Journal of Civil Engineering
The Institution of Engineers, Bangladesh
Vol. CE 31, No. 1, 2003

AN APPARATUS FOR TESTING SOIL DECONTAMINATION BY THE CEHIXM PROCESS

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ABSTRACT: An apparatus was designed and constructed to experimentally evaluate the feasibility, viability, practicability and potential costs of the *CEHIXM (coupled electric-hydraulic gradient assisted by ion exchange medium)* process. The test procedure and the auxiliary equipment required to run the experiments are presented. Results obtained from the decontamination experiments demonstrate its simplicity of operation and its ability to measure various experimental parameters at different spatial and temporal intervals.

KEYWORDS: *CEHIXM*, soil decontamination, experimental apparatus, coarse-grained soils.

INTRODUCTION

The treatment process termed *CEHIXM* for removing metal contaminants from coarse-grained soils has been proposed and validated with laboratory experiments and the applicability and effectiveness along with the detail description of this process appear in Karim and Khan (2001) and Karim (2000). In brief, the *CEHIXM* process couples an electric gradient with a suitable hydraulic gradient along with an ion-exchange medium to remove and subsequently recover the heavy metals from sandy/silty soils. To evaluate the proposed process, an apparatus was designed and constructed at Cleveland State University, Cleveland, Ohio, USA. The main purpose of this paper is to present the design and operation of the apparatus and to illustrate the types of information that can be obtained by using the apparatus.

PURPOSE OF THE APPARATUS

The apparatus has the following capabilities:

- Produces acidic environment at the anode by electrolysis.
- Produces hydraulic-gradient in the contaminated soil.
- Removes metal contaminants from an aqueous stream by ion exchange.
- Measures electroosmotic flow and determines *zeta-potential* indirectly.
- Measures the variation of soil voltage and electric current with time.
- Compares soil decontamination under hydraulic and electric gradients.

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APPARATUS DESIGN CONSIDERATIONS

The experimental instrument was designed and developed from the following considerations:

1. Soil specimen should be separated from the electrode by a water interface.
2. A semi-permeable barrier, independent of the soil sample, should be provided to isolate the acid producing electrode-pair.
3. The apparatus should have electrodes for applying *DC* voltage gradient across the semi-permeable barrier or the test specimen.
4. A peristaltic pump should be provided for generating a hydraulic gradient in the soil.
5. Ports should be provided for water sampling.
6. The apparatus must be hydraulically leak proof and electrically insulated.
7. Valves & fittings in contact with waste solution must be corrosion and chemical resistant.
8. Vents should be provided to release the gas generated by electrolysis of water.
9. A chamber should be provided to contain an ion-exchange medium.

APPARATUS CONFIGURATION

The apparatus has a 'L' shaped configuration and consists of two segments with a common chamber. In addition to the common chamber, the first segment has two additional chambers and the second segment has four additional chambers. A schematic representation of the apparatus is shown in Fig. 1. As shown in the figure, chamber 1, the anode chamber, is the common chamber which holds the primary and the secondary electrodes which act as anodes during the experiment. Chamber 2 is filled with compacted kaolinite clay to create a semi-permeable barrier. The purpose of such a barrier is to prevent intermixing of acidic and basic solutions generated in the anode and cathode chambers. Chamber 3 is the cathode chamber and holds the primary cathode electrode. Application of voltage across the primary anode and the primary cathode generates acid in the anode and base in the cathode. Chamber 4 holds the sample tube and also accommodates the secondary electrodes. These electrodes may be used to apply a voltage gradient across the soil sample.

A pre-treatment chamber (chamber 5) follows the sample tube where the effluent from the soil is collected. Next to the pre-treatment chamber is the ion-exchange chamber (chamber 6) followed by a chamber (chamber 7) that detains the effluent from the ion-exchange chamber. Clear Plexiglas (*1.25*-cm thick) and Eaton Product brand porous stones of *1.25*-cm thick and grade *55* microns are used for constructing the apparatus.

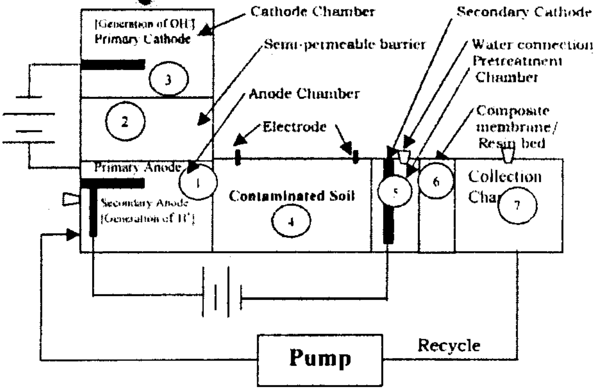

Fig. 1. Schematic representation of the CEHIXM apparatus

COMPONENTS OF THE APPARATUS

The apparatus has a number of chambers as shown in Fig. 1. The physical descriptions and functions of the chambers are briefly described below:

(1) Anode chamber: The dimensions of the anode chamber are *6* cm x *6* cm x *6* cm giving a total volume of *216* cm^3. Two graphite electrodes of surface area about *10.4* cm^2 each, are placed in this chamber. This chamber has two power connections; a water connection; and a gas expulsion port or sample injection/extraction port. The overall liquid capacity of the anode chamber is *186* mL. The top cover can be removed for sampling and cleaning.

(2) Semi-permeable barrier chamber: Compacted kaolinite is used to make the semi-permeable barrier. Kaolinite is placed between two porous stones to form this barrier. The dimensions of the barrier are *6* cm x *3* cm x *6* cm giving a volume of *108* cm^3.

(3) Cathode chamber: The dimensions of this chamber are *6* cm x *5* cm x *6* cm giving a total volume of *180* cm^3. A graphite electrode of surface area about *10.4* cm^2 is placed inside this chamber. This chamber has a water connection; a power connection and a gas expulsion port. Its overall liquid capacity is around *167* mL. This chamber has a combined cover plate and a combined base with the anode and the semi-permeable barrier chambers.

(4) Sample chamber: This chamber holds the sample tube. A *3.45*-cm *ID* and *10*-cm long clear acrylic plastic tube is provided for the soil sample. This tube accommodates two tertiary electrodes at the ends through which the voltage in soil is measured during the experiment. The sample tube is assembled between the anode and the pretreatment chambers with 'O' rings at the ends.

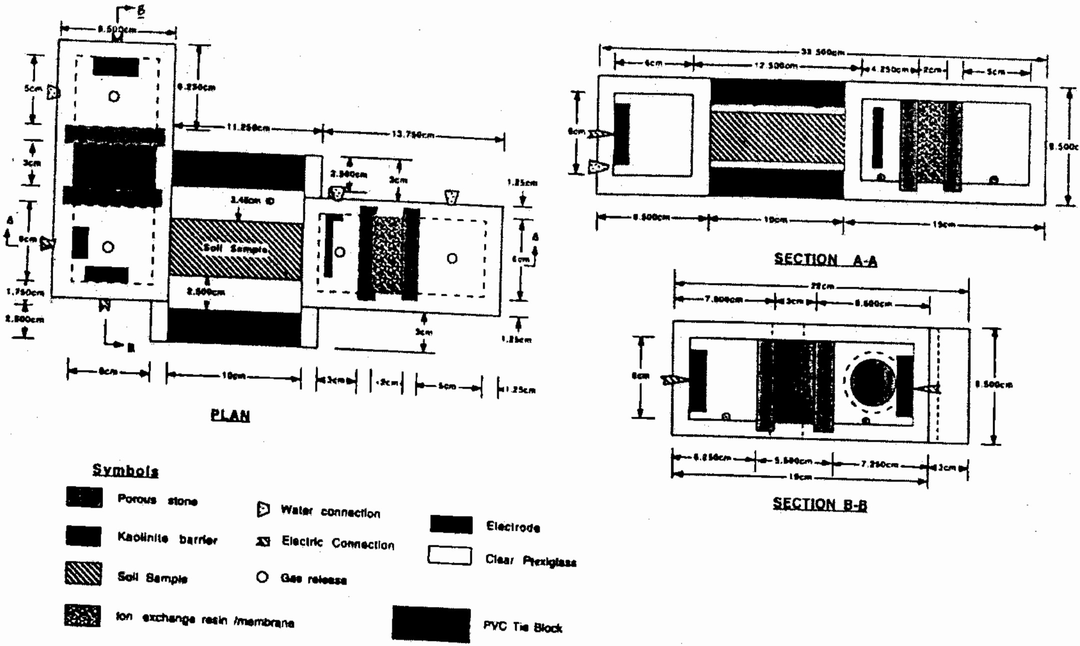

Fig. 2. Detail design representation of the CEHIXM apparatus

(5) Pretreatment chamber: The pretreatment chamber is *3* cm x *6* cm x *6* cm giving a total volume of *108* cm^3. It has a water connection; a power connection and a gas expulsion port. A graphite electrode with a surface area of about *24.5* cm^2 is placed inside this chamber to facilitate the application of voltage across the soil specimen. The overall liquid capacity of this chamber is around *95* mL.

(6) Ion exchange chamber: Heavy metal selective ion-exchange resin is used in this chamber to remove metal ions from the effluent. Like the semi-permeable barrier, the resins are also placed between two porous stones. Composite membrane can also be used as an ion exchange medium. The dimensions of the ion exchange chamber are *5* cm x *6* cm x *2* cm.

(7) Collection chamber: The dimensions of this chamber are *5* cm x *6* cm x *6* cm giving a total volume of *180* cm^3. Its overall liquid capacity is also *180* mL. This chamber has a water connection part and a gas expulsion port. The chamber also has a combined cover plate and a combined base with the pre-treatment and the ion exchange chambers. The top cover can be removed for sampling and cleaning.

Other important accessories of the apparatus include graphite electrodes, water connection, porous stones, gas expulsion and sample extraction/injection ports. These are briefly described below.

Graphite electrodes: High-grade graphite electrodes are used for economic and practical reasons. Graphite electrodes undergo very slow deterioration due to electrolysis reactions and prevent introduction of additional chemical species, which may also be contaminants.

Power connection: Sockets for banana plugs are provided at the outer walls of anode, cathode and pre-treatment chambers for electric connection.

Water connection: Plastic quick-connects from the *CPC* manufacturing company is used in the chambers to connect the chambers to the flow measuring tubes and the pump. This allows the apparatus to be completely detachable from the system while the chambers are charged with water.

Porous stones: Permeable carborundum porous stones of grade *55* microns, manufactured by Eaton Products International, Inc., are used to contain the semi-permeable barrier (compacted kaolinite) and the ion exchange medium. The thickness of the stones is *1.25*-cm with a permeability of greater than $1.33x10^{-2}$ cm/sec. The stones are washed with dilute nitric acid to ensure the removal of metal impurities and particles. The stones are boiled in distilled water before each usage.

Gas expulsion port or sample extraction/injection port: Pressure valves are provided on the top cover plate over each chamber. These valves have metal surfaces that are coated with paint to make them non-conductive and chemical resistant.

Cover plate: A *1.25*-cm thick clear Plexiglas plate with gas expulsion ports mounted is used to seal the chambers air-tight.

PVC tie bar: Anchor plates were provided with *2.54*-cm (*1*-inch) thick gray *PVC* blocks.

AUXILIARY EQUIPMENTS

- (i) Water connections from the chambers: Water connections were provided with *0.25* cm internal diameter chemical resistance flexible Teflon tubes.
- (ii) Volume measuring tubes: Graduated burettes with *50*-cc capacity with *0.1*-cc accuracy were used for flow volume measurement.
- (iii) Multimeter: Radio Shack auto range digital multimeter of model # 22-166B with range of *0* to *200* volts and *0* to *200* mA.
- (iv) DC converter: Consort *E332 DC* converter with a capacity of *200* volts.
- (v) Pump: Ismatec *IPC-8* (*8*-channel) cassette tubing pump with a capacity of 0 to *40* cc/min.

EXPERIMENT PROCEDURE

Sample preparation: The soil sample for the *CEHIXM* tests can either be prepared by compaction or consolidation. The soil can be compacted directly in the sample tube according to *ASTM D2850* procedure. For consolidation of a sample, a pneumatic consolidation apparatus can be used. The detailed description and operation of the pneumatic consolidation device can be found in Khan et al. (1991). Porous stones and O-rings are placed at each end of the sample and glued with silicon glue to prevent water leaks.

Instrument assembly: Kaolinite is compacted in the semi-permeable barrier chamber. An appropriate ion-exchange resin is placed in the ion-exchange chamber. The sample tube was then placed between the anode and the pretreatment chambers by loosening and tightening the screws at the tie bars. The chambers were filled with tap water through the water connections. The cover plates are closed using allen screws. . The instrument is ready for operation after the apparatus is connected to the pump, the measuring tube and the *DC* electric supply. The instrument is ready for operation.

Readings: The outflow tube connected to cathode chamber is filled with tap water and initial volume reading is taken. Power is turned on and the voltage is adjusted to a desired value. The initial current reading is taken. Readings are taken at different time intervals for the duration of the experiment.

Experiment termination: At the end of the experiment, the pH of water in all the chambers was measured. Samples for desired chemical analysis and metal concentration determination were collected following the procedure described in the next section.

SAMPLE COLLECTION

Waters samples are collected through the gas expulsion/sample collection port. A syringe is pressed down on the tip of the sample collection/gas vent valves of the chambers to withdraw the required volume of water. At the end of the experiment, the soil sample is taken out of the sample tube and divided into five equal segments for chemical analysis and water content determination.

APPARATUS CALIBRATION

Experiments are conducted with pure kaolinite clay in the semi-permeable barrier chamber to calibrate the instrument. Tap water is used as influent and the water samples in each chamber are analyzed for heavy metals. The concentrations of various heavy metals in the tap water and the chamber water are typically found to be less than the detection limit.

PRELIMINARY EXPERIMENT RESULTS WITH THE APPARATUS

Preliminary experiments were conducted using the apparatus developed to obtain some typical results. The experiments were performed with a constant electric potential of *100* volts across the semi-permeable barrier (i.e., across the primary electrodes) and a constant flow-rate of *10* cm^3/min. The experiments were terminated after *100* hours. The spent foundry sand with a specific gravity *2.59* and negligible organic content was used in the preliminary experiments. The average particle size, D_{50}, was *0.55* mm. The concentrations of heavy metals in the residual wastes were *Pb~3* mg/kg, *Cd~1* mg/kg, *Zn~6* mg/kg and *Mn~113* mg/kg [Khan and Alam, 1994] which are not high enough to represent the prevailing conditions in the superfund sites. Lead carbonate, zinc chloride, cadmium chloride and manganese chloride salts were used to artificially contaminate the soil samples. The initial concentrations of *Pb, Cd, Zn* and *Mn* in the sample, with respect to the dry weight of the soil, were *482* mg/kg; *502* mg/kg; *1,930* mg/kg and *765* mg/kg, respectively.

The experimental samples were prepared by compacting the soil in the sample tube in three layers, with *15* blows per layer. After compaction the hydraulic conductivity of the soil sample was in the range $1.39x10^{-2}$ cm/sec and the porosity of the sample was about *45%*. The Sybron *IONAC C-249* cation exchange resin with a functional group of $R\text{-}SO_3^{-}\text{-}Na^{+}$ was used as ion medium in the ion exchange chamber during the experiments (Karim and Khan, 2001). The preliminary results presented here were intended to demonstrate some of the capabilities and uses of the apparatus. During the experiment, water samples were collected from all the chambers at different time intervals for the analyses of pH and metal concentrations. The electric current through the kaolinite barrier was also monitored with a Radio Shack auto range digital multimeter. The pH was measured with a Cole-Palmer digital pH meter and standard bulb probe. Metal concentrations were measured according to Standard Method of Examination of Water and Wastewater using Atomic Absorption Spectrophotometer (*AAS*) of Perkin Elmer model *M3100*.

At the end of the experiment, the soil sample was taken out of the sample tube and divided into five equal segments. The wet weight and pH of each segment was measured. The sample was placed in an oven at *110°C* for *24* hours according to *ASTM D4959*. The water content of all samples was calculated. The heavy metals remained in the soil samples were also measured by digesting the contaminants in the soil by the *USEPA 1310* method (USEPA, 1986).

The water content along the length of specimen after *100* hours of the experiment was in the narrow range of *20.9* to *23.9%* throughout the specimen, as shown in Fig. 3. It could be estimated from the void ratio and the water content that the specimen was fully saturated at the end of the experiment. The variation of soil pH along the length of specimen after *100* hours is presented in Fig. 4. Figure 5 presents the variation of soil pH with time at mid section of the soil specimen. A uniform pH of approximately *1.5* after *100* hours was measured throughout the specimen.

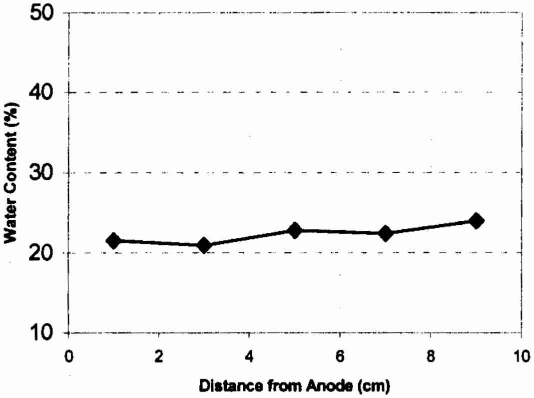

Fig. 3. Typical variation of water content along the length of the specimen after 100 hours of decontamination

The normalized concentration (*C/Co*) of metals in the pore fluid measured after *100* hours as a function of the distance from the anode is presented in Fig. 6. The *C/Co* for *Pb* was in the range of *0.06* to *0.11*. The *C/Co* for *Cd* and *Zn* were in the vicinity of *0.01*. The *C/Co* for *Mn* was in the range of *0.02* to *0.03*. The percentage of metals removal with time is shown in Fig. 7. It is seen that more than *90%* of *Cd* and *Zn* and more than *80%* of *Pb* and *Mn* were removed within the first *50* hours of processing. The order of metal removal rate at the equilibrium pH appeared to be *Zn>Cd>Mn>Pb.* The optimum duration for the decontamination seemed to be approximately *40* hours.

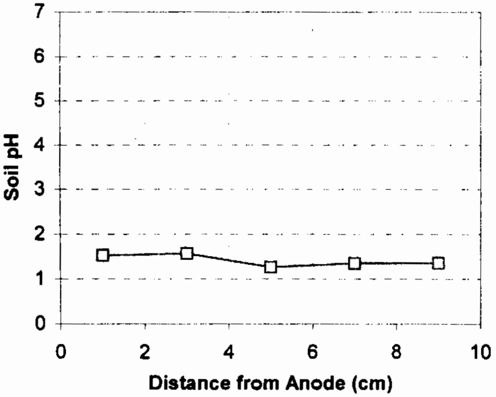

Fig. 4. Typical variation of soil pH along the length of the specimen after 100 hours of decontamination

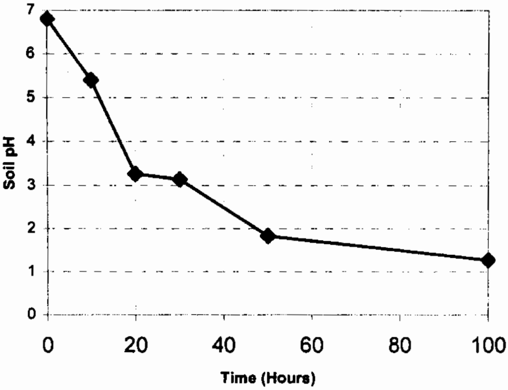

Fig. 5. Variation of soil pH with time at mid section of the specimen at 100V

The typical pH environment in the anode, the cathode, the pre-treatment and the collection chambers, as a function of time is shown in Fig. 8. Lower pH *~2.0* were attained in the anode chamber within approximately the first *12* hours of the processing. The same pH levels were attained in the pre-treatment and the collection chambers within approximately the first *18* and *20* hours of the processing, respectively. The delay in pH changes, in the pre-treatment chamber, might be due to the buffering capacity of the soil. The delay in pH changes, in the collection chamber, with respect to the pre-treatment chamber, might occur because the ion exchange resin captures some hydrogen (H^+) ions along with the metal ions. The pH of the cathode chamber was above *12.0* after *10* hours and remained constant thereafter. This high pH solution may be used to neutralize the treated soil at the end of the process.

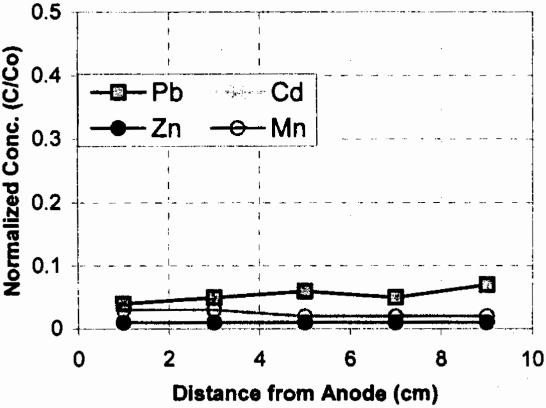

Fig. 6. Variation of metal concentrations along the length of the specimen after 100 hours of decontamination

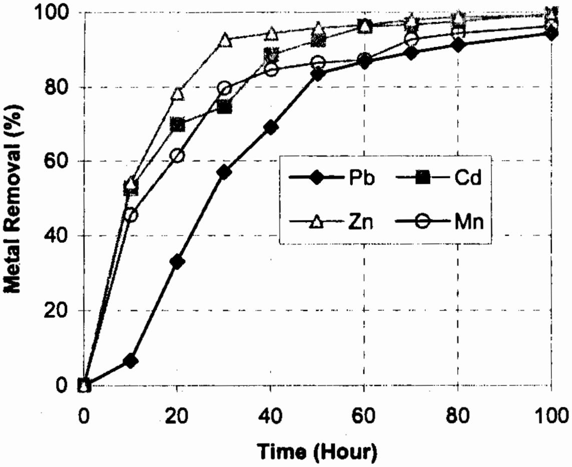

Fig. 7. Perecentage of metal removal as a function of time at 100V

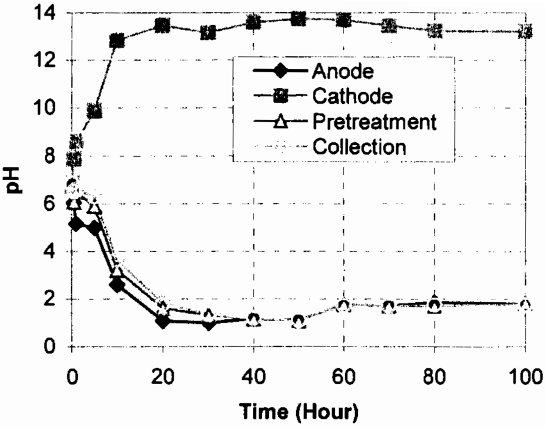

Fig. 8. Typical variation of chambers pH as a function of time at 100 V

The secondary electrodes are included in the instrument with the expectation that they will create hydroxyl (OH^-) ions in the pre-treatment chamber and reduce the H^+ ions. As a result, the competition of adsorption of the cations by the ion exchange medium would be minimal. The second purpose of the secondary electrode is to compare the cost with the primary electrode configuration. The main purpose of the tertiary electrodes in the sample tube is to monitor the distribution of soil voltage along the length of the specimen and the decay of electrical current passing through the specimen with time. In order to compare the cost of the secondary electrode with the primary electrode configuration, two sets of experiments are conducted with a test specimen composed of a blend of residual wastes, spent foundry sand, and millpond sludge (*90:10 w*/w ratio). The characteristics of the residual wastes are presented in Karim (2000). The experiments were conducted at *50* volts and with a constant hydraulic flow-rate of *4* cm^3/min for a period of *200* hours. The experimental values with the primary and the secondary electrode conditions are shown in Table 1. Typical variation of current passing through the test specimen and the kaolinite, as a function of time, at different electrode conditions, is presented in Fig. 9.

In Fig.9, it is seen that the initial current passing through the soil sample was around *20* mA at both the secondary electrode (*SE)* alone and the primary electrode (*PE)* and *SE* conditions. In the case of kaolinite barrier (*PE* alone condition), initial current was about *70* mA. In case of *PE* alone, the current increased to a high value of *90* mA. Afterwards, the current fluctuated and varied between *10* mA and *80* mA throughout the processing. In case of the *SE* alone (current passing through the soil sample), the current initially increased to about *60* mA and varied between *20* mA and *70* mA in the time period of *50* to *120* hours. The current passing through the soil sample (*PE* and *SE* situation) was higher than that of passing through the soil sample in case of *SE* alone, up to *75* hours of the experiment run. After *25* hours, the current passing through the soil sample at *SE* alone was higher than that of in case of the *PE* and *SE* situation to the end of the experimental run.

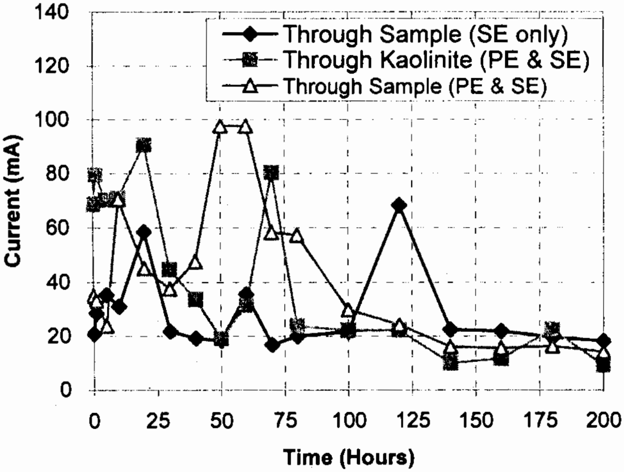

Fig. 9. Typical variation of current passing through the kaolinite and the soil sample with time at different electrode conditions at 50 V

Table 1. Typical energy expenditure with metal concentrations remaining in the soil at different electrode conditions after *200* hours of processing at 50V

Metals	Primary Electrode Only		Secondary Electrode Only	
	Metal remaining Mg/kg (%)	Energy kWh/m³	Metal remaining mg/kg (%)	Energy kWh/m³
Pb	41 (6.41)	~ 480	81 (12.66)	~ 522
Cd	21 (4.16)	~ 480	42 (8.32)	~ 522
Zn	116 (3.17)	~ 480	172 (4.70)	~ 522
Mn	74 (6.49)	~ 480	109 (9.56)	~ 522

From Table 1, it is seen that the concentration of *Pb* remaining in the soil at the primary electrode (*PE)* was about *41* mg/kg *(~6.41%)* and at the secondary electrode (*SE)* was about *81* mg/kg *(~12.66%)*, as twice the amount as at the *PE* situation. A similar trend was observed for *Cd* and *Zn*. The concentration of *Mn* remaining in the soil at the *SE* was about

109 mg/kg *(~9.56%)*, whereas at the *PE*, it was about *74* mg/kg *(~6.49%)*. After *200* hours of processing, energy expenditures were *480* and *522* kWh/m^3 of soil at the *PE* and *SE* respectively. Energy expenditure was higher at the *SE* than that of at the *PE*. Therefore, the electrode configuration with primary electrodes appears to be cost effective and advantageous over the use of the *SE*, in removing heavy metal contaminants from soils. The other expected effect of minimizing the competition of adsorbing cations by the ion exchange medium is still under investigation.

SUMMARY AND CONCLUSIONS

An apparatus was designed and constructed to investigate the potential performance of the *CEHIXM* process in hazardous waste site remediation containing coarse-grained soils. The performance of the apparatus was evaluated experimentally. A comprehensive, large-scale laboratory test program for coarse-grained soils using this apparatus was performed and presented in Karim and Khan (2001), and Karim (2000). The initial experimental results demonstrated the viability of the apparatus. The apparatus produced the acidic environment at the anode, and provided the information to compare the flow under hydraulic and electric gradients. The pressure valves allowed release of the gas produced at the electrode chambers. The electrode configuration with primary electrodes was more cost effective and advantageous over the use of secondary *electrodes* in removing heavy metal contaminants from soils.

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