

Mercury Removal Using Fe –Fe Electrodes by Electrocoagulation

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ABSTRACT: The performance of electrocoagulation is a promising electrochemical technique with iron sacrificed anode for waste water treatment on laboratory scale was done. Several parameters like initial metal concentration, pH, voltage, agitation and electrolyte concentration at different time were investigated to achieve a higher removal rate of mercury. Experiments were carried out with different initial metal concentration ranging from 30 ppm, 50 ppm and 100 ppm, pH ranging from 2.5 pH, 4.5 pH and 7.0 pH, Voltage vary from 6V, 9V and 12V, electrolyte concentration changes from 0.6667 g/lit., 1.333 g/lit. and 2.0 g/lit., agitation vary from 200 rpm, 400 rpm and 600 rpm with a constant spacing between two electrodes is 1.0 cm. Maximum mercury removal was achieved at the end of 40 minute operation. Total operating cost also calculated from the power cost and cost of electrode material. The method was found to be highly efficient and relatively fast compared to conventional existing techniques.

Keywords: Electrocoagulation, Heavy metals, Iron electrodes, Operating cost.

I. Introduction

Legislative regulation concerns with the discharge of waste water are strictly increase drastically, due to higher toxicity of metal ions. Different methods like precipitation, coagulation, sedimentation, adsorption, ion exchange and reverse osmosis [1, 2 and 3]. The difference between electrocoagulation and chemical coagulation is based on the delivered of iron ions [4]. In the electrocoagulation, coagulation and precipitation are not conducted through chemicals – known as coagulants – to the process, but via electrodes in the electrolytic cell [5]. Electrochemically is the most promising technologies to a clear environment with a broad range of technology to treat wastewater. Electrochemical consist of electrocoagulation and electrolysis, have been successfully developed for the treatment of different kind of industrial waste waters [6, 7 and 8]. In electrolysis oxidation, reduction and decomposition whereas electrocoagulation coagulation, adsorption, precipitation and floatation [9, 10 and 11] mechanisms employed to remove pollutant from waste waters. Since the turn of the 19th century, electrocoagulation widely applied for the treatment of waste water [12]. Electrocoagulation process bears merits such as safety, selectivity, versatility, amenability to automation, environmental compatibility and energy efficiency.. Electrocoagulation process is also characterized by simple and easy operated equipment, short operation time, none or negligible amounts of chemicals and low sludge production [13 and 14]. Electrocoagulation is a generation process of metallic hydroxide flocks by electro dissolution of sacrificial anode generally made of iron or aluminum [11]. The metal ion generation takes place at the anode and hydrogen gas is released from the cathode. Also, hydrogen gas is also float the flocculated particles from the water. The mechanism of electrocoagulation is highly dependent on the chemistry of aqueous medium, pH, particle size and chemical constituent concentrations will also influence the electrocoagulation process. Electrode assembly is the heart of the electrolytic cell. Different electrodes are utilized like, iron [15], aluminum [16], graphite, titanium [17], carbon [18] and mild steel [19]. Although, aluminum and iron are extensively used to clarify waste water [20]. They are cheap, readily available and proven effective [21].

The electrocoagulation has successfully been applied to the treatment of removal of arsenic [22], phosphate [23], sulfide, sulfate and sulfite [24], boron [25], fluoride [26], nitrate [27], chromate [28], restaurant waste water [29], Alcohol distillery waste water [30], treatment of copper, lead and cadmium in natural water and simulated waste water [31].

In addition, the following physico-chemical reaction may also took place in the electrocoagulation cell [31]. (i) cathodic reduction of impurities present in the waste water (ii) Discharge and coagulation of colloidal particles (iii) migration of ions due to electrophoretic action (iv) Due to generation of oxygen and hydrogen bubble electrofloatation of coagulated particles took place (v) reduction of metal ions at the cathode (vi) other chemical and electrochemical process.

The main objectives of the present work were to gain into some fundamental mechanisms and possible interactions influencing the removal process of mercury by electrocoagulation. The process was examined under different values of initial pH, initial concentration, electrolyte concentration, applied potential and agitation in order to determine optimum conditions.

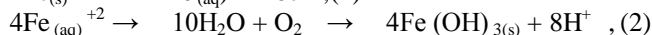
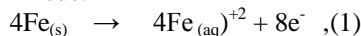
II. Electrocoagulation Mechanism

This technique has recently attracted a great deal of attention. Electrocoagulation or enhanced coagulation accompanied through electrofloatation is an efficient electrochemical water and waste water treatment technology. Electrocoagulation consists of situ formation of coagulants due to electrodisolution of sacrificial anode Coagulants are responsible for aggregation as well as precipitation of suspended particles and simultaneously adsorption of dissolved pollutants. Tiny bubbles of hydrogen and oxygen generated at cathode and anode respectively collide with air bubbles and float the pollutant particles.

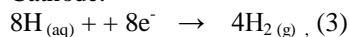
In electrocoagulation, iron is oxidized to iron hydroxide, Fe (OH) n , where n is 2 or 3. The following mechanisms have been proposed for the formation of Fe (OH) n [32]:

Mechanism: 1

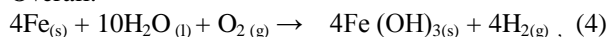
Anode:



Cathode:

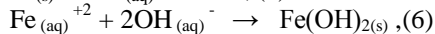
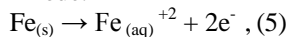


Overall:

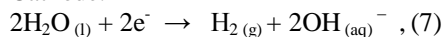


Mechanism: 2

Anode:



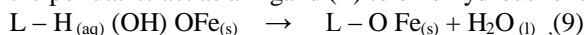
Cathode:



Overall:



The generated $\text{Fe}(\text{OH})_n$ remains in the aqueous solution as a gelatinous suspension, which can remove the pollutants either by complexation or electrostatic attraction followed by coagulation [32]. In the case of surface complexation the pollutants act as a Ligand (L) to bind hydrous ions:



Also, in many wastewaters which contain chlorides, so another strong oxidant of hypochlorite may be developed which is given as per following equation [33 and 34]:



Iron electrode oxidize and ferric ions generated and forms monomeric ions $\text{Fe}(\text{OH})_3$, and hydroxyl complexes like $\text{Fe}(\text{H}_2\text{O})_6^{3+}$, $\text{Fe}(\text{H}_2\text{O})_5^{2+}$, $\text{Fe}(\text{H}_2\text{O})_4(\text{OH})_2^{+}$, $\text{Fe}(\text{H}_2\text{O})_5\text{OH}^{2+}$, $\text{Fe}_2(\text{H}_2\text{O})_8(\text{OH})_2^{4+}$, $\text{Fe}_2(\text{H}_2\text{O})_6(\text{OH})_4^{4+}$ [35]. Generation of these complexes strongly depend upon pH [36]. The formation of these hydroxides, polyhydroxides, and polyhydroxy metallic are responsible for cause of coagulation which have affinity with dispersed as well as counter ions.

III. Experimental and Set up Procedure

3.1 Materials and methods

Electrocoagulation was carried out by preparing synthetic solution of 30 ppm, 50 ppm and 100 ppm concentration by dissolving the require quantity of mercury chloride in a conventional 1000 ml glass beaker. The net spacing between the electrodes are 10 mm in order to minimize the IR drop [36] which is shown in fig. (1). Fe plates of 140 mm x 18 mm x 2 mm were used as electrodes for electrocoagulation. The effective surface area of each electrode was 60 mm x 18 mm x 2 mm. The entire electrode assembly was fitted on non conducting wedges and hanged from the electrocoagulation cell. At beginning of the each run, 750 ml of desired concentration was poured into the reactor. Conductivity of the solution was improved by addition of sodium chloride as an electrolyte in order to reduce the IR – drop or solution resistance potential [36 and 37]. The added amount of sodium chloride was 0.6667 gm/lit, 1.333 gm/lit and 2.0 gm/lit. All chemicals were of analytical grade and supplied by Merck, Germany. All the experiments were carried out at room temperature. The electrodes were connected to D.C. power source in a monopolar mode. The electrical contacts were established with crocodile clips.

The electrode assembly was placed in the cell and the electrodes were connected to respective anode and cathode for a different time interval from 5 minute to 60 minute and voltage vary from 6V, 9V and 12V. All runs were performed at different stirring speed of 200 rpm, 400 rpm and 600 rpm. In each run 750 ml. HgCl_2 solution placed in the electrolytic cell with a pH range of 2.5 pH, 4.5 pH and 7.0 pH for a different electrolyte concentration of 0.6667 g/lit., 1.333 g/lit., 2.0 g/lit. The treated sample collected at different time interval. After each run, the power was switched off and electrodes were dismantled. Before each run, electrodes were washed thoroughly with water to remove any solid residues on the surfaces, dried and reweighed to calculate sacrificial electrodes consumption.

3.2 Chemical analysis

The pH was monitored with a high precision pH meter (micro process based pH system, model 1012 - E) which was equipped with a combined glass electrode. The initial pH of the solutions was adjusted by adding NaOH or H_2SO_4 solutions. Conductivity of the solution measured with electronic conductivity meter (micro process based conductivity meter, model 1601 - E) equipped with an immersion measurement probe. The total concentration of mercury in solution was determined with (ELCO, SL – 159) UV - vis spectrophotometer at 575 nm according to Beer – Lambert law. The mercury removal efficiency is calculated as follows: [15]

$$\text{CR} (\%) = \frac{\text{Co} - \text{Ce}}{\text{Co}} \times 100 \text{ , (11)}$$

Where CR is the removal efficiency, Co – mercury concentration at initial, Ce – mercury concentration at equilibrium.

IV. Result Analysis

4.1 Influence of electrocoagulation time

To explore the effect of operating time for electrolysis, optimum parameter maintained for initial pH 4.5, initial metal concentration 50 ppm, stirring rate 400 rpm, applied potential 9V and electrolyte concentration 1.333 gm/lit. to achieved 94.5% at 40 minute demonstrated in fig (2). The electrocoagulation process is controlled many parameters like pollutant concentration and composition, initial pH and conductivity. Additionally, electrode material applied potential, charge loading and coagulation time effect the process. During electrocoagulation, anodic and cathodic reduction occurs at positive and negative electrode respectively. Lively evolution of oxygen responsible of reduction the anode dissolution which is related with dissolved amount of iron. The released ions neutralized the particles charges so initiate the coagulation.

Initially, minor formation of oxygen at sacrificial anode competes with iron dissolution which reduced the dissolved amount of iron at the sacrificial anode which cause co-precipitation and sweeping effect and attributed to slower removal rate of mercury shown in table (1). Optimum removal efficiency gained at 40 minute with 94.5 % as formation of enough coagulant iron hydroxides concentration. But due to continuous electrocoagulation, some of hydroxides ions oxidized at anode. So generation of iron is reduced [38] and efficiency of removal of mercury decrease by about 12.2 % after 20 minute.

As electrocoagulation proceeding, is responsible for cause of increase of concentration ions and their hydroxide flocks [35] lead to increase of pH. Initially, in the electrocoagulation, slower increased of pH observed due to slower generation of hydroxide ion with increasing the solubility. But as electrocoagulation proceed, reduction of solubility was took place which cause ultimate rise of 43 % in pH of the solution with increasing the hydroxide ion generation. But rate of pH decrease slightly up to 6.44 units due to incremental improvement in the average OH/Fe mole ratio due to decline of solubility.

4.2 Effect of applied potential

It is well known that during electrocoagulation, current passing through the circuit has raised due to increased applied potential. To evaluate the optimum mercury removal efficiency, experiments were conducted at different applied potential such as 6V, 9V and 12V with the different optimum conditions such as initial pH 4.5, initial metal concentration 50 ppm, stirring rate 400 rpm and electrolyte concentration 1.333 gm/lit. to achieved as high as 94.5 % removal efficiency of mercury at 40 minute demonstrates in fig (3) with data in table (2). Also, it is fact that current not only determines the coagulant dosage rate [2, 7, 27] but also the bubble production rate, size and flocks growth which closely affect the mercury removal efficiency of mercury. As per [7, 27, 39, 40, 41] rise of mercury removal efficiency incurred as increase of current and current density. When applied potential rate is increased from 6V to 9V mercury removal efficiency increased from 85 % to 94.5 and then decreased at 12V about 27.2 % of optimum value of 9V. It seems that increasing the applied potential, more Fe^{+2} and OH were generated in the electrochemical cell which enhance the coagulant dosage cause accelerated mercury removal at a optimum value of 94.5 % at 40 minute. Above the critical value, dissolution of Fe^{+2} exceeded, leads to excessive generation of oxygen with consequently heat generation. Therefore, no or more Fe^{+2} ions remain in the solution and decreased mercury removal about 14.7 %, 12.2 % and 13.4 % of critical value of 6V, 9V and 12V respectively [42].

4.3 Effect of agitation

Batch electrocoagulation experiments were conducted for different time and optimize the mercury removal efficiency at a different time interval from 5 minute to 60 minute with different rpm at 200, 400 and 600 illustrated in fig. (4). As stirring rate was increased from 200 rpm to 400 rpm rate of collision and mobility of ions were increased slowly increased with agitation. At critical value of 400 rpm flocks formation was higher which intimately associated with each other and precipitation became easier cause [41] significantly maximum mercury removal as high as 78.3 % and 14.3 % for 200 rpm and 600 rpm respectively. But at higher agitation, solubility of precipitates increase or some time unsuitable flocks formation took place which was leading reasons for decreasing 23.6 %, 12.2 % and 14.2 % the mercury removal efficiency for 6V, 9V and 12V respectively after critical value shown in table (3).

4.4 Cost estimation

Economic analysis includes consist different operating cost like electrodes cost, energy consumption cost, as well as labor, sludge transportation and disposal, maintenance and fixed costs. The later costs are largely independent of the type of the electrode material. For calculation of the operating cost (US \$/m³), energy consumption cost (US \$ KWh/m³) and electrodes material cost (US \$ Kg/m³) have been taken into account as a major cost for economic investigation.

Operating cost = a Cenergy + b Celectrodes

Where 'a' is the electrical energy price (US \$ KWh/m³) and 'b' is the electrode material price (US \$ /Kg Fe). Cost of electrode material (Kg Fe/m³ HgCl₂ solution) is calculated as per following equation by Faraday's law:

$$\text{Celectrodes} = \frac{I \times t \times Mw}{Z \times F \times v} \quad (12)$$

Where: I = Current (A)

t = Electrolysis time (s)

Mw = Molecular mass of Fe (g/mol)

F = Faraday's constant (c/mol)

Z = number of electron transfer

v = volume of HgCl₂ solution (m³)

The major cost of electrocoagulation is associated with electrical energy consumption during electrocoagulation shown in table (4). It is calculated in (KWh/m³ HgCl₂ solution) as per following equation :

$$C_{\text{energy}} = \frac{V \times I \times t_{\text{EC}}}{v} \quad (13)$$

Where: V = Cell voltage (V)

I = Current (A)

t_{EC} = electrolysis time (s)

v = Volume (m³) of HgCl₂ solution

Unit price a, b given for the Indian market April – 2008 was as follows:

- (1) electrical energy price 0.5570 US \$/ KWh
- (2) electrodes material price 1.7722 US \$/Kg for Fe

Cost due to electrical energy consumption and electrode material are calculated for different time shown in fig. (5) follows the data mention in table no. (4) in which both are increasing with time. In fig. (6), it seems that the operating cost shown in table (5) is also increased almost linearly with time due to increase in consumption of electrical energy as well as electrode material.

V. Conclusion

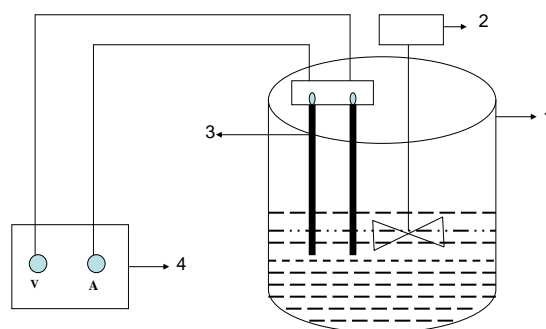
In this study, behavior of iron electrode in the electrocoagulation process has been investigated. On the base of wide range of experiments, different evidence was concluded. The removal rate of mercury was affected by different factors like – initial pH of the solution, electrolyte concentration, applied potential, initial concentration and agitation. By keeping optimum pH – 4.5, ppm – 50, voltage – 9V, agitation – 400 rpm and electrolytic concentration 1.333 g/lit., 94.5 % mercury removal achieved within 40 minute. In the electrocoagulation cell, due to formation of hydroxyl ions forms at the cathode. Removal rate of mercury start from slow rate and achieved a peak value then rate falls. During electrocoagulation, quick and effective mercury removal achieved.

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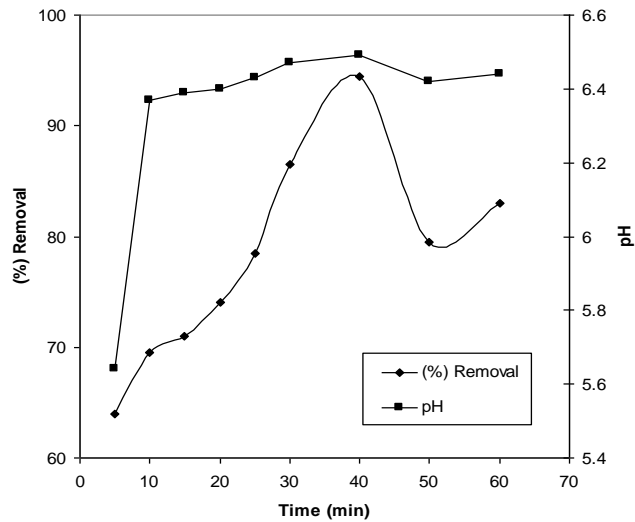
FIGURES AND TABLES



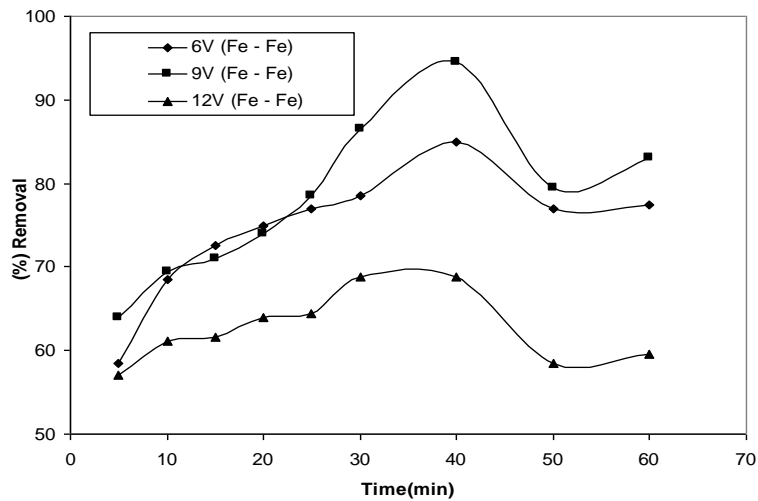
Schematic process arrangement

1. Electrolytic cell
2. Stirrer
3. Mild steel electrode pair
4. Power supply (V, Voltage regulator and A ,current regulator)

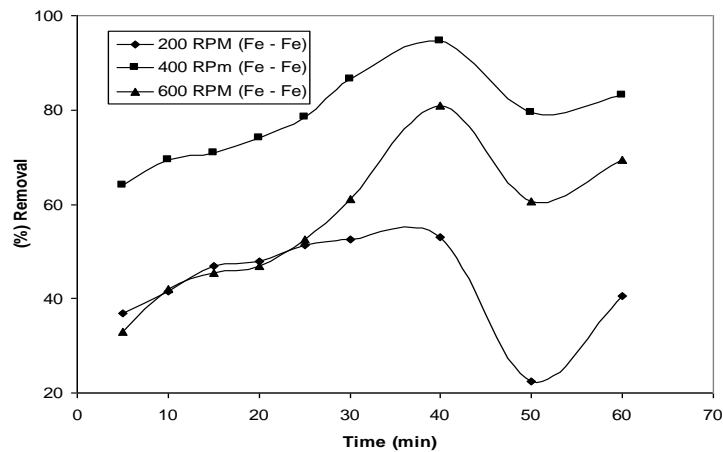
“Fig. (1) Electrolytic cell”



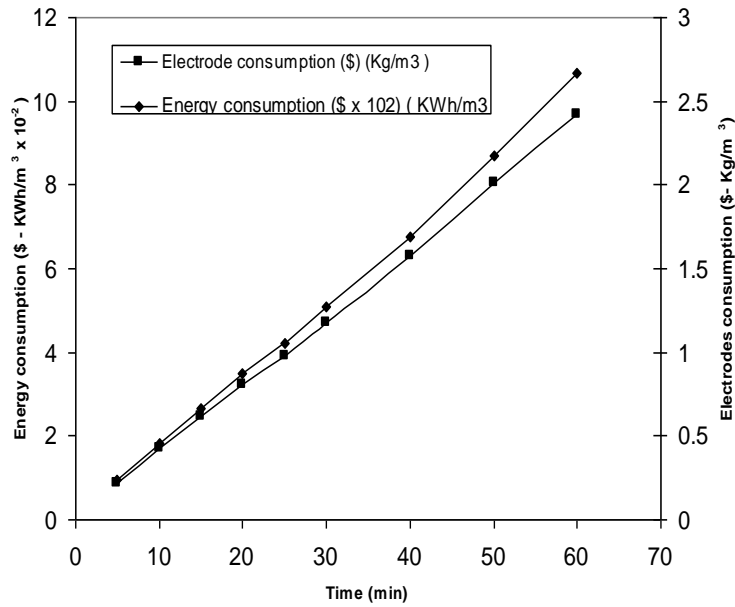
“Fig. (2) % Removal efficiency of mercury and pH with electrocoagulation Time ppm – 50, Voltage – 9V, Agitation – 400 rpm, Electrolyte concentration – 1.333 g/lit., pH – 4.5”



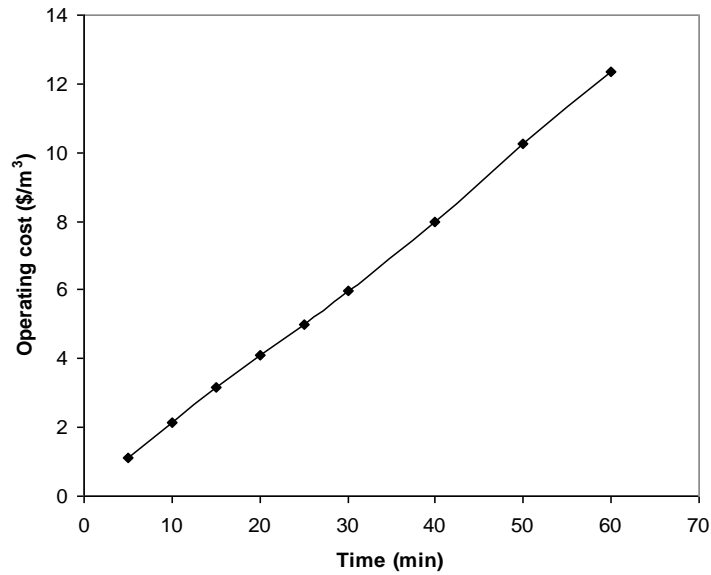
“Fig. (3) Effect of applied potential on the removal efficiency of mercury with Time. ppm – 50, Agitation – 400 rpm, Electrolyte concentration – 1.333 g/lit., pH – 4.5”



“Fig. (4) Effect of agitation on the removal efficiency of mercury with time. ppm – 50, electrolyte concentration 1.333 g/lit., Voltage – 9V, pH – 4.5”



“Fig. (5) Effect of electrolysis time on energy consumption (\$ x 10²) (KWh/m³) and electrode consumption (\$) (Kg/m³ of HgCl₂ solution). ppm – 50, Voltage – 9V, Agitation – 400 rpm, Electrolyte concentration – 1.333 g/lit., pH – 4.5”



“Fig. (6) Effect of electrolysis time on operating cost (\$/m³). ppm – 50, Voltage – 9V, Agitation – 400 rpm, Electrolyte concentration – 1.333 g/lit., pH – 4.5”

TABLE (1) % Removal efficiency of mercury and pH with electrocoagulation Time

<i>Time (min)</i>	<i>(%) Removal</i>	<i>pH</i>
5	64	5.64
10	69.5	6.37
15	71	6.39
20	74	6.4
25	78.5	6.43
30	86.5	6.47
40	94.5	6.49
50	79.5	6.42
60	83	6.44

TABLE (2) Effect of applied potential on the removal efficiency of mercury with Time

<i>Time (min)</i>	<i>(%) Removal</i>		
	<i>6 Volt</i>	<i>9 Volt</i>	<i>12 Volt</i>
5	58.5	64	57
10	68.5	69.5	61.2
15	72.5	71	61.6
20	75	74	64
25	77	78.5	64.4
30	78.5	86.5	68.8
40	85	94.5	68.8
50	77	79.5	58.5
60	77.5	83	59.6

TABLE (3) Effect of agitation on the removal efficiency of mercury with time.

<i>Time (min)</i>	<i>(%) Removal</i>		
	<i>200 RPM</i>	<i>400 RPM</i>	<i>600RPM</i>
5	37	64	33
10	41.6	69.5	42
15	46.8	71	45.5
20	48	74	47
25	51.4	78.5	52.5
30	52.5	86.5	61
40	53	94.5	81
50	22.5	79.5	60.5
60	40.5	83	69.5

TABLE (4) Effect of electrolysis time on energy consumption ($\$ \times 10^2$) (KWh/m³) and electrode consumption (\$) (Kg/m³)

<i>Time (min)</i>	<i>Energy consumption (\$ x 10²) (KWh/m³)</i>	<i>Electrode consumption (\$) (Kg/m³)</i>
5	0.2338	0.8686
10	0.4566	1.6989
15	0.6684	2.4818
20	0.8688	3.2263
25	1.0583	3.9295
30	1.27	4.7154
40	1.6931	6.2872
50	2.1729	8.0658
60	2.6668	9.679

TABLE (5) Effect of electrolysis time on operating cost (\$/m³).

Time (min)	Operating cost (\$/m ³)
5	1.074
10	2.1525
15	3.1502
20	4.0957
25	4.9879
30	5.9854
40	7.9803
50	10.2387
60	12.3458