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Optimization of lead removal by electrocoagulation from aqueous solution using response surface methodology

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ABSTRACT

Lead removal from aqueous media continues to be an important environmental issue. In this study, a batch system for lead removal from polluted water via electrocoagulation (EC) using an aluminum electrode was evaluated. Box–Behnken design for response surface methodology was applied for modeling and optimizing the effects of main operational variables such as current density, initial pH, and initial lead concentration. According to the ANOVA results, current density and lead concentration have a linear effect, while the pH has a quadratic effect on lead removal efficiency. The high R^2 value of 94.12% obtained showed that the experimental data and model predictions agreed well. A removal efficiency of 94% was predicted by the model using the following optimal parameter values: current density of 33 A/m^2, pH of 7.25, and lead concentration of 5 mg/L. The results showed that EC is an efficient method for lead removal from aqueous solution.

Keyword: Electrocoagulation; Lead removal; Response surface methodology; Aluminum electrode

1. Introduction

In recent years, public awareness about the long-term effect of water containing dissolved heavy metal ions has been rising [1]. Toxic heavy metals are released into the environment from a number of industries such as mining, plating, dyeing, automobile manufacturing, and metal processing. The presence of heavy metals in the environment has caused a number of environmental problems. To meet the water quality standards of most countries, the concentration of heavy metals in wastewater must be controlled. Lead, a heavy metal, can cause central nervous system damage. In addition, it can damage the kidney, liver, and reproductive system, as well as affect basic cellular processes and brain functions. The toxic symptoms are anemia, insomnia, headache, dizziness, irritability, muscles weakness, hallucination, and renal damages [2,3].

Techniques for the separation of heavy metals, such as lead, chromium, cadmium, copper, zinc, and nickel, from industrial wastewater include precipitation, ion

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exchange, adsorption, electrodialysis, and filtration. However, these techniques have limitations in selective separation and the associated investment and operation costs are high [4]. Electrocoagulation (EC) is an alternative technology for water and wastewater treatment. The main advantages of EC over other conventional techniques, such as chemical coagulation and adsorption, are "*in situ*" delivery of reactive agents, lack of secondary pollution, and compact equipment. EC is a simple and efficient method in which the flocculating agent is generated by electro-oxidation of a sacrificial anode, generally made of iron or aluminum [5,6]. This process has attracted considerable attention within the wastewater treatment domain because EC has been used successfully in the removal of different kinds of pollutants such as organic compounds, heavy metals, important inorganic pollutants, as well as for the treatment of dairy wastewater, textile wastewater, biodiesel wastewater, paper industry wastewater, municipal wastewater, leachate, petroleum refinery wastewater, etc. [7–16].

The contaminant-removal mechanisms in EC involves three stages: (i) formation of coagulating ions by electrical oxidation of anode electrode, (ii) destabilization of contaminants and suspended particles and breaking of emulsions, and (iii) aggregation of destabilized particles to form flocs [3,17]. The main reaction at the aluminum anode is dissolution, which leads to coagulant formation via electrolytic oxidation of the sacrificial anode:

$$\mathrm{Al(s)} \rightarrow \mathrm{Al^{3+}} + 3\mathrm{e^-} \tag{1}$$

Additionally, water electrolysis occurs at the cathode and anode:

$$2\mathrm{H_2O} + 2\mathrm{e^-} \rightarrow \mathrm{H_{2(g)}} + 2\mathrm{OH^-} \quad \text{(cathodic reaction)} \tag{2}$$

$$2\mathrm{H_2O} \rightarrow 4\mathrm{H^+} + \mathrm{O_{2(g)}} + 4\mathrm{e^-} \quad \text{(anodic reaction)} \tag{3}$$

The hydroxide ions formed at the cathode increase the pH of the wastewater, thus inducing precipitation of metal ions (Me^{n+}) as the corresponding hydroxides and co-precipitation with aluminum hydroxides:

$$\mathrm{Me}^{n+} + n\mathrm{OH^-} \rightarrow \mathrm{Me(OH)}_{n\mathrm{(s)}} \tag{4}$$

In addition, anodic metal ions and hydroxide ions generated at the electrode surfaces react in the bulk wastewater to form various hydroxides and built-up polymers:

$$\mathrm{Al^{3+}} + 3\mathrm{OH^-} \rightarrow \mathrm{Al(OH)_{3(s)}} \tag{5}$$

$$n\mathrm{Al(OH)_{3(s)}} \rightarrow \mathrm{Al}n\mathrm{(OH)}_{3n\mathrm{(s)}} \tag{6}$$

However, depending on the pH of the aqueous medium, other ionic species, such as dissolved $\mathrm{Al(OH)^{2+}}$, $\mathrm{Al_2(OH)_2^{4+}}$, and $\mathrm{Al(OH)_4^-}$ hydroxo complexes may be present in the system as well. The suspended aluminum hydroxides can remove pollutants from the solution by sorption, co-precipitation, or electrostatic attraction, followed by coagulation [4,18,19].

In contrast, when a direct current is applied to the electrodes, the anode is dissolved by electrolysis, and it generates metallic ions which are good coagulants [20]. At the cathode, $\mathrm{OH^-}$ ions are produced during water electrolysis, and they can react with the metallic ions to produce metal hydroxides [21]. Usually, iron or aluminum is used as the electrode in this process [22]. The metal hydroxides producing $\mathrm{Al(OH)_3}$ or $\mathrm{Fe(OH)_3}$) can adsorb and settle both soluble and colloidal contaminants [5,23].

When aluminum electrodes are used in the EC process as both anode and cathode, the concentration of hydrolyzed aluminum species depends on the aluminum concentration and the solution pH. The hydrolysis constants for aluminum span a very narrow range, and the entire aluminum deprotonation process is "squeezed" into an interval of less than 2 pH units. Therefore, apart from a narrow pH region approximately 5–6, the dominant soluble species are $\mathrm{Al^{3+}}$ and $\mathrm{Al(OH)^{4-}}$ at low pH and high pH, respectively [24].

This work aimed to investigate lead removal from polluted water by EC and to optimize the effects of current density, pH, and initial lead concentration on the removal efficiency using the response surface methodology (RSM).

2. Materials and methods

2.1. Experimental setup

The experiments were conducted in a Plexiglas reactor (measuring 40 × 17 × 12 cm with an effective volume of 6 L) in the batch mode. The EC reactor was equipped with a stirrer. Four 8 × 35 cm electrodes made of aluminum plates were connected to a digital DC power supply (jps-330D) via bipolar-series connections. The inter-electrode distance in the EC cell was 10 mm in all experiments. The electrodes were cleaned manually by abrasion followed by washing with 15% hydrochloric acid and distilled water prior to every run. All experiments were carried out under the potentiostatic condition at ambient temperature. Conductivity and pH of the solution were measured using conductivity and pH meters (If90 (WTW), BEL-W3D), respectively.

The pH of the influent solution was adjusted using sulfuric acid solution and sodium hydroxide 0.1 M). Samples were collected from the middle of the cell at regular time intervals; these were filtered immediately through a mixed cellulose acetate membrane (0.45 μm). The residual lead concentration was determined using the atomic absorption method according to the standard method [25].

2.2. Experimental design and data analysis

The effects of different parameters such as current density (11, 22, and 33 A/cm^2), electrolysis time (5, 10, 15, 20, 25 and 30 min), pH (5, 7 and 9), and initial lead concentration (5, 10 and 15 mg/L) were investigated. Minitab 16.1 software was used for the statistical design of experiments and data analysis. The four most important operating variables including current density, initial pH, initial lead concentration, and operating time were optimized for wastewater treatment. The optimum operating time was determined using the one-factor-at-a-time method in the 5–30 min range. The other variables were optimized using the statistical fractional factorial design method. Their actual and coded values are listed in Table 1. The independent variables and their levels were determined from the literatures and pretest runs.

RSM was used in this study to determine the optimum lead removal conditions. RSM is a collection of mathematical and statistical techniques, commonly used for upgrading and optimizing processes. It can be used to determine the relative significance of several important factors in the presence of complex interactions. RSM uses an experimental design method such as central composite design or Box–Behnken design to fit a model following the least squares technique. RSM makes it possible to represent independent process parameters a quantitative form as expressed by (Eq. (1)):

$$y = f(x_1, x_2, x_3, \ldots, x_n) \pm \varepsilon \tag{7}$$

where y is the response (yield), f is the response function, ε is the experimental error, and x_1, x_2, x_3,…, x_n are independent parameters. By plotting the expected response of y, a response surface is obtained. The full quadratic model (Eq. (2)), a higher order polynomial was used in this study. Analysis of variance (ANOVA) was used to determine the interaction between the process variables and the responses. The quality of the fit polynomial model was expressed using the coefficient of determination R^2, and its statistical significance was checked using the Fisher F-test in the same program. Model terms were evaluated using the p value (probability) at the 95% confidence level.

Table 1
Independent variables and Box–Behnken design table for lead removal by EC process

Level	Current density (A/m^2)	pH	Initial lead concentration (mg/L)	X_1	X_2	X_3
High	33	9	15	+1	+1	+1
Center	22	7	10	0	0	0
Low	11	5	5	−1	−1	−1
Run (standard order)		X_1		X_2		X_3
1		−1		−1		0
2		1		−1		0
3		−1		1		0
4		1		1		0
5		−1		0		−1
6		1		0		−1
7		−1		0		1
8		1		0		1
9		0		−1		−1
10		0		1		−1
11		0		−1		1
12		0		1		1
13		0		0		0
14		0		0		0
15		0		0		0

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 \tag{8}$$

The predicted response (y) is therefore correlated to the following set of regression coefficients (β): intercept (β_0), linear β_1, β_2, β_3), interaction (β_{12}, β_{13}, β_{23}), and quadratic (β_{11}, β_{22}, β_{33}). Box and Behnken (1960) developed a family of efficient three-level designs for fitting second-order response surfaces. The experiments carried out in this study were based on their design, and a quadratic model was employed to study the combined effects of three independent variables (current density, initial pH, and initial lead concentration). The optimization experiments were based on 15 combinations with two replicates at center points. Table 1 represents the design matrix of the trial experiments. All experimental designs were randomized to exclude any bias [26,27].

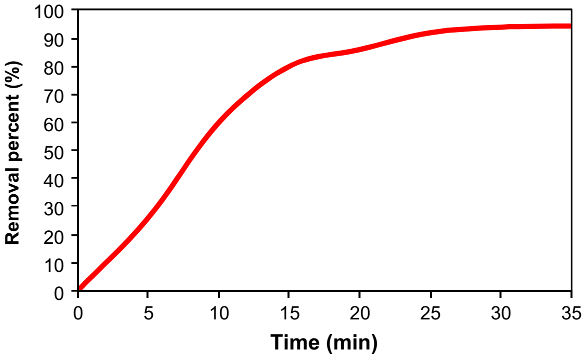


Fig. 1. Effect of EC treatment time on lead removal efficiency.

3. Results and discussion

3.1. Effect of operating time

Before conducting optimization runs, experiments were carried out with different operating times and the following fixed parameters: current density = 33 A/m^2, pH 7, initial lead concentration = 5 mg/L. The results are plotted in Fig. 1, which shows that the optimum time for lead removal is 30 min.

3.2. Effect of variables on lead removal

Process optimization was conducted using statistical design of experiments based on the Box–Behnken design. The most important factors that affect the EC process are current density (X_1), pH (X_2), and initial lead concentration (X_3). To study the combined effects of these factors, experiments were carried out with different combinations of these parameters. The uncoded (actual) values and the corresponding lead removal efficiencies along with the predicted fits and residuals are listed in Table 2.

Table 2
Experimental data, fits, and residuals

Run	Current density (A/m^2)	pH	Initial lead concentration (mg/L)	Lead removal (%)	Fit for lead removal (%)	Residuals
1	11	5	10	74.8	74.90	−0.10
2	33	5	10	85	84.08	0.92
3	11	9	10	77.7	78.63	−0.93
4	33	9	10	84.5	84.40	0.10
5	11	7	5	83.7	84.14	−0.44
6	33	7	5	91.9	93.36	−1.46
7	11	7	15	77.6	76.14	1.46
8	33	7	15	82.3	81.86	0.44
9	22	5	5	83	82.46	0.54
10	22	9	5	87.5	86.14	1.36
11	22	5	15	73	74.36	−1.36
12	22	9	15	74.2	74.74	−0.54
13	22	7	10	81.4	83.90	−2.5
14	22	7	10	84	83.90	0.1
15	22	7	10	83.8	83.90	−0.1
16	22	7	10	86	83.90	2.1
17	22	7	10	85.3	83.90	1.4
18	22	7	10	82.9	83.90	−1

The statistical significance of the ratio of mean square owing to regression and mean square residual error were tested. ANOVA, a statistical technique, subdivides the total variation in a set of data into component parts associated with specific sources of variation for testing hypotheses of the model parameters. The "*p*" values obtained were less than 0.05, thus indicating that the factors played a significant role, and the model is statistically significant. The ANOVA results are summarized in Table 3. The *p* value of the lack of fit was significantly high at 0.37, which implied that the fit achieved using the model was close to the experimental results.

The lead removal percentage (Y) can be predicted using the coefficients listed in Table 4. All terms, regardless of their significance in lead removal, are included in the following regression equation (Eq. (3)) for lead removal using EC.

$$Y = 83.9 + 3.74\ X_1 - 4.88\ X_3 - 3.93\ X_2^2 \qquad R^2 = 94.12\% \tag{9}$$

Before the conclusions from the ANOVA are adopted, the adequacy of the fitted model should be checked to ensure that it provides an adequate approximation of the true system. To this end, the primary diagnostic tool is residual analysis.

A normal probability plot of the residuals is shown in Fig. 2(a). There is no severe indication of normality, nor any evidence pointing toward possible outliers. Fig. 2(b) shows a plot of the residuals vs. the fitted values. There should be no relationship between the size of the residuals and the fitted values. This plot reveals nothing unusual [28]. Furthermore, the coefficient of correlation ($R = 0.97$) and the *p* value ($p < 0.05$) of the regression term in the analysis of variation table refer to adequacy and significance of the model.

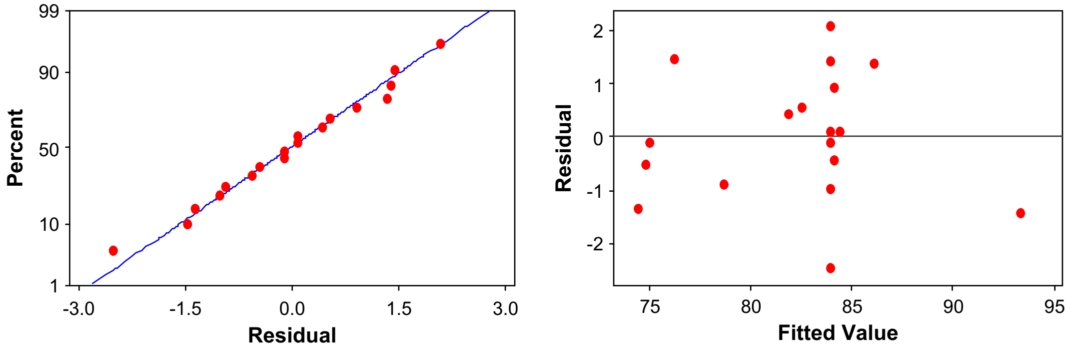
Table 4
Coefficients, and standard error of coefficients, *T* and *p* for lead removal

Term	Coefficient	SE coefficient	*T*	*p* value
Constant	83.90	0.71	117.86	0.000
X_1	3.74	0.62	6.06	0.000
X_2	1.01	0.62	1.64	0.139
X_3	−4.88	0.62	−7.91	0.000
${X_1}^2$	0.53	0.83	0.63	0.547
${X_2}^2$	−3.93	0.83	−4.70	0.002
${X_3}^2$	−0.55	0.83	−0.66	0.528
$X_1 \cdot X_2$	−0.85	0.87	−0.98	0.358
$X_1 \cdot X_3$	−0.88	0.87	−1.00	0.345
$X_2 \cdot X_3$	−0.83	0.87	−0.95	0.372

According to the ANOVA table, the linear and quadratic effects of factors are significant ($p < 0.05$). Thus, current density and initial lead concentrations have linear effects and pH has a second-order effect on lead removal efficiency (Fig. 3). As can be inferred from Table 4, the main effects of these variables are 7.48, −9.75, and −7.85, respectively. Moreover, the results show the absence of any interaction effect on the EC process ($p < 0.46$). In addition, R^2 represents

Table 3
ANOVA results for lead removal via electrocoagulation

Source of variation	Degree of freedom	Sequential sum of squares	Adjusted sum of squares	Adjusted mean of square	*F*	*p* value
Regression	9	389.62	389.62	43.29	14.24	0.001
Linear	3	310.08	310.08	103.36	34.00	0.000
X_1	1	111.75	111.75	111.75	36.76	0.000
X_2	1	8.20	8.20	8.20	2.70	0.139
X_3	1	190.13	190.13	190.13	62.53	0.000
Square	3	70.87	70.87	23.62	7.77	0.009
${X_1}^2$	1	0.03	1.20	1.20	0.40	0.547
${X_2}^2$	1	69.52	67.22	67.22	22.11	0.002
${X_3}^2$	1	1.32	1.32	1.32	0.43	0.528
Interaction	3	8.68	8.68	2.89	0.95	0.461
$X_1 \cdot X_2$	1	2.89	2.89	2.89	0.95	0.358
$X_1 \cdot X_3$	1	3.06	3.06	3.06	1.01	0.345
$X_2 \cdot X_3$	1	2.72	2.72	2.72	0.9	0.372
Residual error	8	24.32	24.32	3.04		
Lack-of-fit	3	10.68	10.68	3.56	1.31	0.370
Pure error	5	13.64	13.64	2.73		
Total	17	413.94				



(a): Normal Probability Plot **(b)**: Residuals versus Fits

Fig. 2. Residual plots for checking model adequacy.

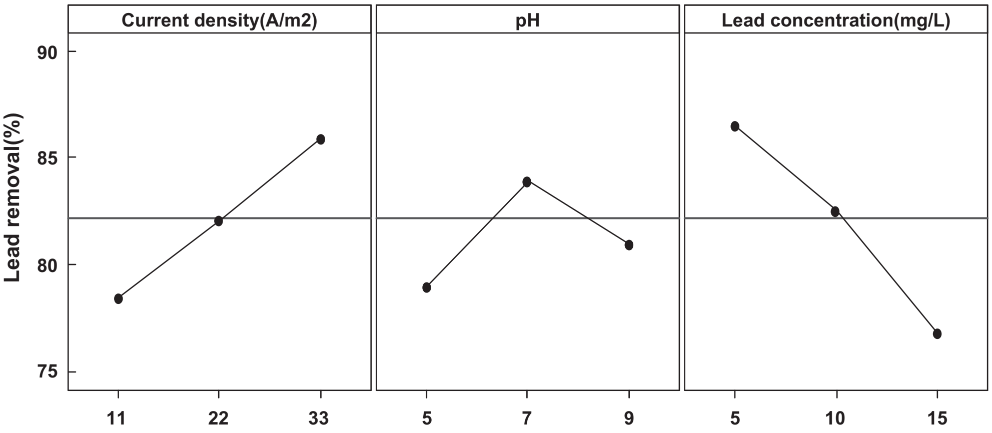


Fig. 3. Main effect of independent variables on lead removal in EC process.

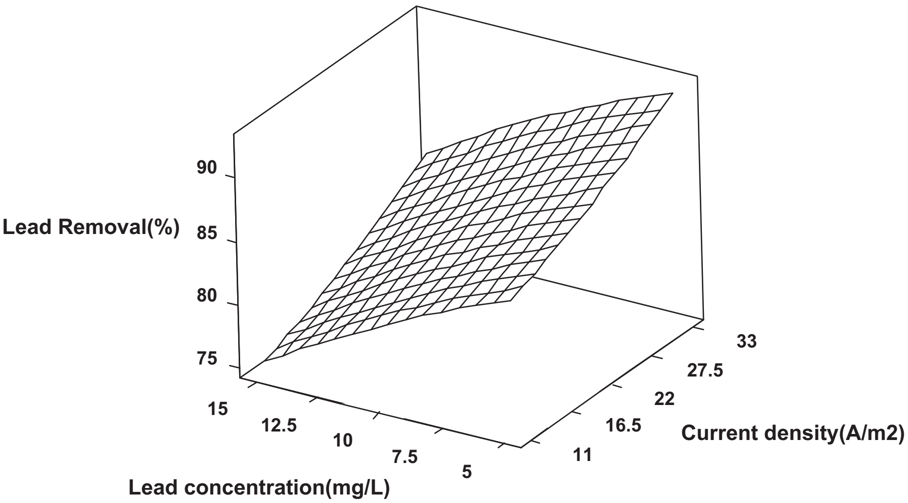


Fig. 4. Surface plot of lead removal vs. current density and initial lead concentration with hold value of pH 7.

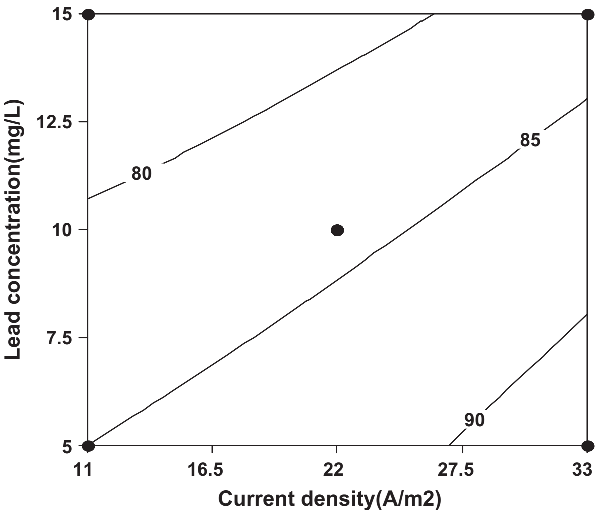


Fig. 5. Contour plot of lead removal vs. current density and initial lead concentration with hold value of pH 7.

that the regression model accounts for 94.12% of the variability in lead removal. The lack-of-fit, too, shows no evidence exists that the model does not accurately fit the data ($p < 0.37$).

3.3. Response to optimization of lead removal

The optimization target for lead removal was set at 94%. The optimal EC conditions determined using the response optimization procedure were as follows: current density = 33 A/m^2, pH 7.25, and initial lead concentration = 5 mg/L. The actual efficiency of lead removal under the optimized conditions was found to be 92%, which is close to the response predicted using RSM. Figs. 4 and 5 show surface and contour plots in the maximum response region with a hold value of pH 7. Moreover, the process efficiency improved with increasing current density and decreasing initial lead concentration. These results show that EC is rather promising at low lead concentrations.

4. Conclusion

RSM is a collection of statistical and mathematical techniques useful for analyzing, improving, and optimizing environmental processes. In this study, RSM was applied for developing an empirical statistical model to optimize lead removal. The efficiency of EC is strongly influenced by current density, pH, and initial lead concentration. It was found that increasing current density and decreasing initial lead concentration have linear effects on the process of lead removal, whereas pH has a quadratic effect. In addition, there is no important two-factor interaction between any two of three variables. The actual optimal values of current density, pH, and lead concentration resulting in 94% lead removal were 33 A/m^2, 7.25, and 5 mg/L, respectively.

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