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Electro-decolorization of Reactive Red 198 from aqueous solutions using aluminum electrodes systems: modeling and optimization of operating parameters

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ABSTRACT

During recent years, electrochemical (EC) techniques have been applied as an effective process to treat industrial and domestic wastewater. Thus, Reactive Red 198 dye was selected, as a model of azo dyes group, to evaluate electrocoagulation decolorization potential. In the present study, the removal model and the related optimal operating conditions were developed using Response Surface Methodology (RSM) and the central composite. Regarding the design of the method (RSM) about 50 time modeling of considerable parameters were analyzed and optimized. In the variants analysis a relatively high correlation coefficient of about 0.77 was observed. p value for the dye decolorization model proved to be significant ($p < 0.0001$).The results related to the optimal parameters were obtained under optimal operating parameters pH = 6.45, current density 7.2 mA/cm^2, operating time 57.4 min, and distance 1.9 cm. The most and the least decolorization efficiency was 69 and 91.3%, respectively. Therefore, EC can successfully be applied to the treatment of effluents containing dyes.

Keywords: Decolorization; Reactive Red 198; Electrocoagulation; Response surface Methodology

1. Introduction

Today, various industries affect human life and the environment directly or indirectly. Among these industries is the textile industry, which is regarded as a major potential source of pollution. Azo Dyes such as RR198 are abundant in textile industries. Because of toxic impact of dye on aquatic life, carcinogenic and mutagenic effects [1] should be treated before disposing of these dyes. Different methods have been used for decolorization of dyes existing in wastewater, so far; among which, adsorption, coagulation, ozonation, fenton, and photo-Fenton processes are worth mentioning [2–4]. UV/NaOCl, electrochemical oxidation, ultrasonic

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irradiation, UV/H_2O_2 are among these methods. Conventional biological processes are often ineffective in removing dyes with highly structured polymers, which are considered as low biodegradable polymers [5]. Also, biological treatment is less efficient due to the toxicity of the dyeing wastewater [6]. Recently, many researchers have considered the electrocoagulation technique as an effective and environmentally friendly process for wastewater treatment. Contaminant removal with electrochemical methods offers several advantages: these methods have no chemical requirements, do not produce sludge, enjoy strong oxidation ability, fast reaction rate, and do not need too much space [3,4,7]. The Electrocoagulation process has been used for treating various effluents from food and protein industry [8], restaurant wastewater [9], electroplating [10], and domestic wastewater [11]. In addition, elimination of various pollutants have been reported, from a real wastewater, to be such as heavy metals (Cr, Zn, Ni, and Cu), chemical oxygen demand, total organic carbon, total dissolved solids, oil and grease, phosphate, fluoride, nitrate, and sulfate, all being removable through electrocoagulation treatment [4,12,13]. In the study of aluminum anodes, anodic and cathode mechanisms have been suggested for the production of metal hydroxide as mentioned in the following [12,14].

Anodic reaction:
In acidic pH:

$$4OH^- - 4\bar{e} \rightarrow 2H_2O + O_{2(g)} \quad (1)$$

In alkaline pH:

$$2H_2O - 4\bar{e} \rightarrow O_{2(g)} + 4H^+ \quad (2)$$

$$Al_{(S)} - 3\bar{e} \rightarrow Al^{3+}_{(aq)} \quad (3)$$

$$Al^{3+}_{(aq)} + 3H_2O \rightarrow Al(OH)_3 + 3H^+ \quad (4)$$

Cathodic reaction:
In acidic pH:

$$2H_2O^+ + 2\bar{e} \rightarrow H_{2(g)} + 2H_2O \quad (5)$$

In alkaline pH:

$$2H_2O + 2\bar{e} \rightarrow H_{2(g)} + 2OH^- \quad (6)$$

$$2H_2O + 2\bar{e} \rightarrow H_{2(g)} + 2OH^- \quad (7)$$

$$O_2 + 2H_2O + 4\bar{e} \rightarrow 4OH^- \quad (8)$$

$$Al_{(S)} + 4OH^- \rightarrow [Al(OH)_4]^- + 3\bar{e} \quad (9)$$

In the present study electrochemical technique and Response Surface Methodology (RSM) were used to evaluate the decolorization efficiency of Reactive Red 198. In addition, a model was developed to promote the efficiency of RR198 and concomitant optimal operating conditions. Decolorization equation model and optimum values enable the estimation and simulation of bench and pilot Scale, estimation of the cost of operation advantages, and application of current density, as a standard for the assessment of dye effluent percentage.

2. Material and methods

2.1. Material

In order to prepare synthetic colored wastewater, Reactive Red 198 was dissolved in deionized water and was used as a stock solution. Characteristics of Reactive Red 198 have been presented in Fig. 1. All reagents (NaCl, NaOH, HCl, H_2SO_4, etc.) having analytical grade were prepared. Their pH was restored to the desired value, using 1 M HCl and 1 M NaOH. The NaCl solution at 0.1% (w/v) was prepared as a supportive electrolyte.

2.2. Analysis

In order to remove solid material, the collected samples were filtered and their respective pH was

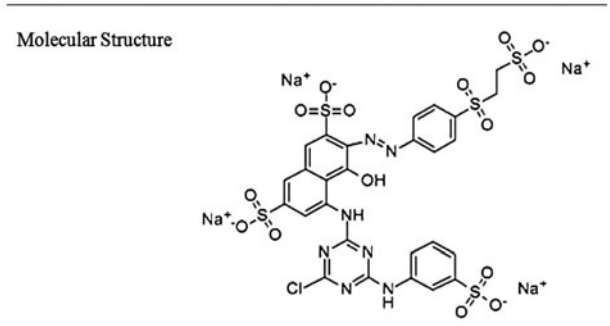

Fig. 1. Chemical structure of Reactive Red 198.

measured by means of a pH-meter (Eutech). Decolorization rate of RR198 in the solution samples was evaluated by measuring light absorbance of the samples at a wavelength of 518 nm using a UV/vis T + 80 spectrophotometer [15]. Scanning electron microscope (SEM) image from the electrocoagulation sludge was taken by means of KYKY-EM3200. Moreover, all the experiments were performed at room temperature (23 ± 1 ℃).

2.3. Reactor setup

Decolorization unit (Fig. 2) consists of the glassy graduated cylinder (250 cc) as electrochemical reactor, DC Power Supply (TEK-8051), peristaltic pump (Watson Marlow 101U/R) equipped with aeration tubes. In the Electrocoagulation process, two electrodes were used in mono-polar state as aluminum–aluminum arrangement. Aluminum electrodes with 65 × 20 × 2 mm dimensions were applied. Before the start of decolorization practices, both electrodes with 0.1 M H_2SO_4 were cleaned and rinsed with deionized water to eliminate impurities from the surface of the electrodes. At the time of analyzing, all the samples were filtered and their absorbance was determined.

2.4. Response Surface Methodology

RSM is a stage of experimental design which includes a collection of mathematical–statistical methods for modeling and determining model equations. This is also used to optimize experimental responses and decrease the number of tests. In the first stage, we found out that the responses properly function with the independent variables. This approximate function must be polynomial of the independent variables. Also the behavior of the system is explained by the following quadratic equation [16,17];

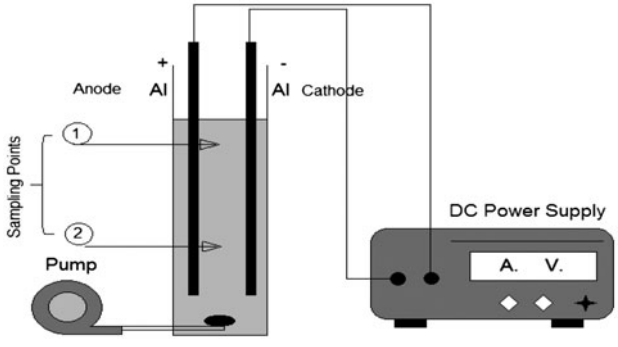

Fig. 2. Schematic figure of decolorization unit.

$$Y = \beta_i + \sum_{i=1}^{k} \beta_1 x_1 + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i=1}^{k} \sum_{j=1}^{k} \beta_{ij} x_i x_j + \varepsilon \quad (10)$$

where Y: the considered response, β_i β_1 β_{ii} β_{ij}: the coefficients of regression, ε: the error value of the system, and X_j: coded variables.

The least squares method was used to estimate polynomial approximation. The central composite design (CCD) was used to introduce this model as the most famous design. CCD of the main parameters (A: Time, B: Dye Concentration, C: pH, D: Current density, and E: Distance between electrodes) has been shown in Table 1. More details such as low axial point, high axial point, factorial point, and central point and their ranges have also been provided.

3. Results and discussion

3.1. Analysis of variance

Regarding the experiment design in modeling, analysis, and optimizing the parameters under consideration were defined 50 times. Table 2 shows the number of experiments with coded factors using the CCD. Also, decolorization efficiency in each condition is shown in two categories. The experimental data were obtained under designed conditions, and model percentages were calculated based on the provided equation and coded factors (Eq. (2)).

Final equation based on the coded factor as following:

$$\begin{aligned}\text{Decolorization } (\%) = {} & 84.37 + 2.50A - 0.60B - 1.31C \\ & + 2.06D - 0.033E + 0.70AB \\ & + 0.27AC - 0.98AD - 0.21AE \\ & - 0.27BC + 0.32BD - 0.25BE \\ & + 0.28CD - 0.27CE - 0.22DE \\ & - 0.85A^2 + 0.22B^2 - 0.23C^2 \\ & - 0.072D^2 + 0.67E^2\end{aligned} \quad (11)$$

Table 2 indicates Analysis of variance (ANOVA) for the RSM model of decolorization percentage of the RR198. In all the experiments, the confidence interval has been set at 0.05, resulting in the verification of the accuracy and reliability of the model. The p-value of the model proved to be significant ($0 < 0.0001$). Sum of squares, mean squares, and F-value for the quadratic model have been achieved to be 691.23, 34.56, and 4.84, respectively. Statistical values of quadratic model have been shown in Table 3. R^2 (0.7695), adjusted R^2 (0.6106), coefficient of variation (3.18%), and the mean

Table 1
The Natural and coded parameters

Factor	Name	Units	Type	Low axial	Low factorial	Center point	High factorial	High axial
A	Time	min	Numeric	2	24.5	41	57.5	80
B	Dye conc.	mg/L	Numeric	25	75.5	112.5	149.3	200
C	pH	–	Numeric	5	6.45	7.5	8.55	10
D	Current den.	ma/cm^2	Numeric	2	7.2	11	14.8	20
E	Distance	cm	Numeric	1	1.9	2.5	3.10	4

removal efficiency (84.14%) have been measured based on ANOVA. Difference between R^2 and adjusted R^2 should not be more than 0.2. In addition, the coefficient of the variation (CV) is the value of the reproducibility of the model and should be lower than 10% [18]. Therefore, the CV 3.18% indicates the reliability and high precision of the experimental data. Adequate precision is the ratio of signal, and the figure should be greater than 4 in order to be desirable [18]. The ratio obtained stood at 9.22, which is indicative of an adequate signal. Moreover, maximum and minimum decolorization percentages were determined to be 91.3 and 69%, respectively. Photographic Fig. 3 shows the color change before and after the electro-decolorization of RR198 at its optimal efficiency (91.3%).

According to this figure, the EC process is a powerful and suitable technique for treating dye existing in wastewater. Fig. 4 shows the predicted values vs. the actual (experimental) values. The predicted values were calculated by the model equation (Eq. (11)). The respective R^2 0.9669 and its fitted equation indicates the reasonability of experiments.

3.2. Effect of main parameters

Fig. 5 illustrates the three dimensional response surface plots of the main parameters and interactions between time, dye concentration, initial pH, applied current density, and distance between electrodes. As seen in Fig. 5, the interaction between operation time, pH, and decolorization efficiency is represented. According to the plot, a higher decolorization rate occurred under lower pH and short duration time. Decolorization is a fast process that occurs during a short exposure time after start-up. Close to 80% of the decolorization of RR198 can be observed in 41 min. According to Can et al. study, decolorization of Reactive dyes occurs in less than 20 min [19]. Aleboyeh et al. reported the maximum efficiency (>91%) of Acid Red 14 (azo dye) at the optimal time of 4.47 min [20]. Fig. 5(b) shows the effect of distance between electrodes and time. Generally speaking, changes in the distance are not followed by a significant effect. Nevertheless, internal resistance of the solution increases and, as a result, the electrical current decreases because of the widening of the space between the electrodes. In addition, less interaction between ions and hydroxyl polymers is expected due to an increase in interference reactions [21]. Fig. 5(c) illustrates the effect of pH and current density on electro-decolorization. The applied current or current density is one of the important factors affecting the electrocoagulation process. As can be seen from Fig. 5(c), the decolorization percentage is proportionate to current density, which can be due to the coagulant dosage rate increase during electrocoagulation [22,23]. According to Faraday's law, direct increase of current intensity provides higher amounts of coagulants produced by the anode which are dissolved into the solution. It also leads to providing more oxidants. On the other hand, higher current density generate higher levels of oxidative bubbles having smaller size [24], providing more contact surface area between oxidant units and pollutants, subsequently. The effect of dye concentration and time on decolorization has been shown in Fig. 5(d). Regarding the changes in the efficiency of different concentrations of RR198, it is seen that higher percentages of efficiency are obtained in lower concentrations. This phenomenon occurs due to the constant value of the surface electrode and oxidative and coagulant species for different amounts of dyes. Thus, a lower amount of dye can be removed quickly.

3.3. Decolorization optimization and validation of the experimental model

To optimize RR198 decolorization, some factors including maximum dye concentration (200 mg/l), natural pH (6.5) of the solution, low current density (7.2 mA/cm^2), and maximum decolorization efficiency were taken into consideration. All of the above factors were applied between 2 and 80 min time and distances 1–4 cm. The following optimal operating conditions (high desirability 0.952), treatment time of 57.4 min, and distance of 1.9 cm were obtained at high concentration of RR198 and low current density with

Table 2
Experimental results of CCD designed experiments

Run	*A*	*B*	*C*	*D*	*E*	Experimental eff.%	Model eff.%
1	1	−1	−1	−1	1	87.8	87.45
2	1	1	1	1	−1	88.3	87.90
3	−1	−1	1	−1	−1	80.1	76.98
4	−1	1	−1	−1	−1	77.4	78.82
5	−1	−1	−1	−1	−1	80.8	79.62
6	1	−1	1	1	−1	85.5	88.50
7	−1	−1	1	−1	1	82.2	77.73
8	−1	1	1	1	1	84.4	82.77
9	1	−1	−1	1	1	89.1	89.05
10	1	−1	−1	1	−1	91.3	88.94
11	0	0	0	0	0	83.4	84.37
12	−1	−1	1	1	1	87.2	84.37
13	0	0	0	0	0	83.4	84.37
14	1	1	1	1	1	87.3	85.93
15	−1	1	−1	−1	1	82.8	79.65
16	1	−1	−1	−1	−1	85.1	86.46
17	0	2	0	0	0	81.8	84.05
18	0	0	0	2	0	85.5	89.28
19	1	1	−1	−1	1	87.4	85.65
20	0	0	0	0	−2	88.3	87.11
21	1	−1	1	1	1	89.3	87.53
22	0	0	0	0	0	84.4	84.37
23	1	1	−1	1	1	88.9	88.53
24	−1	1	1	−1	−1	75.5	75.10
25	0	0	0	0	0	83.6	84.37
26	−1	−1	−1	−1	1	84.8	81.45
27	0	0	0	0	0	84.3	84.37
28	1	−1	1	−1	1	83.5	84.81
29	−1	−1	−1	1	−1	86.7	86.02
30	−1	1	1	1	−1	83.8	83.90
31	−1	−1	1	1	−1	85.5	84.50
32	−2	0	0	0	0	69.9	75.97
33	1	1	−1	1	−1	87.6	89.42
34	−1	1	−1	1	1	87.7	86.45
35	−1	1	−1	1	−1	88.1	86.50
36	1	1	−1	−1	−1	86.4	85.66
37	1	1	1	−1	1	84.9	81.93
38	0	0	0	0	0	84.4	84.37
39	0	0	−2	0	0	85.3	86.07
40	0	−2	0	0	0	84.5	86.45
41	0	0	0	0	2	83.1	86.98
42	2	0	0	0	0	85.2	85.97
43	0	0	2	0	0	75.9	80.83
44	−1	1	1	−1	1	72.8	74.85
45	0	0	0	0	0	85.9	84.37
46	1	−1	1	−1	−1	84.8	84.90
47	−1	−1	−1	1	1	85.5	86.97
48	1	1	1	−1	−1	87.7	83.02
49	0	0	0	0	0	83.8	84.37
50	0	0	0	−2	0	77.9	78.88

Table 3
ANOVA and statistical values

Source	Sum of squares	Mean square	*F* value	*p*-value	
Model (quadratic)	691.23	34.56	4.84	<0.0001	Significant
A-Time	270.74	270.74	37.92	<0.0001	
B-Dye conc.	15.59	15.59	2.18	0.1503	
C-pH	74.43	74.43	10.42	0.0031	
D-Current density	184.64	184.64	25.86	<0.0001	
E-Distance	0.047	0.047	6.59E-03	0.9358	
AB	15.76	15.76	2.21	0.1481	
AC	2.3	2.3	0.32	0.5746	
AD	30.81	30.81	4.32	0.0467	
AE	1.35	1.35	0.19	0.6665	
BC	2.33	2.33	0.33	0.572	
BD	3.29	3.29	0.46	0.5026	
BE	2.04	2.04	0.29	0.597	
CD	2.57	2.57	0.36	0.5536	
CE	2.35	2.35	0.33	0.5702	
DE	1.56	1.56	0.22	0.6439	
A^2	40.17	40.17	5.63	0.0245	
B^2	2.65	2.65	0.37	0.5471	
C^2	2.98	2.98	0.42	0.5234	
D^2	0.28	0.28	0.04	0.8432	
E^2	24.95	24.95	3.49	0.0717	
Lack of fit	205.07				
Pure error	1.97	9.32	33.16	<0.0001	
Cor total	898.27	0.28			
R-squared (R^2)	0.7695				
Adj R^2	0.6106				
Std. dev.	2.67				
Adeq precision	9.22				

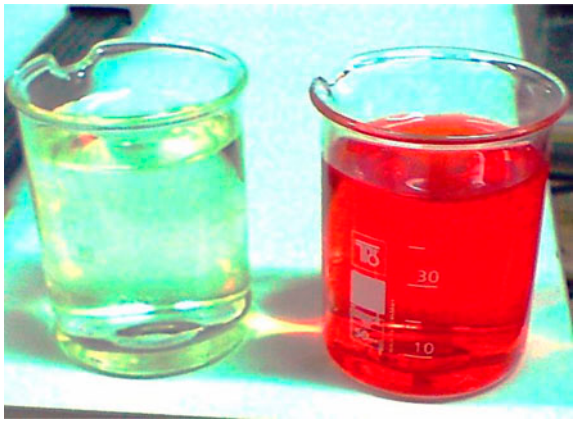

Fig. 3. Photographic image of color change during EC process.

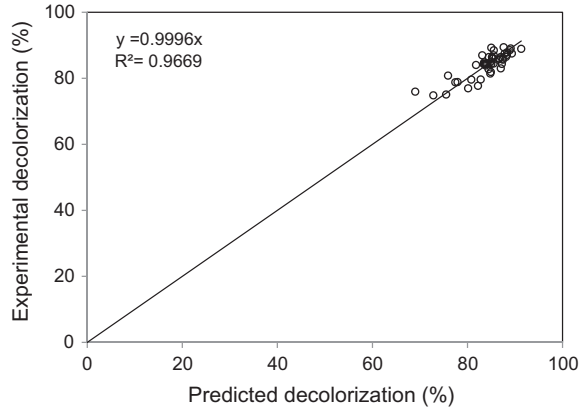

Fig. 4. Predicated value vs. actual value for the decolorization efficiency.

neutral pH. In optimal conditions, decolorization efficiency was estimated at about 88.23% at treatment time of 57.4 min and the distance of 1.9 cm. Predicted and statitical values of the models are presented in Table 4. Using optimum parameters and experimental conditions, decolorization efficiency was estimated at

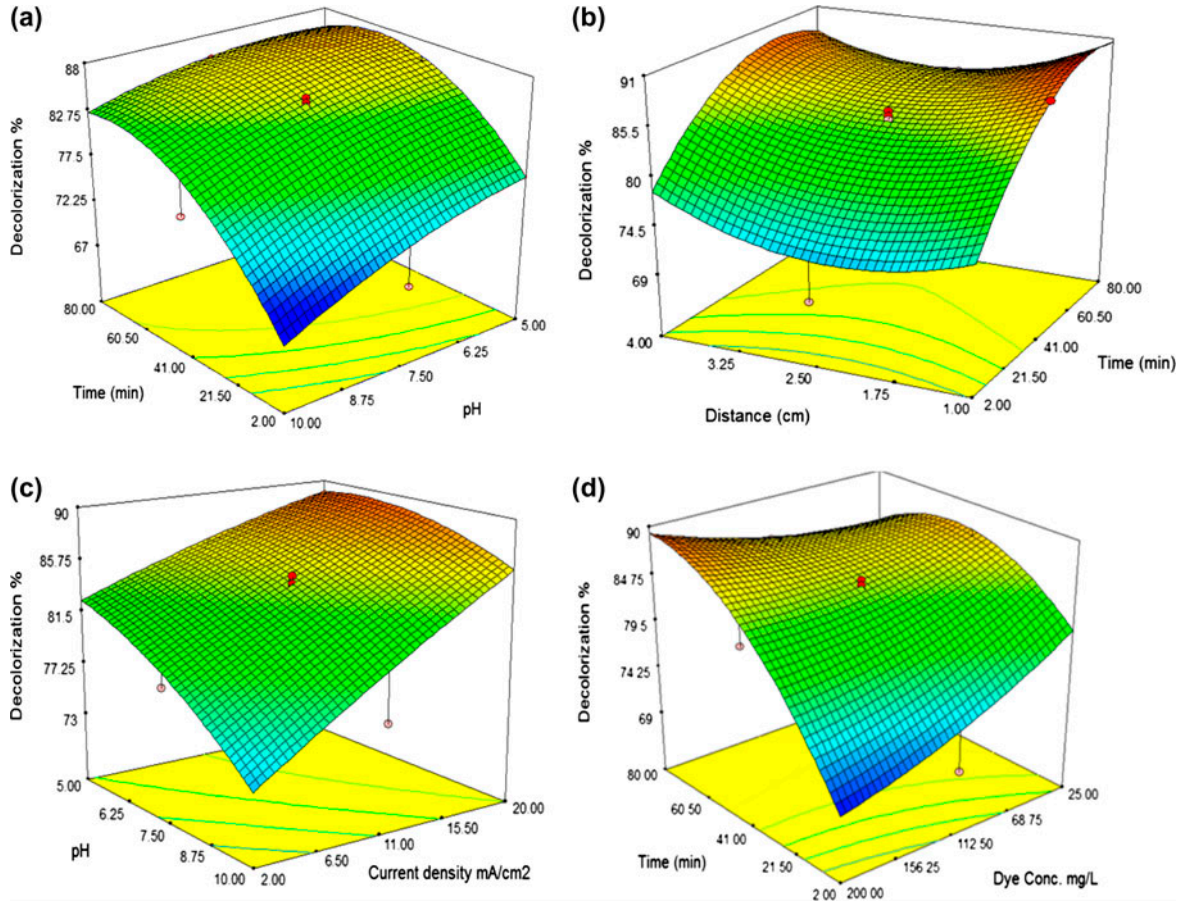

Fig. 5. (a, b, c, d) three dimension response surface plots for natural.

89.08%. Regarding to the model prediction and experimental efficiency in contrast to CI and PI values, the precision of resulting data and models is revealed.

3.4. SEM analyses

SEM of a portion of sludge from the decolorization process is shown in Fig. 6. With respect to sediment morphology, it was found that high levels of porosity and surface area are provided by nanopore and micropore spaces at amorphous structures. These figures range from 1 to 10 μm magnification, showing that a proper space for adsorption of dye and their degradation products is provided thanks to desirable pore sizes (up from 50 to 500 nm).

3.5. Applications and economic aspects

Moreno-Casillas et al. have reported the EC is an enigmatic technology, and despite than widely applications for over a century, the basic science and engineering behind EC design and modeling is still largely empirical and heuristic [25]. Holt et al. have documented a number of major deficiencies of electrocoagulation as following: (i) there is any systematic approach to electrocoagulation reactor design and operation, (ii) lack of published data on the

Table 4
Prediction and Experimental values with statistical Confidence (CI) and Prediction Interval (PI)

			95% CI		95% PI	
Response	Model prediction	Experimental	Low	High	Low	High
Decolorization%	88.23	89.08	81.57	95.05	79.63	96.99

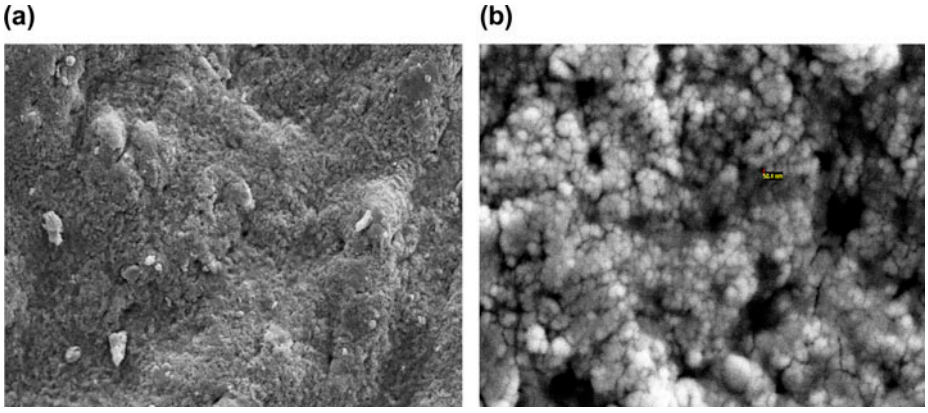

Fig. 6. SEM of the electrocoagulation sludge, 10 μm (a) and 1 μm (b).

operation of batch, and (iii) little available guidance for a priori reactor design or performance prediction [26].

Because of the large variability of the composition of textile wastewaters, most of these conventional methods are becoming inadequate and insufficient. However, the EC can be used as a simple and efficient method for the treatment of textile wastewaters. Nevertheless, because of high initial capital costs the EC process has not been widely accepted compared to other treatment technologies [27]. The treatment costs and electricity consumption of the EC process by Kuokkanen et al. were found to vary greatly depending on the type of solution being treated, being around 0.0047–6.74 /m^3 (treatment costs) and 0.002–58 kWh/m^3 (electricity consumption), but in general they were rather low (typically between 0.1 and 1 /m^3 and 0.4–4 kWh/m^3) [28].

4. Conclusions

In the present study, the electro-decolorization efficiency and operating parameters optimization for RR198 dye removal were evaluated. CCD and RSM were used to design the experiments and investigate the interactive effects of five effective parameters. Important parameters such as time, initial pH, dye concentration, applied current density, and distance effects were evaluated. Variance analysis showed a correlation coefficient (R^2) of about 0.77. p Value for decolorization model proved to be significant ($p < 0.0001$). Also, the following optimal operating conditions were obtained: RR198 200 mg/L, pH 6.45, current density 7.2 mA/cm^2, operating time 57.4 min, and distance 1.9 cm to achieve optimum conditions, decolorization efficiency was found to be 89.08%.

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