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EDTA-ENHANCED ELECTROKINETIC EXTRACTION OF Pb (II) AND Zn (II) FROM MILLPOND SLUDGE

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ABSTRACT : A laboratory investigation was carried out using electrokinetics to remove heavy metal contaminants from residual wastes. Millpond sludge containing high levels of pb (11) and Zn (11) was used as the contaminated source. An electric gradient was applied in a specially constructed eletrokinetic decontamination cell, to generate a low PH solution by electrode reactions. Electroosmosis, the inheren, flow process of electrokinetics, carries the low pH solution through the residual wastes. This low PH solution tends to reduce the soil PH, which ultimately dissolves the heavy metals in soils. Different concentrations of EDTA were used as an influent fluid in the electrode chambers. Experiments were conducted under a constant electric voltage of 30 volts. As much as 76% of pb and 79% of Zn was removed with 0.125M EDTA within 168 hours of the decontamination process.

INTRODUCTION

Electrokinetic soil processing is an innovative in situ technique to remove contaminants from soils and ground water, This process is an alternative to conventional decontamination processes with significant economical and technical advantage. Unite States Environmental Protection Agency (USEPA) has recently designated electrokinetic method as a viable in-situ process and interested parties are attempting to apply this method at contaminated sites which have inherently low permeability soils and otherwise difficult but not impossible to remediate. Electrokinetic flows occur when an electric gradient is applied on a soil-fluid contaminant system due to existence of the diffuse double layer at the soil particle surface-pore fluid interface. There electrokinetic flows include the flow of fluid, which is known as electroosmosis, electric current, dissolved undissolved contaminants and fine praticles. The detailed description of these flow processes and the associated teatures generated by electrochemical reactions are given by Yeung et al. (1997); Acar and Hamed (1992): Datla and Yeung (1994); Eykholt and Daniel (1994): Yeung (1994): Yeung and Datla (1995); and Acar et at (1993).

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Potential applications of electrokinetics in geotechnical and geoenvironmental engineering are described elaborately by Acar et al. (1993). However, presence of significant amount of heavy molecular weight organics (humus substances) within the soil pores may reduce the mobility of the heavy metals due to the formation of insoluble organometallic compounds.. There has been some research in removing heavy metals and low concentration organic matter from soil by the electrokinetic method (Pamukcu et al. 1991: and Shapiro and Probstein, 1993)

This paper concentrates on the use of Ethylenediaminetetraacetic acid (EDTA) to extract contaminants from polluted soils by electrokinetics. An attempt has been made to explore the effect of various concentration of EDTA in the removal of selected heavy metals such as Zinc and Lead from polluted fine-grained soil with a significant organic matter content. A foundry millpond sludge from North East Ohio was chosen as a high organic metter containing matrix. It is a fine grained soil with low permeability ($\approx 10^{-7}$ cm/sec) which satisfies the basic conditions for electrokinetic treatment. Upon burning at 400°C, it turns into reddish brown silty material. Table I lists the various typical characteristics of the millpond sludge.

Table 1. Typical Characteristics of Millpond Aludge

Parameters	Concentrations	Parameters	Concentrations
lead (ρb)	1,600 mg/kg	Water content	72% (average)
Zinc (Zn)	17.00 mg/kg	Initial pH	6.5
Organic content	25%	Appearance	Black, fine-grained
Plasticity	Low, primarily silty	Specific Gravity	2.33

ELECTROKINETIC EXTRACTIONS

The extraction technique, variably called electrokinetic remediation, electroremediation, electroreclamation, electrorestoration, electrochemical soil processing or electrochemical decontamination, uses low level direct current on order of mA/cm^2 between the electrodes or the electric potential difference on the order of a few volts per centimeter across the electrodes placed at the end of the soil sample. When an electric field is imposed to a wet soil mass, positive ions are moved toward the cathode (the negative electrode) and the negative ions toward the anode (positive electrode). Because of the isomorphous substitution and the presence of broken bonds is the soil structures, excess mobile cations are required to balance the negative fixed charges on the soil particle surfaces. Therefore, mobile cations exert more momentum to the pore fluid than do mobile anions. As a result there is net movement of fluid relative to soil particles under the influence of imposed electric potential gradient which is called electroosmosis (Casagrande, 1983). Unlike water flow under pressure, electroosmosis depends on the electric current through the soil, the flow resistance of soil, and the frictional drag exerted by the migrating ions the water molecule; and this flow originates at the electric double layer of the soil pores. The electrokinetic flow rate q_{eo} in a porous medium of length L, porosity n, area A and degree of saturation S, may be presented by the following equation (Khan, 1991):

$$q_{eo} = \frac{\psi d \varepsilon_0 D}{\eta} I_s \left(\frac{R_s}{L}\right) (nAS) \qquad (1)$$

Where, ψd is the potential at the slipping plane, ε_0 is the permeability of free space D is the dielectric constant of the pore fluid. η is the pore water viscosity, Is the current carried by surface conductance and Rs is the surface resistance of the porous medium i.e. soil.

The values of hydraulic conductivity of different soils can differ by orders of magnitude; however, those of coefficients of electro-osmotic conductivity are generally between 1.0×10^{-5} and 10.0×10^{-5} $cm^2/(V.s)$ and are relatively independent of soil type (Casagrande, 1983). Thus, an electric gradient is much more effective driving force than a hydraulic gradient for moving fluid through fine-grained soils of low hydraulic conductivity.

When the electrokinetic technique is applied without conditioning the process fluid at the electrodes (unenhanced electrokinetic remediation), the applied electric current leads to electrolysis reactions at the electrodes, generating an acidic medium at the anode and alkaline medium at the cathode (Hicks and Tondorf 1994). The acid medium (H_3O+ ions) generated at the anode advances through the soil toward the cathode by ion migration due to electrical gradient, pore fluid flow due to any externally applied or internally generated hydraulic gradient and diffusion due to the chemical gradients developed in the system. At the acid buffer capacity of soil is low, acid medium moves through the soil during electrokinetic decontamination and lowers the system PH. Since most heave metals are soluble in acidic environment, this lowering of pH promotes description of heavy metals from the soil and solubilization of metal ions. Ions in dissolved phase can be removed effectively by the combined actions of electroosmosis and ion migration, However if organic matters are present within the soil pores, the metals may not be easily extracted by a low PH solution die to metal-organic matter bonding. Under this circumstances, enhanced clectrokinetic remediation could be necessary.

METAL-SOIL INTERACTION

The aqueous concentration of an inorganic contaminants in contact with a soil can be predicted from appropriate thermodynamic measurements. At high concentrations, cationic metals will form insoluble hydroxide, carbonate or sulfide precipitates (Stumm and Morgan, 1981). The solubility is a function of a concentration of metal and precipitant, the solubility pH and the concentrations of other interaction metal ions and ligands.

Often trace elements are present in the solid phase as a result of adsorption to components of the soil, The most important factor in controlling the partitioning of a metal to soil is the solution PH. As the pH increases, the concentration of soluble metal decreases (Shuman, 1975; Harter, 1983; and Elliot et al. 1986). For anions, such as chromate, a reverse trend is found in which adsorption is maximum at low pH and decreases with increasing pH.

The reaction of metal ions with solid phases has a dependency on pH resulting from surface chemistry of soil materials. Protons can react with anions capable of forming precipitates, to compete with the reaction of metals, thus increasing solubility, Soils have pH-dependent or variable charge associated with reaction of protons with oxide and hydroxide minerals and with certain functional group of humic substances. Protons and metal ions compete with each other for available surface adsorption/ binding sites, $\equiv S\text{-}OH^{\circ}$, on a soil. The following equation represents the reaction for a divalent metal ion,

$$M^{2+} \cong S\text{-}OH^{\circ}+M^{2+} \Leftrightarrow \cong S^{-}OM^{2+}+H^{+} \qquad (2)$$

Where $\cong S\text{-}OM^{2+}$ is the adsorped metal. For homogenous surface sites of fixed total concetration, the concentration of sorbed metal can be determined using the following equation (Bachelor, 1998)

$$\cong S^{-}OM^{2+}= \frac{S_t\frac{K_s}{[H^+]}[M^{2+}]}{1+\frac{K_s}{[H^+]}[M^{2+}]} \qquad (3)$$

Where S_t= total concentration of surface adsorption sites. k_s= equilibrium formation coefficient for sorption.

METAL-ORGANIC MATTER INTERACTION

The organic phase of the soil may be humus and nonhumus. The high molecular weight humus organic substances have a high affinity for metals and form water insoluble metal complexes. Nonhumus substances of low molecular weight, such as organic acid and bases, are relatively soluble when complexed with metals.

Metal ions are bonded to organic molecules by complexation and chelation. A complex is formed when an electron rich atom in an organic molecule shares a pair of electrons with a metal ion having empty outer shell resulting in a coordination compound. Chelation occurs when two or more coordination positions are occupied by two or more donor groups from the same orgainc molecule. The resulting organometallic ring gives the complex a high degree of stability. The presence of humus substances in soils would therefore greatly reduce the mobility of metals. The metals, so bonded, can not be easily ionized by an acidic environment.

The metal organic matter bonded within the soil pores may by broken by the action of a sequestering ligand. EDTA is such a ligand that was used in this study for ρb and Zn extraction. The action of metal and ligand has been discussed in the next section.

Metal-EDTA Complexation

EDTA is a chelating agent that is readily avilable and environmentally benign and does not interact with soils. Many of the chelating agents other than EDTA are ionic and can. in principle, be introduced into the soil by ionic migration, Extensive evaluations of numerous chelating agents (Allen and Chen, 1993) have shown that EDTA is an excellent solubilizing agent for many metals including lead and zinc. It is of interest that EDTA has been used medically to promote removal of lead from the human body and also as an additive to render floor polishes with zinc binders amenable to detergent washing (Joseph et at. 1997)

EDTA is a tetraprotic acid abbreviated as H_4Y, where Y denotes the ethylenediaminetraacetate ion $EDTA^4$. It is slightly soluble water and the our stepwise dissociation constants of the parent acid to yield H_3Y^-, H_2Y^{2-}, HY^{3-} and Y^{4-} ions are 1.00×10^{-2}, 2.16×10^{-3}. 6.92×10^{-7} and 5.50×10^{-11}, respectively (Allen and Chen, 1993). It implies that H_2Y^{2-}, and HY^{3-} Species are major EDTA anions adsorbed (Huang et at. 1988). Each $EDTA^4$ ion can attach to a metal ion at six different sites since each of four acetate groups and the two nitrogen atoms have free electron pairs available for coordinate bond formation as shown in Fig. 1.

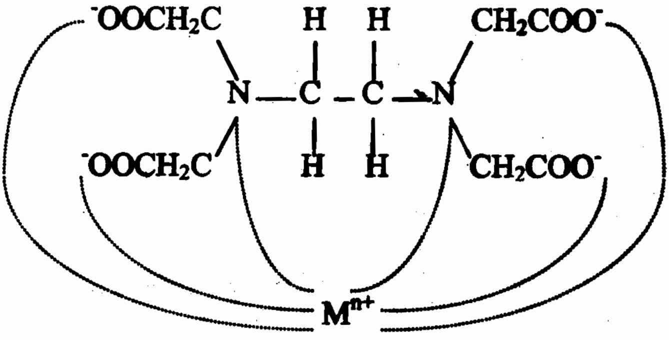

Fig 1. Configuration of Metal-EDTA Complexes (Yeung et. at. 1996)

Unless the pH is very high, the EDTA will not be completely deprotanated. In fact, this is the reason ofr the high solubility of metal-EDTA complexes (Snoeyink and Jenkins, 1980) The complexation of metals by EDTA is dependent on pH. With a metal ion M, it can form a complex MY, a protonated complex MHY, a hydro complex $MY(OH)_n$

and a mixed complex of the form MYX, where X is a unidentate ligand. The complexation reactions involved are

$$M^{n+}Y^{4-} \Leftrightarrow MY^{n-4} \qquad (4a)$$

$$M^{n+}H^{+}+Y^{4-} \Leftrightarrow MHY^{n-3} \qquad (4b)$$

$$M^{n+}OH^{-}+Y^{4-} \Leftrightarrow MY(OH)^{n-5} \qquad (4c)$$

And the solubility constants of these reactions are defined by,

$$K_{MY} = \frac{[MY^{n-4}]}{[M^{n+}][Y^{4-}]} \qquad (5a)$$

$$K_{MHY} = \frac{[MHY^{n-3}]}{[M^{n+}][H^{+}][Y^{4-}]} \qquad (5b)$$

$$K_{MY}\ ({}_{O}H) = \frac{MY(OH)^{n-5}}{[M^{n+}][OH^{-}][Y^{4-}]} \qquad (5c)$$

Where the unit of concentration as indicated by [-] is mol/L. The $\log K_{MY}$, $\log K_{MHY}$ and $\log K_{mY(OH)}$ for different metals are presented in Table 2 (Ringbom, 1963)

Table 2. Stability Constants for Metal-EDTA Complexes.

Element	Cation	Logk_{MY}	LogK_{MHA}	$\mathrm{LogK}_{MY(OH)}$
lead	pb^{+2}	18.0	2.8	....
Zinc	Zn^{2+}	16.5	3.0	1.95
Cadmium	Cd^{+2}	16.5	2.9	....
Calcium	Ca^{2+}	10.7	3.1	....
Chromium	Cr^{3+}	23.0	2.3	29.6
Copper	Cu^{2+}	18.8	3.0	....
Iron	Fe^{2+}	14.3	2.8	....
	Fe^{3+}	25.1	1.4	
Magnesium	Mg^{2+}	8.7	3.9	....
Manganere	Mn^{2+}	14.0	3.1	....
Mercury	Hg^{2+}	21.8	3.1	....
Nickel	Ni^{2+}	18.6	3.2	3.2

Release of Metals from Soils by EDTA.

Yeung et al. 1996) studied the basic ρb-EDTA complexation reactions and their influence on electrokinetic extraction process. Their main focus was on EDTA enhanced electrokinetc extraction of lead from Milwhite and Georgia kaolinite and the acid/base buffer and sorption capacities of these soil minerals. Their study revealed that more than 90% of lead was migrated toward the cathode with a lower voltage applied across the sample within a shorter duration of treatment. Allen and Chen (1993) investigated the extraction of lead from the contaminated New Jersey and Delaware soils with EDTA. The investigation found almost 100% extraction of lead from New Jersey soil at a 10^{-3} M concentration of EDTA and at 10^{-3} M or lower concentration of EDTA, the recovery of lead that had been added to the Delaware soil was greater than that of New Jersey soil that had been previously contaminated at level of pH-4. 3±.0.1. Huang et al. (1988) found that the removal of Zn (11) from solids is independent of types of solids. The addition of EDTA resulted in a shift of maximum Zn (11) adsorption to the acidic pH range, and reduction of zeta potential and overall Zn (11) removal in the presence of EDTA was significantly reduced at alkaline pH range and slightly enhanced in the acidic pH range. Joseph et al. (1997) investigated the feasibility of mobilizing precipitate heavy metals from soil by ionic migration using EDTA. They used EDTA solution to catholyte where it solubilizes the precipitated metals, The resulting complexes are then transported to the anode. The removal efficiencies were found to be very close to 100% for Zn and ρb. Reed et al. (1996) investigated the flushing of a ρb (II) synthetically contaminated sandy loam using 0.1 N HCI, 0.01 M EDTA and 1 M $CaCL_2$ in the continuous-flow (column) mode. Initial concentration of pb ranged from 500 to 600 mg/kg of dry soil. Their study found that ρb removal efficiencies (and final soil ρb concentrations) for HCL was about 85% (≈77mg/kg ρb). for EDTA almost 100% (≈O mg/kg ρb) and for $CaCL_2$ 78% (≈135 mg/kg ρb) HCL and $CaCL_2$ could not reduce the ρb to background levels (≈25 mg/kg) for the synthetically contaminated soil. This brings into doubt their effectiveness in remediating actual contaminated soils where metal retention would be much stronger. While EDTA removed ρb substantially less than the background levels. The final pH for HCI was near 1.0 while the final solution pHs for EDTA and $CaCL_2$ ranged from 4.85 to 5.20.

ELECTROKINETIC EXPERIMENTS

Apparatus: The removal of primarily two heavy metals ρb and Zn from millpond sludge was investigated in a specially constructed apparatus, as shown in Fig 2. The apparatus was constructed with a 1.25 cm thick clear plexiglas. The outer dimension of the rectangular chamber was 10 cm ×7.5 cm ×6.5 cm. The circular chamber containing soil sample was 7.5 cm long and 3.5 cm in internal diameter. The graphite electrodes were attached with the banana socket through the wall and fixed with epoxy and carbon glue. The end chambers (Electrode chambers) were provided with acid resistance CPC quick connection inlet/outlet valves and gas release ports to release gases produced by the electrode reactions or to collect water samples from the electrode chambers. A direct current (DC) power source was used to provide a constant voltage source during the experiment. Details of an eleatrokinetic apparatus are available in Khan (1991)

Sample Preparation : The samples were perpared by consolidation using consolidation apparatus. The detail description of the consolidation apparatus is available in Khan (1991). Millpond sludge was mixed with water and a slurry was made. The slurry was then poured into the sample and guide tube assembly of apparatus. The top of the apparatus was assembled and a pressure of 20 psi was applied for 24 hours . Excess water of the slurry was expelled during consolidation the slurry. The final dimension of the soil sample was 7.5 cm in length and 3.5 in diameter.

Sampling : Water samples from the electrode chambers were collected at certain time intervals. The pH of the water samples was measured. At the end of the experiment, the soil sample was taken out of the sample tube and divided into five segments. The pH and the metal concentrations in these segments were measured. Perkin Elmer Atomic Absorption Spectrophotometer (AAS) of model M3100 was used to determime the metal concentrations.

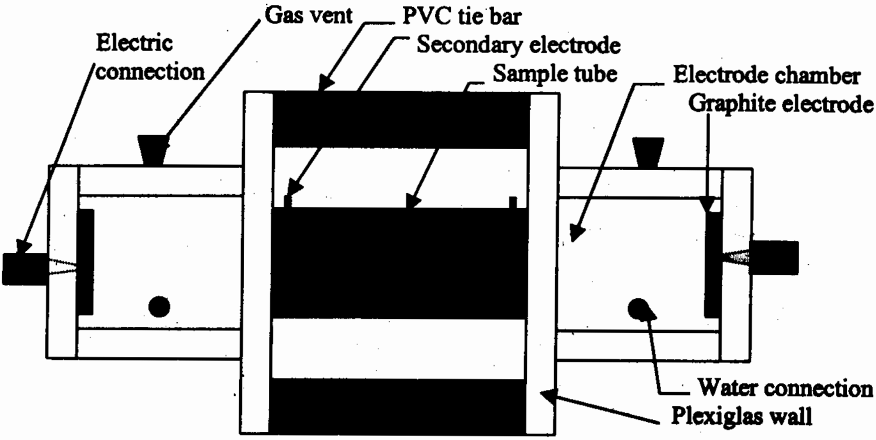

Fig 2. Schematic diagram of electrokinetic apparatus.

RESULTS AND ANALYSIS

Initial experiments were conducted to evaluate the feasibility of the enhancement of electrokinetic decontamination (EKD) with EDTA. Fig. 3 shows the variation of electrokinetic (EO) flow rate as a function of time. This figure indicates that EO flow rate increased with the increase of EDTA concentration. The effect of EDTA on the dissolution of ρb and Zn from millpond sludge is presented in Fig. 4. These tests were conducted under batch reactor conditions with a constant pH of 5.25. It is observed that the concentration of dissolved metal increased with increased EDTA concentration. The fact that EDTA increased both the EO flow and the dissolution of the precipitated metals in the millpond sludge provided sufficient reason to expect enhanced decontamination of millpond sludge by EKD with EDTA.

As one of the process variables is voltage gradient, experiments were conducted at different electric gradients. Fig 5. represents the variation of EO flow as a function of voltage gradient. It is observed that as the supply voltage increased the flow also increased. But the increased voltage also produced higher volume of gas in the electrode chambers. An optimum supply voltage for sufficient flow condition and moderate gas production was about 30 volts with 0.05 M and 0.125 M EDTA along with distilled water (DW). The experiments were terminated after 7 days (-168 hours).

The distribution of normalized concentration (ratio of concentration retained in soil to the initial concentration = C/Co) of ρb and Zn , along the length of the specimen, is presented in Figs. 6 and 7, respectively. From these figures, it is seen that ρb was accumulated after 2 cm from the anode with DW. The accumulation was advanced to 5.5 cm with 0.05 M EDTA while no significant accumulation was observed with 0.0125 m EDTA. Zn showed similar behavior as ρb with both the EDTA concentrations and DW. The accumulation of Zn ($C/C_0 \sim 1.8$) was more than that of ρb ($C/C \sim 1.4$). At the end of 7 days of the decontamination process, the average ρb removal was found to be about 45% both with DW and 0.05M EDTA and 75.8% with 0.125M EDTA. Whereas the average Zn removal was about 23.7% with DW, 68.6% with 0.05M EDTA and 78.7% with 0.125M EDTA. The removal of ρb was lower than the removal of Zn with higher EDTA concentration. For the same duration of the decontamination process. both pb and Zn removal were about 2 to 3 times with 0.125M EDTA to those of with DW. Therefore, the time of EKD process may be significantly reduced with the use of EDTA.

A representative profile of the soil pH during the decontamination process is presented in Fig. 8. Soil pH was close to 4.25 up to 2 cm from the anode and raised to about 8.0 at the cathode end of the sample. No accumulation of metals was observed at the cathode end of the sample at 0.125M EDTA though the pH at the cathode end was about 8.0 as shown in Fig. 9. This observation indicates that EDTA is even capable of dissolving metals at pH slightly greater than neutral pH. Fig 10. represents the variation of anode and cathode pH with the progress of time. Lower pH was attained in the anode chamber with both DW and EDTA solutions.

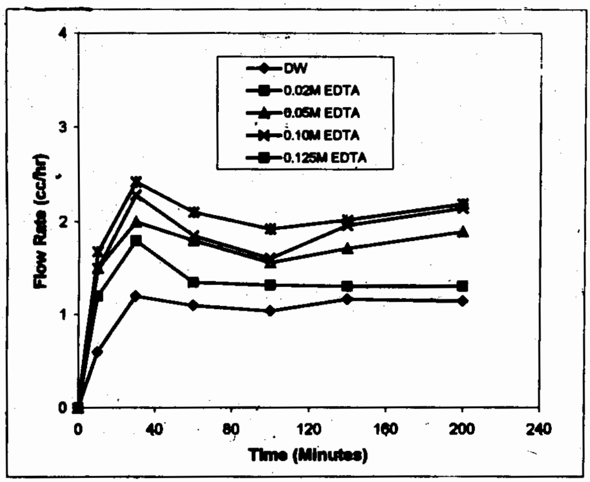

Fig 3. Variation flow rate as a function of time at different EDTA concentrations

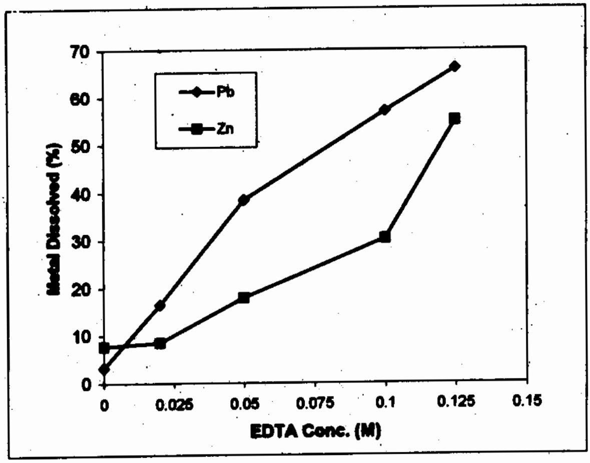

Fig. 4. Variation of percentge of metal dissloved as a function of EDTA concentration pH = 5.25.

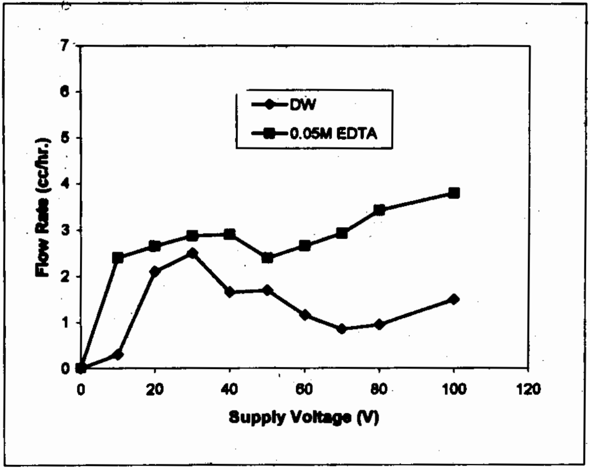

Fig 5. Variation of flow rate as a function of supply voltage

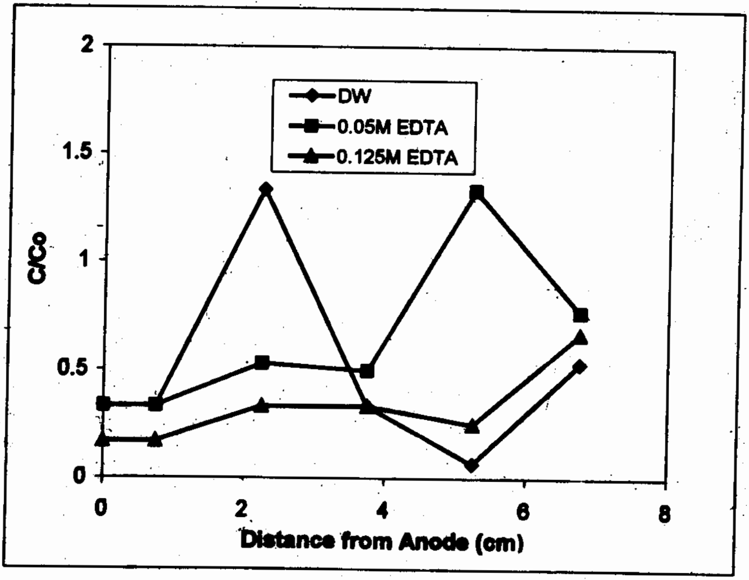

Fig 6. Distribution of lead concentration along the lenght of the specimen

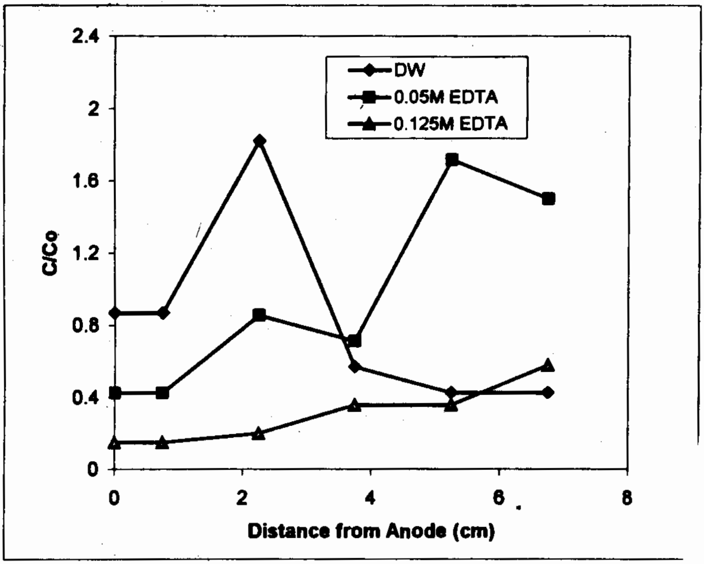

Fig 7. Distribution of zine concentration along the length of the specimen

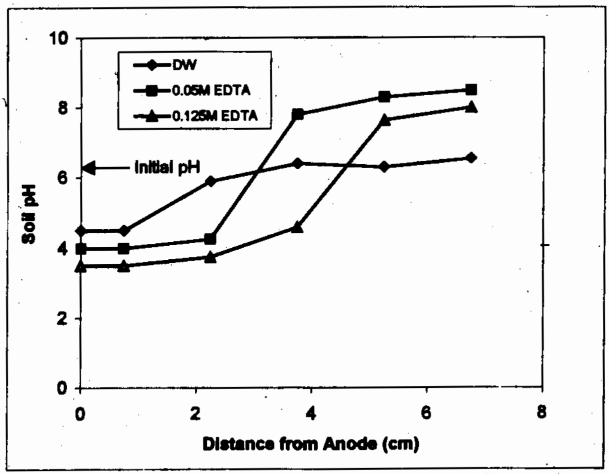

Fig 8. Distribution of pH along the length of the specimen

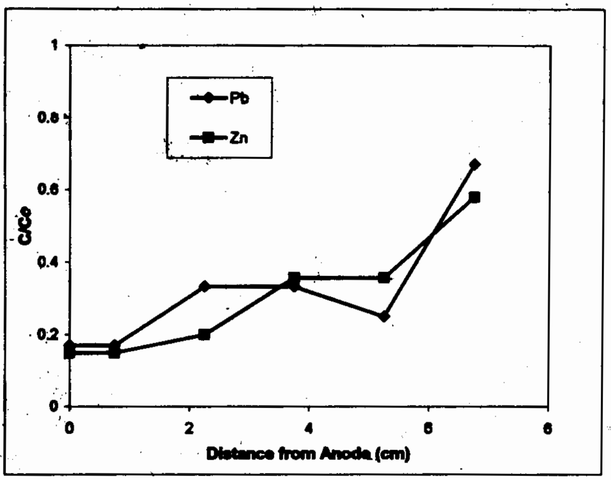

Fig 9. Comparison of pb and Zn concentration distribution long the lenght of the specimen at 0.125M EDTA

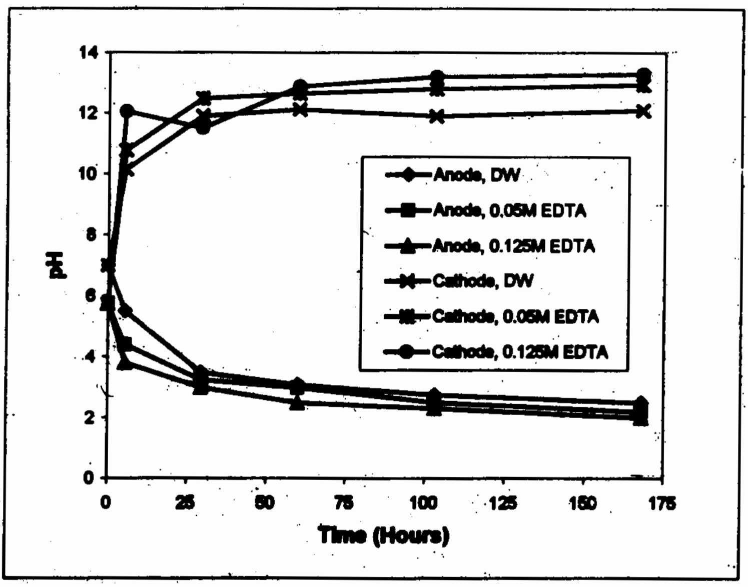

Fig 10. Variation of anode and cathode pH as a function of time

CONCLUSIONS

EKD is proven to be effective in removing contaminats from fine grained soils. EKD becomes more effective with EDTA as an influent for the contaminated source with high organic content. EDTA can enhance EO flow when used in the anode. Higher concentration of EDTA produce higher flow rate. EDTA is also capable of lowering the soil pH slightly, beyond that is not possible by EO flow with DW. As much as 45% of ρb was removed both with DW and 0.05M EDTA and 75.8% with 0.125M EDTA. 23.7% of Zn was removed with DW, 68.6% with 0.05M EDTA and 78.7% with 0.125M EDTA within 7 days of the decontamination process. The removal of pb was slightly lower than the removal of Zn with higher EDTA concentration. For the same duration of the decontamination process, both pb and Zn removal were about 2 to 3 times with 0.125M EDTA to those of with DW. The time of EKD process may be significantly reduced with the use of EDTA.

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