D. Some of the holes that made on the surface of the cathode chamber was covered by the filter paper to prevent soil from entering the electrode compartments and each a slotted graphite electrode depending on the previous work (Faisal and Rashid, 2015). The soil sample in this cell is 13 cm long. It is divided into four sections starting from the anode chamber as S1, S2, S3 and S4, the same division of sample from anode towards the cathode that was adopted by Zhang et al. (2014), the length of each section was equal to 3.25, as shown in Figure 1a. During the test, a multi-meter was used to monitor the voltage applied to the electrodes from a DC power supply and to measure the current flowing through the soil sample, according adopted by Faisal and Rashid (2015). Three experiments were carried out with using fixed anode and the approached anode technique to enhance removal of zinc from the contaminated soil. According to Wan et al. (2019), the approached anode method is similar to the fixed anode method, the anode move 3 cm towards the cathode every 24 hr., while adjusting the voltage to control the electric field strength. In the present study, two first tests was represented as Fixed Anode (FA), that EK process is operated with fixed anode (i.e., the voltage 19.5 V), which was included the experiments EK-1 and EK-2 for study the removal of zinc ion from contaminated soil. In turn, EK-3 represented the experiments for approaching anode electrode, the anodes were located at distance equal to 3.25 cm from S1 (i.e., the anode were switched from S1 to the S2 electrode after 50 hour operation and the voltage (14.625 V) used was adjusted accordingly to maintain 1.5 V/cm), as seen in Table. 2. This is consistent with the work of several research, including Zhang et al. (2014), Wan et al. (2019), Ng et al. (2016), Cai et al. (2016), and Wei et al (2016). In the experiment known as EK-1, distilled water was utilized in both the anode and the cathode compartments. This experiment serves the purpose of establishing a baseline. The experiments [EK-2 and EK-3] were conducted using 1M acetic acid as catholyte for buffering the hydroxide ions and maintaining a low pH (~3), according to procedure adopted by Rashid (2015). At the end of each experiment, the soil sample was collected from each section and then performing acid digestion to measure the concentration of zinc according to the procedure that reported by Faisal and Rashid (2015).

Table 2. Experiments on electro-kinetic remediation in detail

Experiment designation	Conc. (mg/kg)	Time (hours)	PS (pH)	
			Anode	Cathode
EK-1 (FA)	850	100	DW	DW
EK-2 (FA)	850	100	DW	1 M AA (~3)
EK-3 (AAs)	850	100	DW	1M AA (~3)

Note: AA – acetic acid, AAs – approach anode, FA – fixed anode, DW – distilled water.
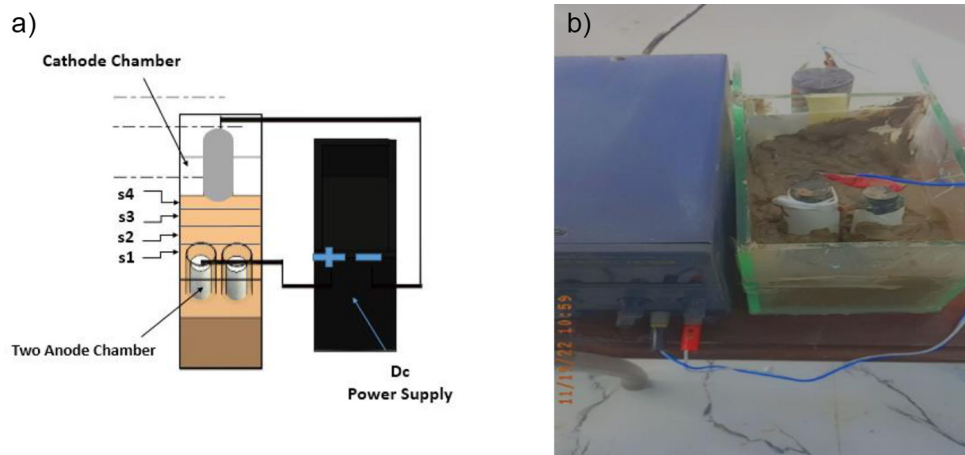
Figure 1. The experimental setup of electro-kinetic cell utilized in the current research electro-kinetic experiments

RESULTS AND DISCUSSION

Results of the contaminated soil artificially analysis

Figure 2 shows the scanning electron microscope (SEM) together with EDS Spectra. For 20 μm magnification scale (the arrangement figure according to Wang et al., 2017), the scanning electron microscopy (SEM) has turned up

to depict such comparisons to reveal the surface shape, particles distribution, contaminant distribution, morphological alterations (Hayder and Ayad, 2018), presented in Figure 2a, are important for identification of the morphological alterations for adopted the soil amended with zinc ions before electro-kinetic remediation. It can be seen that the shape of the initial soil amended with zinc ions is separated aggregates with large pore spacing. Similar results were obtained by Safia et al.
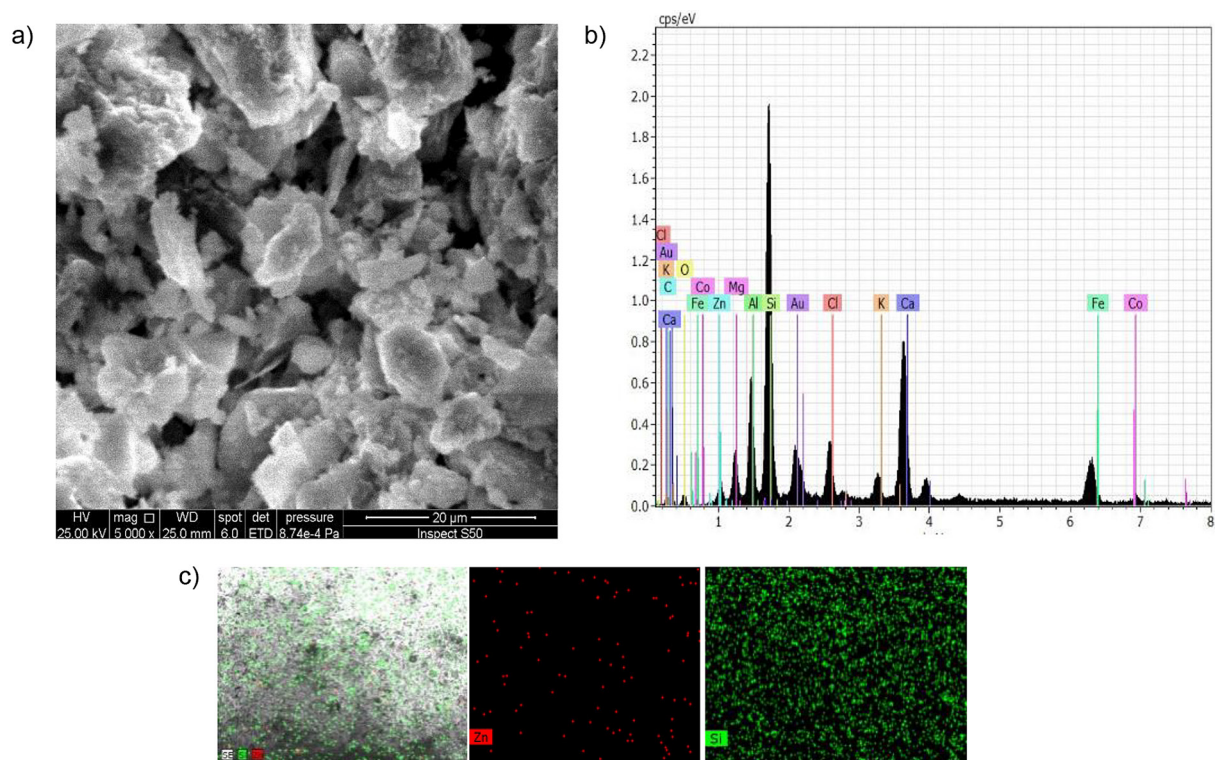
Figure 2. Mapping spectra, EDS and SEM of the soil amended with zinc ions before electro-kinetic remediation: (a) SEM, (b) EDS and (c) mapping of Si and Zn

(2021) who showed that the SEM photographs for the natural soil revealed separate aggregates with large pore spacing. Figure 2b elucidates that silicon (Si), antimony (Au), aluminum (Al) and calcium (Ca) forms the predominant element in the composition of the initial soil amended with zinc ions. Figure 2 (c) shows EDS mapping for Si and Zn. It was noticed that the Si heavily distributed, while Zn was randomly distributed. These results are consistent with finding by Wang et al. (2017), the majority of the Cu and Zn in the sediment was not linked to the components containing S and P. SEM-EDS mappings of the Fe, S, and P were heterogeneously dispersed, whereas Cu and Zn were homogeneously distributed, and most Cu and Zn in the sediment were not associated with the components containing S and P.

Behavior of the metals migration under approached anode electrode technique:

Electric current

The experiments that used of the electrokinetic fixed anode technique to remove zinc from the contaminated soil are conducted using distilled water (DW), 1 M acetic acid (AA) as purging solution (leaching solution). In these experiments, clean soil was spiked with 850 mg/kg zinc. The potential gradient of 1.5 V/cm was chosen in this study, according to Zhang et al., (2021) stated that the higher the voltage, the greater the soil system current. High voltage is beneficial to improving the migration rate of pollutants, but it also increases the power consumption, thus it is suitable to find the optimal voltage gradient. In order to investigate the effect of fixed anode technique (FA) and approached anode technique (AAs) on the electric current variation with time. The experimental results of EK-3 (AAs) are compared with results of EK-2 (FA), as shown in (Fig. 3). These experiments were carried out at the same operating parameters (i.e., used distilled water in anode chamber in addition 1 M AA in the cathode chamber with control pH (~3)). For EK-1, the electric current in the process varied from 29 to 429 mA within ranged between 1 to 46 hr. At 46 hr., electrical current was at its highest, then decreased gradually, and finally stabilized to 71 mA. Similar the result by Faisal and Rashid (2015) showed the current increased from an initial value of 34 mA to peak values of 60 mA in approximately 12 and 24 h.

The current then decreased to a low value and stabilized at approximately 16 mA. In EK-2 and EK-3, when used 1 M AA as catholyte solution in order to maintain a low pH (~ 3) by cathode compartment, according to the procedure adopted by Rashid (2015). The reason for using of acetic acid that it is often adequate to reduce soil pH, In addition, acetic acid is a weak acid that is incapable of highly dissociating; hence, adding acetic acid to the soil will not result in a significant increase in soil electrical conductivity, as cited by Saeedi et al. (2013). The quantity of ion electromigration reveals the electric current (Shen et al., 2007). The electric current variations for EK-2 and EK-3 experiments as a function of the time are depicted in Figure 3. It is seen that the current increase in the first hours of the experiments (first 26 and 30 hours for EK-2 and EK-3, respectively) is greater than later during the experiments. With time, the electrical current seems to approach more stable levels (92 mA and 190 mA for EK-2 and EK-3, respectively). During the experiments, the dissociation of certain chemicals, such as acetic acid, may also increase the electrical conductivity of the soil. Thus, acetic acid positively enhanced electrical current during the testing, according to Saeedi et al. (2013). The experiment of EK-3, where the two anode electrodes were inserted in S2, the distance between anode and cathode was short (9.75 cm from cathode) and the voltage between electrodes applied was equal to 14.625 V, which was slightly higher than in the EK-2 and decreased again until it attained its steady state. The results of this part had provided significant agreement with the previous study by Shen et al. (2007) that exhibited electrical current in the

Figure 3. Effect of FA and AAs on variation of current of electro-kinetic with time for EK-1, EK-2 and EK-3 experiments

CEM-EK (cation exchange membrane (CEM) enhanced electro-kinetic (EK)) with approaching anode electrodes (AAs) which was higher than in the fixed anode electrodes (FA) experiments. This phenomenon partly explained the possible mechanism of enhanced Cd removal in the AAs experiments. Furthermore, Wan et al. (2019) reported that, after the experiment began, the anode was moved a certain distance towards the cathode at regular intervals, and the repair voltage was adjusted to control the electric field strength. Despite the fact that the current variation in the approaching anode method is similar to that in the fixed anode restoration method, the current value is higher in the approaching anode method.

Removal metals from contaminated soil

Figure 4 and Figure 5 illustrate pH and zinc distributions profile of the soil as a function of distance from anode for section S1, S2, S3 and S4 after the electro-kinetic treatment. With the help of electro-migration and electro-osmosis, the acid front formed by electrolysis of water in the anode compartment moved toward the cathode, where it dissolved or desorbed pollutants adsorbed and/or complexed on soil particles. Dissolution and desorption of species in the soil cell occurred concurrently with the acid front migration during treatment, and the contaminant species appeared to be gradually transported toward the cathode under the influence of the electric field, as cited by Rasha (2012). As shown in Figure 4, it was noticed that soil pH for all tests at region near the anode was lower than its region near the cathode. Soil conductivity was reduced, because Cu $(OH)_2(s)$ precipitated in the areas close to the cathode sections, which formed in the high pH environment. The high buffering capacity (the high-pH environment) of soil is caused due to the presence of carbonate or calcite in the contaminated soil. Therefore, the efficiency of electro kinetic remediation process has decreased due to the heavy metal retained in the precipitated hydroxide/carbonate forms, as cited by Faisal and Hussein (2015). In addition, Faisal and Rashid (2015) showed that pH values were restored following electrokinetic treatment. The pH profile shows that the soil around the cathode achieved a value of nearly 8, while the pH in the cathode compartment is kept at roughly 3. These pH values indicate that the cathode reaction may not have been completely depolarized during the experiment.

The remaining concentrations of Zn^{+2} in each section that were obtained for the EK-1, EK-2 and EK-3 experiments after remediation are shown in Figure 5. It was clear that the residual concentrate of Zn^{+2} in S1, S2, S3 and S4 for EK-1 and EK-2 decrease from an initial value of 850 mg/kg. The amount of Zn^{+2} observed for the EK-1 and EK-2 (represented a case of fixed anode), varied from 580–403.46 mg/kg at section S1 (near the anode region) to 722.5–543.2 mg/kg at section S4 (near the cathode region). This result was in agreement with that reported in a previous study (Faisal and Rashid (2015) reported that when distilled water is used as the purging solution, the removal lead is severely affected by the precipitation of these metal in the soil near the cathode region. This precipitation can be attributed to development of high pH conditions which result from the water electrolysis at the cathode compartment. The cathode compartment was purged with 1 M acetic acid to inhibit the fixing process. Acetic acid's production of H⁺ ions allows the pH of the solution to be lowered, which is useful for dissolving metal precipitates. The amount of acetic acid needed to keep the pH in the cathode compartment at a low value (2.5–3) has been calculated. When 1 M acetic acid was used to purge solution in the cathode compartment, lead removal efficiency rose to 37%. Since acetic acid can produce H⁺ ions that can reduce the pH of the solution and dissolve metal precipitates, its usage in the cathode compartment while maintaining a pH of around 3 has a significant impact on the migration of pollutants.

In turn, the concentration of Zn^{+2} in the EK-3 experiment increased towards the cathode from S1 (near the anode region) (250 mg/kg) to S4 (360 mg/kg) (near the cathode region). The reason for this, when the distance between anode and cathode was short as in the EK-3 test, the H⁺ produced by the anode was transferred to the cathode more quickly. The faster the migration of H⁺ was, the faster the desorption and dissolution of lead from soil was, and better the corresponding removal effect (Zhang et al., 2021). It is obvious that the highest removal rate of Zn^{+2} appeared at section S1 because the pH value in the soil near the anode as shown in the same Figure 4 was lower. In turn, the lowest removal rate of Zn^{+2} appeared at S4. Due to the high pH at the S4. In addition, Faisal and Rashid (2015) reported that the removal of lead from the soil system in this test, since all of the samples are below the
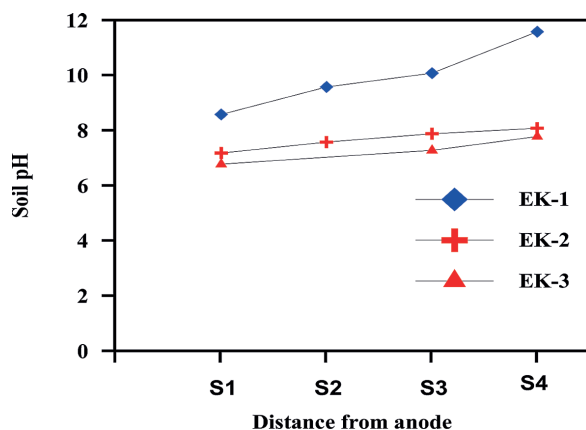
Figure 4. The pH profiles in the treated soil by electro kinetic technique

initial concentration (1500 mg/kg). A higher concentration of contaminants was detected close to the cathode. In addition, the pH distribution is demonstrated to further the observed heavy metal distribution. Starting with the anode, the pH of two-thirds of the soil decreased, whereas the pH of the soil increased in the region of the cathode. The observed pH fluctuations are generally comparable with earlier reported electro-kinetic experiments. Because the smaller ionic radius, the mobility of an H^+ ion is approximately twice the mobility of an OH^- ion. In accordance with a previous study (Wan et al., 2019), this result demonstrated that the removal rate of Cd at A1 and B1 type sampling points that are 4 cm from the anode reached approximately 65%, whereas the removal rate of Cd at A3 and B3 type sampling points that are close to the cathode was relatively lower. This is because H^+ was created and moved from the anode, and the closer it was to the anode, the lower the soil pH.
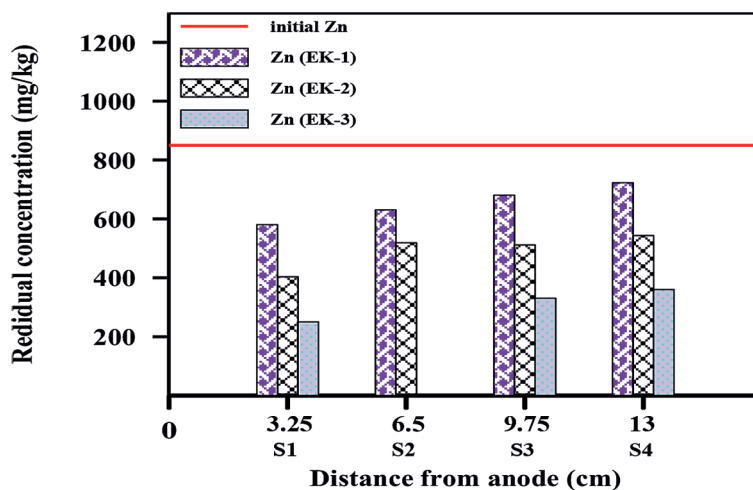
Figure 6 (a and b) shows the effect different types of the leaching solutions (when DW and 1 M AA were used as catholyte solution in the EK-1 and EK-2 tests, respectively), on the decreasing of residual carbonate across the length of the soil specimen. Accordingly, the increasing removal of the salts from the specimen with using 1 M AA of leaching solution in cathode chamber can be distinguished by acceptable quantities from these salts that accumulated on the surface of the cathode electrode. The reason might be to use the soil were consisted from 39.9% sand, 36.4% silt and 16.2% clay (see Table 1). Greičiūtė and Vasarevičius (2007) explained that sandy loam and sand have the coarsest grain structure compared to other types of soil, which allows for a more efficient movement of metal ions and consequently better cleaning results.

Figure 7 (a and b) show the scanning electron microscope (SEM) of sample points S1 (near the anode) and S4 (near the cathode) for EK-2 (when used 1 M AA as catholyte solution), was magnified to a 20 μm scale. Figure 7a shows the shape of the surface of soil in S1 for the region near the anode and Figure 7b presents it S4 for the region near the cathode. This revealed that the treated soil included stacks of huge platy-shaped particles with large spaces after being treated using the Ca-DW system. It is suspected that the coating materials were dissolved near the anode due to the acidic environment or other chemical processes. The huge interconnected soil particles in the background besides with randomly arranged tiny nodules on the foreground of the Ca-DW treated sample near the cathode, according to report by Ahmad et al. (2006).

Figure 5. The zinc profiles in the treated soil by electro kinetic technique for EK-1, EK-2 and EK-3
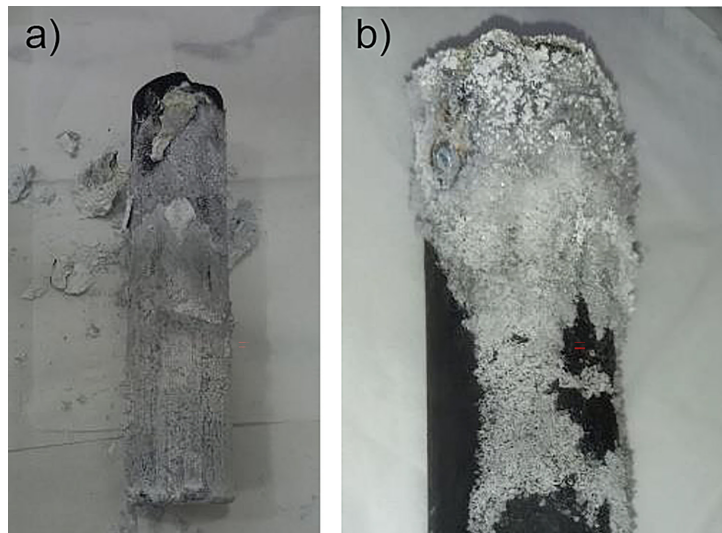
Figure 6. Salt accumulation on the surface of the cathode electrode end time of the electro-kinetic process: (a) in EK-1 test and (b) in EK-2 test
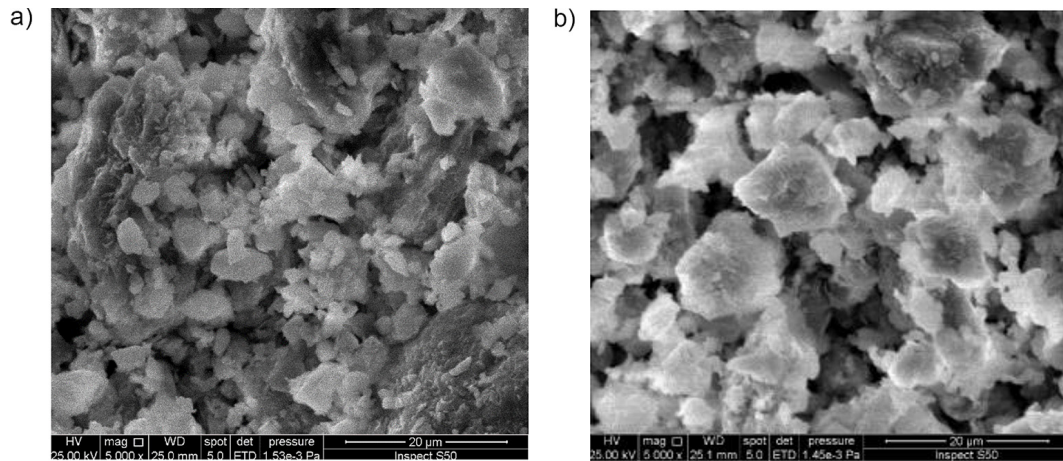
Figure 7. SEM images for the (a) S1 (near the anode) and S4 (near the cathode) samples soil for EK-2 test

CONCLUSIONS

It appears that the residual concentration of zinc in S1, S3 and S4 for EK-3 (approached anode technique) were lower than those in the same sections for EK-1 and EK-2. It was observed that the soil pH for all tests in the region near the anode was lower than its region near the negative electrode.

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