Int. J. Electrochem. Sci., 7 **(2012)** 7809 - 7822

International Journal of
ELECTROCHEMICAL
SCIENCE
www.electrochemsci.org

An Economical Means for Remediating a Multiple-Metal Contaminated Soil Using Electro-Kinetic Technology Under Natural Conditions

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Received: 18 April 2012 / *Accepted:* 23 May 2012 / *Published:* 1 September 2012

A bench-scale study on the improved electro-kinetic remediation of heavy metals from soil collected near an operating electroplating site in Pakistan is reported. Influence of operating parameters such as electrode material, current density, soil temperature and inter-electrode spacing is investigated. A maximum of 61.8% removal of cadmium is achieved using titanium electrodes (under natural conditions simulating rain water present in soil). Removal efficiency improves with the increase in current density and soil temperature. About 73% of copper removal is achieved within 20 hours when the soil temperature is constant at 29°C as compared to 32% removal at 18°C. Rate of metal removal increases as the inter-electrode spacing decreases to 10 cm, which demonstrates the highest copper removal of 88.1% at a current density of 8.9 mA/cm^2. Other heavy metals are well below their maximum permissible limits and thus are not studied in detail. A maximum energy consumption of 5 kWh/m^3 of soil means the process can be economically scaled up. Removal efficiencies may be improved by carefully controlling the catholyte pH and by introducing zero-valent iron particles as a permeable reactive barrier in the electro-kinetic process.

Keywords: Electro-kinetic remediation; heavy metals; industrial electroplating plant site; low energy consumption.

1. INTRODUCTION

Soil may be viewed as a natural resource because it is essential for the production of food and fiber and for proper functioning of the ecosystem. Degradation of soil shows that soil is a finite resource that requires sustainable management [1]. In many parts of the world, especially in chemical and electroplating industry, soil contamination is increasing at an alarming rate [2]. Metals, in addition to other contaminants, present in the nearby soil pose a great threat to plants, animals and especially to human health if their concentration reaches above the maximum permissible level allowed by enforcing agencies [3]. Soil may be contaminated with metals during routine operations and/or accidental spills in the industry. In addition to this, during decommissioning and dismantling operation in industries significant damage to the soil environment may occur [2].

In general, metals represent potential long-term sources for continued soil contamination. Bioaccumulation of these toxic metals may enhance the hazards associated with these metals and necessitate the removal of these metals from the fragile environment [4]. Maximum Permissible Concentration Limits (MPCL) of some metals in soil and in the discharge effluent from industries is presented in Table 1. It can be seen from Table 1 that the MPCL for metals is very low which in turn reflects the toxicity level of these contaminants and their associated environmental hazards [3].

Table 1. Maximum Permissible Concentration Limits (MPCL) of some metal in soil and in the discharge effluent from industry.

Metals	MPCL of Metals in Soil Used for Land Applications (mg/kg)		MPCL in Industrial Effluents to be Discharged on Land or Water Body (mg/l)
	*US-EPA [5]	**Dutch [3,6,7]	#NEQS [8]
Cadmium (Cd)	1.6	0.8	0.1
Chromium (Cr)	120	100	1
Copper (Cu)	100	36	1
Lead (Pb)	60	85	0.5
Mercury (Hg)	0.5	0.3	0.01
Selenium (Se)	1.6	0.7	0.5
Nickel (Ni)	32	35	1
Zinc (Zn)	220	140	5
Arsenic (As)	14	29	1

A number of remediation technologies have been developed and are available for the remediation of contaminated soil [9-12]. However, most of the established technologies being used are demonstrated to have very low efficiencies especially when dealing with fine-grained soil.

The remediation of soil with electro-kinetic process is a promising technique. The electro-kinetic phenomenon is a combination of three processes, which include electro-osmosis, electrophoresis and electrochemical processes [13]. The degree to which each process occurs depends on the properties of the soil/pore fluid matrix including the degree of saturation, ionic strength of pore fluid, types of ions/charged particles present, pH of pore fluid, temperature, porosity, soil parameters (percentage of clay, type of clay, etc.) and zeta potential of the soil particles [14]. Largely, the in situ remediation of a contaminated soil is an exercise in mass transfer limitations. The challenge is to mobilize the contaminated soil and transport it to a treatment/collection zone. One of the main applications of the electro-kinetic process is in the use of low level direct currents to extract heavy metals from soils and slurries [14,15].

Karachi, Pakistan has electroplating plants that result in the discharge of heavy metals such as copper and cadmium to the soil environment. Exposure to excessive levels of both metals can result in a number of adverse health effects including liver and kidney damage, anemia, immune-toxicity, and developmental toxicity [2,14]. Excessive amounts of cadmium may even be carcinogenic. Hence it is imperative that electro-kinetic remediation techniques are used to decontaminate Karachi’s soil environment so that these toxic heavy metals do not find their way into the drinking water.

This paper provides the results of bench scale experiments on improved extraction of copper and cadmium from contaminated soil samples collected from an operating electroplating industrial site in Karachi. Such studies are reported to a limited extent in the literature [3,14,15] and this may be one of the few investigations where contaminated soil has been treated directly using electro-kinetic technology without spiking it with copper and cadmium or combining it with phytoremediation processes [12] or adding any sort of chelating agents [13] or nitric acid [7] to improve the efficacy of the process. The effect of important operating variables on the performance of the decontamination process is discussed in detail.

2. MATERIAL AND METHODS

A bench scale electro-kinetic setup made up of Plexiglas box, electrodes and other accessories were arranged as shown in Fig. 1. The electro-kinetic Plexiglas cell is a rectangular box having dimensions of 320 mm × 300 mm × 200 mm as shown in Fig. 1. The cell had two electrodes (anode and cathode) made up of 3-mm thick plates each having a size of 280 mm × 180 mm. Electrodes were placed in the soil zone and connected to a constant DC power supply source (Hitachi Model-17858) along with a rheostat (FVRB, MFPR, USA). Electrode spacing of 10 cm was used in all experimental runs, based upon previous results obtained for nickel removal [3], except during the study of inter-electrode spacing. Porous ceramic plates of 5-mm thickness (size 300 mm × 200 mm) were provided at the end of the cell adjacent to a coarser sand layer (Fig. 1), which provided drainage through the cell-porous media. The current and voltage were adjusted and monitored with a multi-meter (8846A, Fluke, USA). A separate electrolyte tank was used to provide a controlled electrolyte flow of 0.6 ml/hr.

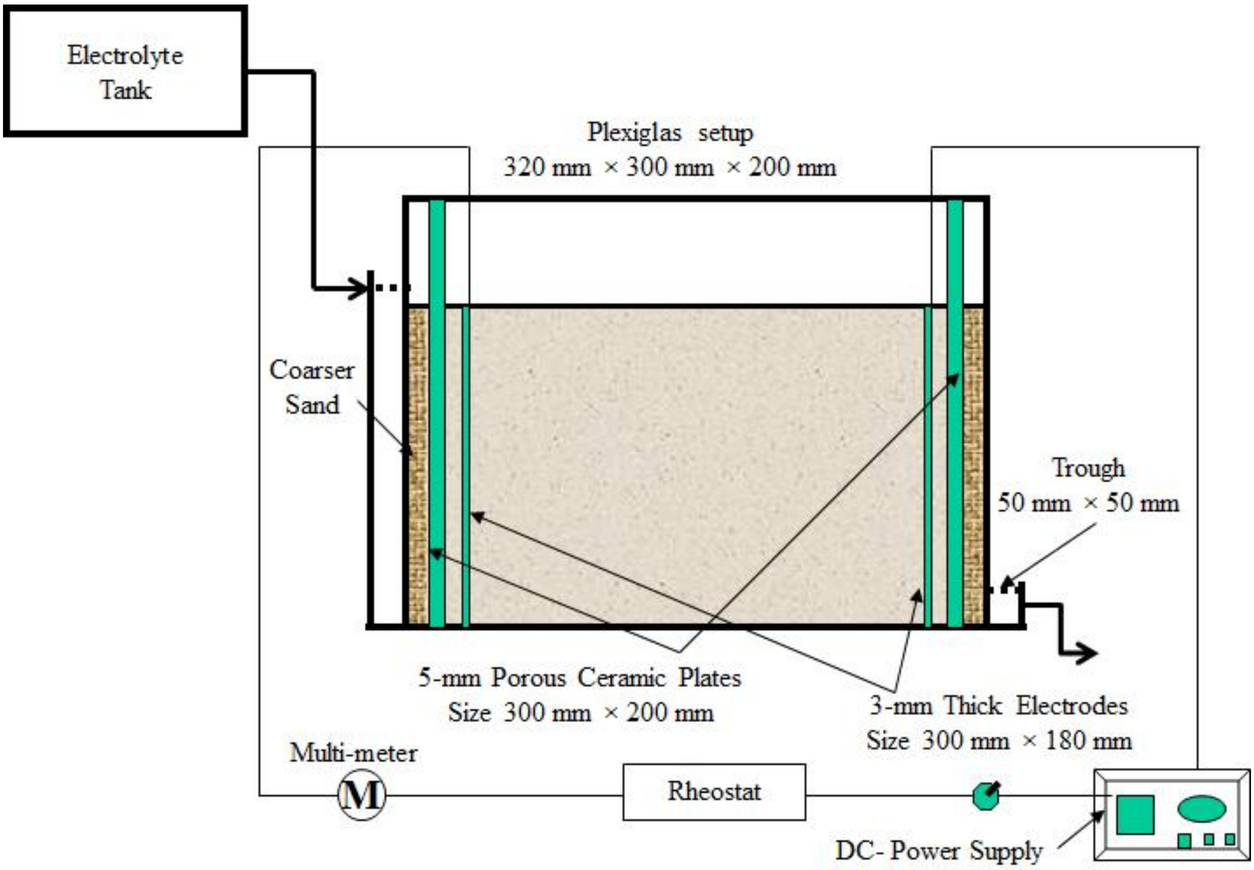

Figure 1. Schematic diagram of electrokinetic soil decontamination cell

Low permeability soil was collected as per standard procedures described elsewhere [15], from an industrial area (SITE), Karachi which is known to be contaminated with copper and cadmium due to uncontrolled discharge from a nearby plating industry and the soil had a similar classification as that reported in a previous work [3]. Clean soil samples were prepared by sieving through a 2 mm sieve to remove stones, branches or any other coarse material. Soil was then washed with clean water followed by washing with 2% commercial sulphuric acid (Merck, Pakistan) to remove organic components from the sample. Again the soil was rinsed with distilled water to obtain an impurity free soil sample. Properties of soil such as pH (340i, WTW pH meter) and moisture content were determined in the laboratory using standard methods [16]. An initial concentration of metals was determined to be about 4938.4 mg/kg for copper and 917.2 mg/kg for cadmium and was way above the MPCL (Table 1). Moisture contents were in the range of 21.9 to 38.6% and determined by gravimetric analysis technique [16]. Electro-kinetic remediation treatment was run for at least 100 hours following the rationale of previous workers [14,17] as well as to lower total energy consumptions. Grab sampling of soil had been done manually to monitor the mass transfer and treatment performance. Samples were collected with the help of a hollow cylindrical auger near the anode, cathode and at the middle of the reactor. Collected samples were analyzed (in triplicate) for concerned metals in the laboratory. Acid digestion was carried out for the extraction of metals as per standard methods [16]. Concentration of metals was determined using Hitachi/Z-200 Zeemen flame/furnace Tandem Atomic Absorption

Spectrophotometer [16]. Only results sampled from the middle of the reactor is shown in this work as maximum removal was observed there. Mineralogical XRD analysis of the studied soil was performed in the X-Ray Diffraction Laboratory of Materials at PCSIR, Karachi. All precautions were taken to ensure the accuracy and repeatability of the test results in a similar manner to that reported before [14].

3. RESULTS AND DISCUSSION

Table 2. XRD mineralogical analysis results of soil sample**.**

Mineral	Percentage
Quartz	71.3
Calcite	9.8
Kaolinite	1.8
Albite	4.1
Muscovite	2.1
Thuringite	1.4
Cristobolite	1.3
Montmorilonite	7.5

Table 3. Geotechnical and physicochemical characteristics of soil sample

Parameter	Value
Moisture content	21.9-38.6 %
pH	6.8 ±0.5
Buffering capacity	0.08±0.01 mM/g
Density	1.16±0.02 g/cm3
Porosity	49.2±1.5 %
Sp. Gravity	2.46±0.2
Organic content	0.16±0.5 %
Electrical conductivity	66.3±2.2 □S/cm
Hydraulic conductivity	10-4 – 10-3 cm/s
Liquid limit	26.1±1.5%
Plastic limit	16.2±0.8%
Plastic index	9.9±1.3

Results of XRD analysis on the soil sample are presented in Table 2. Results show that the clayey fraction is mainly consisting of montmorilonite, muscovite and kaolinite. Thus, the clay present in the soil is partially a swelling type of clay. Presence of calcite (9.8%) in the soil provides buffering during electro-kinetic remediation treatment. Soil samples were also studied by conducting standard geotechnical and physicochemical analysis during the course of the electro-kinetic remediation process. Results of this analysis are presented in Table 3. Soil samples were also analyzed for background metal concentration. Results of metal analysis are shown in Table 4. It can be seen from Table 4 that in addition to high copper and cadmium (4938.4 mg/kg and 917.2 mg/kg respectively) major alkaline earth elements such as Na and K are present in the soil sample along with some significant concentrations of Mn and Fe. However, concentrations of toxic heavy metals other than copper and cadmium in soil sample were far below the allowable limits (Table 1). Hence electro-kinetic remediation of those metals was not considered essential.

Table 4. Results of metal analysis showing background metal concentration present in soil sample**.**

Metal	Concentration (mg/kg)
Na	4321.5±12.6
K	1292.3±3.7
Mn	769.2±0.8
Cr	21.6±0.2
V	17.9±0.2
Zn	10.4±0.3
Ni	4.1±0.1
Pb	1.3±0.06
Cu	4938.4±10.6
Co	0.2±0.01
Cd	917.2±0.8
Fe	62.8±0.4

3.1. Effect of electrode material

As the selection of electrode materials has vital importance in electro-kinetic processes, at least from the process efficiency and economic point of view, a detailed study has been performed to evaluate different electrode materials for soil treatment contaminated with metals based on their removal efficiency. Four materials, namely 304 stainless steel, nickel, aluminum and titanium were tested for possible candidate electrode materials based upon i) high electro catalytic activity, ii) selectivity and current efficiency, iii) resistance to erosion and chemical /electrochemical corrosion, and iv) having a longer life, as compared to other available materials [18-20]. Other electrode materials were tested, such as graphite, carbon steel, zinc, and tin but heavy metal removal was poor and thus results from such work were omitted.

Temporal concentration of copper and cadmium at the middle of the contaminated soil was noted while applying current density of 8.9 mA/cm^2 (this value was an order of magnitude higher than previous reports [7,14] because of higher soil content but the voltage across the cell was lower and the energy consumption was thus compensated for as reported elsewhere for nickel removal studies [3]). All studied electrodes were tested and the results are shown in Fig. 2. It can be seen from Fig. 2 that the removal of metals is increasing with time. It is to be noted that the removal efficiency of all electrode materials are higher for copper as compared to cadmium. This may be attributed to the higher mass number of cadmium as compared to copper which is a significant factor in the mass transport through low permeability soils [21]. Efficiency of studied electrode materials is about 30 to 43% higher (average) for copper removal as compared to cadmium.

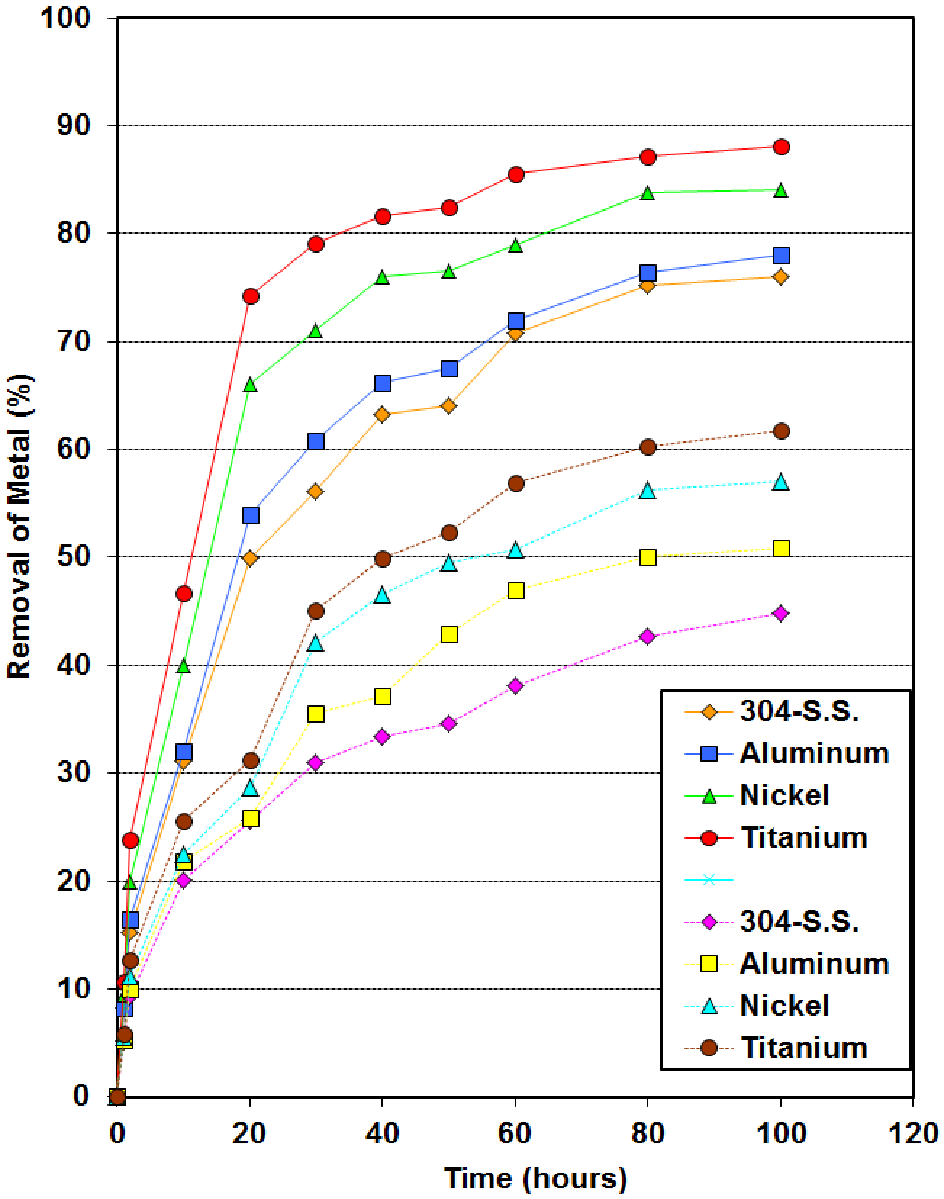

Figure 2. Effect of electrode material on the removal of copper (--- continuous lines) and cadmium (- - - dotted lines)

In the case of stainless steel electrode, during electrolysis rusty color streaks appeared in the soil, which may be attributed to the dissolution of consumable electrode material [22]. In addition to stainless steel electrodes color production was also observed in the case of aluminum electrode. These electrodes were less efficient especially the anode since its oxidation potential was much lower than water and began to corrode. After several hours the soil near the anode turned a rusty brown color and iron "trails" could be seen migrating through the soil. Ultimately the soil near the cathode became opaque and took on a rusty brown color as well. This significantly degraded the effectiveness of the remediation process, as the iron particles became the dominant charge carriers over the contaminant copper ions. As the consumption of electrode is directly related to the economics of the process one should consider this factor in the optimization of operating parameters. Among all four types of studied electrode materials titanium and nickel showed the highest metal removal. However, removal of metals using titanium electrode was highest.

Removal efficiency of titanium for copper and cadmium removal reached up to 88.1% and 61.8% respectively at the end of 100 hours run. Furthermore, the removal efficiency for copper reached up to 73% within initial 20 hours of experimental run. Results show that a significant amount of metal can be removed from contaminated soil during first 20 hours. As titanium showed maximum removal efficiency for copper and cadmium removal, remaining experiments were performed with titanium electrodes.The efficiency for copper removal was higher than other reported values in the literature [12-15], whereas that for cadmium was exactly as reported by Rawat [17]. Rawat showed that without the use of zero valent iron (ZVI) powder 62% removal efficiency of cadmium could be achieved while the implementation of ZVI as permeable reactive barrier raised up the removal efficiency to 83% [17]. However, Rawat didn’t use real soil samples contaminated by a variety of heavy metals and thus his results cannot be considered to apply under the same conditions as reported in this research.

Material balance for the electro-kinetic removal of copper is given in Table 5 while that for cadmium is given in Table 6. The discrepancies in mass balance for the removal of both copper and cadmium were less than 1%, thereby confirming the soundness of the material balance calculations. In addition, energy dissipation calculations showed that a maximum value of 5 kWh/m^3 was obtained when using stainless steel as electrodes.

This value was lower than energy dissipation results reported by other workers using simulated soil samples (kaolin) [7].

Table 5. Material balance of copper removal experiments, total energy consumption and current efficiency

Electrode	Cu collected in anolyte (mg)	Cu remaining in cell (mg)	Cu collected in catholyte (mg)	Total Cu (mg)	Discrepancy in material balance (%)	Energy usage (kWh)	Current efficiency (%)
Titanium	< 0.1	585.3	4351	4936	0.04	0.0457	100
Nickel	< 0.1	788.7	4148.3	4937	0.03	0.0589	100
Aluminum	< 0.1	1086	3852	4938	0.00	0.0735	75
304 S.S.	< 0.1	1232.2	3703.8	4936	0.05	0.0855	75

Table 6. Material balance of cadmium removal experiments, total energy consumption and current efficiency

Electrode	Cd collected in anolyte (mg)	Cd remaining in cell (mg)	Cd collected in catholyte (mg)	Total Cd (mg)	Discrepancy in material balance (%)	Energy usage (kWh)	Current efficiency (%)
Titanium	< 0.1	347.8	568.7	916.5	0.08	0.041	100
Nickel	< 0.1	394.2	522.8	917	0.02	0.057	97
Aluminum	< 0.1	448.3	467.8	916.1	0.12	0.065	85
304 S.S.	< 0.1	503.3	412.7	916	0.13	0.091	73

3.2. Changes in pH between electrodes of reactor

Electrolysis of water in the vicinity of the electrodes results in the production of acidity at the anode and alkalinity at the cathode, as follows [23]:

Anode: $$2H_2O \rightarrow O_2 + 4H^+ + 4e^- \quad (1)$$

Cathode: $$2H_2O + 2e^- \rightarrow 2OH^- + H_2 \quad (2)$$

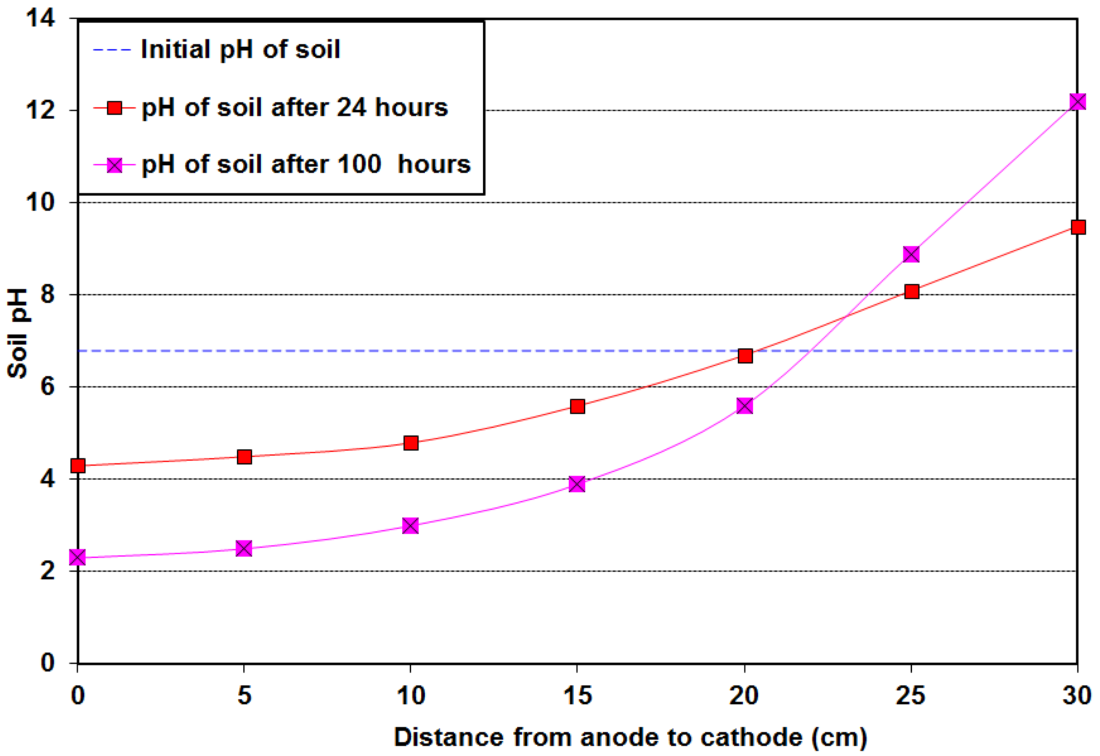

Figure 3. Temporal variation of soil pH at different points between anode and cathode of electro-kinetic cell

The acid formed at the anode is highly mobile and moves in the applied field towards the cathode at a rate of at least twice that of any metal ions and also of the hydroxide ions formed at the cathode which migrate to the anode. Its movement is further enhanced by the electroosmotic flow of water towards the cathode. Therefore, the acid, which can be regarded as a leachant for absorbed metals in the soil, mobilises metal ions as it moves to the cathode [23]. The metals also migrate towards the cathode and are removed from the catholyte chamber effluent. Fig. 3 shows the distribution of pH between both electrodes during electrokinetic treatment of soil using titanium electrodes. It is clear that the high pH near the cathode had limited the complete removal of copper and cadmium from the soil. Future investigations may look into ways of keeping the pH at an optimum value to maximize heavy metal removal without compromising on the energy consumption and thus the cost.

3.3. Effect of current density

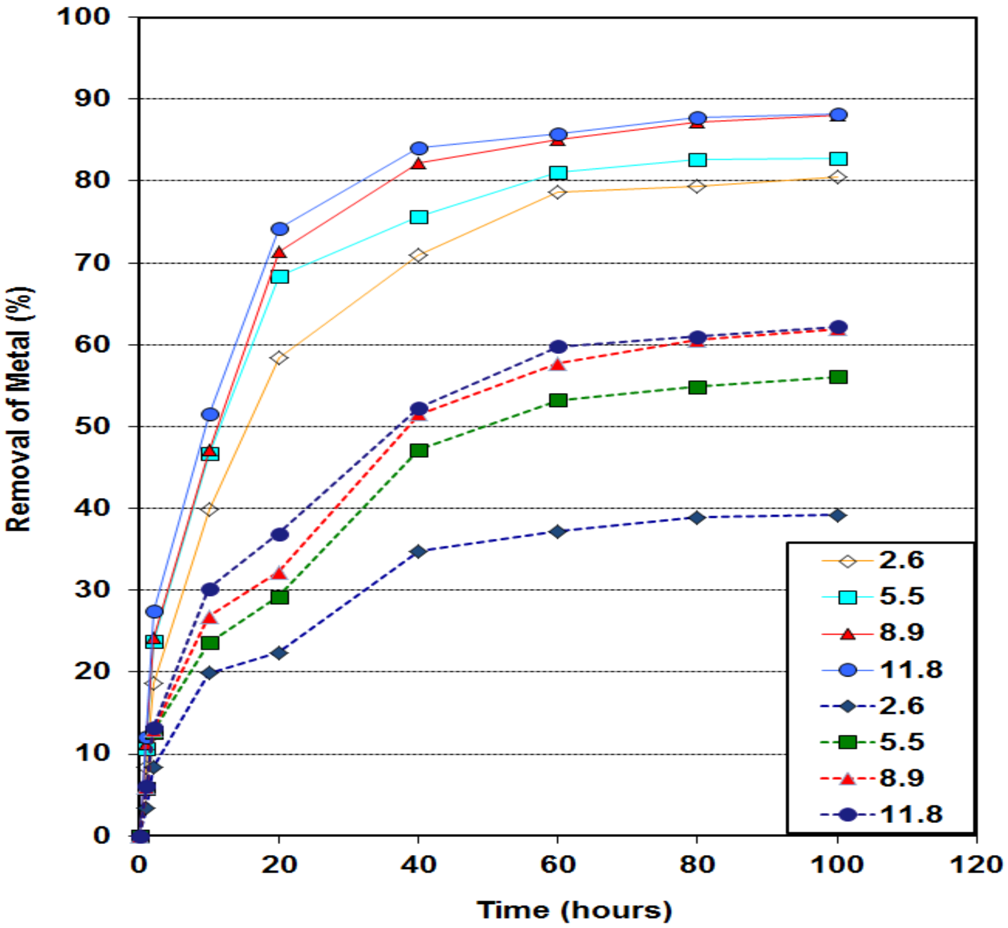

Figure 4. Effect of current density on the removal of copper (--- continuous lines) and cadmium (- - - dotted lines). All current densities are given in units of mA/cm^2.

In this part of the study supplied DC current was adjusted in a controlled manner to achieve current densities ranging from 2.6 mA/cm^2 to 11.8 mA/cm^2 during experimental runs (2.6 mA/cm^2, 5.5

mA/cm^2, 8.9 mA/cm^2 and 11.8 mA/cm^2). The results of the temporal variation in the copper and cadmium removal at different current densities are presented in Fig. 4. It is depicted in Fig. 4 that the metal removal increases with the current density however; increase in copper removal is marginal (8.2% as compared to cadmium removal of 22.6% when current density increased from 2.6 mA/cm^2 to 11.8 mA/cm^2).Results show that a current density of 8.9 mA/cm^2 is capable of bringing the copper concentration below 600 mg/L from 5000 mg/L within 100 hours of experimental run. However, further increase in current density does not improve the removal efficiency of the process for both metals. As an increase in current density augments the cost of treatment one may use an optimum value of current density for efficient removal and minimum cost. Therefore, a current density of 8.9 mA/cm^2 is a reasonable value for an efficient removal for both heavy metals.

3.4. Effect of soil temperature

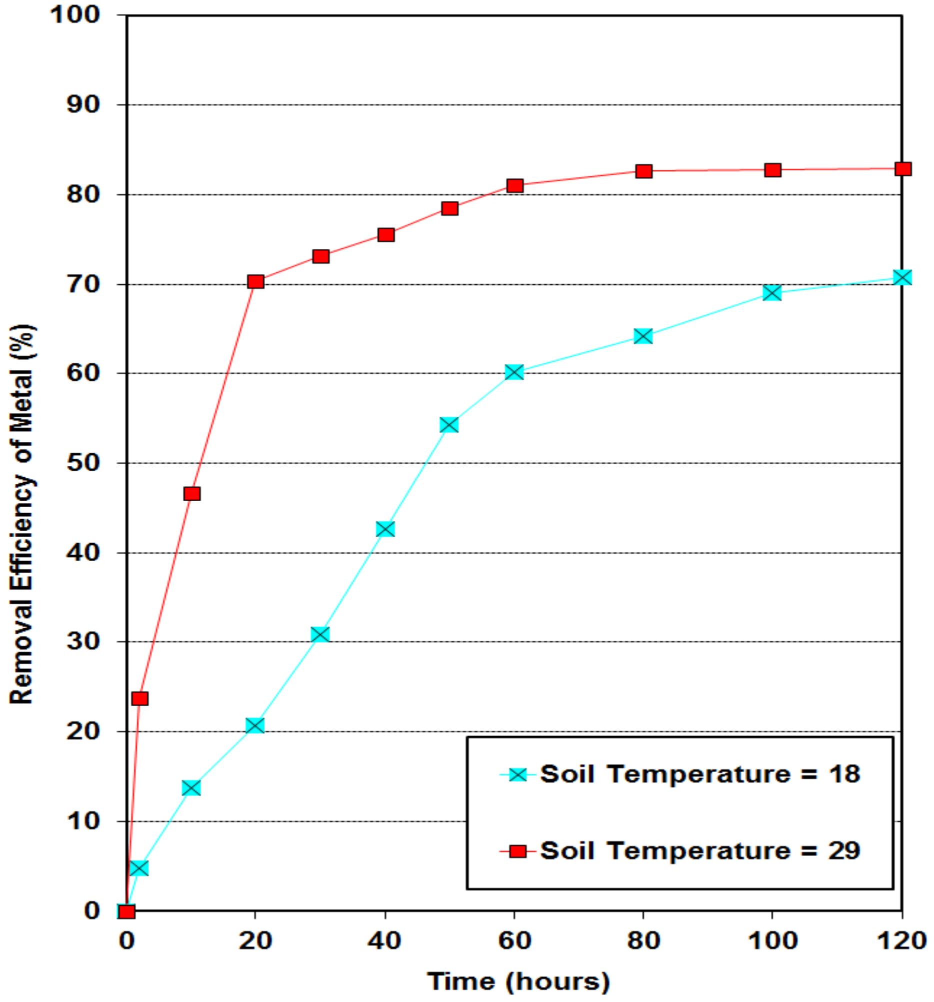

Figure 5. Effect of soil temperature on the removal of copper (both temperatures in °C)

In order to investigate the effect of soil temperature on copper removal efficiency two experimental runs were performed while keeping temperature around 18°C and 29°C. Temperature of soil was maintained at the desired value by keeping the electrochemical cell in a continuous watercirculating bath (Precision). Maximum difference in the temperature of the outer and middle of the soil mass was monitored and found to be about 1 – 0.5°C. Effect of soil temperature on the copper removal efficiency at 18°C and 29°C is presented in Fig. 5. It can be seen from Fig. 5 that the removal efficiency increases with the increase in temperature. Influence of temperature on removal efficiency is significant during the first few hours of run. One can see from Fig. 5 that the removal efficiency during the initial 20 hours reached to about 70% when the temperature was 29°C as compared to a value of 20% when the temperature was 18°C (difference of about 50%). However, when the time difference reduced the efficiency reached to about 12% at the end of 120 hours. Results show that the effect of soil temperature is very important during the first few hours and it may significantly improve the removal efficiency of the process. Therefore, by increasing the soil temperature by a few degrees one may augment the process efficiency. However, some of the researchers remained vigilant about the desiccation of soil which could have caused problems, particularly on large scale samples and in field studies [22,24]. Further study is suggested to see the consequences of soil drying out which may cause shrinkage and cracking of the soil, development of uneven flow paths, and eventually cessation of fluid flow.

3.5. Effect of inter electrode spacing

Inter-electrode spacing is an imperative parameter in electro-kinetic treatment method. Increasing the electrodes spacing will reduce the capital cost of treatment but may reduce the treatment efficiency. Hence, an optimization of this parameter is critical. Temporal variation in the copper removal efficiency as a function of inter-electrode spacing is presented in Fig. 6. Analysis of Fig. 6 revealed that the rate of copper removal increased as the inter-electrode spacing decreased. Its maximum removal was achieved for a spacing of 10 cm. A reduction of inter electrode spacing from 30 to 10 cm may increase copper removal efficiency from 70.9% to 88.1%. However, further reduction in inter electrode spacing does not increase the copper removal. It can be seen from Fig. 6 that the removal rate of copper at a spacing of 5 cm is approximately equal to that obtained at a spacing of 20 cm and the trend showed some nonlinear behavior. This nonlinear behavior may be attributed to the transitions in overcoming double layer forces that became effective near certain inter-electrode spacing (in the case of these experiments the spacing was 5 cm). Therefore, beyond a gap of 10 cm, the copper removal dropped.

This observation is in line with the idea that a shorter gap would favor to minimize the potential drop, and lead to a higher current density. But after an optimum spacing electrostatic double layers formed since the cations swarmed near the soil particle surface and may have dominated and suppressed the copper removal [25,26].

This phenomenon was also pointed out by other researchers [10]. One may conclude that for the studied operational conditions an inter electrode spacing of 10 cm is feasible to obtain best heavy metal removal efficiency.

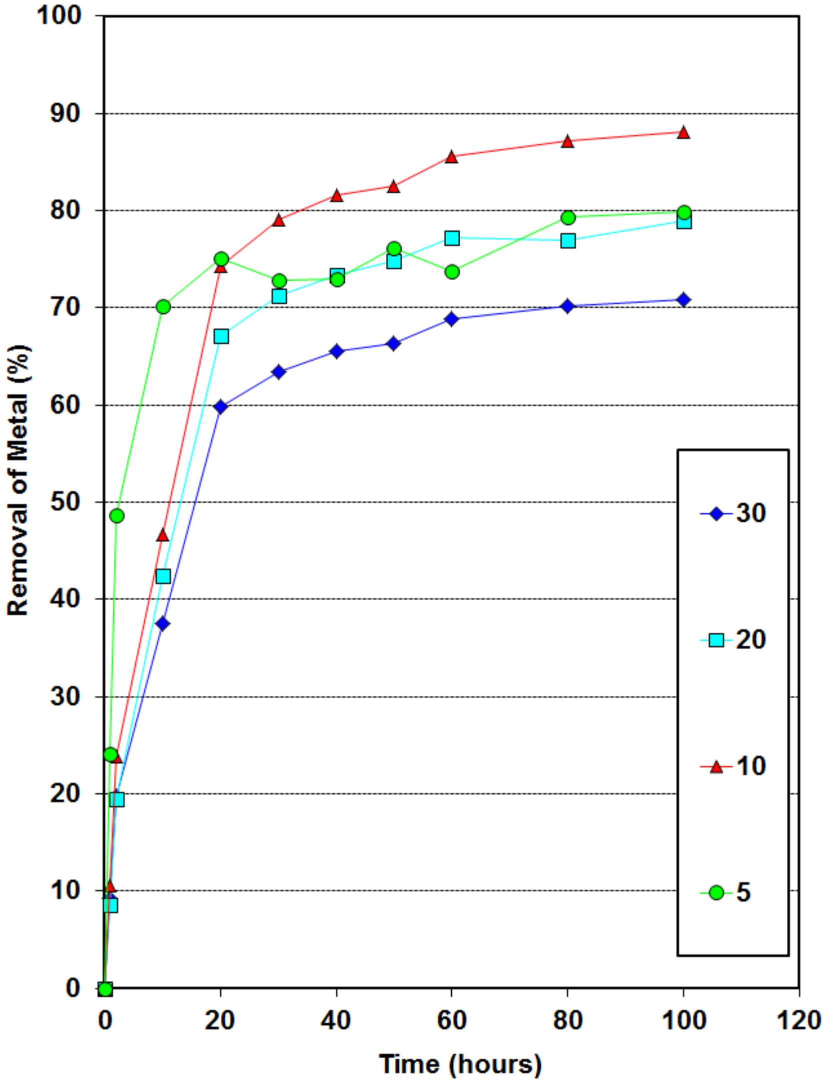

Figure 6. Effect of inter electrode spacing (in cm) on the removal of copper

4. CONCLUSIONS

An efficient and economical means for treating a soil contaminated with heavy metals from a discharge of an operating electroplating plant was investigated. No acids or chelating agents were used during the electro-kinetic remediation, thereby saving costs and allowing the process to run under natural conditions. Titanium electrodes gave highest removal efficiency in comparison to 304 stainless steel, nickel and aluminum electrodes. Best current density for extracting heavy metals is 8.9 mA/cm^2. An increase in temperature from 18 ^{0}C to 29 ^{0}C enhanced removal efficiency by 50%. Finally an optimum inter-electrode spacing of 10 cm is found to give higher copper removal efficiencies than recently reported values in the literature [15]. Removal efficiencies may be improved by washing the soil with sodium dodecyl sulphate or by introducing zero-valent iron particles as a permeable reactive barrier in the electro-kinetic process. The energy dissipated during operation using stainless steel electrodes (lowest removal efficiency of heavy metals was achieved with these electrodes) came to about 5 kWh/m^3 of soil, which was significantly below that reported in previous studies [7], even though the current density employed here was 10 times higher.

ACKNOWLEDGEMENTS
The authors acknowledge support from JUC, the University of Malaya (UM) Bright Sparks Unit {Bsp App462/11(K)} and the Ministry of Higher Education High Impact Research project UM.C/HIR/MOHE/ENG/18.

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