

Electrocoagulative treatment of mercury containing aqueous solutions

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ABSTRACT

In recent years, electrocoagulation has been successfully used for wastewater treatment and is efficient in heavy metal ions removal. In the present work, electrocoagulation has been used for the removal of Hg(II) from synthetic wastewaters containing up to 20 mg/L of mercury. The electrode materials used are stainless steel (SS) and aluminum (Al). The effects of operating parameters, viz., current density, time of electrocoagulation, distance between electrodes, initial pH of the solution, presence of electrolyte in the solution and temperature have been studied. It was observed that more than 99% Hg(II) removal can be achieved by keeping the distance between SS and Al electrodes from 2 to 6 cm and initial pH range from 3 to 7. The results show that the pseudo second-order kinetics fits the data well. Also, preliminary cost estimation was reported.

Key words | electrometallurgy, hydrometallurgy, pollution, wastewater

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INTRODUCTION

Various pollutants present in wastewater generated from different types of chemical process industries are necessary to be treated before they are discharged into natural water sources. Various metals like Zn, Cu, Ni, Pb, Hg, Cr, B, Cd, etc. are present in effluents generated from industries like metal plating, mining, smelting, battery manufacturing, tanneries, petroleum refineries, paint and pigment manufacturing, pesticides manufacturing, printing and photographic, etc. Among all the metals present in the pollutants, treatment of mercury is most important because it is considered to be one of the most toxic metals in wastewater (Dong *et al.* 2008). Mercury is considered as carcinogenic, teratogenic and promotes tyrosinemia. If a high amount has been taken, it can create impairment of pulmonary and kidney function, chest pain and dyspnoea (Zhang *et al.* 2005). The permissible discharge limit as per the United States Environmental Protection Agency (EPA) for total mercury present in wastewater is 10 µg/L and the limit for drinking water (Zhang *et al.* 2005) is 2 µg/L. The World Health Organization (WHO) suggests maximum uptake of 0.3 mg/week and 1 µg/L as maximum acceptable limits in drinking water. Generally, mercury is present in organic and inorganic forms and both are cumulatively toxic (Tennakone & Ketipearachchi 1995). We can find mercury in effluents from industries like

chlorine manufacturing, switchgear/battery manufacturing, fluorescent lamp manufacturing, paint manufacturing, fertilizers manufacturing, pharmaceuticals, agrochemicals manufacturing and dental wastes (Hassan *et al.* 2008). Mercury is also used in the mercury cell for the production of caustic soda and chlorine and is generating 50% NaOH with 30 ppm salt concentration, which is higher than membrane cell and diaphragm cell (Austin 1984). But, a small loss of mercury in the effluent can create extreme pollution problems for the environment. Emission limitation for mercury is 0.01 mg/L provided by the Central Pollution Control Board (CPCB) of India for different industries (CPCB 2010).

There are numerous studies available in literature which used biomaterials, activated sludge, bacteria, algae, yeasts, fungi, etc. to treat wastewaters containing metal ions. Lipczynska-Kochany & Kochany (2009) treated wastewater containing heavy metals (Cr, Cu, Fe, Mn, Ni and Zn) using biological treatment and they achieved 90% removal efficiency. Ahluwalia & Goyal (2007) in their work concluded that biomass-based technologies need not necessarily replace the conventional treatment routes but may complement them. At present, information on different biosorbent materials is inadequate to accurately define the parameters for process scale-up and

design perfection including reliability and economic feasibility. To provide an economically viable treatment, the appropriate choice of biomass and proper operational conditions has to be identified. Wagner-Döbler *et al.* (2000) used applied microorganisms for the removal of mercury from chemical wastewater. They have taken an initial concentration in the range of 3–10 mg/L and achieved removal efficiency from 95 to 99%.

Pagnanelli *et al.* (2009) studied Cd and Pb removal using activated sludge in sequencing batch reactors and suggested mechanism of separation. Özbelge *et al.* (2005) reported about the uptake of Cu^{2+} and Zn^{2+} by activated sludge at ambient conditions (pH 7 and 25 °C). Arican *et al.* (2002) have observed that the activated sludge exhibits both active and passive uptake of Ni^{2+} , and concluded that the *d* passive uptake was the dominant mechanism in Ni^{2+} sorption. Luo *et al.* (2006) studied removal of heavy metal ions, such as Cu^{2+} , Cd^{2+} and Zn^{2+} , from municipal sewage using waste activated sludge and found 96.47, 80 and 90% removal, respectively. Bux *et al.* (1999) reported Zn removal from metal plating effluent using waste activated and digested sludges. Bakkaloglu *et al.* (1998) reported a comparative study of various types of waste biomasses and bacteria in the removal of Zn, Cu, and Ni from aqueous solutions. Dilek & Gökçay (1996) reported laboratory scale results on the removal of Ni and Cr from simulated wastewaters. Javaid *et al.* (2011) reported use of a macro-fungus to remove Cu, Ni, Zn and Cr from water. There are good review articles published on the removal of metal ions by various biomaterials, including activated sludge, bacteria, algae, yeasts, fungi, etc. (Basha & Murthy 2007; Wang & Chen 2009; Fu & Wang 2011). But biological treatment methods are insufficient for the treatment of pollutants containing heavy metals, since they are non-biodegradable (Ngah & Hanafiah 2008). So, one has to go for alternatives like chemical treatment methods, electrochemical treatment methods, membrane separations, etc.

Various chemical treatment methods like chemical coagulation, adsorption, solvent extraction etc. are very efficient but generate secondary pollutants after treatment. In this case we have to go either for membrane separations or electrochemical treatment methods like electrocoagulation, electrofloatation, electro-oxidation, etc. Experimental studies on various metals removal by electrocoagulation show that the metals removal efficiency is very good and can be more than 99% in many cases (Bektas *et al.* 2004; Escobar *et al.* 2006; Gomes *et al.* 2007; Heidmann & Calmano 2008a, b; Chou *et al.* 2009; Nanseu-Njiki *et al.* 2009; Murthy & Parmar 2011).

Open literature survey shows that there is only one article available on the treatment of mercury containing water treatment by electrocoagulation (Nanseu-Njiki *et al.* 2009). Effect of operating parameters like distance between electrodes, current density, charge loading and initial pH have been studied; but effects of parameters like electrocoagulation time, presence of electrolyte, temperature and cost estimation have not been reported. In our work all these parameters have been studied and preliminary cost estimation has been reported. In addition, study of adsorption kinetics has also been reported.

THEORY

Electrocoagulation has the potential to replace conventional wastewater treatment plants (Mollah *et al.* 2004; Emamjomeh & Sivakumar 2009). In the electrocoagulation experiment a pair of anodes and cathodes made of iron and aluminum (Al) can be used. Electrical potential is applied from an external power source. Oxidation of the anode material takes place, while the cathode will be subjected to reduction or reductive deposition of elemental metals. The following chemical reactions have been proposed to be taking place during the process (Mollah *et al.* 2004; Emamjomeh & Sivakumar 2009; Moreno *et al.* 2009):

At the anode:



At the cathode:



where M is either Fe or Al, depending on which type of electrode is being used in the process, and *n* is the number of electrons. If we use Fe electrodes, we will get reddish brown corrosion product of iron which is known as rust (Moreno *et al.* 2009). During the electrocoagulation process metal hydroxides, polyhydroxides and/or polyhydroxymetallic compounds of the electrode material will be generated. These materials contain strong affinity for dispersed particles and counter ions which results in coagulation (Hansen *et al.* 2008).

MATERIALS AND METHODS

Materials and instruments used were stainless steel (SS) and Al electrodes (commercial grade, total immersed surface area = 5.655 cm²), multiple DC power supply device (0–30 V, 2 Amp. Scientech Technologies, Indore, India), magnetic stirrer (Labstar, Mumbai, India), UV-VIS Spectrophotometer (Hach, DR5000, Loveland, Colorado, USA), mercury (II) chloride (99.5% pure, Merck, Mumbai, India), dithizone (C₁₃H₁₂N₄S, S.D. Fine-chem, Mumbai, India), ammonia buffer solution (Finar, Ahmedabad, India), chloroform (CHCl₃, Finar, Ahmedabad, India), and distilled water (pH 5.9 ± 0.2, conductivity 1.0 μS/cm produced by Millipore, Elix, Bangalore, India). All the experiments were performed in a 500 mL beaker in batch mode. Each batch contained 200 mL of feed. After each run, solution obtained was collected and filtered using filter paper (Sonar[®] 1, Delhi, India). Filtrate was collected and analyzed. Analysis has been done by a first-order derivative spectrophotometric method using dithizone as reagent (Mathew *et al.* 1995). Absorbance has been measured at 500 nm.

Laboratory-scale experiments were performed for the removal of Hg(II) from synthetic wastewaters containing up to 20 mg/L of Hg(II) in batch scale mode using SS and Al electrodes. Effects of operating parameters, viz., electrocoagulation time, current density, presence of electrolyte, distance between electrodes, initial pH and temperature on the Hg(II) removal efficiency have been studied. The Hg(II) removal data have also been used to find the process kinetics.

RESULTS AND DISCUSSION

Effects of electrocoagulation time, current density, concentration of NaCl, distance between electrodes, initial pH and temperature have been investigated. Percent Hg(II) removal has been calculated using the relation $[(1 - (C/C_0)) \times 100]$, where, C₀ and C are concentrations of Hg(II) before and after treatment, respectively.

Effect of time of electrocoagulation

Time of electrocoagulation is one of the most important parameters. Optimization of time of electrocoagulation is essential because finding the minimum required time for desired removal is necessary. In the present work, it can be observed from Figure 1(a) that the removal of Hg(II) increased drastically at the initial stage of the process

up to 10 min (99% removal) in the case of SS electrodes and 15 min (98% removal) in the case of Al electrodes. In the case of SS electrodes after 15 min electrolysis time the observed removal percent is 99.7%. At the end of 30 min process time SS electrodes removed 99.83% Hg(II) and Al electrodes removed 99.37% Hg(II). Hence, 15 min process time has been taken as optimum time for further experiments.

Effect of current density

Current density is defined as the current applied per unit surface area of the electrode. The current density has to be optimized because higher current density does not mean higher removal after certain limit. From Figure 1(b), it can be observed that an increase in current density results in increased Hg(II) removal. Here, optimum current density is considered to be 1.77 A/dm², as after this value the percent separation of Hg(II) was marginal.

Effect of electrolyte concentration

Addition of electrolytes to solutions will increase the conductivity of the medium. Electrolytes like NaCl, KCl, Na₂SO₄, K₂SO₄, NaNO₃, etc. can be used for this purpose. Since, NaCl is the most commonly and cheaply available electrolyte, researchers have widely used it to increase the conductivity of the system solution. It was mentioned that the presence of 20% Cl⁻ in the solution is required for the normal operation of electrocoagulation and reduce the adverse effect of other ions present in the solution, and also the electrochemically generated Cl will be effective in water disinfection (Chen & Hung 2007). In the present work, without adding electrolyte only 54% Hg(II) removal by SS electrodes and 51.7% Hg(II) removal by Al electrodes were observed. 10 mL of 5, 10, 15 and 20% NaCl solution by weight has been added to the medium and their effect on percent removal of Hg(II) has been observed, as shown in Figure 1(c). It was observed that the percent removal is more than 99% with both the electrodes at 5% NaCl concentration, and thereafter the percent increment rate was negligible. Hence, 5% NaCl was used as optimum electrolyte concentration in further experiments.

Effect of distance between electrodes

Increase in distance between electrodes increases the path of the electrons to flow and hence more ions will be generated which results in higher rate of generation of

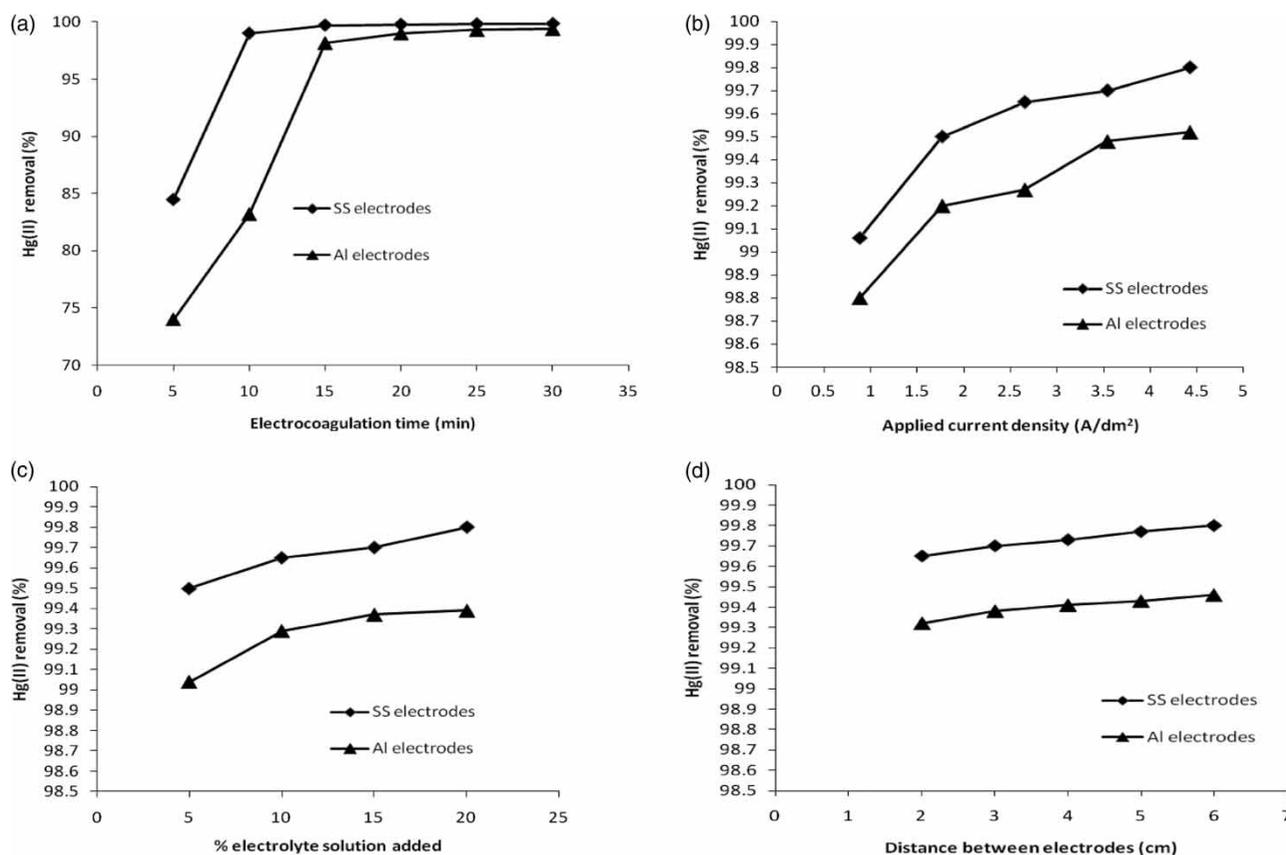


Figure 1 | Effects of operating conditions on the electrocoagulation process. (a) Mercury removal as a function of electrocoagulation time. (Operating conditions are current density = 1.77 A/dm², distance between electrodes = 6 cm, initial Hg(II) concentration = 10 mg/L, initial pH = 5. Also, 10 mL of 5% (w/v) NaCl solution was added). (b) Mercury removal as a function of applied current density. (Operating conditions were time = 15 min, initial pH = 5 and distance between electrodes = 6 cm, initial Hg(II) concentration = 10 mg/L, 10 mL of 5% NaCl solution (w/v) was added). (c) Mercury removal as a function of externally added electrolyte concentration. (Operating conditions: electrocoagulation time = 15 min, applied current density = 1.77 A/dm², distance between electrodes = 6 cm, initial Hg(II) concentration = 10 mg/L, initial pH = 5). (d) Mercury removal as a function of distance between electrodes. (Operating conditions: electrocoagulation time = 15 min, applied current density = 1.77 A/dm², 10 mL of 5% NaCl solution has been added, initial Hg(II) concentration = 10 mg/L, initial pH = 5).

metal hydroxides and, in turn, slightly increases the removal efficiency (Nanseu-Njiki *et al.* 2009). Effect of distance between electrodes is observed by varying distance between electrodes inside the solution and is shown in Figure 1(d). From the experimental observations, the highest distance possible in the reactor is 6 cm and at this distance the percent removal of Hg(II) is found to be highest. Hence, 6 cm distance between the electrodes is taken as optimum in all subsequent experiments.

Effect of initial pH

It is well known that pH influences the electrocoagulation process and the pollutant removal efficiencies were best found near neutral pH (Chen & Hung 2007). In the present work the effect of pH from 3 to 8 has been studied by applying 1.77 A/dm² current density for 15 min and keeping 6 cm distance between electrodes. HCl (0.1 N) and NaOH (0.1 N)

solutions were used to increase and decrease pH, respectively. It can be observed from Figure 2(a) that pH has an effect on the percent removal of Hg(II). The percent removal has increased up to a pH value of 7; this is because of evolution of hydrogen at cathode would be higher in the acidic pH (Chen & Hung 2007) and thereafter it started to decrease with both the electrodes. The reason for this downward trend may be that at higher pH values less generation of H⁺ ions will lead to less generation of metal hydroxides. In acidic range percent removal remains more than 99%. But there has been a drastic change in percent removal for a pH value higher than 7.

Effect of temperature

Studies on the effect of temperature on the electrocoagulation process are feeble (Chen & Hung 2007). Generally higher temperature is not desired, as it requires an additional amount of heat which results in increasing

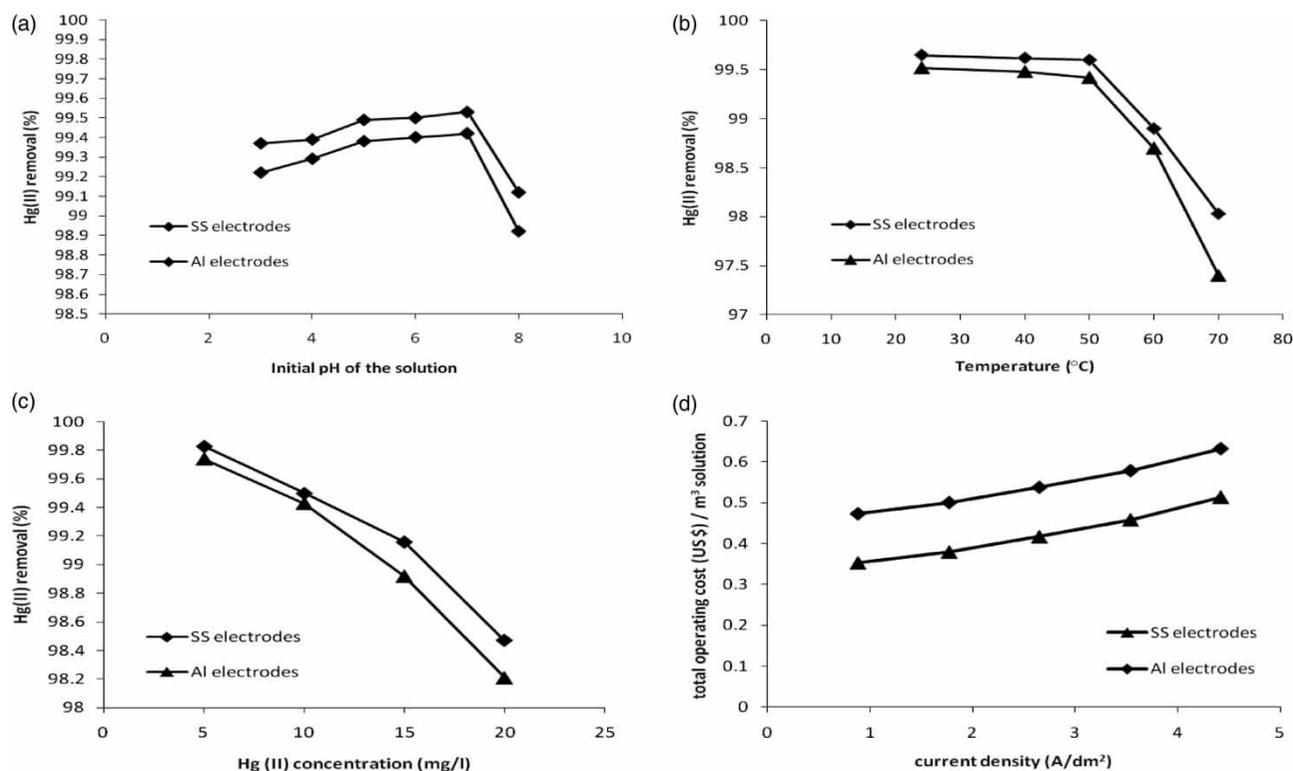


Figure 2 | Effects of operating conditions on the electrocoagulation process. (a) Mercury removal as a function of initial pH of the solution. (Operating conditions: electrocoagulation time = 15 min, applied current density = 1.77 A/dm², distance between electrodes = 6 cm, initial Hg(II) concentration = 10 mg/L, 10 mL of 5% NaCl solution has been added). (b) Mercury removal as a function of temperature. (Operating conditions: electrocoagulation time = 15 min, applied current density = 1.77 A/dm², distance between electrodes = 6 cm, initial Hg(II) concentration = 10 mg/L, 10 mL of 5% NaCl solution has been added). (c) Plot of percent removal vs. concentration. (Operating conditions: electrocoagulation time = 15 min, applied current density = 1.77 A/dm², distance between electrodes = 3 cm, 10 mL of 5% NaCl solution has been added). (d) Total operating cost (US\$) per m³ solution vs. current density.

operating cost. In the present work, effect of temperature from 24 to 70 °C has been studied for the Hg(II) removal efficiency and is shown in Figure 2(b). As can be seen from Figure 2(b) that increase in temperature resulted in decrease in percent Hg(II) rejection.

Effect of Hg(II) concentration

Effect of Hg(II) concentration from 5 to 20 mg/L on the removal efficiency has been shown in Figure 2(c). It can be observed that increase in concentration for same electrocoagulation time and current density results in decrease in removal efficiency.

Operating cost analysis

Operating cost for electrocoagulation process mainly involves utility cost (electricity) and material cost (electrodes) and other costs like maintenance and labor costs (Bayramoglu *et al.* 2004). Here we have considered

energy and electrode cost only for our analysis. The cost of electricity has been taken as US\$0.105 per kWh, cost of SS electrodes US\$3.42 per kg and cost of Al electrodes US\$4.6 per kg. Based on these we have calculated the total operating cost per m³ of solution treated as a function of applied current density, which is shown in Figure 2(d). From Figure 2(d) it is clear that the SS electrodes will be more economical than Al electrodes, mainly because of material cost.

Adsorption kinetics

The metal hydroxides generated during the electrocoagulation will be acting as adsorbents in the removal of solutes from the solution (Moreno *et al.* 2009). In the present work, adsorption of Hg(II) on these metal hydroxides is interpreted with Lagergren pseudo-first-order and Ho's pseudo-second-order kinetic models (Ho 2004, 2006). Pseudo-first-order rate equation for the liquid–solid adsorption system can be given as

Table 1 | Comparison between the experimental and calculated q_m values for different initial mercury concentrations

Sr. No.	$q_{m,exp}$	Pseudo-first-order kinetics			Pseudo-second-order kinetics		
		q_m	k_1	r^2	q_m	k_2	r^2
1.	4.991	0.825	-0.2091	0.8042	4.998	5.921	0.9810
2.	9.95	1.650	-0.2162	0.8068	9.987	5.970	0.9872
3.	14.88	2.475	-0.2183	0.8429	14.973	6.121	0.9919
4.	19.694	3.300	-0.2189	0.8632	19.957	6.227	0.9923

(Ho 2004, 2006):

$$(dq_m/dt) = k_1(q_m - q_t) \quad (5)$$

where q_m = adsorption capacity at equilibrium (mg g^{-1}); q_t = adsorption capacity at time t (mg g^{-1}); k_1 = rate constant of pseudo-first-order adsorption (min^{-1}).

Equation (5) can be simplified as (Ho 2004, 2006):

$$\ln(q_m - q_t) = \ln(q_m) - k_1 t \quad (6)$$

The plot of $\ln(q_m - q_t)$ vs. t gives the values of q_m and k_1 , which is compared with the experimentally obtained q_m and hence used to decide whether pseudo-first-order kinetics is suitable for it or not. Pseudo-second-order rate equation can be given as (Ho 2004, 2006)

$$(dq_m/dt) = k_2(q_m - q_t)^2 \quad (7)$$

where k_2 = rate constant of second-order adsorption ($\text{mg g}^{-1} \text{min}^{-1}$).

Equation (7) will give the simplification (Ho 2004, 2006):

$$1/(q_m - q_t) = 1/q_m + k_2 t \quad (8)$$

The plot of $1/(q_m - q_t)$ vs. t gives values of q_m and k_2 for the above equation. The parameters estimated from both the rate equations suggest (see Table 1) that the pseudo-second-order kinetics fits the data well in terms of q_m and correlation coefficient (r^2).

CONCLUSIONS

In the present work removal of Hg(II) from synthetic aqueous solutions by electrocoagulation, with SS and Al electrodes, has been reported. Effects of various parameters, viz., current density, electrocoagulation time, initial pH,

distance between electrodes, presence of electrolyte in the solution and temperature, on Hg(II) removal efficiency have been studied. SS electrodes (99.7% Hg(II) removal) are proved to be better than the Al electrodes (98% Hg(II) removal) in the present case. The optimum operating parameters are electrocoagulation time of 15 min, current density of 1.77 A/dm^2 , pH 7 and distance between electrodes 6 cm. Mercury removal kinetics follows the pseudo-second-order kinetics.

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