

Nickel removal from low permeable kaolin soil under unenhanced and EDTA-enhanced electrokinetic process

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Abstract. This paper represents a set of experimental tests on remediation of nickel-contaminated kaolin by Electrokinetic method. For this purpose, we conducted unenhanced and EDTA-enhanced Electrokinetic tests in one, three, and five days of treatment. In unenhanced tests, we used deionized water as an electrolyte in the anode and the cathode compartments. In the EDTA-enhance tests, we used ethylenediaaminetetra acetic acid 0.1 Molar in the cathode and sodium hydroxide 0.1 Molar in the anode. The average nickel removal for unenhanced tests after three and five days of treatment was 19 and 23 percent, respectively. High buffer capacity of the soil is responsible for low removal efficiency in the unenhanced tests, which maintained pH close to the initial amount that restrained nickel as an adsorbed or precipitated forms. The average nickel removal for EDTA-unenhanced tests after three and five days of treatment was 22 and 12 percent, respectively. Lower ionic mobility of EDTA-Ni complex in comparison with Ni⁺², which is the main transportation mechanism for this complex, could be responsible for less removal efficiency in EDTA-enhanced test.

Keywords: acid enhanced; bench-scale experiment; electrokinetic treatment; kaolin; nickel

1. Introduction

Soil pollution is one of the worldwide problems, which has involved many environmentalists and governments. Heavy metal contamination is a type of soil pollutions that attracts more concern recently. Lead, chromium, nickel, cadmium, and arsenic are the significant heavy metal contaminants at numerous industrial sites (Taylor *et al.* 1997). In low permeable soils such as clays, the traditional methods, like *in-situ* soil flushing and chemical treatment, are high-priced and inefficient (Yeung *et al.* 1996). Electrokinetic remediation (EKR) is a technique which has considerable potential for *in-situ* remediation of low permeable soils (e.g., clay and silt) (Reddy *et al.* 2001).

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EKR involves applying a direct current (DC) electric field, across the soil medium. The contaminants are removed by the combination of different transport phenomena (1) electro-osmotic advection of the pore fluid flushing the contaminants; (2) ionic migration or electromigration of contaminants carrying charges; and (3) electrophoresis of charged particles and colloids that carry contaminants (Gholami *et al.* 2014, Yeung 2006). These transport mechanisms are operated by injecting a clean solution or enhancement agents such as surfactants, nutrients and complexation agents which cause to improve the performance and efficiency of the process (Yeung *et al.* 1996, Yeung 2006, Acar and Alshawabkeh 1996, Acar *et al.* 1997, Budhu *et al.* 1997, Wong *et al.* 1997, Reddy *et al.* 2003, Reddy and Cutright 2003, Thevanayagam and Rishindran 1998, Rabbi *et al.* 2000, Khodadadi *et al.* 2011, Kim and Han 2003, Håkansson *et al.* 2008). Ethylenediminetetra acetic acid (EDTA) is one of these complexation agents used for removal purpose in a wide range of heavy metals. Different studies have been conducted to investigate the effect of injecting EDTA into the contaminated soil on the efficiency of EKR (Amrate and Akretche 2005, Yeung *et al.* 1996, Reddy *et al.* 2004, Reddy and Chinthamreddy 2004, Reddy 2000). Because EDTA has a substantial ability to form strong water soluble chelates with most cationic metals, it has been used to extract heavy metals from the contaminated soil as an enhancement (Altin *et al.* 2004). EDTA added to the cathode compartment can be easily injected into a soil and it can increase the solubility of precipitated metals like lead and zinc (Wong *et al.* 1997).

Several laboratory tests have been performed to investigate the feasibility of the EKR to remove nickel from fine-grained soils like kaolin (Taylor *et al.* 1997, Guaracho *et al.* 2009, Giannis *et al.* 2010, Saleem *et al.* 2011, Saeedi *et al.* 2009). The direction of the migration and overall removal efficiency of contaminant depend on three factors: polarity of the contaminant, the presence of co-contaminants, and the type of soil (Reddy *et al.* 2001). Previous studies have disclosed that Ni (II), Cd (II), Pb (II) migrate towards the cathode in kaolin; however, the migration was mainly retarded in the presence of sulfides due to increased pH through the soil (Reddy and Chinthamreddy 1999, Huang *et al.* 2015). In the case of mixed contaminants soil composition, the type and amounts of different contaminants designate the initial speciation of contaminants (Reddy *et al.* 2001). In the EKR, adsorption, precipitation, and reduction are the most important obtrusive mechanisms for the removal of heavy metals (Reddy *et al.* 2001). The combination of electrokinetic with activated carbon barrier could increase nickel's migration and effectively prevent the opposite electroosmotic flow (Saeedi *et al.* 2009). About coexisting of nickel and organic contaminants in clay soils, integrated EKR has a potential to remediate contaminated soils by degrading the organic contaminants through the chemical oxidation process and at the same time removing the heavy metals through electromigrations (Cameselle and Reddy 2013). Giannis *et al.* (2010) evaluated the feasibility of the chelating agents' nitrilotriacetic acid (NTA), diethylenetriaminepenta acetic acid (DTPA), and diaminocyclohexanetetracetic acid (DCyTA) in desorbing cadmium, nickel, and zinc from contaminated soils and they proved that binding forms of these metals changed from the difficult type to easier extraction type. The simultaneous application of bipolar electrodes and a longer cathodic chamber length without agents addition was investigated as an enhanced EKR of nickel, cadmium, and lead contaminated sediment (Rajić *et al.* 2013).

In the present study, we investigated the EKR of nickel from artificially contaminated kaolin. Kaolin does not have enough buffering capacity, and the clay mineral has a low surface area and cation exchange capacity (CEC). However, kaolin polluted with Ni is a problematic soil in some part of Iran. Therefore, we selected kaolin as a soil model. We conducted a set of experiments to

understand the effect of using the EDTA solution and pH conditioning at anode chamber to enhance removal process. The aim of this research was to compare between unenhanced and EDTA-enhanced EKR tests for nickel-removal. Other researchers have not investigated this form of experiment.

2. Materials and methods

2.1 Electrokinetic cell

Fig. 1 indicates the electrokinetic apparatus. This cell was made of Plexiglas and had three parts, anode and cathode chambers and the soil cell. The soil separated from chambers using a perforated Plexiglas plate, a porous stone, and a filter paper (Whatman No. 40 middle speed). Platinum plate electrodes were used as both anode and cathode electrodes.

Table 1 Soil configuration

Properties	Value
Permeability (cm/s)	6.32E-08
Initial water content (%)	1.18
Liquid limit (LL %)	45.8
Plastic Limit (PL %)	26.6
Plasticity Index (PI %)	19.2
Specific gravity (gr/cm ³)	2.68
USCS Classification	ML

Table 2 soil chemical and mineralogical characteristics

Types of Analysis	Chemical Compound	Percent
Chemical Analysis	SiO ₂	63±1
	Al ₂ O ₃	24±1
	Fe ₂ O ₃	0.55±0.1
	TiO ₂	0.04±0.01
	CaO	1.2±0.2
	MgO	0.55±0.06
	Na ₂ O	0.4±0.1
	K ₂ O	0.3±0.1
Mineralogical Analysis	Kaolinite	64±2
	Quartz	27±2
	Calcite	2.1±0.5
	Others	6±1

2.2 Soil

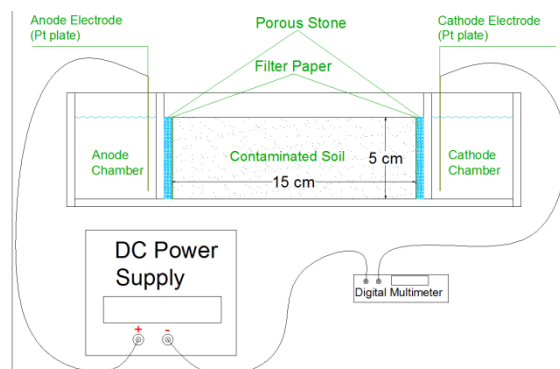


Fig. 1 Schematic of electrokinetic reactor

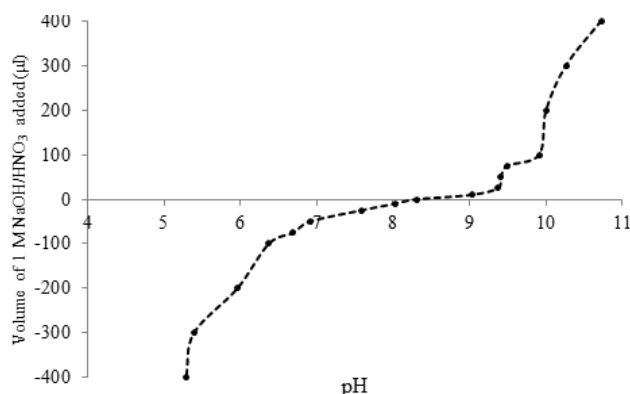


Fig. 2 Acid/base buffer capacity curve of kaolinite soil

Tables 1 and 2 indicate the kaolin properties. Kaolin spiked artificially at 500 mg/kg of dry soil concentration by nickel (II) nitrate salt ($(\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O})$, for analysis EMSURE® ACS 99 percentage reagent grade salt supplied by Merck Millipore Co. Germany).

We carried out acid/base buffer capacity test of determination to understand the buffer capacity of kaolin, which was used in the present EKR tests. The acid/base buffer capacity of a soil sample depends on many variables such as soil concentration, the ionic strength of the solution, and presence of exchangeable cations, (Yeung 2006). Fig. 2 presents the result of the experimental test of acid/base buffer capacity. The slope of the curve represents the buffer capacity of the soil used in the EKR experiment (Chen 2009). As illustrated in Fig. 2, the soil has a high acid/base buffer capacity.

2.3 Contaminated soil preparation

For each experiment, about 700 grams of dry kaolin was taken and 280 ml deionized water that mixed with 1.7342 grams nickel nitrate salt was added to achieve soil with 40% water content and to reach a final concentration of 500 mg/kg of nickel. We mixed the soil thoroughly and allowed to equilibrate for 24 hours. Afterward, we placed in the Electrokinetic cell in layers and compacted uniformly. The initial water elevations in both of the electrode cells kept to the same level in order to prevent a hydraulic gradient forming across the soil sample. Then the Electrokinetic cell

connected to a power supply, and we applied a constant voltage drop of 0.5VDC/cm electric field to the soil sample. We monitored the current, which passed through the soil sample, the pH, and electrical conductivity (EC) of the aqueous solutions in both chambers during the experiments. Aqueous solutions from both cathode and anode compartments were collected at the end of the tests to determine nickel concentration.

2.4 Unenhanced and EDTA-enhanced EKR tests

We conducted six EKR experiments (Table 3). Three experiments were unenhanced and the rest were enhanced tests. We used deionized water in both anode and cathode chambers for unenhanced experiments. In enhanced tests, we used 0.1 M solution of EDTA ($C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$), with a molecular weight of 372.24 gr/mol, ACS reagent grade salts supplied by Merck Millipore Co. Germany, as catholyte and 0.1 M NaOH as anolyte. We conducted each experiment for one, three, and five days. All experiments were performed under constant voltage drop by applying 0.5 (VDC/cm) electric field.

Table 3 the conducted experiments' properties

No.	Test type	Treatment Duration (hour)	Potential Gradient (VDC/m)	Catholyte	Anolyte
1	Unenhanced	24	50	deionized water	deionized water
2	EDTA-enhanced	24	50	EDTA 0.1 M	NaOH 0.1 M
3	Unenhanced	72	50	deionized water	deionized water
4	EDTA-enhanced	72	50	EDTA 0.1 M	NaOH 0.1 M
5	Unenhanced	120	50	deionized water	deionized water
6	EDTA-enhanced	120	50	EDTA 0.1 M	NaOH 0.1 M

2.5 Analytical methods

We measured the pH of the soil before and after treatment at each section. For this measurement, 1:2.5 soils to deionized water slurry were prepared and agitate by a wrist action shaker for 30 minutes, and after the soil sample centrifuged for 15 minutes at 6000 RPM and pH determined in the supernatant. The pH and electrical conductivity of anolyte and catholyte measured before, during, and after the treatment by directly immersing pH and conductometer probes into these solutions.

In each experiment, we selected six samples (one sample before and five samples after treatment). We sectioned the soil specimens into five different parts after remediation process. We prepared two samples from each section, one for pH and another for concentration analysis. Each sample preserved in a separate glass jar. Soil samples (1-2 gram of wet soil) were acid digested according to U.S.EPA 3050B method. For detailed descriptions of acid digestion procedure, readers refer to the work of Reddy and Chinthamreddy (Reddy and Chinthamreddy 1999). We measured nickel concentration using ICP-AES Spectrometer (SPECTRO ARCOS, Ametek Co. Germany).

3. Results and discussions

3.1 Current

Figs. 3 and 4 indicate current density through the soil specimen for unenhanced and EDTA-enhanced experiments, respectively. The soil conductivity depends on the concentration of the ionic species in pore fluid which is responsible for the current conduction through the soil (Reddy *et al.* 2001). Consequently, by increasing the concentration of the ionic species, the passing current through the soil will be increased (Reddy *et al.* 2001). Current density expresses as the proportion of passing current to the cross-sectional area of the electrodes. We conducted these experiments under constant voltage drop condition and allowed current variation.

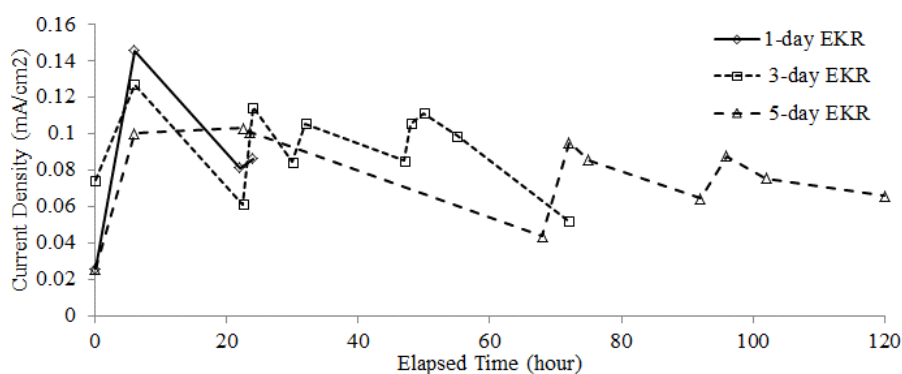


Fig. 3 Current density variation in unenhanced tests

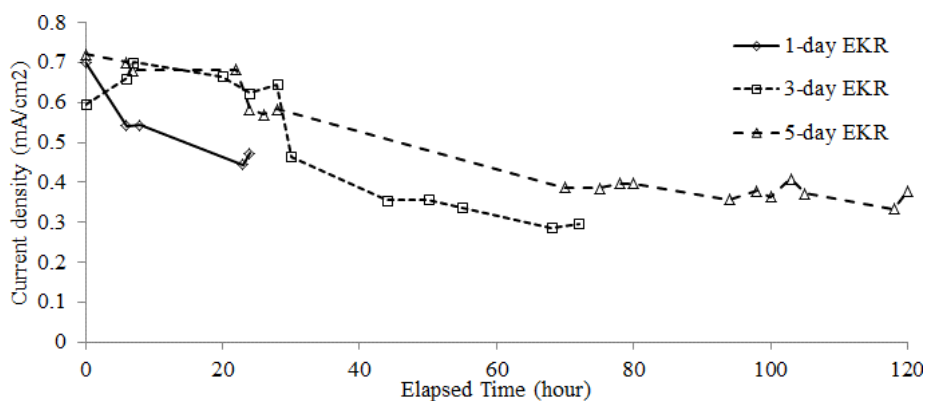


Fig. 4 Current density variation in EDTA-enhanced tests

As depicted in Fig. 3 current density increased initially. This increase may be due to low pH condition, resulted from water electrolysis reactions which were mentioned by some researchers (Reddy *et al.* 2001, Rajić *et al.* 2013). Having increased concentration of dissolved ionic species, the soil conductivity remained high and current density increased (Rajić *et al.* 2013, Cameselle and Reddy 2013). Close to the cathode chamber with high pH the rate of adsorption and precipitation increase which these phenomena are responsible for high resistance that developed near the

cathode, resulting in a much lower current at the end of the process similar to other researchers' work (Reddy *et al.* 2003, Rajić *et al.* 2013). As well as, this pattern repeated in EDTA-enhanced test (Fig. 4), current density increased at the beginning, and it diminished and oscillated around a constant value at the end of the process.

3.2 pH changes and electrical conductivity

In unenhanced tests the electrodes' pH are not manipulated during the test and can change because of electrolysis reactions (Asadollahfardi *et al.* 2015). As Fig. 5 illustrates pH changes in unenhanced tests, pH increased up to 11 at the cathode owing to OH⁻ generation, and decreased to a value around 2 because of H⁺ generation at the anode, which was mentioned by other researchers (Rezaee *et al.* 2015, Asadollahfardi *et al.* 2016). In all unenhanced experiments, pH reached its final value after 2 hours at the cathode and 5 hours at the anode chamber.

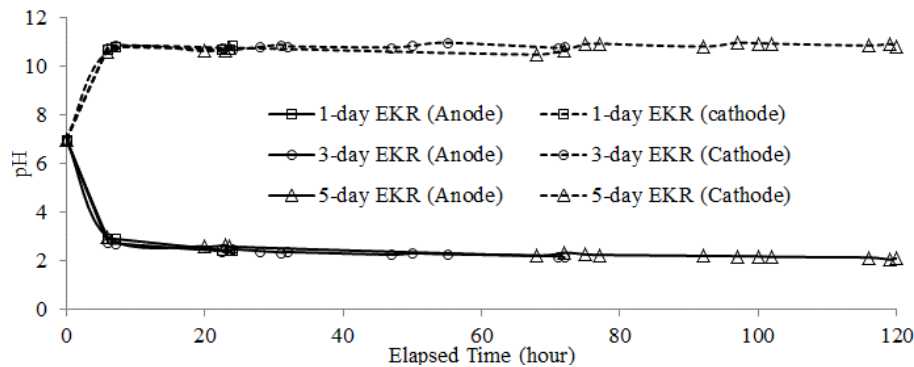


Fig. 5 pH changes in electrode chambers in unenhanced tests

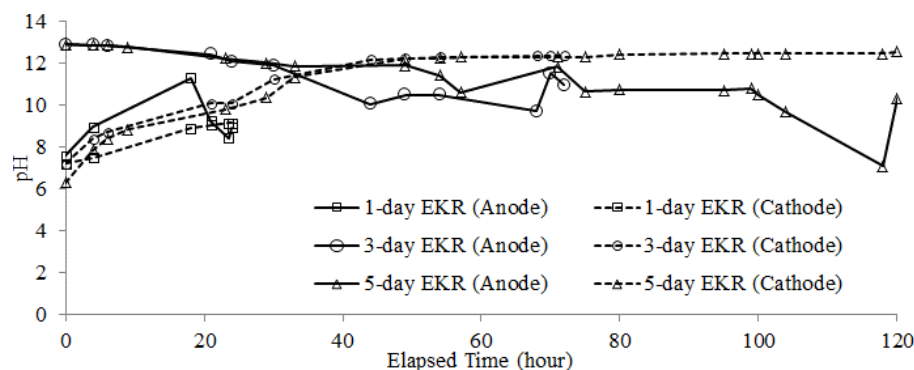


Fig. 6 pH changes in electrode chambers in EDTA-enhanced tests

In the EDTA-enhanced experiments (Fig. 6), pH of catholyte (EDTA 0.1 M) modified before starting treatment at pH > 7 in order to facilitate forming anionic complexes with nickel (Cameselle and Reddy 2013). Consequently, pH in cathode chamber started from an amount above 7 and reached to 9.62, 12.35, and 12.55 after 1, 3, and 5 days of treatment, respectively. At the anode, (sodium hydroxide 0.1 M as anode solution) pH was 12.89 at the beginning and it decreased

during the process. The diagram's oscillation attributes to adding primary solution whenever the fluid level decreased in the chamber to prevent hydraulic gradient.

Figs. 7 and 8 indicate the pH distribution in the unenhanced and the EDTA-enhanced tests through the soil sample respectively. H^+ and OH^- ions produced in the chambers, migrated through the soil specimen and affected the soil pH. According to Fig. 7, in the unenhanced EKR process, pH in the soil profile did not change rigorously after 24 hours of treatment. In longer experiments (3-day and 5-day experiments) the influence of pH variations in the soil profile is also negligible.

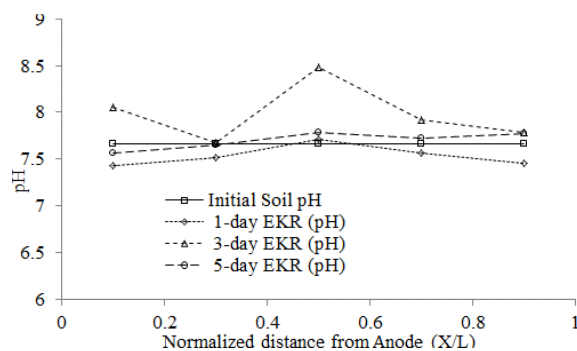


Fig. 7 pH in soil specimen in unenhanced tests

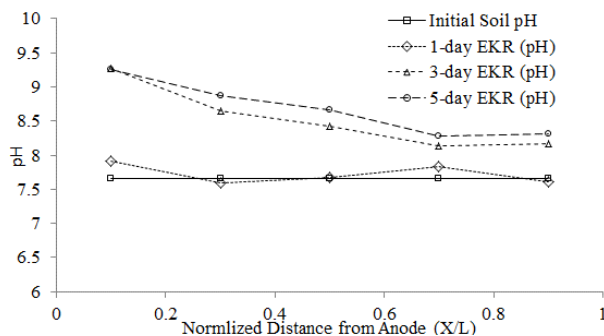


Fig. 8 pH in soil specimen in EDTA-enhanced tests

As indicated in Fig. 2, the high buffer capacity of the soil prevents pH changing, increasing or decreasing, in the soil sample during the EKR tests.

Considering EDTA-enhanced experiment (Fig. 8), similar to unenhanced 1-day treatment, pH did not change drastically. At 3-day and 5-day tests, pH changed slightly and increased from the cathode side because of OH^- ions influx from the cathode chamber. From anode side due to the reaction between $NaOH$ and H^+ , instead of the proton, counter ion (Na^{2+}) transports into the soil medium and prevents decreasing pH near the anode zone.

Figs. 9 and 10 indicate the EC of the electrode solutions. The EC depends on the ionic strength of the pore solution in the soils and represents the amount of dissolved ionic species (Reddy *et al.* 2001). For unenhanced tests, as indicated in Fig. 9, the EC of solutions started from a negligible amount (because of using deionized water in both chambers) and increased gradually. In contrast, the growth rate of the anode was more than cathode reservoir. This may be due to the migration of nitrate ions (nitrate salts of nickel used as sources of nickel) into the anode chamber. In enhanced

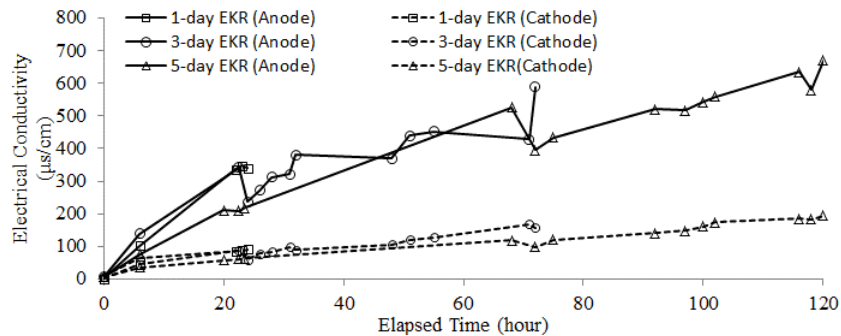


Fig. 9 Electrical conductivity variation in electrode chambers in unenhanced tests

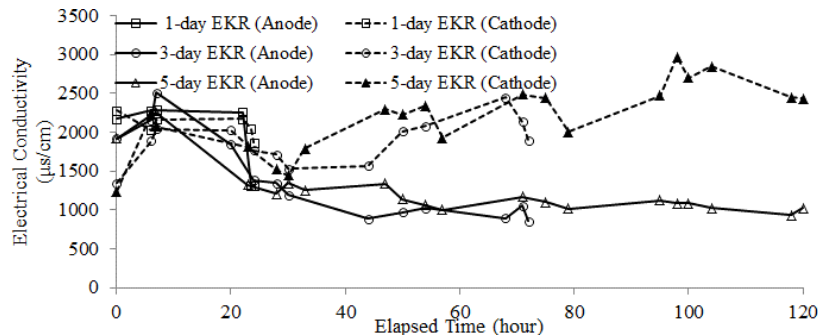


Fig. 10 Electrical conductivity variation in electrode chambers in EDTA-enhanced tests

experiments (Fig. 10), the EC of catholyte increased from an initial value of $2000 \mu\text{S}/\text{cm}$ to $3000 \mu\text{S}/\text{cm}$ and decreased from $1000 \mu\text{S}/\text{cm}$ to $2000 \mu\text{S}/\text{cm}$ at the anode. These variations may be owing to the presence of ionic species at cathode rather than anode chamber.

3.3 Nickel migration and removal

Figs. 11 and 12 illustrate nickel concentration for unenhanced and EDTA-enhanced experiments at the end of the remediation, respectively. As illustrated in Fig. 11, nickel removal for the one-day long experiment is low because of the short remediation duration. By increasing the remediation duration, we observed that the removal efficiency improved in 3-day and 5-day experiments. Generally low removal efficiency may attribute to the high pH condition of the soil medium which results from high acid/base buffer capacity of the soil, which renders the nickel ions to adsorb or precipitate.

In the EDTA-enhanced tests (Fig. 12), the direction of nickel migration changed, because EDTA forms anionic complexes with Ni under basic conditions (Chen 2009, Amrate and Akretche 2005). These complexes migrated towards the anode. In alkaline condition, the EDTA causes heavy metals to dissolve as anionic complexes and increase removal efficiency (Yeung *et al.* 1996). The average removal efficiency of nickel in the enhanced test in 3-day and 5-day tests is 19 and 23 percent and for the EDTA-enhanced test in 3-day and 5-day tests is 22 and 12 percent, respectively. The lower removal efficiency in EDTA-enhanced test could be due to lower ionic mobility of Ni-EDTA in comparison with Ni^{2+} (Amrate and Akretche 2005). Therefore, lower ionic mobility results in lower complex movement. Consequently, more EKR duration could be

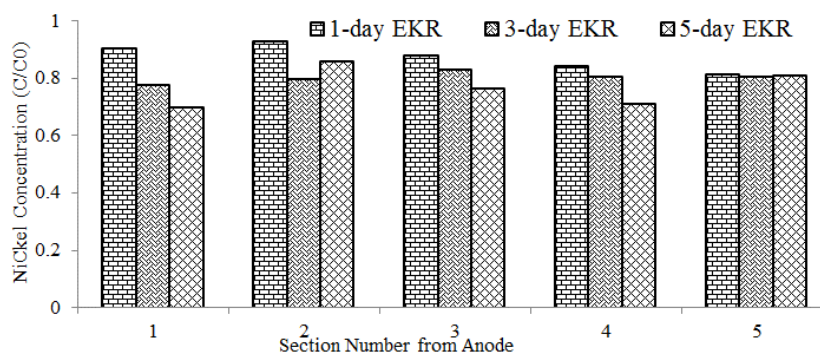


Fig. 11 Nickel concentration in kaolin after unenhanced EKR tests

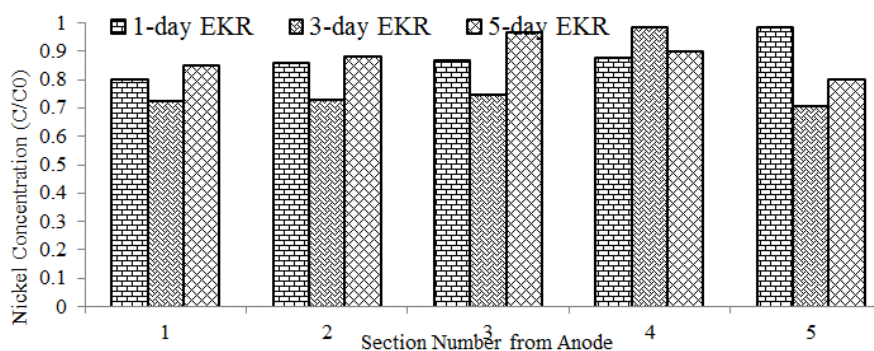


Fig. 12 Nickel concentration in kaolin after EDTA-enhanced EKR tests

necessary to increase the removal efficiency in the EDTA-enhanced test.

4. Conclusions

In the present study, we conducted unenhanced and EDTA-enhanced experimental tests for nickel removal from artificially contaminated kaolin. The results indicated that pH along the soil in unenhanced and EDTA-enhanced tests did not change drastically, which we ascribe this phenomenon to the high buffer capacity of the kaolin used in the present study. In unenhanced experiments, most of the nickel ions were in the form of adsorbed or precipitated phase due to the basic condition resulted from the high buffer capacity of the soil. Using EDTA as a complexing agent did not increase removal efficiency in comparison with unenhanced test after 5 days of treatment. The low ion mobility of Ni-EDTA complex led to less migration rate of the complex; therefore, increasing time of the EKR process could improve the removal efficiency significantly.

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