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The using of response surface methodology to optimize methyl orange removal from textile dyeing wastewater

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ABSTRACT

The enormous demand for water and the creation of effluent that is dye-rich make the textile industry a hazard to water resources. Electrocoagulation (EC) is considered an appealing approach for treatment, which offers effective color removal in a quick, dependable, and affordable manner. Using 3^3 full factorial design trials, the performance of EC in the treatment of synthetic wastewater including textile coloring, such as methyl orange, was examined using the response surface methodology (RSM). In the presence of iron electrodes, wastewater was electrolyzed at room temperature with a pH of 7 under various working parameters, including initial dye concentration, applied voltage, and electrolysis time. The findings suggested that utilizing EC has been a successful strategy for removing color from textile dyeing effluent. At an applied voltage of 15 V, an electrolysis period of 30 min, and an initial dye concentration of 50 mg/L, the maximum color removal efficiency was 96.6%. The comparable Electrical Energy Consumption (EEC) for these circumstances was 23.7 kWh/m^3. It was noticed that 87.5%–91.0% of the color was eliminated during the first 20 min of the process.

Keywords: Electrocoagulation; Methyl orange; Wastewater; Textile; Dye

1. Introduction

The textile industry is a key sector in a nation's economy, and it presents several work prospects [1,2]. Egypt has a sizable textile sector that exports and produces a lot of textiles globally. In 2013, Egypt's textile sector consisted of well over 3,000 businesses, ranging from modern, highly automated plants to small, traditional units for handmade goods. The textile industry has a major impact on the economy of Egypt, this constituted by about 34% of total exports [3].

Large amounts of water are contaminated by the synthetic colors used in the textile industry. Textile colors are released into the aquatic environment as effluent because they do not adhere to the fabric securely [4]. As a result, the environment and public health are significantly harmed by the ongoing untreated outflow of wastewater from several textile industries. From an environmental viewpoint, textile and dyeing industries are characterized by being extremely unfriendly owing to the obnoxious nature of the polluting species present in the drained wastewater. These include dyes, organic moieties and diverse suspended solids [5,6].

There are several physical, chemical and biological methods used in decolorization of wastewater, such as: coagulation–flocculation [7], oxidation–ozonation [8], microalgae-bacteria consortium [9], membrane technologies [10] and adsorption techniques [11].

For the effective, dependable, and affordable removal of color from textile dyes, electrocoagulation (EC) is a desirable treatment option. However, it is necessary to analyze its

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process variables in order to improve removal effectiveness and lower operating costs [12,13].

While coagulation is one of the most successful wastewater treatment methods, the combination of coagulants and electrochemical processes has resulted in much higher pollutant elimination [14–16]. The reason for such improvement is related to the much higher adsorption level of onsite metallic hydroxides on mineral surfaces when using EC compared to that when hydroxides are used as coagulants [17,18].

Several studies related to the use of EC for the treatment of textile effluent have been documented. In this context, new treatment methods have been investigated, with great emphasis on electrocoagulation by Lach et al. [19] to evaluate the efficiency of implementing the EC process in the treatment of synthetic textile effluents for removing azo dye, in relation to carbon oxygen demand, total organic carbon, true color and acute toxicity. Experimental tests were performed for the EC process to investigate the effects of current intensity, electrolyte concentration and dye concentration regarding the removal of the dye into the synthetic textile wastewater. The results indicated color removal of 96.5% with a 4 A current intensity and 20-min electrolysis time applied.

In their paper, Yaqub et al. [20] have applied electrocoagulation process for the decolorization of synthetic wastewater containing Reactive Blue-2. Experiments for decolorization of Reactive Blue-2 solutions were performed by varying the types of Aluminum and steel electrode connected with power supply. Optimum experimental conditions were found at 95% color removal efficiency using electrolysis time of 6-min and at current density = 16.6 mA/cm^2 using steel electrodes.

In another work, the possibility of using EC for efficient removal of pollutants in the industrial liquid waste of a textile industry was studied by Núñez et al. [21]. The performance of the process was evaluated through the analysis of color, turbidity, and chemical oxygen demand (COD). After the treatment, 86% of the color, 82% of the turbidity, and 59% of the COD could be eliminated. It was shown that the quality of the dyed cloth is not adversely affected by the reuse of treated water during the wool dyeing process. As a result, the procedure can be used in the textile industry to lower water use.

Two stainless steel anodes and pure iron were compared in an electrocoagulation study by Dura and Breslin [22] for the simultaneous removal of phosphates, orange II and zinc ions from a synthetic wastewater. High removal efficiencies were observed with steel anode and pure iron, reaching values between 88% and 99%.

The treatment of wastewater containing two textiles' dyes (disperse and reactive types) by EC using aluminum electrodes was studies by Criado et al. [23]. The effects of the operating parameters pH, current intensity and electrolysis time on the percentage of color removal were investigated. Maximum percentages of color removal of 92.48% and 91.34% were achieved simultaneously, under the optimum operating conditions, for the disperse and reactive dyes, respectively. This study demonstrated that the EC process is an efficient way to treat wastewater. Also, since wastewater can be recycled during the wash stages of the dyeing process, it is a prospective alternative for usage in the textile sector.

In a study carried out by Tyagi et al. [24], EC process was used to treat a synthetic wastewater containing basic red dye. The effect of different parameters like pH and current density on color and chemical oxygen demand removal was investigated using iron electrodes. Their results indicated that chemical oxygen demand removal from solution was 76% and color removal was 95% during 1 h operation.

The main objective of this study was to investigate the removal of methyl orange (MO) dye from synthetic wastewater by electrocoagulation cell. MO removal from textile effluent using EC has not been investigated much by researchers. In this research, the novelty was to study response surface methodology (RSM) to develop an experimental program in order to know the optimum treatment process conditions for MO removal in the presence of iron electrodes.

2. Materials and methods

2.1. Materials and chemicals

- Iron electrodes.
- Sodium chloride, hydrochloric acid and sodium hydroxide were supplied from El Nasr Pharmaceutical Chemicals Company.
- Methyl orange was supplied from: El Nasr Pharmaceutical Chemicals Company for chemicals.

Methyl orange dye general characteristics are illustrated in Table 1.

2.2. Experimental apparatus

The used EC reactor components consist of a cylindrical glass beaker (500 mL) with two identical electrodes, iron anode and iron cathode with dimensions of (60 mm × 120 mm × 2 mm) connected in parallel mode to a DC power supply (RXN-305A(D) (0–30 V/0–5 A) and inter-electrode distance was 20 mm. The EC system was placed on a magnetic stirrer as shown in Fig. 1 [25].

2.3. Experimental procedure

500 mL of dye solution was placed into the electrolytic cell for each run through batch mode of operations. The synthetic dye solution was prepared by dissolving 10, 30, 50 mg of MO in 1 litre of distilled water and mixed by a magnetic stirrer at stirring speed of 300 rpm. The mixing of the solution was applied during the whole process at various electrolysis times 10, 20, 30 min in order to achieve complete mixing. The position of the stirrer was so adjusted

Table 1
General characteristics for methyl orange

Characteristic	Methyl orange
Chemical formula	$C_{14}H_{14}N_3NaO_3S$
Molecular weight	327.33 g/mol
λ_{max} (nm)	465

to ensure proper mixing while its speed was regulated so as to keep flocks from breaking down. The conductivity of the solution was enhanced by adding 1.6 g/L sodium chloride as supporting electrolyte. Sodium hydroxide or Hydrochloric acid was added to adjust the initial pH of the solution before each run and initial pH was measured by HANNA, HI 2211 PH/ORP Meter to get initial solution pH at 7.3. All experiments were carried out at room temperature. At the end of the EC experiments, all samples were leaved for settling for 10 min. Then, the samples were filtrated using double rings filter paper circles (125 mm) as shown in Fig. 2. Before each test, the electrode plates were cleaned manually by scrubbing with sandpaper and by treatment with 5% (v/v) HCl acid for 5 min, followed by washing with distilled water [26]. Each test was conducted two times and the average removal percentage was used in the calculations.

At the end of each run, the dye concentrations were determined using a UV-visible spectrophotometer (Jenway 6305) at a wavelength corresponding to the maximum absorbance of the MO (λ_{max} = 465 nm).

2.4. Calculations

2.4.1. Color removal efficiency

The color removal efficiency R (%) was calculated according to Eq. (1):

$$R(\%) = \frac{C_i - C_f}{C_i} \times 100 \qquad (1)$$

where C_i is the initial dye concentration (mg/L); C_f is the final dye concentration (mg/L).

2.4.2. Electrical energy consumption

The consumption of electrical energy represents an important economic parameter in the EC process. The electric energy consumption was calculated using Eq. (2):

$$E = \frac{V \cdot I \cdot t}{V_s} \qquad (2)$$

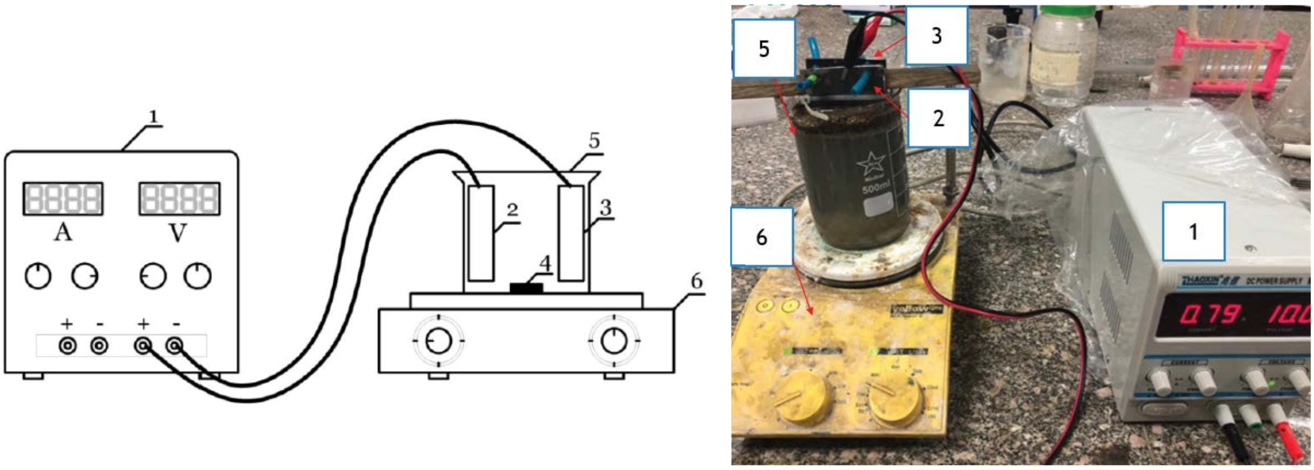

Fig. 1. Schematic view of electrochemical reactor: 1 – source of electric power; 2 – anode; 3 – cathode; 4 – magnetic stir bar; 5 – electrochemical cell; 6 – magnetic stirrer.

(a) (b)

Fig. 2. (a) Settling step and (b) filtration step.

where E is the electrical energy consumed per unit volume (kWh/m^3); V is the cell voltage (V); I is the current (A); V_s is the volume of solution (L); t is the time of EC process (h).

2.4.3. Full factorial experimental design

A 3^3 full factorial design method was applied to reduce the number of experiments. Table 2 shows the different factor level settings to determine the best conditions for maximum color removal. The calculations were done using XLSTAT 2020 module and Table 3 shows all three variables information which were entered to the module. As shown in Table 4 the set of experiments were designed to test successively different operating independent parameters such as voltage (10–20 V), electrolysis time (10–30 min), initial dye concentration (10–50 mg/L).

3. Results and discussion

A starting point in the present investigation was to determine the relative effect of each of the three enunciated parameters on the % color removal. This was carried out using the DATA ANALYSIS module present in EXCEL program. The reported numerical figures represent the extent of correlation between the % color removal and each parameter, through the individual correlation coefficient R.

Three-dimensional (3D) response surface plots are designed to analyze the individual and interactive effect between process variables on the responses and also to determine the optimal condition for maximum efficiency in color removal. These were generated through the software used.

Also, 2-dimensional plots obtained as contour mapping of the three-dimensional ones can be deduced that better emphasize the effect of each investigated parameter on the % dye removal.

Table 5 shows the obtained correlation matrix on applying the aforementioned module on the experimental data obtained for methyl orange. It appears from the table that the most influencing parameter is the initial dye

Table 2
Experimental parameters and their levels

Parameter	Level		
	Low	Center	High
Initial dye concentration (mg/L)	10	30	50
Electrolysis time (min)	10	20	30
Applied voltage (V)	10	15	20

Table 3
Variable information

Short name	Long name	Unit
F_1	Initial dye concentration	mg/L
F_2	Operation time	min
F_3	Applied voltage	V

Table 4
3^3 Full factorial design matrixes with the studied independent variables

Run order	Concentration (mg/L)	Time (min)	Applied voltage (V)
1	10	10	10
2	30	10	10
3	50	10	10
4	10	20	10
5	30	20	10
6	50	20	10
7	10	30	10
8	30	30	10
9	50	30	10
10	10	10	20
11	30	10	20
12	50	10	20
13	10	20	20
14	30	20	20
15	50	20	20
16	10	30	20
17	30	30	20
18	50	30	20
19	10	10	30
20	30	10	30
21	50	10	30
22	10	20	30
23	30	20	30
24	50	20	30
25	10	30	30
26	30	30	30
27	50	30	30

Table 5
Correlation matrix for % color removal for methyl orange dye

Variables	Initial dye concentration	Operation time	Applied voltage	Color removal
Initial dye concentration	1.000	0.000	0.000	0.513
Operation time	0.000	1.000	0.000	0.127
Applied voltage	0.000	0.000	1.000	0.190
Color removal	0.513	0.127	0.190	1.000

concentration, followed by a large margin by applied voltage whereas the electrolysis time seems to be the least factor of influence. Fig. 3 which represents the standardized coefficients at 95% confidence interval confirms the same result of the most influencing parameter. Also, it illustrates the interactive effect between process variables.

3.1. Regression equation

The program used enabled to predict the relation between % color removal of MO and the three operating variables in the form of a second-order regression equation involving interaction terms. This equation takes the form:

$$\%C = 91.867 + 3.217C_0 + 0.794t + 1.194V - 5.237C_0^2 + 0.253t^2 + 2.747V^2 + 1.648C_0 \cdot t + 2.137C_0 \cdot V - 0.370t \cdot V \tag{3}$$

The validity of that equation can be judged in several ways. A common method is to plot the experimental values of color removal against the values predicted from the regression equation. Such plot is displayed in Fig. 4 referred to a 45° line. The model has a coefficient of determination (R^2) of 0.737 and the calculated multiple correlation coefficient (R) for these data of 0.858, which represents a fair validation of the above equation and means that it is moderately reliable.

3.2. Effect of applied voltage

The two parameters used in electrochemical processes that make it simple to control the reaction rate are applied voltage and electrolysis time. This is since they define the generation rate of the coagulant.

As shown in Figs. 5 and 6, an increase in color removal was detected with increasing applied voltage and operating time values as previously reported from Table 5. However, an increase in both parameters result an optimum zone for effective color removal represented by a dark shade. This occurs at applied voltages higher than 15 V and electrolysis time start from 10 min which represent optimum conditions for color removal as the maximum predicted removal reaches 96%. The highly non-linear character of the response surface explains the low values of correlation coefficients appearing in Table 5 which were due to the fact that these coefficients are calculated assuming a linear correlation.

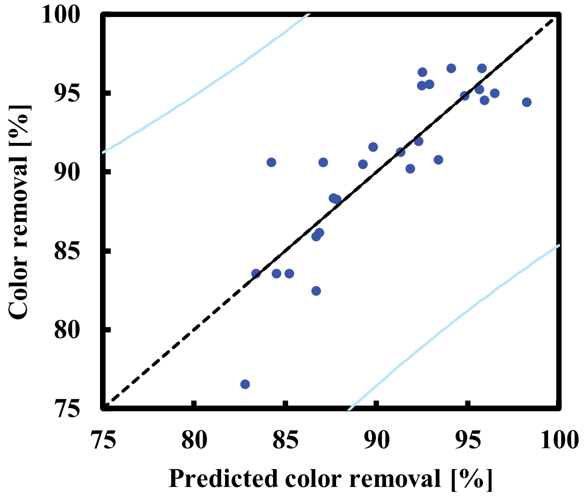

Fig. 4. Comparison between experimental and predicted values of color removal of MO from wastewater.

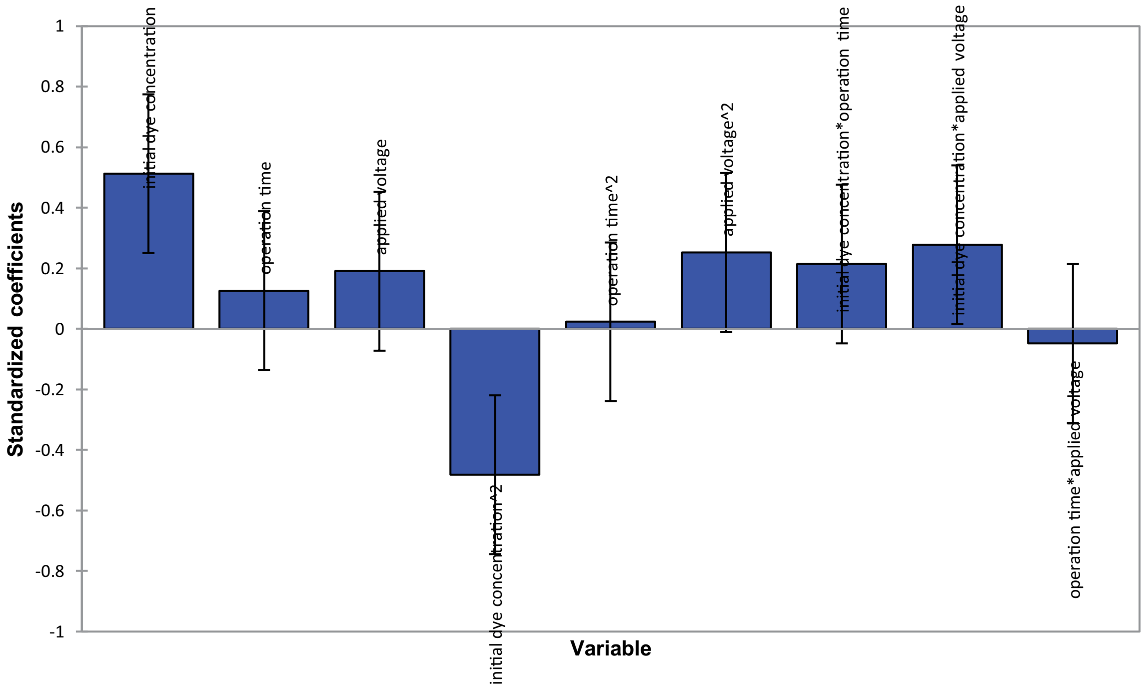

Fig. 3. Color removal/standardized coefficients (95% conf. interval).

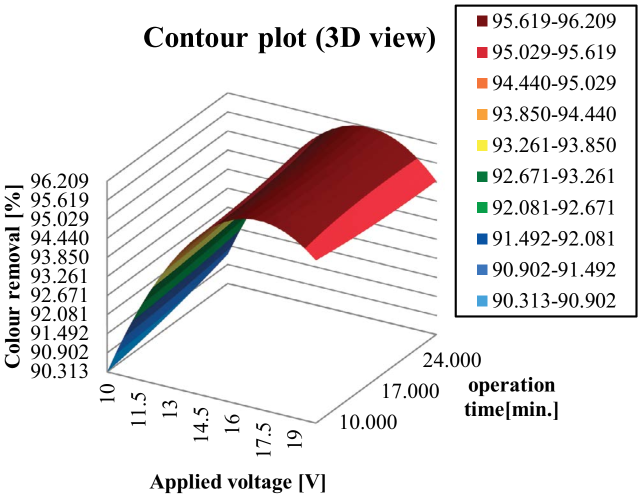

Fig. 5. Response surface plots (3D) for the effects of variables on MO color removal efficiency at constant initial dye concentration.

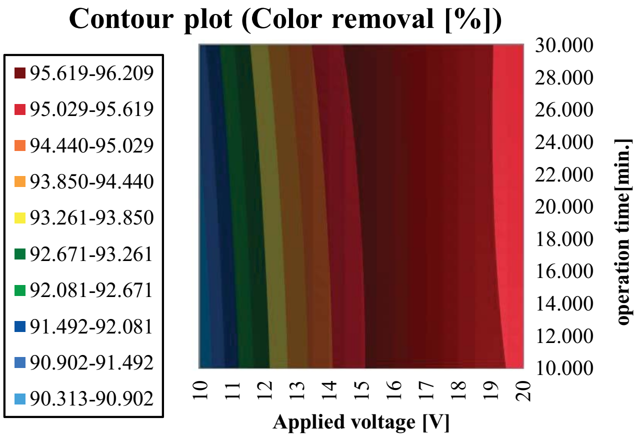

Fig. 6. Contour plot of % color removal of MO representing electrolysis time vs. applied voltage.

As shown in Fig. 7 when the applied voltage increased from 10 to 20 V at the optimum time of 17 min, the % color removal is increased from 90% to 95%. For a solution with a dye concentration of 30 mg/L, the optimum current voltage was in the range of 15–19.5 V.

The previous results reveal that the rate at which the anode is depleted increases with applied voltage, following Faraday's law. This results in a progressive increase in iron hydroxides creating more flocks that enhance the coagulation process. In addition, the bubbles generation rate increased and the size of these bubbles decreases at higher voltage which enhance the mixing of iron hydroxides and MO dye and further enhance the flock separation by flotation [27]. Consequently, the amount of dye adsorption increased with the increased concentration of adsorbents in the reactor.

On the other hand, Fig. 8 shows the effects of voltage and time on electrical energy consumption. Results showed that when electrolysis time increases from 10 to 30 min. at 20 V and any different initial dye concentration the EEC was approximately triple the figure at 10 min. At electrolysis time 30 min and applied voltage 15 V the energy consumption was 21.7, 21.0 and 23.7 kWh/m^3 at dye concentration 10, 30 and 50 mg/L, respectively. Results showed that at electrolysis time 20 min and initial dye concentration 30 mg/L when the voltage increased from 15 to 20 V, the electrical energy consumption increased from 13.4 to 29.6 kWh/m^3 wastewater which was approximately double the figure at 20 V. Since at 15 V, the color removal was approximately at its maximum value (96%), So, 15 V is the recommended voltage due to high dye removal efficiency and low electrical energy consumption.

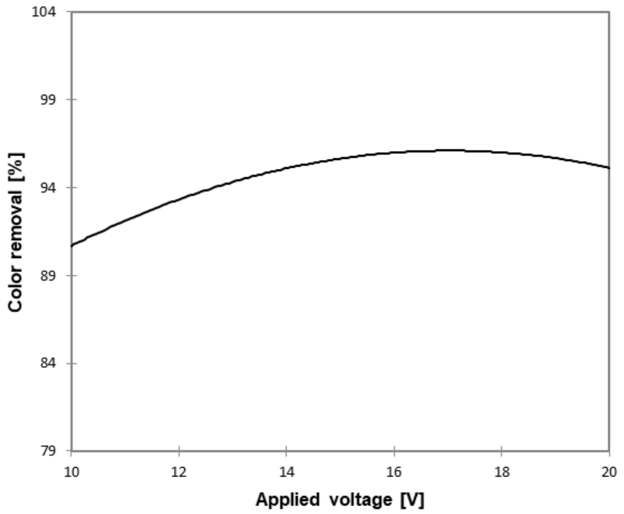

Fig. 7. Effect of applied voltage on MO color removal in EC.

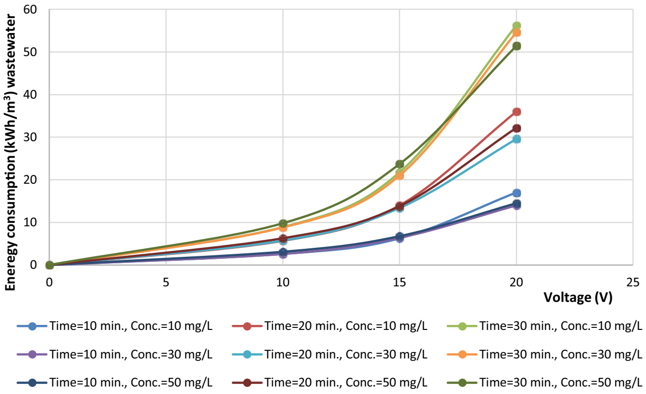

Fig. 8. Effect of voltage on electrical energy consumption in color removal of MO.

3.3. Effect of electrolysis time

Electrolysis time is one of the affecting parameters in the EC. Figs. 9 and 10 display the 3-dimensional response surface and 2-dimensional counterplot to investigate the effect of applied voltage and electrolysis time on MO removal, where the other parameters were maintained constant at 30 mg/L and pH = 7.3.

These figures reveal that discoloration efficiency increases from 87% to 92% when the electrolysis time increases from 10 min up to 30 min at constant dye concentration 30 mg/L and applied voltage 10 V.

This result can be explained by the fact that when the electrolysis time increases the concentration of metal ions (Fe ions), then the concentration of metal hydroxides produced on the electrodes will increase. As a result, the formation of coagulants increases which promotes the color removal [28].

The prediction of the maximum amount of color removal as a function of time of EC and initial dye concentration is shown in the contour plot in Fig. 10. It appears that color removal increases with an increase in the electrolysis time during of the initial dye concentration range studied from 10 to 50 mg/L. The maximum color removal was greater than 93% at operation time 30 min and initial dye concentration of 50 mg/L.

As shown in Fig. 11, there is an increasing trend in color removal with increasing of electrolysis time till 25 min. The removal efficiency almost reached maximum values at this value, remaining almost constant for higher operating times. Increasing the operating time can increase the accumulation of dye residue causing passivation on cathode content. This way, anodic dissolution decreases leading to a corresponding decrease in the formation of metal hydroxide. As a result, dye removal efficiency stops increasing [29]. These results comply with those obtained by Dalvand during the treatment of synthetic textile wastewater containing Reactive Red 198 by using EC process [30].

3.4. Effect of initial dye concentration

To study the influence of initial dye concentration on dye removal efficiency during electrocoagulation, three dye solutions with different initial dye concentrations (10, 30, and 50 mg/L) were used. Figs. 12 and 13 show that when the dye concentration increased from 10 to 30 mg/L, the % color removal increased from 78 to 86%. For an initial dye concentration 50 mg/L it increased to reach 96% when the applied voltage was kept above 10 V.

The color removal increases with the increase in the initial dye concentration, as can be seen in Fig. 14 because

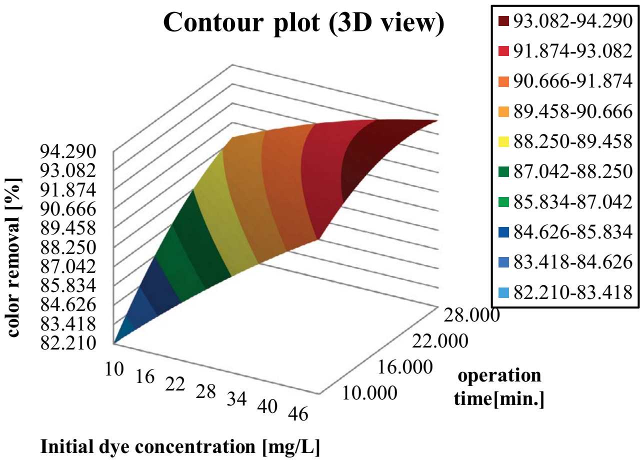

Fig. 9. Response surface plots (3D) for the effects of variables on the color removal efficiency of MO at constant the applied voltage.

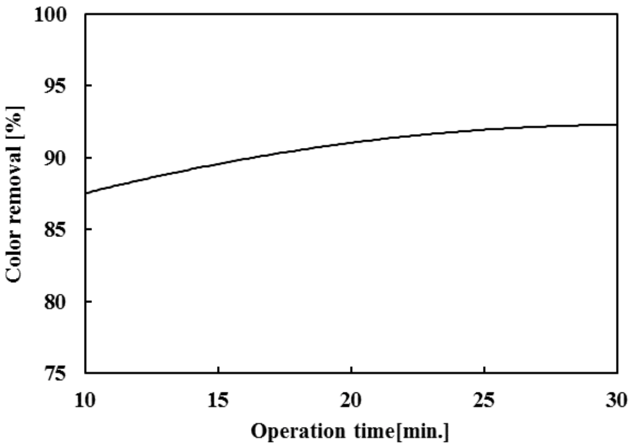

Fig. 11. Effect of electrolysis time on color removal of MO.

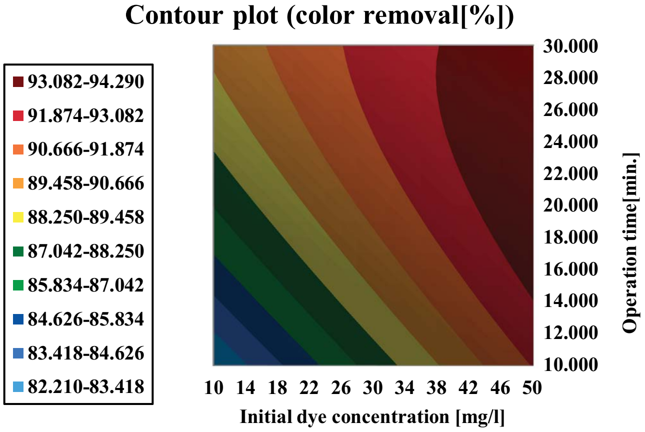

Fig. 10. Contour plot of % color removal of MO representing electrolysis time vs. applied voltage.

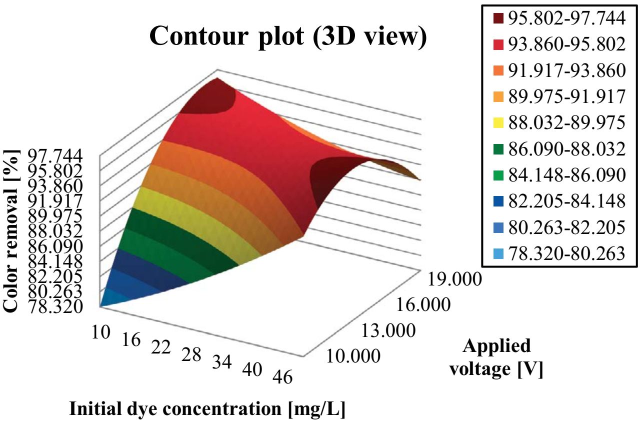

Fig. 12. Response surface plots (3D) for the effects of variables on the color removal efficiency of MO at constant electrolysis time.

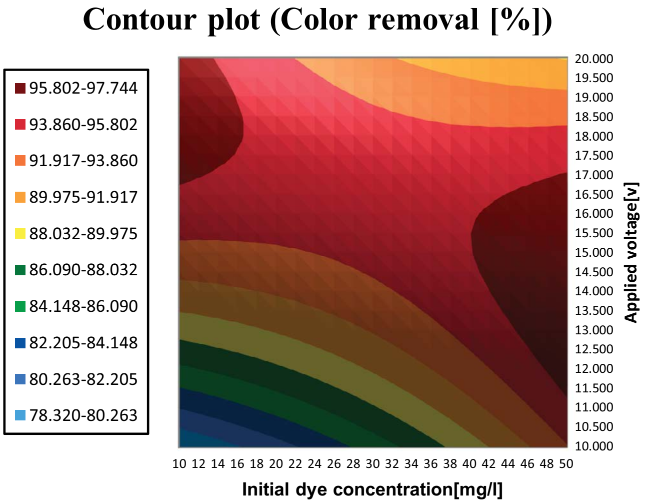

Fig. 13. Contour plot of % color removal of MO representing initial dye concentration vs. applied voltage.

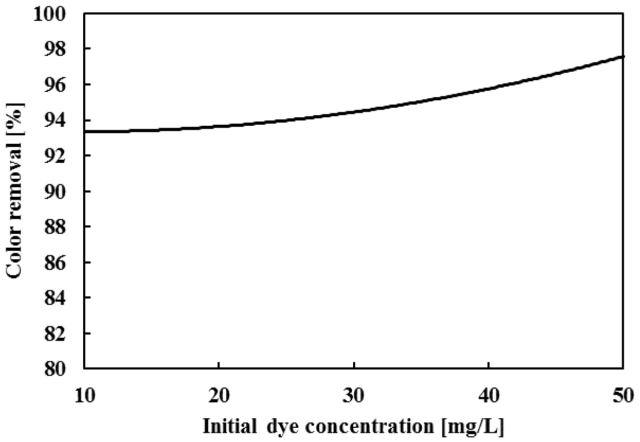

Fig. 14. Effect of initial dye concentration on color removal of MO.

Table 6
Statistics summary

Variable	Observations	Obs. with missing data	Obs. without missing data	Minimum	Maximum	Mean	Std. deviation
Color removal	27	0	27	76.515	96.571	90.376	5.221
Initial dye concentration	27	0	27	10.000	50.000	30.000	16.641
Operation time	27	0	27	10.000	30.000	20.000	8.321
Applied voltage	27	0	27	10.000	20.000	15.000	4.160

Table 7
Analysis of variance

Source	DF	Sum of squares	Mean squares	*F*	Pr. > *F*
Model	9	522.646	58.072	5.302	0.002
Error	17	186.207	10.953		
Lack of fit	17	171.459	10.086		
Pure error	0	14.748			
Corrected total	26	708.853			
Computed against model $Y = 0$					

of the increased possibility of metal hydroxide interaction with dye molecules to form large-sized flocks. In this way, they promote their separation by the bubbles of released gases at electrodes. The same results were obtained when basic dye Rhodamine B was removed from a solution by using EC. When the initial dye concentration increased from 10 to 50 mg/L, the color removal increases from 25.1 to 134.3 mg/g. Also, Durango-Usuga et al. [31] concluded that the discoloration rate increased by increasing the initial concentration of crystal violet dye.

Summary of statistics and standard deviation is shown in Table 6. It is clear that for MO dye, the minimum color removal efficiency was 76.5% while the maximum color removal efficiency was 96.6% with standard deviation of 5.2.

The sufficiency of the statistical significance of the model equation and regression coefficients was evaluated by the analysis of variance (ANOVA). ANOVA showed that the suggested equation is significant at a 2% level of probability (Table 7). Moreover, the reliability of the model was determined through the ANOVA, and values of "Prob. > *F*" display that the model can be moderately valid.

4. Conclusions

In this paper, the objective was to evaluate the performance of using of electrocoagulation in the treatment of textile dyeing wastewater. The three-level full factorial design was applied for determining the optimum experimental conditions. Several experiments were performed using an electrochemical batch reactor at varies electrolysis time (10, 20 and 30 min.), applied voltage (10, 15 and 20 V) and initial dye concentrations (10, 30 and 50 mg/L).

As a result of the experimental work, the following conclusions were drawn:

- Using EC with iron electrodes has proven to be an effective method in the color removal from textile dyeing wastewater.
- The XLSTAT 2020 verified that the initial dye concentration is the most influential factor in the EC removal of color.

- 70%–71% of the color removal occurred during 20 min and for higher operation time, the removal color rate increases to 76%.
- For MO dye, the maximum color removal efficiency was 96.6% and reached at applied voltage 15, time = 30 min and initial dye concentration = 50 mg/L and the corresponding EEC was 23.7 kWh/m^3.
- There is a direct relation between the electrolysis time and applied voltage on the energy consumption, when the electrolysis time increase or applied voltage, the EEC increases.
- At constant electrolysis time and different dye concentration, the energy consumption was approximately double the figure when the applied voltage increased.

Acknowledgement

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