

# Development of a novel electro-dialysis based technique for lead removal from silty clay polluted soil

A.M.O. Mohamed\*

*Department of Civil Engineering, United Arab Emirates University,  
P.O. Box 17555, Al Ain, United Arab Emirates*

Received 21 May 2001; received in revised form 18 September 2001; accepted 6 November 2001

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## Abstract

In this study, a novel electro-dialysis based technique has been developed and used to treat a silty clay soil polluted by lead. The effect of chemical reagents, i.e. tap water at pH 4 (reagent 1) and sodium acetate at pH 5 (reagent 2), on enhancing electro-dialysis extraction of lead from the tested soil was examined. Specimens were prepared by mixing soil with 1000 ppm of lead and were compacted in the dialysate at predetermined dry density and moisture content. Then, specimens were subjected to a predetermined level of current. In the dialysate compartment, anions and cations were removed by charge transport in opposite directions to the anodic and cathodic cells. Meanwhile, in the anodic and cathodic cells, ion concentrations were increased. Thus, concentrated electrolyte streams were produced in alternating cells and cleaned soils were obtained in the dialysate. Both soil pH and lead concentrations were uniformly distributed within the compacted soil specimen during testing. Total lead removal of 80 and 92% was obtained for reagents 1 and 2, respectively. The high removal efficiency was attributed to the separation of electrode reactions from the soil and inclusion of ion selective membranes (ISM), which restrict the movement of counter charged species. © 2002 Elsevier Science B.V. All rights reserved.

*Keywords:* Ion selective membranes; Anions; Cations; Cathode; Anode; Dialysate; Current; Voltage; Adsorption; Speciation

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## 1. Introduction

Heavy metal pollution of soil and groundwater is a worldwide problem. Methods for cleaning up contaminated sites involve either long-term containment systems or treatment

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\* Tel.: +971-3-7051698; fax: +971-3-7623154.

E-mail address: mohamed.a@uaeu.ac.ae (A.M.O. Mohamed).

systems. Containment techniques are interim solutions because of the possibility of future leaks. As the contaminants in such instances are only confined or immobilized, these sites are often restricted for further use.

With in situ treatment systems, such as soil flushing, chemical treatment, and bioremediation, contaminants in fine-grained soils cannot be effectively removed because of low soil hydraulic conductivity. For more than 50 years, engineers have been using electro-kinetic techniques to stabilize soft soils. While electro-kinetic technique can be used to consolidate and increase the strength of soil, it has not been widely used in construction problems due to high costs and time constraints.

Electro-kinetic technique has been used for in situ remediation of heavy metals [1–4]. Field trials have shown considerable promise [1–3] but they have not been completely successful due to inadequate knowledge of the electro-kinetic phenomena associated with the soil-contaminant interactions [4–6]. Although it is impossible for electro-kinetic remediation to solve all contamination problems, with more understanding of the electro-kinetic phenomena and the soil-contaminant interactions, it could be a valuable tool for engineers in dealing with some of the today's complex contaminated sites.

Numerous laboratory studies have been performed to study the feasibility of using electro-kinetics for removing heavy metals from polluted soils [5–14]. Treatment studies performed by Hamed [15]; Hamed et al. [16]; Pamukcu and Whittle [17]; Acar et al. [13] using Georgia kaolinite clay have shown that over 70% of lead or cadmium could be either removed or accumulated within a small portion of the soil near the cathode. In order to mobilize the precipitated heavy metals near the cathode, a strong nitric acid solution (pH 3.1) was injected at the cathode. As a result, a reverse electro-osmotic flow occurring from the cathode to the anode was reported [12,14], hence complicating the electro-kinetic process. Also, in case of reversed flow, injecting a strong anionic complexing agent such as ethylene-diamine-tetra-acetate (EDTA) into the soil from the cathode enhanced electro-kinetic extraction via formation of soluble Pb-EDTA complexes [11].

The preceding discussion poses the following question. What is the relationship between the electro-osmotic flow and clay surface charges? For clay surfaces with negative charges, the direction of electro-osmotic flow is from the anode to the cathode. If the charges on the clay surfaces are changed, a reversal flow in the direction of the electro-osmotic flow will occur. For Georgia kaolinite clay, it is known that kaolinite clay minerals have zero point of charge (zpc) at pH 4.2, which is designated as pH<sub>zpc</sub>. When soil pH is less than pH<sub>zpc</sub>, positive surface charges are produced; while negative surface charges are produced when the soil pH is greater than pH<sub>zpc</sub> [4].

In electro-kinetic standard application, a pH gradient is developed within the soil specimen, with minimum pH of 2 at the anode and maximum pH of 12 or 13 at the cathode. Accordingly, the direction of electro-osmotic flow within the compacted soil specimen, which is placed between the anode and the cathode, is changed. For soil sections with pH less than pH<sub>zpc</sub>, the direction of electro-osmotic flow is toward the anode; while for soil sections with pH greater than pH<sub>zpc</sub>, the electro-osmotic flow direction is toward the cathode. Also, in case of injecting nitric acid solution (pH 3.1) at the cathode, soil pH becomes less than pH<sub>zpc</sub> and electro-osmotic flow direction is reversed.

In order to overcome problems associated with pH variations, clay mineral surface charge reversal, and electro-osmotic flow direction within the compacted soil specimen, an inno-

vative electro-dialytic approach is developed. The approach is similar to that previously reported by Mohamed et al. [18]. Review of the basic elements of electro-dialysis, experimental procedures and results of electro-dialysis extraction, performed on lead contaminated silty clay soil are discussed in this paper.

## 2. Electro-dialysis

Electro-dialysis is a membrane process in which dissolved ions are removed from soil pore fluid through membranes under the driving force of a dc electric field. Electro-dialysis membranes are ion exchange membranes and are of two types: cation exchange membranes that essentially allow only cations to pass through, and anion exchange membranes that allow only anions to pass through [19].

In standard electro-dialysis, a common form of which is shown in Fig. 1, flat membrane sheets are arranged to form parallel channels. The membranes are arranged so that cation and anion exchange membranes alternate, with electrodes at each end. The ion containing solution (as an example sodium chloride) flows through the channels. When an electric field is applied transverse to the membrane, cations such as  $\text{Na}^+$  pass through the cation exchange membranes and anions such as  $\text{Cl}^-$  pass through the anion exchange membranes. With reference to Fig. 1, this reduces the salt content in the channel formed by the left pair of the membranes, termed the dialysate channel, and increases in the channel formed by the right pair of membranes, termed the concentrate channel.

The salt solution is pumped through the dialysate and concentrate channels, with salt removed continuously along the length from the dialysate channel and transferred to the concentrate channel. A dialysate and concentrate channel with the associated membranes are termed a cell pair. A typical electro-dialysis stack may have 50–300 cell pairs between a single pair of electrodes, and a number of stacks may be used in series to achieve the desired level of salt removal.

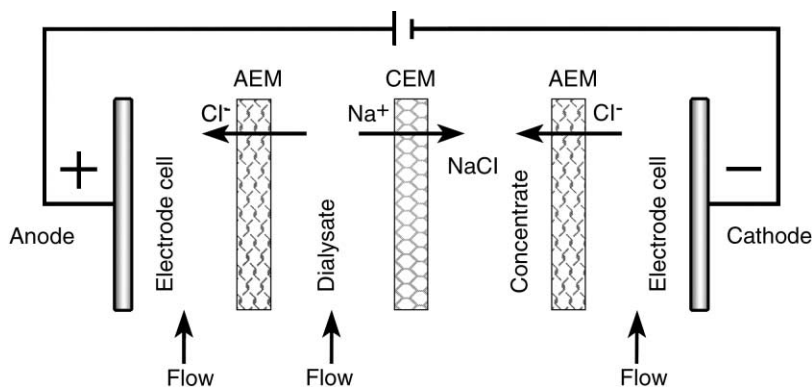


Fig. 1. Standard electro-dialysis demineralization process: AEM, anion exchange membrane; CEM, cation exchange membrane.

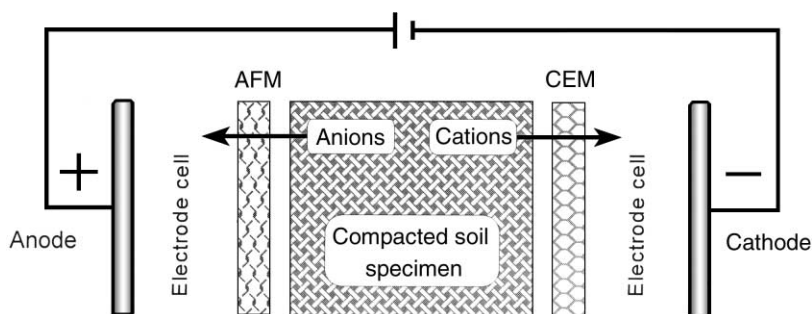


Fig. 2. Schematic showing electro-dialysis soil treatment: AEM, anion exchange membrane; CEM, cation exchange membrane.

Using the same principle, contaminated soil is placed in the dialysate as shown in Fig. 2. Since the generated hydrogen ions at the anode, due to water hydrolysis, are not allowed to cross the anion exchange membrane, acidic solution is formed in the anodic cell. In the same manner, the generated hydroxide ions at the cathode are prevented from crossing the cation exchange membrane; hence, an alkaline solution is formed in the cathodic cell. Both acidic and alkaline solutions are pumped through the anodic and cathodic cells with continuous metal ion removals from the contaminated soil specimen in the dialysate.

### 3. Test apparatus

The electro-dialysis test apparatus, shown in Fig. 3, is composed of the following components:

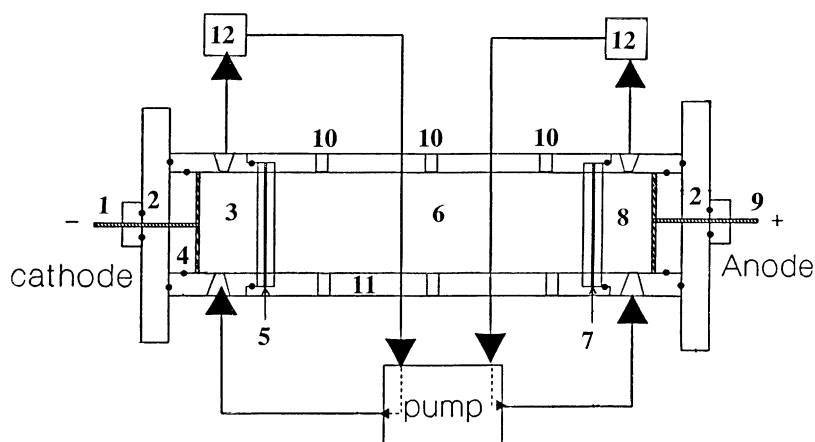


Fig. 3. Electro-dialysis testing apparatus: 1, cathode electrode; 2, lucite cap; 3, cathodic cell; 4, O-ring seal; 5, CSM; 6, contaminated soil (dialysate); 7, ASM; 8, anodic cell; 9, anode electrode; 10, feed solution inlets; 11, lucite tube (76 mm diameter); 12, reservoir.

- (1) Lead contaminated soil compartment (dialysate) made of lucite cylinder with 90 mm in diameter and 150 mm in length.
- (2) A cathodic compartment made of a lucite cylinder with 90 mm in diameter and 30 mm in length. It is used for isolating unfavorable electrodic reactions from contact with contaminated soil specimen and producing concentrated metal hydroxides.
- (3) An anodic compartment made of a lucite cylinder with 90 mm in diameter and 30 mm in length. It is used for preventing acid front movement into compacted soil and producing concentrated acidic solution.
- (4) A cation selective membrane (CSM) sandwiched between two spacers and placed next to the dialysate and before the cathodic compartment.
- (5) An anion selective membrane (ASM) sandwiched between two spacers and placed next to the dialysate and after the anodic compartment.
- (6) Stainless steel electrodes.
- (7) Power supply with current and voltage control options.
- (8) Electric pump for circulating electrolyte solutions in both the cathodic and anodic compartments and for preventing concentration gradient build up across the surface of the CSM and ASM.
- (9) Two tanks for collecting the circulated electrolyte solutions from both the anodic and cathodic compartments.

## 4. Material and methods

### 4.1. Soil material

A mixture of 80 wt.% sand and 20 wt.% illite was used in the laboratory experiments. This ratio was chosen to allow the tests to be conducted in a reasonable period of time. Since illitic clay mineral is frequently found in most natural soils, it was selected for this study. Another essential reason for the selection of this clay mineral was attributed to its high acid buffering capability [4,20]. Furthermore, if it is demonstrated that electro-dialysis extraction of contaminants from illitic soil is effective, the success rate with any other soils found in the field will be very high.

The selected illitic soil is produced by Canada Brick Limited, under the patent name Domtar Sealbond. The clay soil is derived from a marine shale belonging to the Ordovician sedimentary rock formation. As determined from the X-ray diffraction analysis, the Domtar Sealbond consists not only of illite mineral, but also of varying amounts of chlorite, quartz, feldspar, and calcite. Some soil organic matter (0.9 wt.%) and amorphous contents (6.8 wt.%) were also detected in the clay soil. The carbonate content of 14.7% is considered to be significant and probably accounts for the high soil pH value of 8.7. The sand is washed, quarry sand distributed under the patent name Sakrete.

The geotechnical properties of the tested material are shown in Table 1. The grain size of the sand/illite mixture, shown in Fig. 4, is composed of 81% sand, 10.5% silt and 8.5% clay and the mixture is classified as silty clay soil. The optimum moisture content and the maximum dry density of this mixture were determined based on the Standard Proctor

Table 1  
Geotechnical properties of tested soil materials

Properties	Illite	Illite/sand mixture
Liquid limit, ASTM D423 (%)	32.1	21.8
Plastic limit, ASTM D424 (%)	17.9	11.9
Specific gravity, ASTM D854	2.74	2.69
Particle size, ASTM D422 (%)		
Gravel	0	0
Sand	14.6	81
Silt	40.9	10.5
Clay	44.5	8.5
Hydraulic conductivity (m/s) @ 1% above optimum	$2.1 \times 10^{-10}$	$1.6 \times 10^{-7}$
Maximum dry density, ASTM D698 ( $\text{Mg/m}^3$ )	1.85	2.02
Optimum moisture content, ASTM D698 (wt.%)	15.6	10.2

test method [22]. The maximum dry density was found to be  $2.02 \text{ Mg/m}^3$  and the optimum moisture content was 10.2% as shown in Fig. 5. Based on compaction test results, a moisture content of 11% and a dry density of  $1.98 \text{ Mg/m}^3$  were used for placing the soil mixture into the electro-dialysis cell. The measured hydraulic conductivity of the tested soil was  $1.6 \times 10^{-7} \text{ m/s}$ .

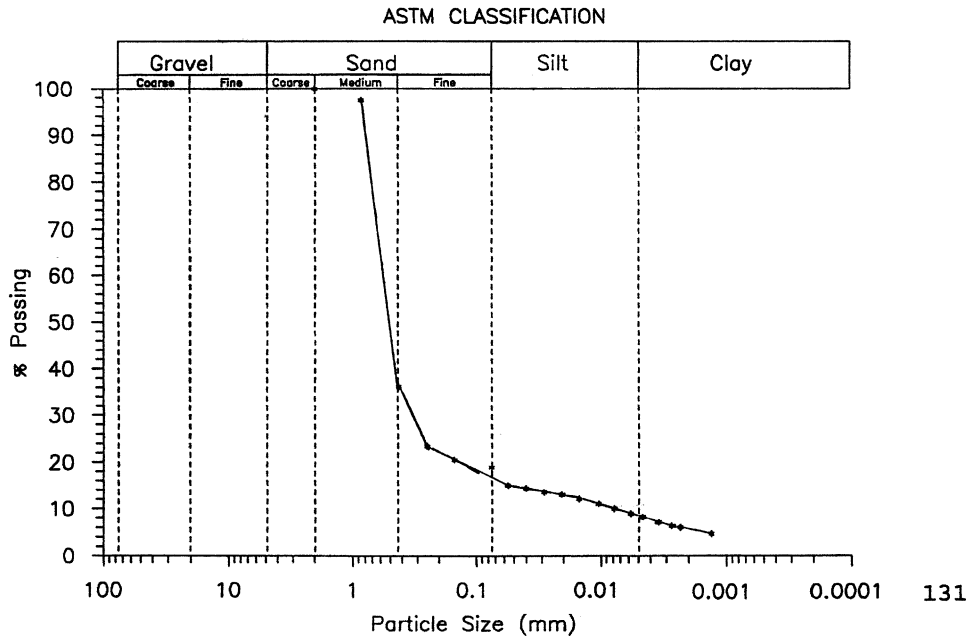


Fig. 4. Particle size distribution of tested sand/illite mixture.

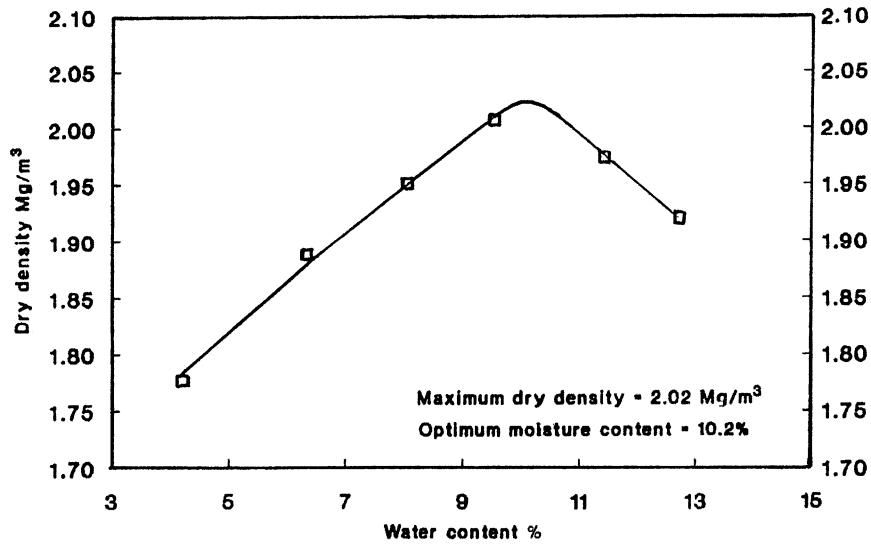


Fig. 5. Compaction characteristics of the tested sand/illite mixture.

#### 4.2. Reagents

The presence of carbonate (14.7%) in the tested soil contributes significantly to the retention capability of the soil as demonstrated in Fig. 6 [4,20,21]. At low pH value (pH < 4), dissolution of carbonates occurs, and hence, retention of lead by carbonates will be minimal. When the pH of the soil solution is greater than 4, lead is retained in the illitic based soils by hydroxide, carbonate and as exchangeable cations. As the soil pH is

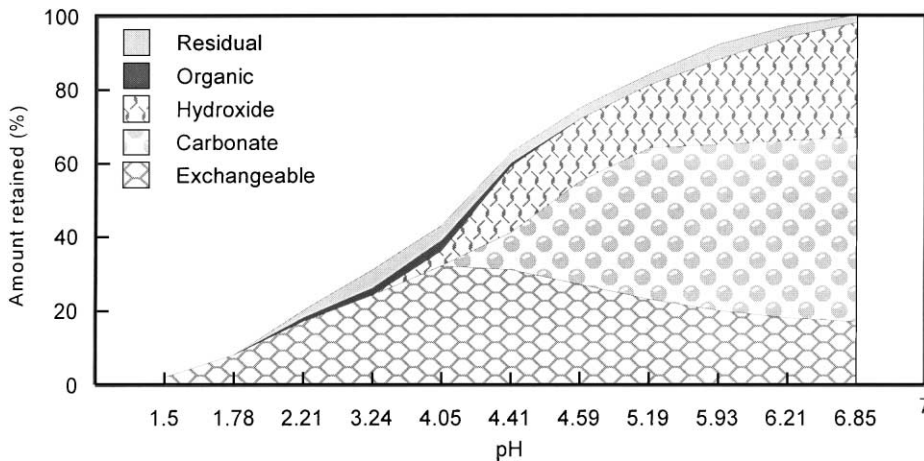


Fig. 6. Amounts of lead retained by the various soil constituents as a function of soil pH.

reduced below precipitation pH of lead, one expects that the dominant mechanism for lead retention would be via sorption processes.

Various chemical reagents have generally been used for extracting lead associated with different soil constituents and phases [4,20,21]. To release the lead precipitated or co-precipitated as natural carbonates, an acidic solution can be applied. The generally used extracting agent is acidified acetate. Therefore, in this study, two reagents were used. The first reagent is tap water acidified with nitric acid adjusted to pH value of 4 while the second reagent is sodium acetate solution adjusted to pH value of 5. These reagents were used to saturate the tested soil and decrease its acid buffering capacity via carbonate dissolution.

#### 4.3. Sample preparation

The sand/illite soil was mixed with  $\text{Pb}(\text{NO}_3)_2$  solution of 1000 ppm concentration of  $\text{Pb}^{2+}$  ions up to a moisture content of 11 wt.%. The soil column was divided into six sections each measuring 25 mm in length. In each section, the soil was statically compacted to a designated wet density of  $2.19 \text{ Mg/m}^3$ . After compaction, filter papers, ASM and CSM, anodic and cathodic compartments, stainless steel electrodes, and lucite caps were placed and assembled together as shown in Fig. 3.

#### 4.4. Test procedures

The compacted soil specimen was placed in the dialysate compartment and saturated by allowing the reagents to seep through it, under a hydraulic gradient of 1, from three locations as shown in Fig. 3. Both anodic and cathodic cells were filled with brine solutions having conductivities of  $15 \text{ mS/cm}$  for starting the process. After saturating soil specimen, cathode and anode electrodes were connected to a dc power supply. Electrolyte solutions in both anodic and cathodic compartments were circulated at a rate of  $80 \text{ ml/min}$  for preventing build up of concentration gradient across the ion selective membranes (ISM).

The variations of both current density and voltage as a function of time during the electro-dialysis treatment are shown in Fig. 7. It can be seen that the current density and voltage were almost constant throughout the tests. The current density, CD, is defined as the current passing through a unit area of membrane ( $\text{A/m}^2$ ). The ability of the solution to carry current is a function of the normality,  $N$ , of the solution. The ratio  $\text{CD}/N$  provides an important characterization of the system. For this study, the calculated  $\text{CD}/N$  values ( $(\text{A/m}^2)/(\text{g eq./l})$ ), for reagents 1 and 2, are  $67.57 (0.05 \times 10^{-3} \times 10^4/0.0074)$  and  $108.11 (0.08 \times 10^{-3} \times 10^4/0.0074)$ , respectively. It should be noted that these calculated values are far below the maximum reported  $\text{CD}/N$  values of 6000 [23]. A large value can result in too much current for ions available in solution. This can lead to concentration polarization (i.e. regions of low ion concentration) which will result in an increase in resistance and a decrease in system efficiency.

During experiments, the consumed voltage and current were recorded as a function of time. For anodic and cathodic cells, solution pH and conductivity were measured at different time intervals. Also, with time, water samples were collected for chemical analysis. The concentration of various cations, i.e.  $\text{Pb}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{K}^+$  and  $\text{Na}^+$ , in each collected water sample, were measured using a GBC double beam atomic adsorption spectrophotometer.



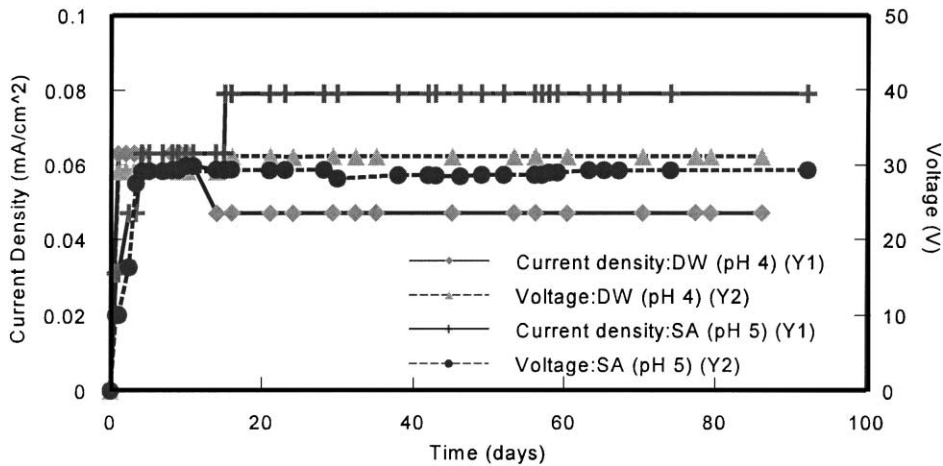


Fig. 7. Current density and voltage variations as a function of time during experiments.

After testing, the soil specimen was extruded and sliced into sections for measuring the concentration of various cations in both liquid and solid phases. The soluble concentrations were measured according to Lavkulich [24] and the total concentrations were measured according to EPA method 3050 [25].

## 5. Results and discussion

Variations of relative lead concentrations ( $C/C_0$ ,  $C$  represents lead concentration variation with time and  $C_0$  the initial lead concentration in the compacted soil specimen in dialysate) in the cathodic cell as function of time and used chemical reagents are shown in Fig. 8. For tap water reagent, the shape of the curve indicates the continuous increase of lead concentration in the cathodic cell with time. However, for sodium acetate reagent, the curve has three distinctive zones. At the end of each zone, lead removal starts to decrease and system efficiency is reduced. To increase lead removals, concentrated electrolyte solution in the cathodic cell was removed and new fresh brine solution was added.

For both chemical reagents, as time increases, relative lead concentrations are increased. At early times, the use of sodium acetate has contributed to 10–30% increase of lead removal than tap water reagent. At the end of the test, lead removal of 92 and 80% was obtained for sodium acetate and tap water reagents, respectively.

Soil pH variations with distance from the cathode, for both tap water and sodium acetate reagents, are shown in Fig. 9. Average soil pH of 7.5 and 6.5 were determined for tap water and sodium acetate reagents, respectively. Unlike the standard electro-kinetic technique, the electro-dialysis extraction process maintains uniform pH variations within the compacted soil specimen as shown in Fig. 9. Hence, with the use of electro-dialysis extraction process, the following problems associated with the standard electro-kinetic technique are eliminated: (1) soil acidification near the anode; (2) clay surface charge reversal in the

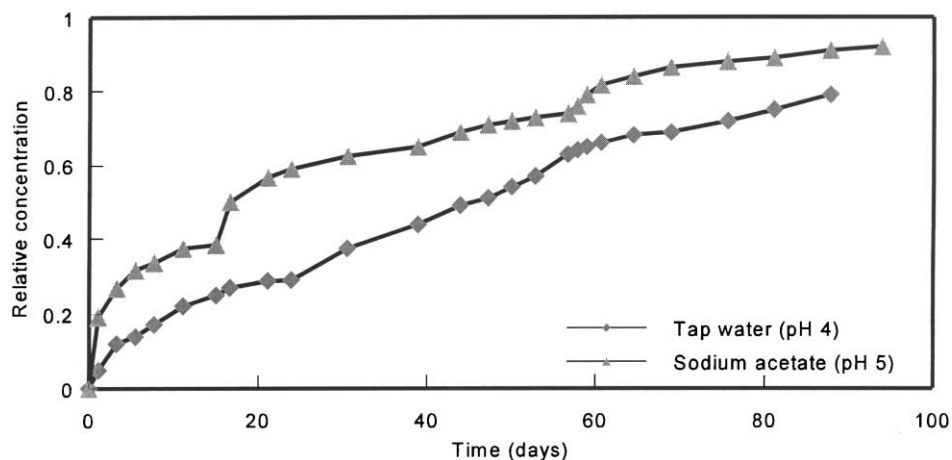


Fig. 8. Relative lead concentrations in the cathodic cell as a function of time during the application of electro-dialysis treatment.

compacted soil; (3) production of alkaline soil near the cathode; (4) metal ion precipitation in the compacted soil specimen near the cathode.

Total lead concentrations with distance from the cathode, for both tap water and sodium acetate reagents, are shown in Fig. 10. For tap water reagent, after a testing period of 90 days, the remaining total lead concentrations in the compacted specimen varies between 20 mg/l of dry soil near the anodic compartment and 30 mg/l of dry soil near the cathodic compartment that is consistent with charge transport theory. The remaining lead concentration in the compacted specimen is below the required background lead concentration in soils by various regulatory agencies. For sodium acetate reagent, the remaining total lead concentration in

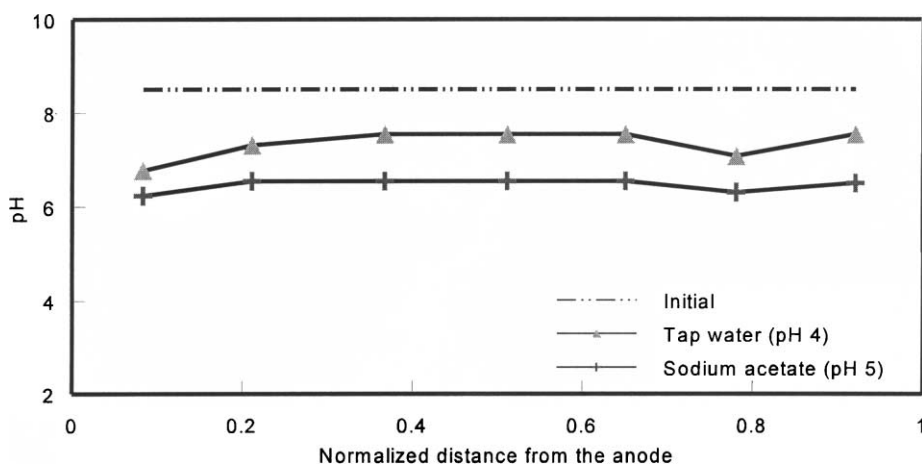


Fig. 9. Soil pH variations within silty clay soil after the application of the electro-dialysis treatment.

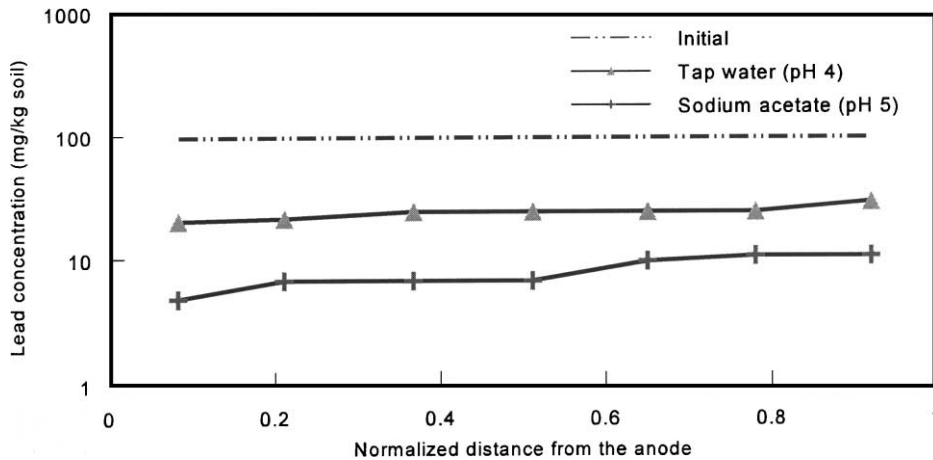


Fig. 10. Lead concentration variations within silty clay soil after the application of the electro-dialysis treatment.

the compacted soil specimen was less than 10 mg/l of dry soil with high concentrations near the cathodic compartment.

## 6. Analysis of the experimental results

### 6.1. Current efficiency

The current required to operate an electro-dialysis unit can be calculated by using Faraday's law, which indicates that to transfer 1 g eq. of a substance by electrical force, a single cell will require 1 F of electrical current. In  $n$  cells, the required current is given by the following equation:

$$I = \frac{FQN E_1}{n E_2} \quad (1)$$

where  $I$  is current (A),  $F$  the Faraday's constant (96,487 C/g eq.) or (A s/g eq.),  $Q$  the feed flow rate (l/s),  $N$  the normality of solution (g eq./l),  $n$  the number of cells between electrodes,  $E_1$  the removal efficiency (fraction), and  $E_2$  is the current efficiency (fraction).

The current efficiency,  $E_2$ , represents the number of gram-equivalents of product actually produced per Faraday applied. Current efficiency is adversely affected by inefficient selectivity of the electro-dialysis membranes and diffusional losses due to concentration gradients.

Based on Eq. (1), the current efficiency can be calculated as follows:

- (1) Faraday's Constant ( $F$ ):  $F = 96,487 \text{ (A s/g eq.)} / 86,400 \text{ (s/day)} = 1.1167 \text{ A day/g eq.}$
- (2) Normality ( $N$ ): It should be noted that during treatment, lead concentration in the dialysate decreases as a function of time. The rate of decrease varies depending on

reagent used as shown in Fig. 8. For sake of simplicity, one considers a constant value equal to the initial lead concentration.

$$\text{Molecular weight of Pb(NO}_3)_2 = 207.2 + 14.0 + 3(16) = 269.2 \text{ g/g mol}$$

$$\begin{aligned} \text{Equivalent weight of Pb(NO}_3)_2 &= \text{Molecular weight}/(\text{g eq.}/\text{g mol}) \\ &= 269.2/2 = 134.6 \text{ g/g eq.} \end{aligned}$$

$$\text{Concentration} = 1000 \text{ mg/l} = 1 \text{ g/l}$$

$$\text{Normality} = \frac{1(\text{g/l})}{134.6(\text{g/g eq.})} = 0.0074 \text{ g eq./l}$$

- (3) Current ( $I$ ): For the case of distilled water reagent, the average current is  $4 \times 10^{-3}$  A while, for the case of sodium acetate reagent, the average current is  $5 \times 10^{-3}$  A.
- (4) Feed flow rate ( $Q$ ): Since the material used is a compacted soil and lead exists in both soil pore fluid and different soil constituents, as shown in Fig. 6, the feed flow rate will be governed by the hydraulic conductivity of the compacted soil. The feed flow rate is calculated as follows:

$$\begin{aligned} Q = vA = KiA &= 1.6 \times 10^{-7}(\text{m/s}) \times 86,400(\text{s/day}) \times 1 \times \frac{3.14 \times (0.15)^2}{4(\text{m}^2)} \\ &= 2.442 \times 10^{-4}(\text{m}^3/\text{day}) \times 10^3(\text{l/day}) = 0.2442 \text{ (l/day)} \end{aligned}$$

- (5) Number of cells between electrodes ( $n$ ): The developed electro-dialysis apparatus is characterized as one cell compartment. Therefore,  $n$  will be assigned a value of 1.
- (6) Removal efficiency ( $E_1$ ): Based on the experimental results shown in Figs. 8 and 10, removal efficiency values of 0.8 and 0.92 will be considered for distilled water and sodium acetate reagents, respectively.
- (7) Current efficiency ( $E_2$ ): The current efficiency can be calculated by using Eq. (1) and the calculated parameters which are described in items 1–6.

$$E_2 = \frac{FQNE_1}{In}$$

- (a) For distilled water reagent, at pH 4,

$$\begin{aligned} E_2 &= \frac{1.1167(\text{A day/g eq.}) \times 0.2442(\text{l/day}) \times 0.0074(\text{g eq./l}) \times 0.8}{4 \times 10^{-3}(\text{A}) \times 1} \\ &= 0.4036 \end{aligned}$$

- (b) For sodium acetate reagent, at pH 5,

$$\begin{aligned} E_2 &= \frac{1.1167(\text{A day/g eq.}) \times 0.2442(\text{l/day}) \times 0.0074(\text{g eq./l}) \times 0.92}{5 \times 10^{-3}(\text{A}) \times 1} \\ &= 0.3713 \end{aligned}$$

The calculated results reveal that the current efficiency in the developed system is lower for the sodium acetate reagent (0.37) than for distilled water reagent (0.4), which are consistent with the experimental results shown in Fig. 8. However, the reported higher removal efficiency for sodium acetate reagent than distilled water reagent could be attributed to the effect of sodium acetate on carbonate dissolution, hence increasing the lead concentration in soil pore fluid.

## 7. Summary and concluding remarks

In this study, a novel electro-dialysis based technique has been developed and used to treat a silty clay soil polluted by lead. The effect of chemical reagents, on enhancing electro-dialysis extraction of lead from the tested soil, was examined. The experimental results have shown that the developed technique is very effective in removing lead from tested soil. Both soil pH and lead concentrations were uniformly distributed within the compacted soil specimen during testing. A total lead removal of 80 and 92% was obtained for tap water at pH 4 (reagent 1) and sodium acetate at pH 5 (reagent 2), respectively. In the dialysate compartment, anions and cations were removed by charge transport in opposite directions to the anodic and cathodic cells. Meanwhile, in the anodic and cathodic cells, ion concentrations were increased. Thus, concentrated electrolyte streams were produced in alternating cells and cleaned soils were obtained in the dialysate. The high removal efficiency was attributed to the separation of electrode reactions from the soil and inclusion of ISM, which restrict the movement of counter charged species.

Furthermore, the experimental results indicated that higher removal efficiency was obtained for reagent 2 than reagent 1, however, the calculated current efficiency was 0.4 for reagent 1 and 0.37 for reagent 2. This indicates that the used chemical reagents played a major role during the treatment process.

Last but not the least, as we know, movement of charged species in compacted soil specimen depends, in large part, on the distribution of species between soil pore fluid and sorbed phases. The extent to which species are retained by soil solids depends on the nature of sorptive surfaces, ionic concentration and solution pH. In electro-dialysis process, charge transport is the major transport mechanism. Transient soil pH conditions, that cause problems during the application of the standard electro-kinetic technique, are eliminated via the developed electro-dialysis based process. Before attempting to apply this technology in the field, further developments are needed to better understand the transient chemical and physical processes within the system.

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