Zn(II) Removal from Wastewater by Electrocoagulation/Flotation Method using New Configuration of a Split-Plate Airlift Electrochemical Reactor

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ABSTRACT

In this paper, split-plate airlift electrochemical reactor as an apparatus with new configuration for wastewater treatment was provided. Two aluminum plates were fixed inside the reactor and present two functions; first it works as split plates for internal loop generation of the airlift system (the zone between the two plates acts as riser while the other two zones act as downcomer) and second it works as two electrodes for electrocoagulation process. Simulated wastewater contaminated with zinc ions was used to test the performance of this apparatus for zinc removal by studying the effect of different experimental variables such as initial concentration of zinc (50-800 ppm), electrical current density (2.67-21.4 mA/cm^2), initial pH (3-11), air flowrate (12-50 LPH), and implicitly the electrocoagulation time. The results have shown the applicability of this split-plate airlift reactor as electrocoagulation cell in the treatment of wastewater such as wastewater containing Zink ions. The Zink removal percent was shown to increase upon increasing the current density and the electrolysis time. Also best removal percent was achieved in the initial pH range between 7 and 9. The minimum electrocoagulation time required for removal of $\geq$ 90% of Zn(II) decreases from 90 to 22 min when operating current density increases from 2.67 to 21.4 mA/cm^2.

Key words: wastewater treatment, zinc ions, electrocoagulation/flotation, split-plate, airlift reactor.

ازالة ايونات الزنك من المياه الصناعية بواسطة طريقة التخثير/التطويف الكهربائي وباستخدام شكل جديد لمفاعل الرفع الهوائي ذو الصفائح الفاصلة

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الخلاصة

تم في هذا البحث توفير شكل جديد لمفاعل كهروكيمياوي وهو مفاعل الرفع الهوائي ذو الصفائح الفاصلة Split-plate airlift reactor في معالجة النفايات السائلة. استخدمت صفيحتين من الالمنيوم مثبتة داخل المفاعل من جهة تعمل على توفير نظام الرفع الهوائي ذو التدوير الداخلي (بحيث ان المنطقة مابين الصفيحتين تتصرف كجزء ناهض riser والمناطق الاخرى تعمل كجزء هابط

downcomer ومن جهة اخرى تعمل هذه الصفيحتين هي نفسها كأقطاب كهربائية لعملية التخثير الكهربائي. تم اختبار هذه المفاعل في معالجة وازالة ايونات الزنك من المياه الملوثة والمحضرة مختبريا عن طريق دراسة تأثير المتغيرات التالية على كفائة ازالة ايونات الزنك وهي التركيز الابتدائي للزنك (من 50 الى 800 جزء بالمليون)، كثافة التيار الكهربائي المسلط (من2,67 الى 21,4 ملي امبير/سم^2)، الاس الهيدروجيني الابتدائي للمحلول (من 3 الى 11)، معدل جريان الهواء (من 12 الى 50 لتر/ساعة) وزمن التخثير الكهربائي. بينت النتائج نجاح وامكانية تطبيق هذا المفاعل ذو الرفع الهوائي والصفائح الفاصلة كخلية تخثير كهروكيمياوية في معالجة المياه الملوثة بايونات الزنك. تزداد نسبة الازالة بزيادة كثافة التيار الكهربائي وزمن التخثير وكانت افضل نسبة ازالة لايونات الزنك تتحقق بحدود اس هيدروجيني بين 7 و9. اقل زمن تخثير لتحقيق اكبر او يساوي 90% نسبة ازالة لايونات الزنك ينخفض من 90 الى 22 دقيقة عند استخدام كثافة تيار كهربائي من 2,67 الى 21,4 ملي امبير/سم^2 على التوالي.

الكلمات الرئيسية: معالجة المياه الملوثة، ايونات الزنك، التخثير/التطويف الكهربائي، الصفائح الفاصلة، مفاعل الرفع الهوائي.

1. INTRODUCTION

Nowadays, environmental pollution by heavy metals has become one of the most serious environmental problems. Many industrial activities generate various wastewater contain several danger heavy metals disposed in the environment which have negative effect on humans, plants and animals, **Srivastava** and **Majumder, 2008**.

With the fast development of industrial activities such as mining industries, metal plating, fertilizer, batteries, tanneries, paper and insecticides industries, wastewaters containing high concentrations of toxic heavy metals include copper, zinc, nickel, mercury, lead, cadmium and chromium may be disposed into the environment increasingly. Heavy metals are not biodegradable and inclined to accumulate in living organisms. Many heavy metal ions are known to be carcinogenic, **Fu** and **Wang, 2011**.

Zinc is important for human health as a trace element. It is essential for the physiological functions of living tissue and regulates many biochemical processes. However, the excessive intake of zinc can cause notable health problems, such as stomach cramps, skin excitement, puke, nausea and anemia, **Oyaro et al., 2007**.

The electrocoagulation (EC) method is one of the most successful processes used for treatment of different type of soluble or colloidal pollutants in the industrial wastewater. Such pollutants as emulsion oils, dyes in textile wastewater, heavy metals, and organics such as phenolic compounds. Because of the simple and low cost operation, excellent removal efficiency, and low sludge production EC process therefore has wide applications and replaces the traditional chemical-coagulation process which uses expensive chemicals, **Emamjomeh, et al., 2009**.

EC includes applying direct electric current to two metal electrodes anode and cathode inside an electrochemical reactor tank. The two electrodes may be made of same or different metal. The coagulants were generated by electrolytic oxidation of the electrode in the first stage then in the second stage the contaminants or pollutants become unstable and aggregate together because of the attraction between the formed coagulants and these contaminants. The aggregated contaminants then either settle or float to be easily separated, **Heidmann** and **Calmano, 2008, Chen, 2004**.

The electrodes are made from either aluminum or iron. In case of aluminum cations dissolve from the anodes according to, **Rincon** and **La Motta, 2014.**

$Al_{(s)} \rightarrow Al^{3+}{}_{(aq)} + 3e^{-}$ and/or
$Al_{(s)} \rightarrow Al^{2+}{}_{(aq)} + 2e^{-}$

Also the following water electrolysis reaction may take place:

$2H_2O_{(l)} \rightarrow 4H^{+} + O_{2(g)} + 4e^{-}$

Simultaneously, at the cathode water is reduced to hydrogen gas and hydroxyl ion OH^{-} according to:

$2H_2O_{(l)} + 2\,e^{-} \rightarrow H_{2(g)} + 2OH^{-}$

Aluminum cations are reacting with hydroxyl to form various aluminum hydroxides:

$Al_3+ + 3OH^{-} \rightarrow Al(OH)_{3(s)}$

Also hydroxides polymers can be built up according to:

$nAl(OH)_{3(s)} \rightarrow Al_n(OH)_{3n(s)}$

Other dissolved ionic species, like $Al(OH)^{2+}$, $Al_2(OH)_2{}^{4+}$, $Al(OH)_2{}^{+}$, $Al(OH)_4{}^{-}$, $Al_6(OH)_{15}{}^{3+}$, $Al_7(OH)_{17}{}^{4+}$ … etc. may also occurr in the system. These ionic hydroxides have very strong attraction force toward destabilized pollutants caused removal of these substances from the system by electrostatic attraction and then coagulation. The continually small bubbles of hydrogen and oxygen gases generated in the EC process make the pollutants floats to the wastewater surface in the reactor tank, **Balla, 2010**. **Fig.1** shows a representation of the electrocoagulation/flotation process.

The interaction between the two phenomena; electrocoagulation (EC) and electroflotation (EF) gives unique technique for removal of several pollutants from wastewater before disposal or reuse, **Deghles** and **Kurt, 2016**.

Airlift reactors have been widely utilized in the wastewater treatments to implement many applications including two or three phase. Airlift reactors are a special case of bubble column. It consists of two distinctive zones the riser and the downcomer. The air or gas may be sparged in the riser zone which leads to a difference in gas holdup between riser and downcomer. As a result of the gas holdup differences and thus densities differences between the two sections, there will be a static pressure difference between the riser and downcomer, therefore this leads the liquid circulation between them. There are two main configurations for airlift: internal loop and external loop airlift reactors, **Chisti, 1989**.

The objective of this research is to utilize internal-loop with two split plates airlift reactor as electrocoagulation/flotation cell. Several experimental parameters were studied on the zinc removal efficiency from water such as initial Zn concentration, electrical current density, and initial pH of the wastewater, contact time and air pumping effect.

2. EXPERIMENTAL WORK

2.1 Materials

Stock solution of 800 mg/L Zn(II), was prepared by dissolving the required amounts of zinc sulfate ($ZnSO_4 \cdot H_2O$, 99.0% purity supplied by Merck) in deionized water. Other required lower initial concentrations were prepared by suitable dilution. Sodium nitrate (NaCL, ≥ 99.0% purity, supplied by Sigma-Aldrich) was added if necessary to increase the solution conductivity. Initial pH of the reactor content was adjusted by adding 0.1 M solutions of HCl and NaOH as needed.

2.2 Experimental setup and procedure

The experimental setup mainly consists of the airlift electrochemical reactor. The reactor designed as an airlift reactor with internal loop with capacity of 2.5 L includes two aluminum plates of rectangular shape. These plates act as electrodes (anode and cathode) and split plates for airlift system generation at the same time. The reactor was made from Perspex material with 10 cm diameter and 33 cm height giving a working volume of 1.8 L. The two aluminum plates were fixed inside the reactor, the plate dimensions were (85 mm* 220 mm with 1 mm thickness giving electrode surface area of 187 cm^2) and the distance between electrodes was 4 cm. A digital DC power supply (model HY3002, Hyelec, India) was used to create electrolysis. **Fig.2** shows the schematic representation experimental setup arrangements.

Five parameters were selected to investigate the zinc removal from simulated wastewater, these parameters include initial concentration of metal (50, 100, 200, 400 and 800 mg/L), electrical current 0.5, 1, 1.8, and 4 A (which correspond to current density of 2.67, 5.34, 9.6 and 21.4 mA/cm^2), air flowrate (12, 25, 37, and 50 LPH), electrocoagulation time (5-75 min) and initial pH (3-11). All experiments were performed at a room temperature (about 30^oC). The experimental work was divided into four parts:

- **Part#1 (initial concentration & electrocoagulation time effect studies):** In this set, the prepared wastewater with desired zinc initial concentration varied from 50 to 800 mg/L was poured into the reactor. The air flowrate, electrical current and initial pH, were fixed to the values 0 LPH, 9.6 mA/cm^2, and 7 respectively. The samples were taken according to the prespecified time intervals.
- **Part#2 (Current density study):** In this set, four runs were performed, the current density was varied as 2.67, 5.34, 9.6 and 21.4 mA/cm^2 while other parameters initial copper concentration, air flowrate, and initial pH were fixed at 200 mg/L, 0 LPH, and 7 respectively. The electrocoagulation time in this runs was varied also according to the prespecified time intervals.
- **Part#3 (Initial pH study):** Finally, the initial pH of the solution was varied from 3 to 11, initial pH value was varied by adding small droplets of 1 M NaOH or 1 M HCl solution as needed. The pH was monitored with a pH meter (model HI110, HANNA Instruments Co., India). Other parameters such as current density, air flowrate, initial zinc concentration and

electrocoagulation time were fixed at 21.4 mA/cm^2, 0 LPH, 200 mg/L and 35 min respectively.

- **Part#4: (Air flowrate study):** the flowrate of inlet air was studied from 12 to 50 LPH while the current density, initial pH, initial zinc concentration and electrocoagulation time were fixed at 21.4 mA/cm^2, 7, 200 mg/L and 35 min respectively.

Every electrocoagulation experiment was started with working volume of 1800 ml of zinc solution. The desired current was applied and 2 mL samples were taken from the bulk solution in the downcomer zone every prespecified min. The total outlet concentration of zinc in the treated solution from the reactor was determined with flame Atomic Adsorption Spectrophotometer AAS (model Perkin Elmer 5000, Italy).

The removal efficiency of zinc was calculated as:

$$\mathrm{R}\ (\%) = \frac{C_{in}-C_{out}}{C_{in}} \times 100 \tag{1}$$

where R (%) is the removal efficiency, C_{in} is the initial zinc concentration (ppm) and C_{out} is the residual outlet zinc concentration (ppm) after electrocoagulation time of t.

Also, the specific electrical energy consumption of zinc treatment via electrocoagulation/flotation process in this reactor was calculated by the following equation, **Konstantinos et al., 2011**:

$$E\left(\frac{kWh}{kg\ Zn(II)}\right) = \frac{\mathbf{1000}\ \boldsymbol{UIt}}{\boldsymbol{V}\ \boldsymbol{C_{in}}\ \boldsymbol{R}\%} \tag{2}$$

Where E is the electrical energy consumption ($\frac{kWh}{kg\ Zn(II)}$), U is the voltage (V), I is the applied current (A), t is the electrocoagulation time, and V is the working volume in the reactor.

3. RESULTS AND DISCUSSION

3.1 Effect of initial concentration and electrocoagulation time

Fig.3 shows the removal efficiency of zinc in the reactor with time (electrocoagulation time) at different initial zinc concentration and at current density of 9.6 mA/cm^2 (1.8 A) without air pumping. The removal efficiency increased almost linearly by time during the first 40 min. The removal percent for 50, 100, 200, 400 and 800 ppm initial Zn concentration in 1800 ml reactor working volume were nearly 90% removed after 25, 35, 45, 50 and 75 min respectively as shown in **Fig.4**. Conversely, the removal efficiency of Zn increase with decreasing initial Zn concentration due to the availability of large amount of coagulation agents required for low initial concentration. Almost, the same results of zinc removal were obtained by **Adhoum, et al., 2004** and **Prica, et al., 2015**.

Generally, a considerable formation of H_2 gas micro bubbles was observed at the cathode during all the EC experiments, whereas at the anodes no O_2 gas bubbles were evolved. The main reactions at the anode were aluminum dissolution while at the cathode, H_2 and OH^- ions were formed.

3.2 Effect of current density

Fig.5 shows the effect of electrocoagulation time on the removal efficiency for different current density values. The current density has a significant impact on Zn removal efficiency. Increasing the current density leads to increase the Al^{3+} and OH^- dosage into the system by time and also the bubble H_2 production rate, in contrast the bubble size decreases with increasing current density, **Khosla** and **Somasundaran, 1991** which is advantageous to the removal process. Thus the process of Zn removal can be accelerated. This effect was also observed by other researchers with Zn(II) such as **Adhoum, et al., 2004** and **Meunier, 2004**. The electrocoagulation time required for removal of more than 90% of Zn(II) decreases from 90 to 22 min when operating current density increases from 2.67 to 21.4 mA/cm^2 as shown in **Fig.6**.

In all cases, it was noted that increasing the current density leads to increase the liquid circulation velocity inside the reactor due to the increase the hydrogen micro bubbles generated at the cathode surface. It was noted that the liquid circulation between the riser and downcomer zones was achieved even by small bubbles of hydrogen gas at the cathode electrode when no air bubbled in the riser.

Liquid circulation velocities were determined by calculating the circulation time in the riser and downcomer zones by a method called neutral-buoyancy flow follower. The method depends on using of plastic particle with a distinctive color. The diameter of tracer particle was 2 mm and specific gravity was 1.05. The time which elapsed in the downcomer or riser is the time taken by this particle to travel a vertical distance equal to the riser height plus half distance between top edge of the plates and liquid level plus half distance between bottom edge of the plates and base of the reactor. The procedure was repeated many times and the velocity in each section was calculated from the average of 5 trials, **Lu, et al., 1994.** Increasing hydrogen generation with small bubbles in the riser leads to increase gas holdup in this zone and therefore increasing liquid circulation due to density difference between riser and downcomer. The double effect of current density on the zinc removal efficiency was resulted from two effects: first, the formation more coagulating agents such as $Al(OH)_3$ due to increasing the rate of anodic dissolution and second, good mixing in the reactor which resulted from increasing circulation velocity inside the reactor by more micro bubbles of hydrogen generated at the cathode surface. This means more coagulating agent is being contacted with Zn(II) ions which leads to improve the removal efficiency without using mechanical stirring.

3.3 Effect of initial pH

The initial pH has a notable influence on the performance of electrocoagulation/flotation process, **Chen et al., 2000**. To study this effect, a set of experiments were performed, using solutions of Zn(II) of 200 ppm each, with an initial pH varying in the range (2–11). As shown in **Fig.7**, the removal efficiency, after 35 min of electrocoagulation at 21.4 mA/cm^2, reached value as high as 93%, when pH exceeded 5. Furthermore, it can be seen that the removal efficiency decreased upon decreasing initial pH. The best removal condition was for the initial pH, which was between 7 and 9. The decrease of removal efficiency of Zn(II) at a pH less than 5 and higher than 9 was attributed to an amphoteric behavior of $Al(OH)_3$ which led to soluble Al^{3+} cations, when the initial pH was low and to monomeric anions such as $Al(OH)_4{}^-$, when the initial pH was higher than 9, **Do** and **Chen, 1994**. When the initial pH was set in the range between 7–9, all aluminum cations formed at the anode are polymeric species and precipitated $Al(OH)_3$ leading to a more effective treatment, **Kobya** and **Bayramoglu, 2003**.

Fig.8 shows that the pH changed during electrocoagulation process. The treatment induced an increase in the final pH when the initial pH was low as shown in **Fig.9**. This might be attributed to the excess of OH^- ions formed at the cathode in acidic conditions and by the liberation of OH^- due to the occurrence of a partial exchange of Cl^- with OH^- in $Al(OH)_3$, **Chen, et al., 2000**. When the initial pH is above 9, the formation of $Al(OH)_4{}^-$ species together with parasite attack of the cathode by OH^- ions which led to a slight decrease in the final pH.

3.4 Power consumption

The electrical energy consumption was calculated in terms of *kWh* per kg of *Zn(II)* of treated effluent using **Equation 2** at operation time for which Zn removal was 90%, initial Zn content was of 200 ppm and initial pH was 7. **Fig.9** shows the electrical power consumption at different current density. It was noted that a continuous increase in energy consumption with increasing of the current density. Similar results of power consumption in the electrocoagulation/flotation process were obtained by many researchers such as **Prica, et al., 2015, Fu** and **Wang, 2011**.

3.5 Effect of air pumping

Fig.10 shows the effect air flowrate on the zinc removal efficiency. The air flowrate was studied from 0 to 50 LPH while the current density, initial pH, initial zinc concentration and operation time were fixed at 21.4 mA/cm^2, 7, 200 mg/L and 35 min respectively. The purpose of this study is to check the effect of increasing the liquid circulation velocity when air is pumped in the riser zone of the reactor. Removal efficiency increase slightly with increasing air flowrate, therefore, this type of reactor can be used as electrocoagulation/flotation cell successfully even without using air in the riser.

4. CONCLUSIONS

In this work, the removal of Zn(II) ions from simulated wastewater by the electrocoagulation/flotation process was investigated. The results have shown the applicability of split-plate airlift reactor as electrocoagulation/flotation unit for the treatment of wastewater containing Zn ions without using mechanical stirring and therefore less power consumption. Several experimental variables (initial zinc concentration, current density, operating time and air pumping) were studied. The Zn removal rate was shown to increase significantly upon increasing the current density. Also, it was observed that zinc removal efficiency increased with the increase of electrolysis time. Best removal capacity was achieved in the pH range between 7 and 9.

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6. NOMENCLATURE

C_{in} = initial Zn(II) concentration, ppm
C_{out} = outlet Zn(II) concentration, ppm
E = electrical energy consumption, $\frac{kWh}{kg\ Zn(II)}$
I = electrical current, A
LPM = liter per min.
M = concentration, molarity
R = removal efficiency, %
t = electrocoagulation time, min.
U = voltage, V
V = volume of the liquid in the reactor, L

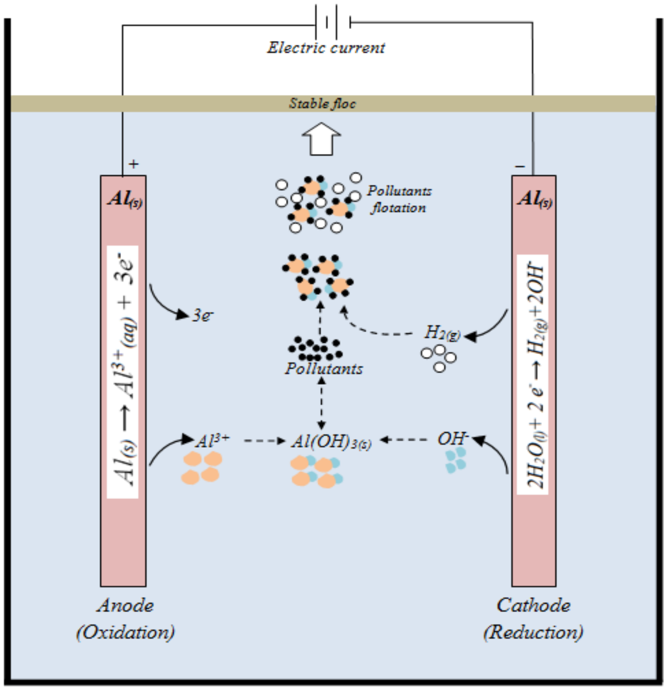

Figure 1. Representation of the electrocoagulation/flotation process.

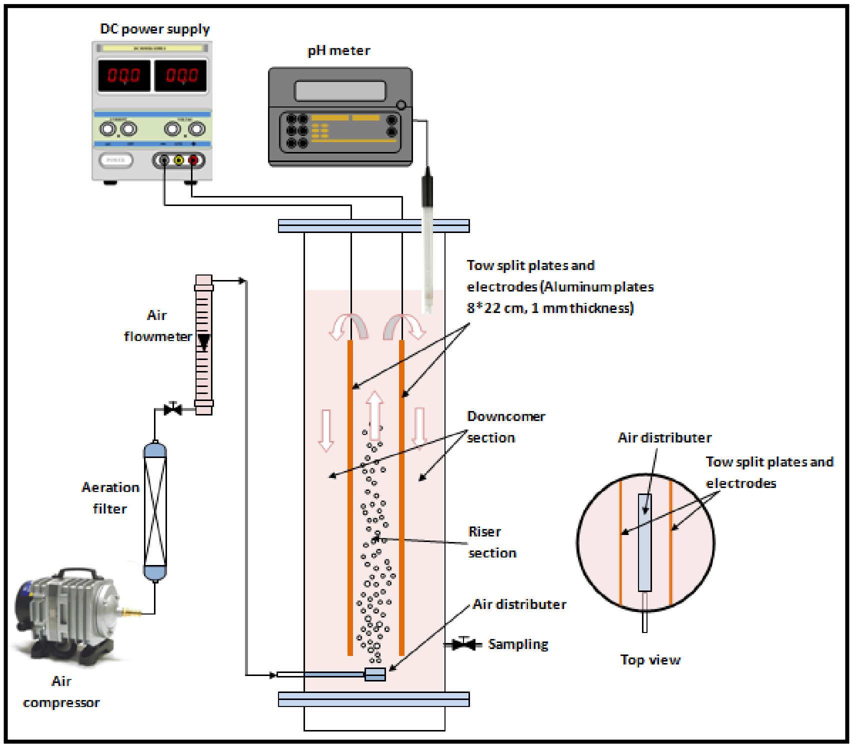

Figure 2. Experimental setup arrangement of the split-plate airlift electrochemical reactor

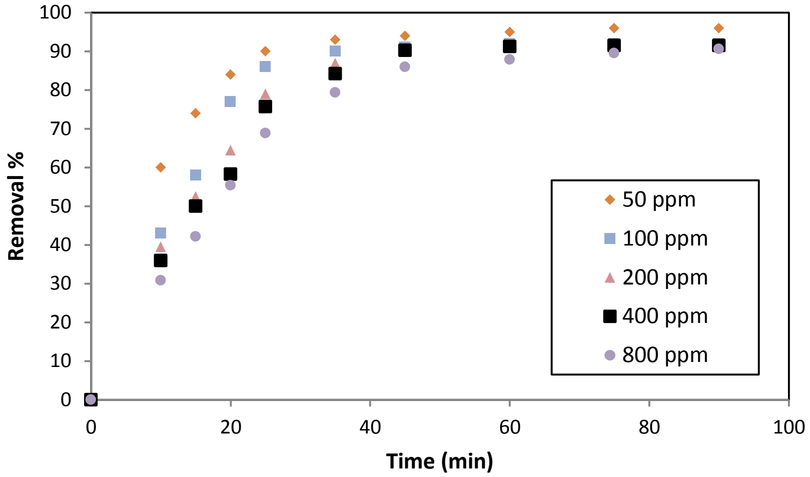

Figure 3. Effect of initial zinc concentration on the removal efficiency (current density: 9.6 mA/cm^2, initial pH: 7).

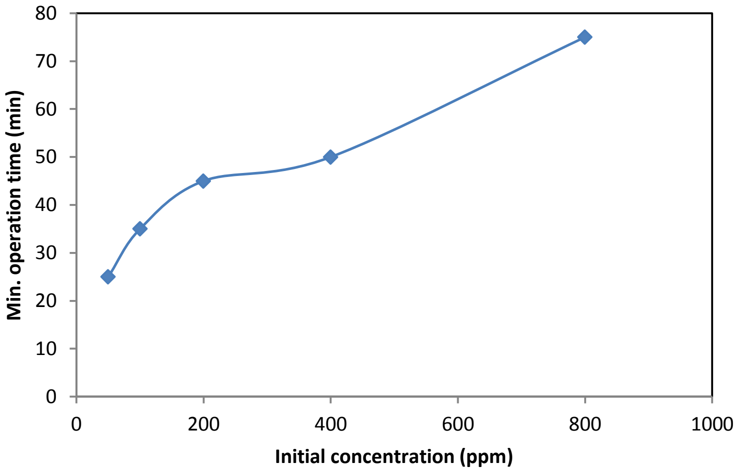

Figure 4. Effect of initial zinc concentration on the minimum operation (electrocoagulation) time value required for ≥ 90% removal efficiency (current density: 9.6 mA/cm^2, initial pH: 7).

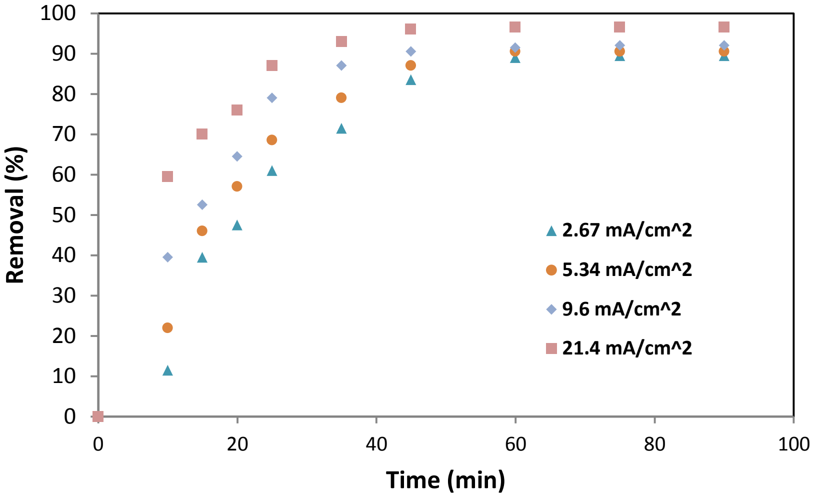

Figure 5. Effect of current density on the removal efficiency (Zn initial concentration: 200 ppm, initial pH: 7).

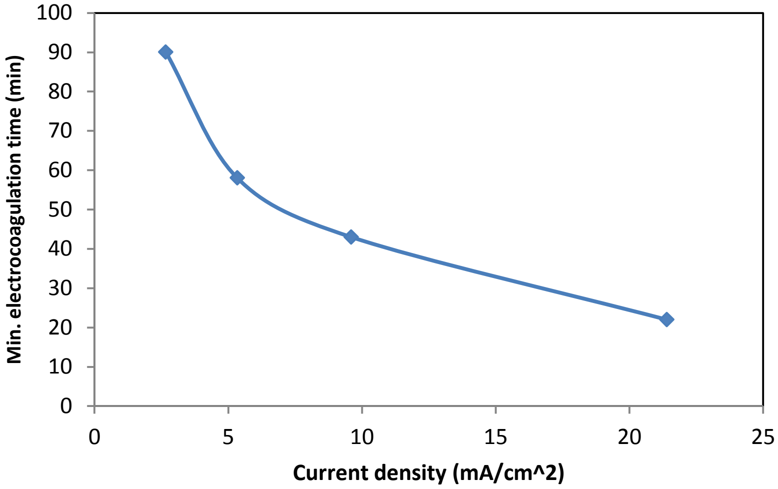

Figure 6. Minimum electrocoagulation time required for ≥ 90% Zn removal at different current density (Zn initial concentration: 200 ppm, initial pH: 7).

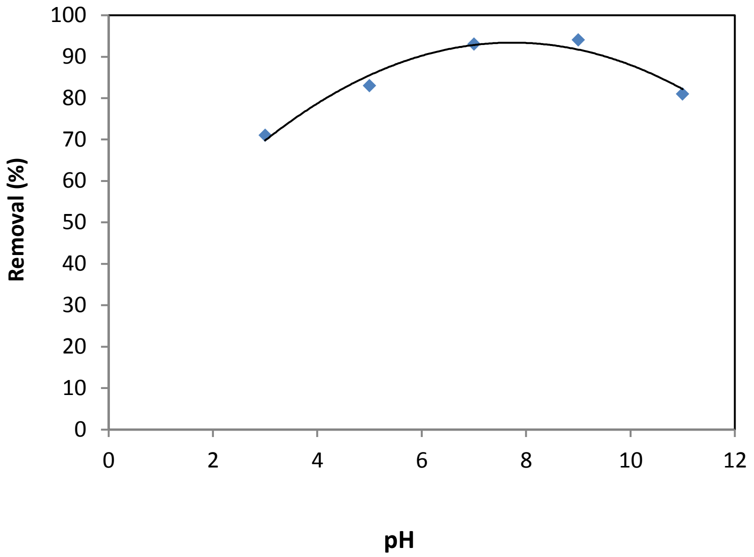

Figure 7. Effect of initial pH on Zn(II) ions removal. (Initial concentrations: 200 ppm, current density: 21.4 mA/cm^2, time of electrocoagulation: 35 min)

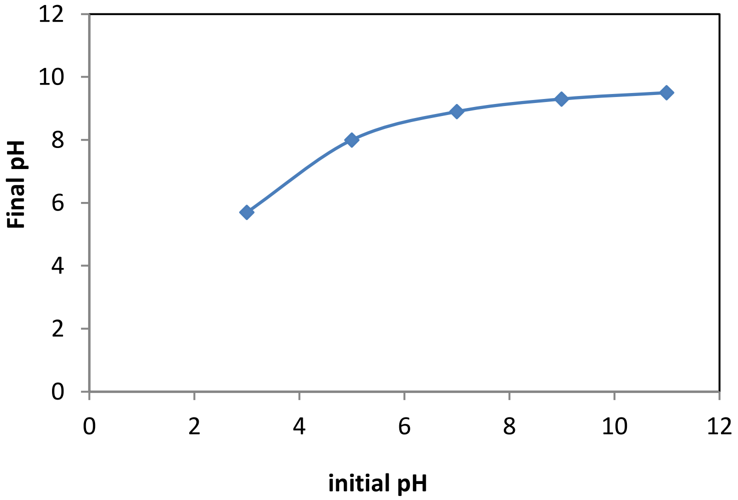

Figure 8. pH variation after electrocoagulation (Initial concentrations: 200 ppm, current density: 21.4 mA/cm^2, time of electrocoagulation: 90 min)

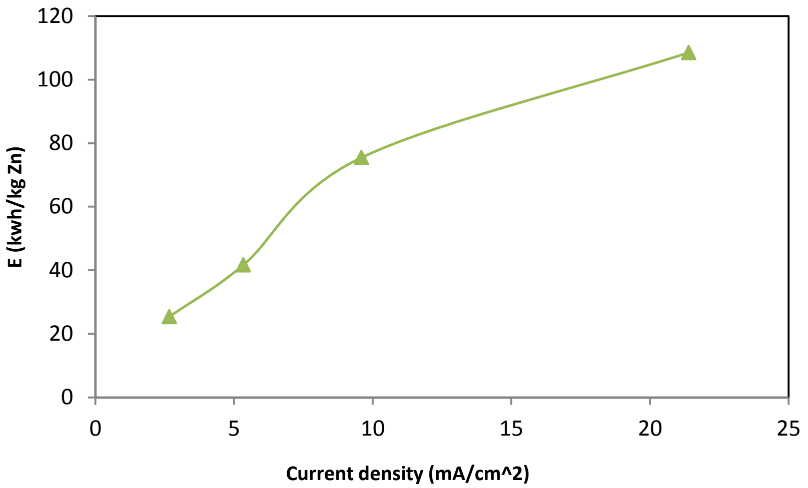

Figure 9. Effect of current density on electrical energy consumption (*E*) at operation time for which Zn removal of 90% (initial Zn concentration: 200 ppm, initial pH: 7)

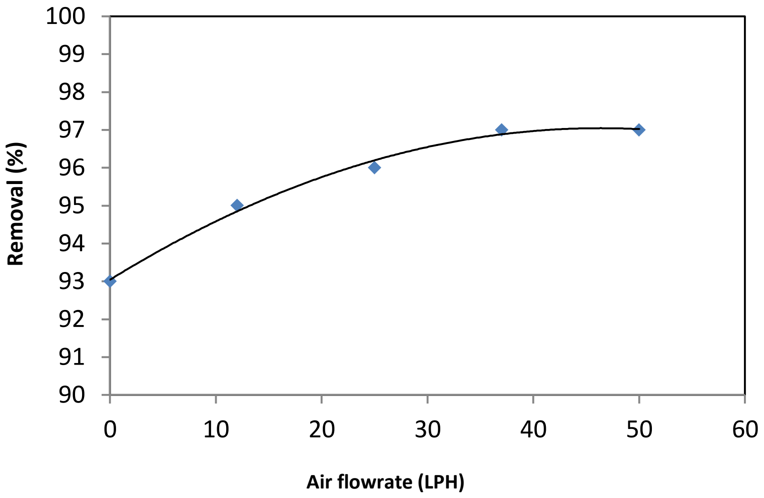

Figure 10. Effect of air flowrate on Zn removal efficiency (operation time: 35 min, initial Zn concentration: 200 ppm, initial pH: 7 and current density of 21.4 mA/cm^2)