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Employing electro coagulation for the removal of Acid Red 182 in aqueous environment using Box-Behenken design method

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ABSTRACT

No study has been performed on the treatment of Acid red 182 (AR182) in aqueous environment up to this time. Therefore, in this research, the removal of AR182, Azo dye, was investigated by electro coagulation (EC) process in an electrochemical batch reactor. A Box–Behnken design (BBD) was employed for optimization of the influencing factors such as current density, time of electrolysis and initial pH in the dye removal efficiency. The graphical counter plots and response surfaces were used to determine the optimum conditions. The Analysis of variance (ANOVA) was presented a high determination coefficient value (R^2 = 0.9817, R^2_{adj} = 0.9487 and R^2_{pred} = 0.7070) and satisfactory prediction second-order regression model. The optimum amounts of current density, time of electrolysis and initial pH of the dye solution were found to be at 16 m A/cm^2, 14.2 min and 8, respectively. The production rate of coagulant was improved with an increase in the current density. The dye removal efficiency was very low either at acidic or very alkaline pH. At optimum conditions, the removal efficiency for Acid red 182 and chemical oxygen demand (COD) were 98.7 and 88.3%, respectively.

Keywords: Electro coagulation (EC); Acid red 182 (AR 182); Analysis of variance (ANOVA); Current density; Box–Behnken design (BBD)

1. Introduction

Several industries such as textile, leather, paper, food, plastic, and so on, generate the large quantity of organic colored waste waters that can cause noticeable environmental problems [1]. It is estimated that about 1–15% of the dye are missing in the dying process and are released into the wastewater. The request of azo dyes in industries is very essential than other dyes. The presence of dyes even at very low concentration is greatly visible and undesirable. The Azo dyes are toxic, mutagenic and carcinogenic, hence their release into the environment would be dangerous to aquatic life and human health [2]. The classic methods such as biological, chemical, and physical techniques have been used for dye removal, but the biological systems have not been very effective because most of the dyes are not biodegradable [3]. The decomposition of chromophores in dyes can be obtained by injecting a strong oxidizing agent such as ozone. But, a large dose of ozone is usually necessary for decolorization of the dye that is not economically reasonable [4]. Therefore, electro coagulation as an electrochemical method was advanced to overcome the disadvantages of conventional methods [5,6]. The foundation theory of EC process is (i) electrode oxidation (ii) creation of gas bubbles and coagulating agents (iii) coagulation ions conjoin with the pollutants and generate flocks, (iv) provide easier removal of pollutants by sedimentation or floatation [7]. The EC includes no addition of chemicals and provides better removal abilities than chemical coagulation. The coagulant in this case is created by dissolution of a sacrificial anode. The EC uses an electrical current to create several metal ions in the solution. The benefits of EC are easy process, simple apparatus and short-term reactive retention period, production of less sludge and accordingly decreasing the sludge disposal cost [8,9]. The mechanism of the EC method was described in our former work [10].

The Box-Behnken design (BBD) in the response surface methodology (RSM) is a main design tool used for the optimization of methods. The BBD makes complete results and detailed information even for a small number of exper-

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iments and positive effects of operating parameters on all responses [11]. Among all the RSM designs, the BBD needs fewer runs and offers calculations of the response function and method efficiency at intermediate levels and any experimental point, respectively within the range studied through accurate analysis [12].

A large number of researches have been done on the treatment of azo dyes in waste waters by the EC, but no investigation has been executed on the remediation of Acid red 182 in aqueous environment using EC or other processes up to this time. Thus, in the present study, the removal of Acid red 182 from aqueous solutions was explored by EC technique. The effects of the operating variables such as current density, initial pH, and electrolysis time on color removal efficiency in a synthetic wastewater containing Acid Red 182 were investigated.

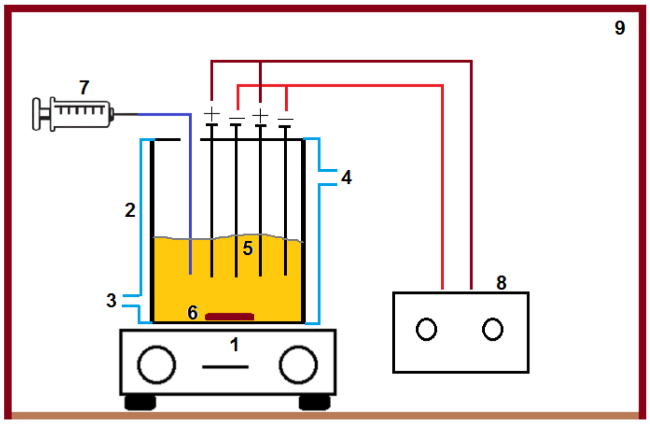


Fig. 1. Schematic diagram of bench-scale EC reactor, Notes: (1) magnetic stirrer, (2) batch electrochemical reactor, (3) cooling water inlet, (4) cooling water outlet, (5) aluminum electrodes, (6) magnetic bar, (7) sampling port, (8) DC power supply, (9) wooden box.

2. Experimental

2.1. Materials

Acid Red 182 (95% purity, CAS number: 61901-42-6), was purchased from the Merck Company of Germany. The dye was used without further purification. The selected amount of the dye (60 mg l^{-1}) was dissolved in distillate water prior to use. H_2SO_4 and NaOH are all provided from Merck. Distilled water was used throughout the experiment.

2.2. Electro chemical reactor

The tests were performed in a glass cylindrical batch electrochemical reactor. The system was equipped with a sampling system (Fig. 1). The water bath, BW 20 G model from Korean company, was used for fixing temperature at 25°C by circulating water in the jacket of the reactor. Two anodes and two cathodes were made from aluminum plates (99.5% purity), with dimensions of 4.6 cm× 5.5 cm × 0.3 cm and connected to a digital DC power supply (Top ward 6306 D, 30 V and 6 A) in mono polar mode. The total immersed and effective surface area of each electrode was 45 cm^2 and the space between the electrodes was 1.5 cm.

2.3. General procedure

The volume of the solution for test in each batch was 1 L. The pH was adjusted through the addition of NaOH or HCl (0.1 M) and measured by pH Meter PT-10P Sartorius Instrument Germany. In all runs, about 1.5 g/l of NaCl was added to the solution to keep the conductivity of the solution and a Jenway conductivity meter (Model 4200) was used to control the conductivity of the solution. The concentration of the dye was tested by UV–Vis spectrophotometer (Agilent, 5453, American) at a wavelength of 550 nm. The solution was stirred at 150 rpm by a magnetic stirrer to let the chemical to be precipitated and grow large enough for separation. The electrodes were washed completely before and after each run, by dipping in HCl solution (5% v/v) for at least 20 min and rinsing again with distilled water.

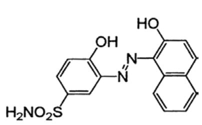
The COD of the samples were measured by the HACH's COD method using a COD reactor (DR/5000U) from HACH Company and the absorbance of samples was estimated by spectrophotometer at 600 nm. The COD was measured by the standard closed reflux and Colorimetric method [13]. The removal efficiencies were calculated with respect to its initial values and the removal percent for Acid red 182 and COD were achieved as in Eqs. (1) and (2):

$$\text{Color removal}\,(\%) = \left(\frac{[C]_0 - [C]}{[C]_0}\right) \times 100 \quad (1)$$

$$\text{COD removal}\,(\%) = \left(\frac{[COD]_0 - [COD]}{[COD]_0}\right) \times 100 \quad (2)$$

where $[C]_0$ and $[COD]_0$ are the concentration of the dye and amounts of COD at the start of the reaction, and [C] and [COD] are the concentration of the Acid red 182 and extents of COD at time t, respectively.

Table 1
The chemical structure and characteristics of Acid Red 182

Pollutant	Molecular structure	λ_{max} (nm)	Molecular Weight (gr/mol)
Acid red 182 ($C_{16}H_{13}N_3SO_4$)		550	343.36

3. Results and discussion

3.1. Design of experiments with Box-Behenken method

The purpose of the optimization was to find conditions that had the maximum dye removal efficiency. The BBD was used to obtain the relationship between the input variables and the response. Three variables, involving current density (mA/cm^2) (X_1), time of electrolysis (min) (X_2), and initial pH

(X_3) are independent variables shown in Table 2. The three levels were anticipated and encoded by (–1, 0, +1).

In this study, by using the BBD and the RSM, the influences of three independent variables on the response function were investigated to get the optimum conditions [14,15]. By statistical analysis of the obtained experimental data, a second-order polynomial equation [Eq. (3)] was achieved as an empirical model that describes the response surface: [16]

$$Y = \beta_0 + \sum \beta_i X_i + \sum \beta_{ii} X_i^2 + \sum \beta_{ij} X_i X_j \tag{3}$$

where Y is the response, β_o, β_i, β_{ii}, β_{ij} and are coefficients of the intercept, linear, square and interaction effects, respectively. The optimum removal efficiency of AR182 (Y) and the process parameters were also obtained.

In all 15 runs, the tests were performed in duplicated and all obtained results from the BBD are shown in Table 3. The observed values and predicted response values with residuals in all 15 runs are displayed in Table 3.

All runs were performed to form a mathematical model for describing the relation between the removal efficiency of Acid red 182 (response) and the three operating variables that they were fitted to a second order polynomial equation. The predicted values of the dye removal efficiency were attained from quadratic model fitting methods using the software Minitab17. The response function with determined coefficients for the removal efficiency is presented by the mathematical equation [Eq. (4)] in terms of the actual variables (confidence surface above 95%).

$$(Y) = -160.9 + 17.37X_1 + 13.12X_2 + 9.06X_3 - 0.6762X_1^2 - 0.631X_2^2 - 0.752X_3^2 + 0.249X_1X_2 + 0.099X_1X_3 + 0.103X_2X_3 \tag{4}$$

The equation discloses that the current density is further significant because it has a higher coefficient ($17.37X_1$) than other two variables.

Table 2
Experimental ranges and levels of the independent test variables

Variables	Range and levels		
Current density (mA/cm²)	Level 1(–1)	Level 2(0)	Level 3(+1)
	6	12	18
Time of electrolysis (min)	5	10	15
Initial pH	4	7	10

3.2. Effect of different variables

The appropriateness of the model was evaluated through the analysis of variance (ANOVA). The ANOVA was used to investigate the influence of three independent variables (X_1, X_2 and X_3) on the removal of AR182 in EC process. As it is obvious from Table 4, the fitness of the model was tested by ANOVA. The suitability of the model in the removal of AR 182 was controlled on the basis of the values of regression coefficient (R^2), adjusted R^2, predicted R^2, F-value and P-values (Table 4). Coefficient of regression (R^2) is a significant parameter for checking the suitability of a model [17,18]. The response function forecasts fitted well with the experimental data (R^2 = 98.17). In this study, values of adjusted R^2 for the removal of AR 182 was 0.9487 and it was above the acceptable level (0.8) which displays good acceptability of the model. High values of adjusted R^2 express that the model terms are highly important. The values of predicted R^2 are the values predicted by the design, which measures the difference in the model predicted data [19,20]. In this project, all values of adjusted R^2 and predicted R^2 are in the satisfactory range.

Table 3
Coded factor levels for a Box-Behnken design of a three-variable system

Observation	Actual values			Conversion		
run	X_1	X_2	X_3	Y_{exp}	Y_{pred}	Residual
1	6	5	7	18.0	10.56	7.43
2	18	5	7	46.3	47.51	–1.21
3	6	15	7	39.0	37.78	1.21
4	18	15	7	97.2	104.63	–7.43
5	6	10	4	28.2	32.71	–4.51
6	18	10	4	85.2	81.06	4.13
7	6	10	10	29.5	33.63	–4.13
8	18	10	10	93.6	89.08	4.51
9	12	5	4	43.0	45.92	–2.92
10	12	15	4	88.3	85.00	3.30
11	12	5	10	44.0	47.30	–3.30
12	12	15	10	95.5	92.57	2.92
13	12	10	7	89.8	90.23	–0.43
14	12	10	7	90.3	90.23	0.06
15	12	10	7	90.6	90.23	0.36

Table 4
ANOVA test for response function

Source	SS	DF	MS	F-value	P-value	Remarks
Model	12192.1	9	1354.68	29.74	0.001	Significant
Linear	8984.7	3	2994.91	65.75	0.000	Significant
X_1	5387.2	1	5387.22	118.27	0.000	Significant
X_2	3557.5	1	3557.46	78.10	0.000	Significant
X_3	40.1	1	40.05	0.88	0.391	Insignificant
Square	2961.6	3	987.21	21.67	0.003	Significant
X_1*X_1	2187.8	1	2187.75	48.03	0.001	Significant
X_2*X_2	917.9	1	917.86	20.15	0.006	Significant
X_3*X_3	169.1	1	3.71	169.06	0.112	Insignificant
2-Way Interaction	245.7	3	81.91	1.80	0.264	Insignificant
X_1*X_2	223.5	1	223.50	4.91	0.078	Insignificant
X_1*X_3	12.6	1	12.60	0.28	0.621	Insignificant
X_2*X_3	9.6	1	9.61	0.21	0.665	Insignificant
Error	227.8	5	45.55			
Lack-of-Fit	227.4	3	75.81	464.14	0.002	
Pure Error	0.3	2	0.16			
Total	12419.8	14				
S	R^2	$R^2_{(adj)}$	$R^2_{(pred)}$			
6.74914	98.17%	94.87%	70.70%			

3.3. Accuracy of the model

The F-value clarifies the distribution of actual data around the fitted model and P-value describes the significance of the model terms. The P-value was used to check the importance of each coefficient and P-value < 0.05 indicates that the model is significant and the model values is not significant in the case that a probability value is larger than 0.1.

The F-value is used to test the significance of the model, distinct variables and their interactions [21]. The statistical significance of the model and the coefficients were explored by the F-test and P-value, respectively [22]. The F-value of 29.74 indirectly mentions that the model is significant and in the present study, F-values were not high for all responses.

The ANOVA data from Table 4, specifies that the second-order polynomial model in term of Eq. (4) was statistically significant and suitable to represent the actual relationship between the efficiency and the significant variables.

The prediction values have been compared with experimental data in the removal of AR 182 using Eq. (4) in Fig. 2. It can be discovered from the figure that the model equation predictions are acceptably matched with the experimental data. The accuracy of the model is demonstrated in Fig. 2, which compares the measured values against the predicted responses by the model.

The results demonstrated in Fig. 3 convey the general impression of a normal distribution of basic errors, since the residuals fall near to a straight line; so, there is no clear indication of non-normality of experimental results. Normal probability plots are a suitable graphical method for judging the normality of the residuals [23,24]. The plots of residual vs. predicted responses are shown in Fig. 4. All points of experimental runs were scattered randomly within the constant range of residuals across the

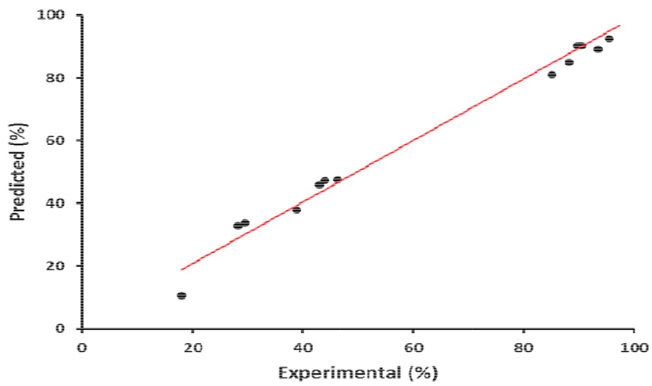


Fig. 2. Comparison of predicted value with experimental data for the removal percentage of AR 182.

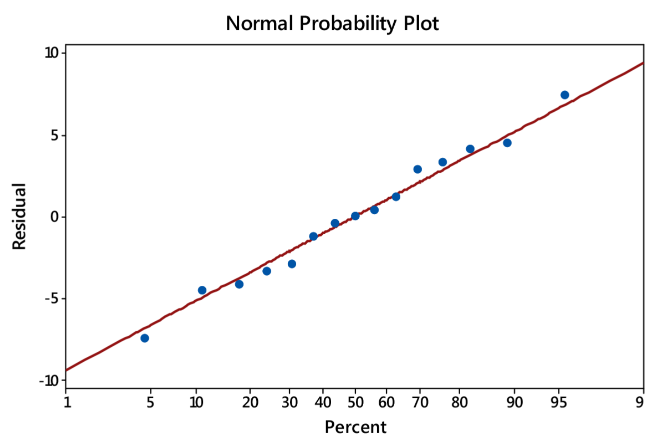


Fig. 3. The normal probability plot of residual for AR 182 removal efficiency.

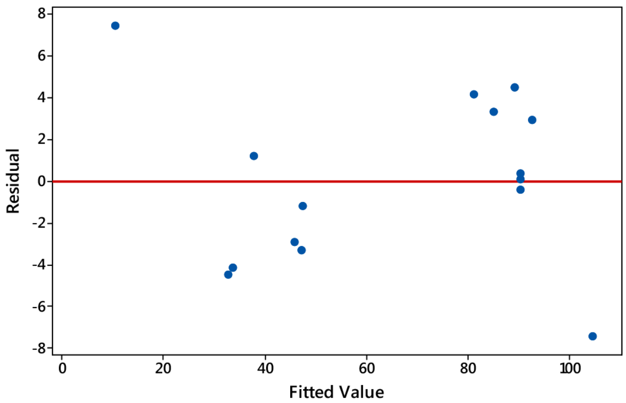


Fig. 4. The plot of residual vs. predicted response for AR 182 removal efficiency.

graph, i.e. within the horizontal lines. This indicates that the models proposed are adequate and that the constant variance assumption was established. Responses from experimental results also fitted well within an acceptable variance range when compared to the predicted values from respective empirical models.

All of the plots in Figs. 2–4, showed that the model is adequate to describe the removal of AR 182 by response surface methodology.

3.4. The contour plots and three-dimensional response surfaces

The three dimensional response surfaces and their corresponding contour plots can simplify the analysis of the experimental variable effects on the response. The contour plots of AR 182 removal efficiency vs. current density (X_1) and time of electrolysis (X_2), was signified in Fig. 5a. It is clear that the removal efficiency was improved with an increase in current density and time of electrolysis.

The current density is the most significant parameter for controlling the reaction rate in the electrochemical reactor. The coagulant production rate can be determined by current density. It was affected on the growth of created flocks which cause the pollutant to be treated [25]. The three different current densities were selected at 6,12 and 16 mA/cm^2 for investigation the influence of current density on the removal of Acid red 182. As it can be seen from Eq. (4), the current density had a higher coefficient and so a more influence on the color removal efficiency, therefore an increase in current density leads to the increase in color removal efficiency. When the current density rises, the number of released aluminum cations by the anode and consequently the particles of $Al(OH)_3$ enhances. Consequently, the amounts of anodic dissolution of aluminum were increased and larger amount of precipitate was produced for the removal of AR 182. Additionally, the bubble production rate was enriched and the bubble size reduced with increasing current density, so a large amount of dye was removed by H_2 floatation [26,27].

As the current density was increased, the required time for the remediation reduced. Consequently, for a distinct time, the removal efficiency was enhanced significantly with an increase in the current density.

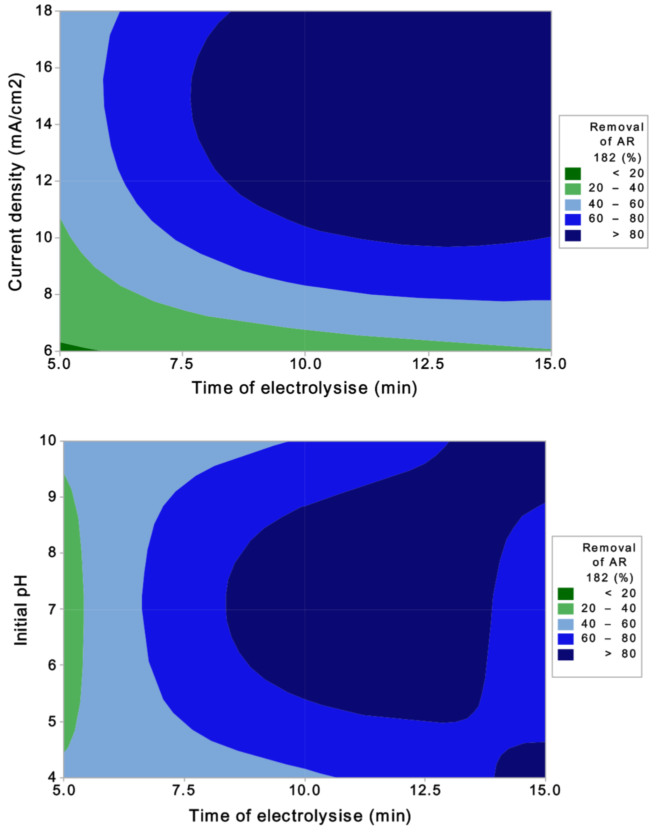


Fig. 5 (a). Contour plot describing the removal efficiency of AR 182 as a function of current density and time of electrolysis. Fig. 5(b). Contour plots describing the removal efficiency of AR 182 as a function of initial pH and time of electrolysis.

Fig. 5b shows the contour plot of initial pH (X_3) and time of electrolysis (X_2). As it is obvious, an increase in the time results in the improvement of color removal efficiency, but both increase and decrease in pH lead to the decrease in the response. So it was observed that high removal of AR 182 was happened at a medium pH. The result of these figures are in agreement with those of Table 3.

The time of treatment is affected on the treatment efficiency. The influence of time of electrolysis was considered at three levels of 5, 10 and 15 min. The removal percent of Acid red 182 vs. the electrolysis time is shown in Figs. 5a and b. The release of coagulating species was originated from anodic electro dissolution throughout the electrolysis process. The dye removal efficiency depends on the concentration of Al ions produced on the electrodes. When the electrolysis time increases, the concentration of Al^{3+} ions and their hydroxide flocks was increased, subsequently the removal of dye was upgraded [28,29].

In order to investigate the influence of pH on the removal of Acid Red 182, a series of tests was performed by regulating the initial pH at the three ranges of 4, 7 and 10. The influence of pH on electro coagulation is shown in Fig. 6a. The results exhibited that when the pH of the solutions was neutral, the removal efficiency of dye was optimum, because the solid flocks of $Al(OH)_{3(s)}$ was shaped at this pH. The flocks of $Al(OH)_{3(s)}$ are essential for adsorption of soluble organic pollutants and trapping of colloidal

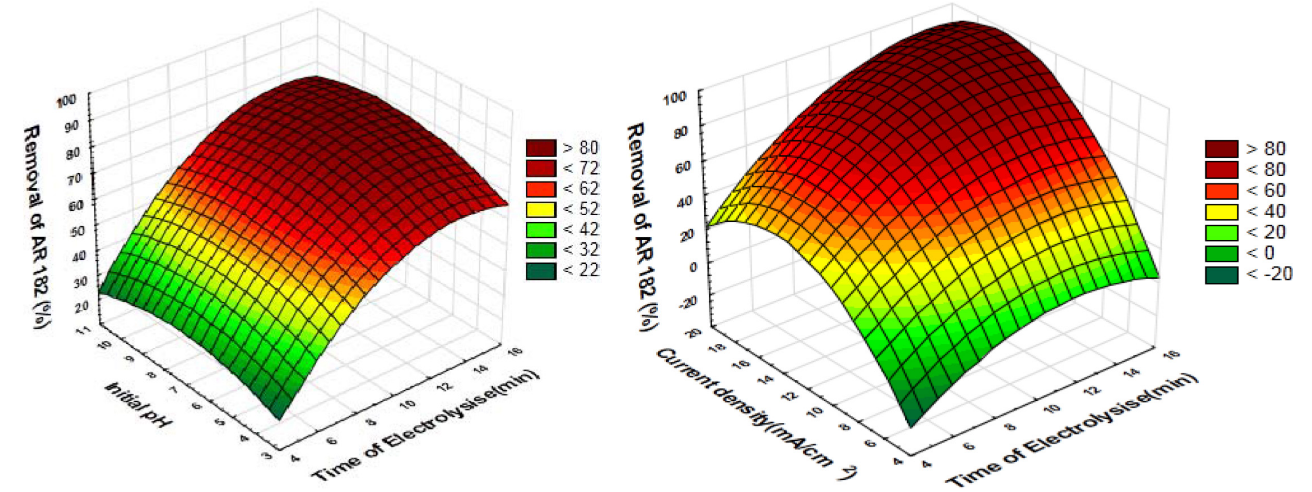


Fig. 6 (a). 3-D plot of Acid red 182 removal efficiency vs. initial pH and time of electrolysis. Fig. 6 (b). 3-D plot of Acid red 182 removal efficiency vs. current density and time of electrolysis.

particles [30,31]. The removal efficiency of AR 182 was less at low and high pHs. When the pH increased from 4 to 7, the removal of dye was enhanced. This trend was related to the amphoteric behavior of aluminum hydroxide, which does not precipitate at low pH [32], because at pH of 4, the soluble cations such as Al^{3+} and $Al(OH)^{2+}$ are predominant. The concentration of soluble anion, $Al(OH)_4^-$, was enriched at pH of 10, therefore the removal of AR 182 was decreased.

The response surface, 3D plots of efficiency as a function of two independent variables are illustrated in Figs. 6a,b. The interaction of each two variables was explored by remaining the other at its central level in the removal of Acid red 182. Eq. (4) is used for drawing the plots. However, the response surface of AR 182 removal shows a clear peak, signifying that the optimum conditions for maximum dye removal are well inside the design boundary.

According to the overlay plot, the optimum conditions for current density, pH and time are, 16 m A/cm^2, 8 and 14.2 min, respectively. These values were also experimentally validated. The removal of Acid Red 14 Azo dye, by the EC process with RSM was investigated by Aleboyeh et al., and they confirmed that the current density, time of electrolysis, their interaction, and the quadratic effect of all variables were the most effective factors. The significance of these cooperative effects between the variables would have been lost if the experiments were performed by conventional techniques [33]. In this project the maximum removal of AR 182 was 97.2% that it was observed in run number 4. For further validation the experiment was performed in the optimum conditions obtained from software and about 98.7% of AR 182 was removed. Therefore, the result (98.7%) were verified the predicted values (100%). The results showed that the maximum removal percent was obtained when the values of each variable were arranged as the optimum values, which was in good agreement with the value fore casted by the model. It exposes that the method used to optimize conditions for treatment of AR 182 by the EC process was positive.

At optimum condition the removal of chemical oxygen demand (COD) was 88.3%, and it was lower than the removal of AR 182 (98.7%). The difference between the removal of COD and AR 182 was low because the creation of intermediate products was low.

4. Conclusions

The EC system can remove Acid red 182, effectively. The Box–Behnken design was employed to investigate the influence of current density, time of electrolysis and pH on the removal percent of AR 182. The three dimensional response surfaces and their resultant contour plots were employed to assess the simple and combined effects of the three main independent parameters on the dye removal efficiency. The ANOVA was presented a high determination coefficient value (R^2 = 0.9817, R^2_{adj} = 0.9487 and R^2_{pred} = 0.7070) and satisfactory prediction second-order regression model. The influences of the main operating parameters on dye removal efficiency have been inspected. The high efficiency is reached in current density at 16 m A/cm^2, time of electrolysis at 14.2 min and pH of 8. The coagulant production rate, the growth of created flocks and the generation of bubbles was increased with enhancement in current density. The dye removal efficiency was very low either at very acidic or alkaline pH. This study clearly exhibited that the RSM was one of the suitable approaches to optimize the operating conditions and maximize the dye removal. At optimum conditions, the removal of Acid red 182 and COD was 98.7 and 88.3%, respectively.

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