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## Chromium speciation some effluents of tannery industries

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### ABSTRACT

*Separation of chromium from Tannery effluents has been attempted. In this various physic chemical characteristics of water contaminated by tannery effluents have been carried out. Electro kinetic remediation of soil contaminated with heavy metals from tannery effluents is the main aim of the present communication and towards this an attempt has been made to treat the simulated chromium spiked artificial soil. Electro kinetic remediation method has been attempted to remove chromium from the chromium spiked artificial soil and the results are presented and discussed in this communication.*

**Keywords:** Speciation, Physico-chemical parameters, Chromium, Electro kinetic method.

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### INTRODUCTION

Traces metal ions play very important roles in our life. Among the trace metals chromium (III) is a necessary species for living organisms. But chromium (VI) is a toxic species, chromium is a major water polluting element and this is inducted into water resources from tannery effluents and industrial electroplating effluents. Due to the levels of chromium species in the natural water samples are generally at mg/L and high matrix contents of the samples, a separation/pre concentration technique is necessary, prior to determination of chromium by an instrumental technique. [1, 2] Many preconcentration methods including liquid-liquid extraction, ion-exchange, electro analytical techniques and membrane filtration have been proposed for the speciation of chromium. Enrichment and separation methods, based on the formation of Cr (III) complexes, have not been applied so far, because of the inert nature of hydrated Cr (III) species. Cr (III) concentration is usually calculated by difference after determination of total chromium and Cr (VI). [1, 3] Also in the speciation studies of Cr (III), Cr (VI) and total chromium in the natural water samples, solid phase extraction procedures have been used by various researchers.[4-7] In these procedures, various solid phases such as artificial adsorbents, activated carbon, sepiolite etc. have been used. Ambersorb adsorption resins are synthetic carbonaceous adsorbents.

They are partial pyrolyzed resin of sulfonated styrene/divinylbenzene polymer. They have good adsorption properties: high surface area, porosity, durability, uniform pore distribution, high surface area. They have been used for the separation, preconcentration and isolation of organic substances from various media especially natural waters. [8-14] Ambersorb 563 is a member of Ambersorb resin family. Its surface area is 580m<sup>2</sup>/g. [8] at our

knowledge, the use of Ambersorb resins for the preconcentration and separation of trace heavy metal ions is yet very limited. Kenduzler and Turker [15] have been used Ambersorb 572 for the preconcentration of iron prior to its flame atomic absorption spectrometric determination. In the present work, atomic absorption spectrometric determinations of chromium (VI), chromium (III) and total chromium species in natural water and sediment samples were performed after their speciation on Ambersorb 563 resin. The influences of various parameters including pH, reagent amounts, and sample volume and flow rates on the speciation of chromium were examined. Separation of chromium from Tannery effluents has been attempted. In this various physico-chemical characteristics of water contaminated by tannery effluents have been carried out. Electro kinetic remediation of soil contaminated with heavy metals from tannery effluents is the main aim of the present communication and towards this an attempt has been made to treat the simulated chromium spiked artificial soil. Electrokinetic remediation method has been attempted to remove chromium from the chromium spiked artificial soil and the results are presented and discussed in this communication.

## MATERIALS AND METHODS

### Collection of Water and Soil samples

Water samples were collected from the lakes of Karai, Pulianthangal, Bharathi Nagar, Tandalam and Maniyambat villages of Ranipet industrial town. Sub soil samples upto 15-30 cm depth, were collected from the agricultural lands from the above mentioned sites. The soil samples were brought to the laboratory and the following physico-chemical characteristics were analysed.

### Physico-chemical characteristics and heavy metals of water samples

Physico-chemical factors such as pH, Electrical conductivity, Total dissolved solids, Turbidity, Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Total alkalinity, Total Hardness, Total Nitrogen, Potassium, Phosphorous, Calcium, Sodium, Chloride, and heavy metals Chromium and Cadmium were analysed as per the methods of [16].

### Physico-chemical characteristics and heavy metals of soil samples

Physico-chemical factors such as pH, Electrical conductivity, Moisture, Porosity, Alkalinity, Salinity, Organic matter, Total Nitrogen, Phosphorous, Sodium, and Calcium were analysed for the five soil samples as per the method of [16]. The heavy metals Chromium and Cadmium concentration were estimated using Atomic Absorption Spectroscopy (Varian AAA 220 FS).

## RESULTS

### (i) Karai

The pH value recorded as 8.1 infers that the soil sample is alkaline. The electrical conductivity was 315  $\mu\text{mhos}/\text{cm}^2$ . The alkalinity was 382 mg/L and the reason for high alkalinity could be due to the percolation of surface pollutants into the soil. The soil sample of Karai showed higher levels of total nitrogen. The Chromium and Cadmium levels were found to be 241 ppm and 12 ppm respectively, showing the high contamination levels of the Karai soil.

### (ii) Pulianthangal

The pH value recorded as 7.4 infers that the soil sample is slightly alkaline. The electrical conductivity was 3110  $\mu\text{mhos}/\text{cm}^2$ . The salinity of Pulianthangal soil (543.9 mg/Kg) was found to be the highest among all the five samples under study and the reason could be attributed to the common salt used in preserving the hides in the tanneries. Salinity within irrigated lands limits productivity in vast areas of the world. The alkalinity was 411 mg/L and the reason for high alkalinity could be due to the percolation of surface pollutants such as basic chromic sulphate for tanning process. The soil sample of Pulianthangal showed moderate levels of total nitrogen and high levels of Phosphorous, Potassium, Sodium and Calcium. The Chromium and Cadmium levels were found to be 1247 ppm and 42 ppm respectively showing the contaminated nature of the soil.

### (iii) Bharathi Nagar

The pH value recorded as 8.1 infers that the soil is slightly alkaline. The electrical conductivity was 3450  $\mu\text{mhos}/\text{cm}^2$ . The alkalinity was 403 mg/L and the reason for high alkalinity could be due to the percolation of surface pollutants. The soil sample of Bharathi Nagar showed higher levels of Total Nitrogen, Phosphorous, Potassium, Sodium and Calcium than all the other four soil samples. The Chromium and Cadmium levels were

found to be 4594 ppm and 35 ppm respectively. The highest level of Chromium in Bharathi nagar sample is due to chemical industries such as TCC, Malladi Drugs and Pharmaceuticals, Thirumalai Chemicals Limited and few other tanneries.

#### (iv) Tandalam

The pH value recorded as 8.1 shows that the soil sample is slightly alkaline. The electrical conductivity was 3721  $\mu\text{mhos}/\text{cm}^2$ . The alkalinity was 441 ppm and the reason for high alkalinity could be due to the percolation of surface pollutants. The salinity of Tandalam soil was found to be 460.1 mg/Kg. The high salinity may be due to the usage of common salt in preserving the hides in the tanneries. The soil of Tandalam showed the highest level of organic matter 9.1%. The soil sample of Tandalam showed high levels of Total Nitrogen, Phosphorous, Potassium, Sodium and Calcium. The high levels of most of the parameters may be due to the presence of clusters of chemical industries supplying allied chemicals for leather industries in this site. The Chromium and Cadmium levels were found to be 2125 ppm and 76 ppm respectively.

#### (v) Maniyambat

The neutral nature of soil is shown by the pH value of 7.4. The electrical conductivity was 3721  $\mu\text{mhos}/\text{cm}^2$ . The alkalinity was 441 ppm. The salinity of Maniyambat soil was found to be 123.4 mg/Kg. The soil sample of Maniyambat showed high levels of Phosphorous, Potassium, Sodium and Calcium. The Chromium and Cadmium levels were found to be 168 ppm and 15 ppm respectively. The results indicate that the leather industries in this site are mostly involved in pre-tanning process.

#### Electro Kinetic remediation of chromium spiked artificial Soil

Fig. 1 show that the contaminated soil sample, selected for this study, was spiked with chromium artificially similar to the one that is collected from Tandlem area. The experimental setup was arranged as shown in the figure. The test setup mainly consists of an electro kinetic cell, two electrode compartments, a DC power source and a mutimeter. Plexi glass cell having a total length of 25 cm, inside diameter of 15 cm and 50cm height was used as the electro kinetic cell. Each electrode compartment included an outlet valve and a wire mesh. The cell was divided into four compartments and labelled as anode, middle anode, middle cathode and cathode respectively. The outlet valves were connected to the reservoirs to collect the liquid that was removed along with the gases. A DC power source was used to apply a constant voltage to the electrodes, and a multimeter was used to monitor the voltage and measure current value in the soil sample during the test.

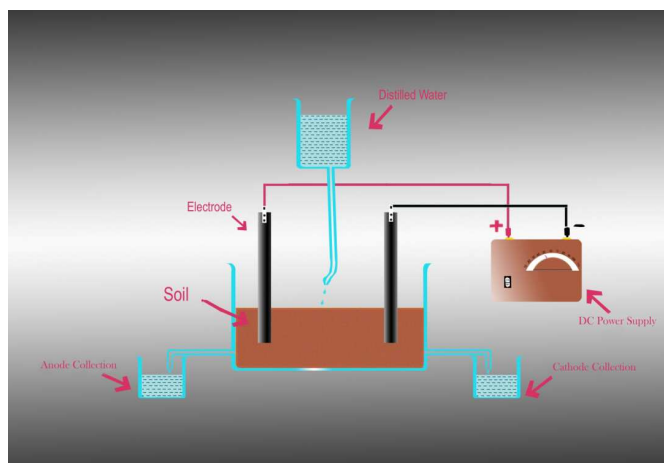


Fig 1. Electro kinetic Remediation Set up

Contaminated soil of about 3 Kgs was saturated with 1.5.L of deionised water and mixed in a polythene container thoroughly for several minutes to achieve homogeneity. Then the homogenized slurry was packed in the electro kinetic cell and pressed using a hand compactor to minimize the amount of void spaces. After compacting the soil was allowed to reach physico-chemical equilibrium for 48 Hours. Deionised water was circulated inside to achieve the mobility of ions. Two graphite electrodes of dimensions 25 cms and 1.5 cm outer diameter were inserted into the

compartments. When DC electric fields are applied to contaminated soil via graphite electrodes inserted into the soil packed in the electro kinetic cell, migration of charged ions occurred.

### Testing Procedure

To determine the dependence of the efficiency of the electrokinetic remediation method on the voltage, the experiment was performed at two voltages (30 V and 50 V). As the Tandalam soil was found to contain more amounts of heavy metals in exchangeable form after the speciation studies, it was subjected to electrokinetic remediation at a voltage of 30V. The soil and water samples were collected at regular intervals of 2 hrs, 4 hrs, 24 hrs, 48 hrs and 78 hrs. After the completion of each test, aqueous solutions from the anode and cathode reservoirs were collected and the volumes were measured. The soil samples collected from the four compartments at regular intervals of time were digested using conc.HNO<sub>3</sub>. The concentration of the heavy metal chromium was analysed in the soil and water samples, using Atomic Absorption Spectroscopy.

The same type of an electrokinetic treatment was carried out at 50 Volts. The soil and water samples were analysed at the same intervals of 2hrs, 4 hrs, 24 hrs, 48 hrs and 78 hrs. As 50 Volts showed high levels of movement of metals in the electro kinetic cell, 50 Volts was chosen for electro kinetic remediation and the treatment time was extended. This provided a voltage gradient of 2 V/cm and a constant current supply. Higher voltage and longer duration of electro kinetic treatment was found to be more effective in removal efficiency, making the contaminants in the soil getting accumulated near the electrodes.

Contaminants from the different soil sections were extracted by acid digestion and the fluid samples were also digested. The total concentrations of Chromium and Cadmium were determined using Atomic Absorption Spectrophotometer (Varian 4S AA). The mass loss due to adsorption onto electrodes and reactor wall was neglected. Removal efficiency for each test was calculated dividing the total mass deposition in anolyte and catholyte by initial mass of contaminant of untreated soil.

Similar type of electro kinetic treatment was carried out with artificially heavy metals chromium and cadmium spiked soils. OECD artificial soil was prepared according to OECD Guidelines (1984) with the composition 70% sand, 20% kaolin clay and 10% finely ground peat. The artificial soil was infused with potassium dichromate to study chromium removal and with cadmium chloride to study cadmium removal. The samples were dried. Contaminants from the different soil sections were extracted by acid digestion in accordance with APHA (1990). Total concentrations of the heavy metals Chromium and Cadmium were determined using an Atomic Absorption spectrophotometer (Varian 4S AA) represented in Table 1 and Fig. 2.

**Table 1. Physico chemical parameters of water samples of the five areas**

Parameters	Karai	Pulianthangal	Bharathi nagar	Tandalam	Maniyambat
pH	7.8	8.5	7.65	7.5	7.5
Electrical conductivity ( $\mu$ mhos/cm)	915	4160	7480	3150	726
TDS mg/lit	749	2212	5443	3405	664
Turbidity mg/lit	2	2	3	2	2
BOD mg/lit	310	755	450	539	420
COD mg/lit	787	1874	745	1290	1326
Total alkalinity mg/lit	188	811	724	936	268
Total Hardness mg/lit	220	2400	1110	920	300
Total Nitrogen mg/lit	85	758	86	710	131.2
Potassium mg/lit	112	543	184	112	117
Phosphorus mg/lit	0.323	0.04	0.04	0.05	0.60
Calcium mg/lit	125	1640	475	912	336
Sodium mg/lit	125	1822	989	1748	158
Chloride mg/lit	210	1750	1370	1540	270
Chromium mg/lit	241	1247	4594	2125	168
Cadmium mg/lit	12	79	78	147	39

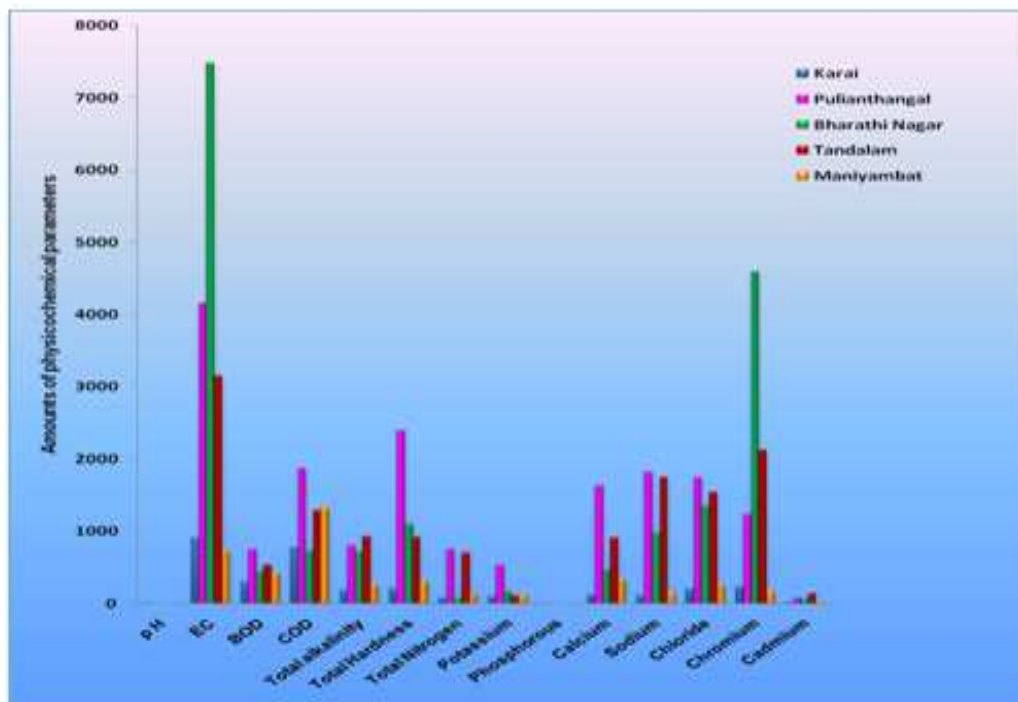


Fig 2. Physico-chemical parameters of water samples of the five areas

Table 2. Physico chemical parameters of soils of the five areas

Parameters	(1) Karai	(2) Pulianthangal	(3) Bharathi nagar	(4) Tandalam	(5) Maniyambat
pH	8.1	7.4	8.1	8.1	7.4
Electrical Conductivity ( $\mu\text{mhos}/\text{cm}^2$ )	315	3110	3450	3721	428
Moisture %	31	32	26	28	35
Porosity	3.15	3.11	3.4	2.24	3.81
Alkalinity mg/Kg	382	411	403	441	381
Salinity mg/Kg	54.2	543.9	252.4	460.1	123.4
Organic matter %	4	6	2.5	9.1	8.1
Total Nitrogen mg/Kg	1128.7	526.7	1569.5	1202.2	1012.6
Phosphorous mg/Kg	123	912	627	396	212
Potassium mg/Kg	91	172	91	301	228
Sodium mg/Kg	442	964	1758	1124	933
Calcium mg/Kg	1045	1031	1322	1010	910
Chromium mg/lit	241	1247	4594	2125	168
Cadmium mg/lit	12	42	35	76	15

### Removal of Chromium by EKR

A sufficient cleaning efficiency could be reached in all soil types, if the duration of cleaning would be longer [18]. Each of these experiments was conducted for 25 days. Treatment time can vary significantly depending on the properties of soil. Literature review showed that extension of process duration would ensure better results. Results shown in Fig 3 & 4 and Table 3 and Table 4 for the experiments performed at 30 V and 50 V showed that efficient removal was observed at 50 V. After 72 hours of EKR treatment, about 3% of chromium migrated towards anode and cathode at 30 V as shown in Table 3. On applying 50 V, the heavy metal deposition on the anode and cathode was 4.4% (Table 4).

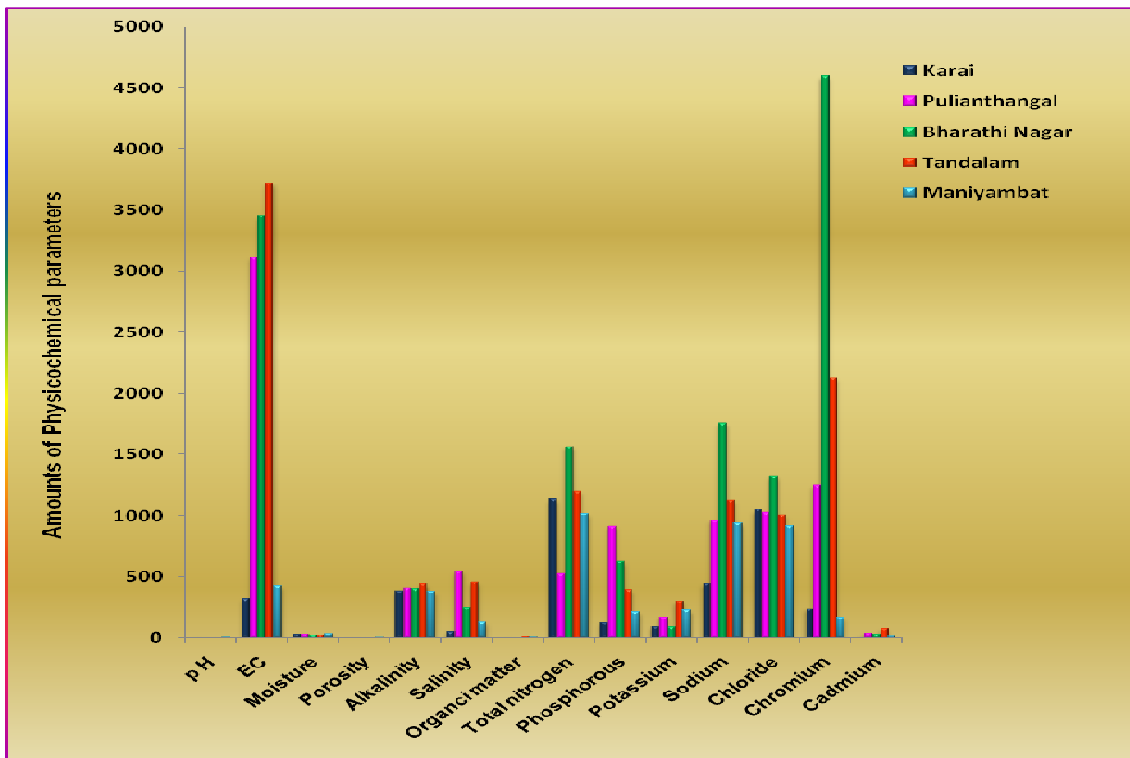


Fig 3. Physico-chemical parameters of soils of the five areas

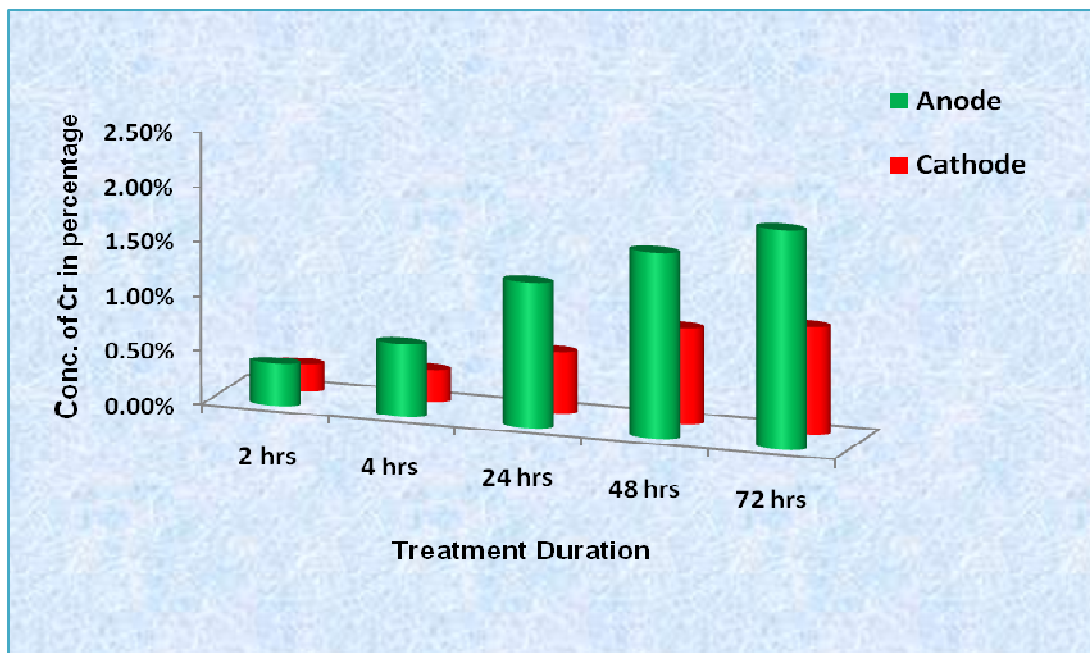


Figure 4. Concentration of Chromium in percentage after Electro kinetic treatment at 30 V

**Table 3. Concentration of Chromium in ppm after Electro kinetic treatment at 30 V**

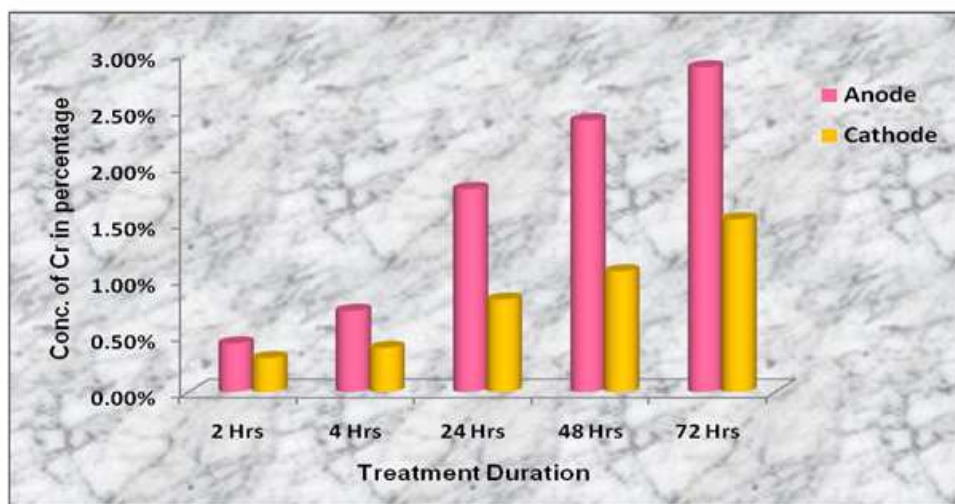
Treatment Duration	Anode	Cathode
2 hrs	8.362(0.39%)	5.435(0.25%)
4 hrs	14.326(0.67%)	6.42(0.30%)
24 hrs	28.162(1.33%)	12.44(0.56%)
48 hrs	36.2(1.7%)	18.64(0.88%)
78 hrs	42.8(2.01%)	21.08(0.99%)

*Values are mean of six individual observations*

**Table 4. Concentration of Chromium in ppm after Electro kinetic treatment at 50 V**

Treatment Duration	Anode	Cathode
2 hrs	9.24(0.43%)	6.43(0.30%)
4 hrs	15.37(0.72%)	8.35(0.39%)
24 hrs	38.35(1.80%)	17.42(0.82%)
48 hrs	51.27(2.41%)	22.64(1.07%)
72 hrs	61.34(2.88%)	32.67(1.53%)

*Values are mean of six individual observations*

**Figure 5. Concentration of chromium after Electro kinetic treatment at 50 V**

The results are compared with heavy metals chromium and cadmium spiked in OECD artificial soil under similar experimental conditions. From the electro kinetic treatment of spiked soil (Table 4 and Figure 5) the following observations were made. On the 25<sup>th</sup> day, 23.13% of chromium was leached out into the water collected from the anode terminal and 7.74% of chromium was detected in the water collected from the cathode terminal. The percentage accumulation of chromium near anode was (490.4 ppm) 23% and at cathode was (242 ppm) 11.4% in the spiked soil. Spiked soil showed remarkable changes compared to experimental soil under the same durations viz., 5<sup>th</sup>, 10<sup>th</sup>, 15<sup>th</sup>, 20<sup>th</sup> and 25<sup>th</sup> days of treatment. The removal efficiency on the 15<sup>th</sup> day was 39.18% and that on the 25<sup>th</sup> day was 65.28%. Results of EKR carried out with spiked soil showed higher removal efficiencies of the heavy metal chromium. This may be attributed to the presence of sand used in the spiked soil preparation and to the absence of other possible contaminants in spiked soil as in the case of experimental soil. Sandy loam and sand have a most coarse grain structure as compared with other types of soil, which ensures a better movement of metal ions and thus better results of cleaning [17]



Table 5. Chromium in spiked soil after Electro kinetic treatment at 50 V

No. of Days	Water from Anode	Anode	Middle Anode	Middle Cathode	Cathode	Water from Cathode
5	75.45(3.55%)	80.02(3.76%)	56.12(2.64%)	44.04(2.07%)	39.24(1.85%)	30.04(1.4%)
10	150.2(7.06%)	206.03(9.69%)	201.24(9.47%)	74.08(3.48%)	50.24(2.36%)	56.32(2.65%)
15	290.37(13.66%)	382.15(17.98%)	256.64(12.07%)	148(6.94%)	85.4(4.02%)	74.65(3.5%)
20	386.3(18.18%)	442.28(20.81%)	297.32(13.99%)	252(11.85%)	220(10.35%)	144.34(6.79%)
25	491.6(23.13%)	490.4(23.07%)	402.4(18.94%)	300(14.17%)	242(11.38%)	164.46(7.74%)

Values are mean of six individual observations

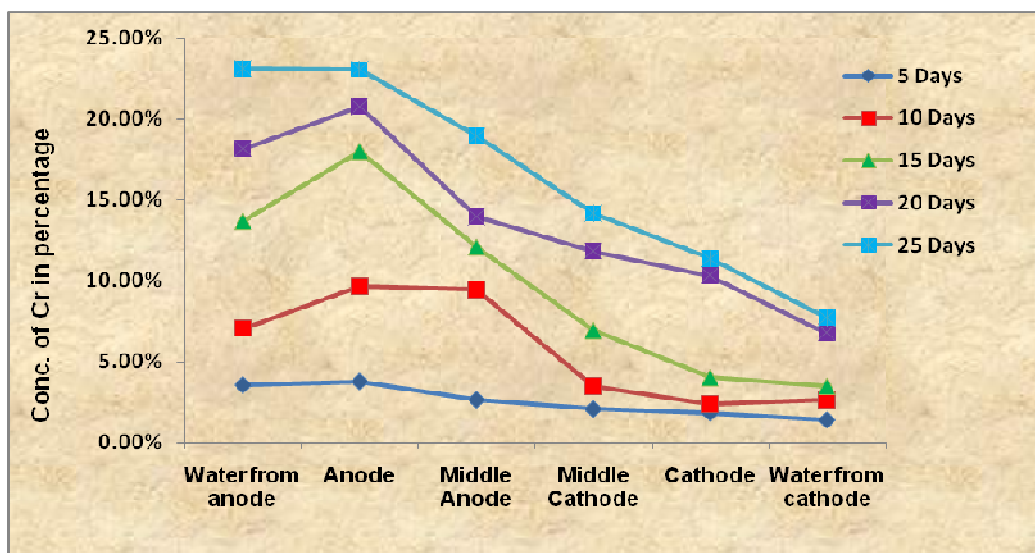


Figure 6. Chromium in spiked soil after Electro kinetic treatment at 50 V

Fig. 6 Show that the EKR treatment carried out on both the experimental and spiked soils, showed that the concentration of chromium in various compartments gradually decreased in the following trend

**Anode > Middle anode > Middle cathode > Cathode**

Previous researches had shown that chromium is available in hexavalent form as soluble chromium and hence it is leached out. On further analysis, it was observed that concentration of chromium is more towards anode than at the cathode confirming the presence of chromium in its anionic forms like  $\text{Cr}_2\text{O}_7^{2-}$ ,  $\text{CrO}_4^{2-}$ ,  $\text{HCrO}_4^-$ . In addition to dissolved metal complexes, metals also may associate with mobile colloidal particles. Colloidal size particles are particles with a diameter ranging from 0.01 and  $10\mu\text{m}$  [18, 19] reported that colloidal particles of intermediate diameter,  $0.1\mu\text{m}$  to  $1\mu\text{m}$ , were the most mobile particles in a sandy medium.

Knowledge of the basic chemistry, environmental and associated health effects of these heavy metals is necessary in understanding their speciation, bioavailability, and remedial options. The fate and transport of a heavy metal in soil depends significantly on the chemical form and speciation of the metal. Once in the soil, heavy metals are adsorbed by initial fast reactions (minutes, hours), followed by slow adsorption reactions (days, years) and are, therefore, redistributed into different chemical forms with varying bioavailability, mobility, and toxicity [20].

The two forms of hexavalent Chromium are pH dependent; hexavalent chromium as a chromate ion ( $\text{CrO}_4^{2-}$ ) predominates above a pH of 6; dichromate ion ( $\text{Cr}_2\text{O}_7^{2-}$ ) predominates below a pH of 6. Hence the metals move more towards anode than towards cathode. The presence of complex species in the soil solution can significantly affect the transport of metals through the soil matrix relative to the free metal ion in the environmental samples. Metals applied with sewage sludge may be retained in the soil as a result of their adsorption on hydrous oxides, clays, and organic matter; the formation of insoluble salts; or the presence of residual sewage sludge particles [21]. The role of carbonates on Cu and Zn retention has been pointed out by other authors and has been partly attributed to the formation of metals carbonates in soils [22].



Many studies were made of electro kinetic removal of heavy metals from spiked model soils - mainly kaolinite [23]. In general, very high removal efficiency was observed with the spiked soils and the duration of the treatment was short.

The leather industry is the main cause for the high influx of Chromium to the biosphere. In India, about 2000 - 32,000 tons of elemental Chromium annually escapes into the environment from tanning industries [24]. The behavior of Cr in the environment is complex. The mobility of Cr is dependent on the speciation of Cr, which is considerably affected by the conditions of the environment. Cr (III) is primarily present in a reducing environment, whereas an oxidizing condition favours Cr (VI) species.

Trivalent Cr (III) and hexavalent Cr (VI) are the two stable species in the environment. Under natural conditions, Cr (III) is readily adsorbed on solid phase, while most Cr (VI) solids are relatively soluble and, therefore, Cr (VI) is generally considered to be more mobile and consequently more bioavailable than Cr (III) in soil-water systems [25]. The concentration of metals in the soil solution, at any given time, is governed by a number of interrelated processes, including inorganic and organic complexation, oxidation reduction reactions, precipitation/dissolution reactions, and adsorption/desorption reactions. Metals will form soluble complexes with inorganic and organic ligands. Common inorganic ligands are  $\text{SO}_4^{2-}$ ,  $\text{Cl}^-$ ,  $\text{OH}^-$ ,  $\text{PO}_4^{3-}$ ,  $\text{NO}_3^-$  and  $\text{CO}_3^{2-}$ . Soil organic ligands include low molecular weight aliphatic, aromatic, and amino acids and soluble constituents of fulvic acids [26].

## RESULTS AND DISCUSSION

The high metal concentration in the soil samples of all the five sites may be due to the tanneries and chemical industries. The tannery effluent is mainly responsible for high levels of sodium and other salts in soils and well waters near these areas. For agricultural sustainability monitoring soil salinity in agricultural lands is to be carried out and this can be easily done by soil electrical conductivity. Soil salinity refers to the presence of major dissolved inorganic solutes, including charged species (e.g.,  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Cl}^-$ ,  $\text{HCO}_3^-$ ,  $\text{NO}_3^-$ ,  $\text{SO}_4^{2-}$  and  $\text{CO}_3^{2-}$ ), nonionic solutes and ions that combine to form ion pairs. Salinity limits water uptake by plants by reducing the osmotic potential. Salinity may also cause specific ion toxicity or upset the nutritional balance. The salinity level influences the exchangeable cation composition, soil permeability and crop productivity.

Studies had been carried out in Ranipet Industrial area and the report showed that several acres of the land area were irreversibly polluted due to the discharge of untreated effluents from the tanneries and leather processing units. Hence from the results of the above water and soil analyses it became evident that all the sites in the Ranipet Industrial area are heavily polluted by various physico-chemical factors and heavy metals due to irresponsible disposal of untreated or partially treated effluents into the surrounding environments by the industries. Maintaining strict pollution control regulating measures alone can prevent further damage of the environment by the industries.

The lands which are already damaged and remain uncultivable as shown in Plate 4 should be remediated by suitable methods so that the farmers get back their lands for cultivation. It becomes the responsibility of the environmental scientists to find the measures which are cost effective, viable and do not cause disposal problems or secondary pollutants.

While total metal content is a critical measure in assessing the risk of a contaminated site, total metal content alone does not provide predictive insights on the bioavailability, mobility and fate of the metal contaminant. From the perspective of risk assessment, speciation of metals from environment samples becomes important. Speciation concerns the identification and quantification of specific forms of an element. As different forms of an element may exhibit differing toxicities and mobilities in the environment, it becomes important to distinguish between the individual species present in a particular sample.

The Cr (VI) fraction decreases towards the anode which is indicative of preferential migration of  $\text{Cr}_2\text{O}_7^{2-}$  into the anode chamber. The opposite trend is true for Cr (III). Monomeric hydroxide complexes of Cr (III) would remain stable in solutions at the pH levels encountering in the region of the anode but above pH 4 they hydrolyze to form poly nuclear complexes and ultimately precipitated from solution [27] mostly the alkaline pH maintained in the clay soil helps the formation of hydrooxo complexes and these are removed due to electromigration and consequently high percentage removal of the trivalent Cr is recorded.

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**CONCLUSION**

The soil contaminated by the tannery industry in the area Ranipet is analyzed and the removal of toxic chromium by electro kinetic method has been suggested. The results of the electro kinetic extraction may be modestly effective in removing Cr from the soil. Electro kinetic extraction thus has several procedural advantages that would help reduce the cost and complexity of extraction of Cr from the contaminated soil.

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