



# Alkali-clearing process optimization of the newly synthesized disperse dye and its promising removal from wastewater using electrocoagulation

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#### ABSTRACT

This paper deals with the optimization of alkali-clearing process of a novel alkali-clearable disperse dye and the investigation of its removal from the dyeing wastewater. The response surface methodology was used for the optimization of alkali-clearing process of the dyed polyester fibers. The results have clearly shown that the synthesized disperse dye has reasonable level of hydrolysis under relatively mild alkaline condition. It was also found that the adsorbed dye molecules would be removed from the fibers using a simple alkaliclearing process rather than the conventional methods being employed. In order to study the environmental aspects of the synthesized disperse dye, its removal process was investigated by the electrocoagulation method using iron as electrodes. The effect of operating parameters such as current density, conductivity, initial dye concentration, and pH on the removal efficiency were examined. Moreover, the results showed that the electrocoagulation process with low cost and reasonable efficiency is a promising method for the removal of the newly synthesized dye from wastewater.

*Keywords:* Alkali-clearable disperse dye; Polyester fiber; Response surface methodology; Dye removal; Electrocoagulation

#### 1. Introduction

The limited solubility of disperse dyes in water may cause some particulate of their molecules to be occluded onto the surface of fibers after the dyeing process is completed. If these adsorbed disperse dyes are not removed from the surface of the fibers, their contamination can undermine the brightness of shade as well as the wash, sublimation, and rubbing fastness results. As a conventional treatment method, the reduction clearing is applied where the dyed fibers are treated in a strong reduction bath usually containing sodium hydrosulphite and caustic soda. On the other hand, the majority of disperse dyes are azo dyes due to the large number of molecular combinations generating by the variation of diazo and coupling components, their relatively simple manufacturing

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processes and, finally, their wide color gamut of high color strength. Therefore, when the conventional reduction clearing process is applied to these disperse dyes, which are mostly used in textile industry for fiber dyeing processes, the azo linkage is broken and, as the case may be, carcinogenic amines are liberated into the effluents which have high biochemical oxygen demand (BOD) values due to the presence of sodium hydrosulfite [1–3].

Textile industry is considered as one of the biggest consumers of water and complex chemicals during various processing stages. The unused materials especially dyes are discharged into wastewater which is high in color, BOD, chemical oxygen demand (COD), pH, temperature, turbidity, and toxic chemicals. The direct discharge of the wastewater into the water bodies like lakes, rivers, etc. pollutes the water and affects the flora and fauna, and if it is discharged into the sewage networks, it will disturb the biological treatment processes [4]. The typical characteristics of textile wastewater generally include a wide range of pH, COD, total suspended solids, dissolved solids, heavy metals, and strong color [5], which may be comparable to moderate municipal wastewater [6]. The major characteristics of the real textile wastewater are presented in Table 1. It is observed that the textile wastewaters exhibit a wide range of pH from 2 to 14, COD from 50 mg/L to approximately 18,000 mg/L, TDS from 50 to over 6,000 mg/L with a very strong color. This wide variation in the characteristics of textile wastewater is due to the complexity of materials used in this industry during the processing of textiles which should be either eliminated from the wastewater before they are released into the environment or be replaced with compounds which do not require the complicated equipment for their treatments. However, the main challenge is to eliminate the color of wastewater, which is due to the remaining dyes.

Dve molecules are comprised of two key components: the chromophores that are responsible for producing the color and the auxochromes, which not only supplement the chromophore but also render the solubility of molecule in water and enhance the affinity of dyes toward the fibers. In our previous study [1], a novel disperse dye containing an azo linkage as the chromophore and a carboxylic acid of ester groups as the auxochrome was synthesized and characterized. The conventional reduction clearing method was successfully replaced by a simple alkali clearing bath to remove the adsorbed disperse dyes from the surface of the polyester fibers. These so-called alkali-clearable disperse dyes obviated the need for sodium hydrosulfite and reduced the cost of effluent treatment, significantly [24]. Furthermore, it is assumed that minimizing the need for reduction clearing can lead to the improvements in substantial productivity, water, and chemical savings as well as the reduction of the effluent load, which are the key

Table 1	l					
Major	characteristics	of real textil	e wastewaters	studied by	various rese	archers

pН	COD (mg/L)	$BOD_5 (mg/L)$	TSS (mg/L)	TDS (mg/L)	Color	Turbidity (NTU)	References
8.8–9.4	$595 \pm 131$	$379 \pm 110$	$276 \pm 76$	_	_	-	[5]
11.2	2,276	660 <sup>a</sup>	_	47.9	-	-	[7]
5–10	1,100-4,600	110-180	_	50	1,450–1,475 (ADMI)	-	[8]
6.5-8.5	550-1,000	-	100-400	-	7.50–25.50 <sup>b</sup>	15-200	[9]
2.7	7,000	-	440	930	-	2,140	[10]
13.56	2,968	-	_	_	3,586 (C.U)	120	[11]
12–14	1,500-2000	-	-	-	Dark blue	-	[12]
10	1,150	170	150	-	1.24 (436 nm)	-	[13]
9	750	160	_	_	-	-	[14]
2-10	50-5,000	200-300	50-500	_	>300 (C.U)	-	[15]
8.32-9.50	278–736	137	85-354	1,715–6,106	-	-	[16]
$8.7 \pm 0.2$	$17,900 \pm 100$	$5,500 \pm 100$	$23,900 \pm 50$	$1,200 \pm 50$	-	-	[17]
9.30	3,900	-	-	-	-	240	[18]
7.8	$810 \pm 50.4$	$188 \pm 15.2$	$64 \pm 8.5$	_	0.15 (669 nm)	-	[19]
$13 \pm 1$	$2,300 \pm 400$	-	$300 \pm 100$	_	-	-	[20]
6.95	3,422	-	1,112	-	-	5,700	[21]
7.86	340	210	300	-	>200 (Pt-Co)	130	[22]
$7.5 \pm 0.3$	$131 \pm 18$	_	$75 \pm 13$	$1,885 \pm 80$	_	_	[23]

<sup>a</sup>BOD<sub>7</sub> and effluent is from reactive dye bath.

<sup>b</sup>Integral of the absorbance curve in the whole visible range (400–800 nm), ADMI: American dye manufacturer institute, C.U: Color unit.

objectives in the implementation of a Rapid Dyeing approach [1]. Therefore, in general, the replacement of reduction clearing method with alkali-clearing process can reduce the COD, BOD, and water pollution as well as the expenditure of production.

In order to investigate the capability of the synthesized disperse dye to be extracted from the effluents, the electrocoagulation (EC) process was selected as one the most efficient treatment processes. EC has proven to be very effective in the removal of color and organic matters as well as heavy metals, oil, and grease from industrial wastewaters [25-28]. EC provides a simple and efficient method where the flocculating agent is generated in situ by electro-oxidation of a sacrificial anode, so there is no need for additional chemicals and/or secondary pollutions. It also reduces the amount of sludge that needs to be disposed [29]. EC technique uses a direct current source between metal electrodes immersed in the polluted water and generates the coagulant species in situ as the sacrificial metal anode dissolves while hydrogen is simultaneously evolved at the cathode. The produced hydrogen gas will also facilitate the floatation of the flocculated particles [30-32].

It is generally accepted that the EC process involves three successive stages: (a) formation of coagulants by electrolytic oxidation of the "sacrificial electrode"; (b) destabilization of the contaminants, particulate suspension; and (c) aggregation of the destabilized phases to form the flocs, and break the emulsions [33].

When an iron anode is utilized in EC,  $Fe^{2+}$  is dissolved in the wastewater from Fe oxidation at the anode [34] (Eq. (1)):

$$Fe_{(s)} \rightarrow Fe^{2+} + 2e^{-} \tag{1}$$

whereas hydroxide ion and  $H_2$  gas are generated at the cathode from the cathodic reaction (Eq. (2)):

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

OH<sup>-</sup> production from reaction (2) increases the pH during the electrolysis. Insoluble  $Fe(OH)_2$  precipitates at pH > 5.5 and remains in equilibrium with  $Fe^{2+}$  up to pH 9.5 or with monomeric species such as  $Fe(OH)^+$ , Fe (OH)<sub>2</sub>, and  $Fe(OH)_3^-$  at higher pH values. The formation of insoluble  $Fe(OH)_2$  can be written as Eq. (3) [35]:

$$\mathrm{Fe}^{2+} + 2\mathrm{OH}^{-} \to \mathrm{Fe}(\mathrm{OH})_{2(s)} \tag{3}$$

In the presence of  $O_2$ , the dissolved  $Fe^{2+}$  is oxidized to insoluble  $Fe(OH)_3$  (Eq. (4)):

$$4Fe^{2+} + 10H_2O + O_{2(g)} \rightarrow 4Fe(OH)_{3(s)} + 8H^+ \tag{4}$$

Protons can be directly reduced to  $H_2$  gas at the cathode (Eq. (5)):

$$8H^+ + 8e^- \to 4H_{2(g)}$$
 (5)

The corresponding overall reaction obtained by combining the Eqs. (1), (4) and (5) results in Eq. (6):

$$Fe + 2H_2O \rightarrow Fe(OH)_{3(s)} + H_{2(g)}$$
(6)

Once the insoluble flocs of  $Fe(OH)_3$  are produced, they can remove the dissolved dyes by surface complexation or electrostatic attraction. The first mechanism considers that dye can act as a ligand to bind to a hydrous iron moiety of the floc yielding a surface complex (Eq. (7)):

$$Dye - H + (OH) \cdot OFe_{(s)} \rightarrow Dye - OFe_{(s)} + H_2O$$
(7)

The second one supposes that  $Fe(OH)_3$  flocs with surface complexes contain areas of apparent positive or negative charges that attract the opposite regions of the dyestuff. The coagulation of these flocs forms particles that are separated from the wastewater by sedimentation or electroflotation [35].

The aim of this research is to optimize the alkali clearing condition of the dyed polyester fabrics using the high temperature dyeing method with the newly synthesized alkali-clearable disperse dye. The optimization of the process variables (alkali clearing bath temperature, time, and pH) was performed by using the well-known statistical method: "Response surface methodology". In this regard, the three-factor and five-level central composite design (CCD) was employed. The alkali clearing was characterized by the optical density of the obtained wastewater. Moreover, the removal of the synthesized disperse dye from the colored wastewater was investigated using the EC process, and the effect of important parameters including pH, initial dye concentration, current density, and salt concentration on the removal efficiency was also studied.

#### 2. Experimental

#### 2.1. Materials and methods

Polyester fabric  $(190 \text{ g/m}^2)$  having twill weave, warp: 136 threads/inch, weft: 72 threads/inch, with the thickness of 0.54 mm was chosen for the present

study. Nonionic detergent (SDL Technologies, UK) was applied to scour the polyester fabrics in order to pretreat them for dyeing process. Sodium hydroxide, monosodium phosphate, disodium phosphate, and trisodium phosphate (Aldrich Chemical) were used to prepare the buffer solution for alkali-clearing process. All other chemicals used elsewhere were also Aldrich Chemical HPLC grade.

Dyeing of polyester fabrics was performed in a laboratory model glycerin bath of metallic beaker-dyeing machine (Nasaj Sanat Yazd). Visible absorption spectra of wastewater obtained from alkali-clearing and the EC process were recorded using a Cecil 9200 double beam UV–visible spectrophotometer.

# 2.2. Synthesis of dye and preparation of dispersion

p-Amino-benzoic acid (2.6 g) was slowly added to a solution containing hydrochloric acid (2 mL) and water (6 mL. The reaction temperature was increased to 75°C and the solution was cooled down to 5°C. Then, hydrochloric acid (3 mL) and ice (10 g) were added to the solution. Sodium nitrite (1.44 g) was dissolved in 2.8 mL of water and this solution was gradually added over 25 min to the reaction. On the other hand,  $\beta$ -naphthyl acetate (3.39 g) was dissolved in sodium hydroxide (10%) and the resulting clear diazonium salt solution was immediately used in the designated coupling component and stirred for 2 h at temperatures lower than 10°C. The pH value was adjusted between 4 and 5 by the addition of sodium acetate solution, and was maintained in this range until the end of the reaction. The solution was stirred for 4 h, at the same temperature, and the resulting dye was filtered and washed with hot water, and finally dried. The crude product was purified using the thinlayer chromatography method on silica gel (solvent: Chloroform).

In order to prepare the dye dispersion, dispersing agent (1 g) (Lyoprint EV, Ciba-Geigy), dye (0.5 g) and water (2 mL) were added to a mortar and milled for 60 min; the ensuing mixture was diluted with 20 mL water, transferred to a ball mill and milled for 20 h. The volume of the dispersion was adjusted to 100 mL and filtered through a 5 mm Micro-Prazisions Sieb Fritsch.

#### 2.3. Dyeing and alkali-clearing process

Polyester fabric (1 g) was pretreated with 5 g/L nonionic detergent at 80°C for 20 min (liquor ratio of 50:1), after which the substrate was rinsed and dried. Dyeing was carried out using the liquor ratio of 50:1,

pH 4–5 (acetic acid) at 0.1, 0.3, 0.5, 0.7, 1, 1.5, 2, and 4% (o.w.f.: on the weight of fabric), according to our previous work [1]. At the end of the dyeing process, the excess dyes were removed by subjecting the fabrics to the alkali-clearing process. Alkali clearing was undertaken using a 50:1 liquor ratio at the CCD experimental conditions. The alkali cleared samples were rinsed and dried in room temperature.

#### 2.4. Dye removal process

The electrocoagulation reactor consisted of a double-walled cylindrical Pyrex cell with 0.4 L capacity of aqueous solution. Water was circulated in the external jacket to keep the temperature constant at  $25 \pm 1$  °C. Two iron electrodes (with 98% purity) with the dimensions of  $1 \times 20 \times 50 \text{ mm}^3$  were used as anode and cathode for the experiments. The distance between the electrodes was set at 10 mm in all experiments. The aeration header was placed at the center bottom of the cell for mixing the solution. A digital DC power supply (Dual-Tracking DC Power Supply, JPS-302D) providing 0-30 V (0-6 A) was used to control the current density. Sodium chloride salt (Merck, 99.9% purity) was used to adjust the initial solution conductivities. The pH of the dye solutions was adjusted by the addition of HNO<sub>3</sub> (1 M) or NaOH (1 M). The removal process started when the current density was adjusted to the desired value. During the experiments, samples were collected at different time intervals, filtered, and analyzed for dye removal. Before each run, the electrodes were dipped in HCl solution to remove grease and impurities on the iron electrode surface, then they were abraded with sand paper and put in oven (110°C) for 30 min. Dye removal efficiency (%) was determined according to Eq. (8):

Dye removal efficiency (DR%) = 
$$[(A_0 - A)/A_0] \times 100$$
(8)

where  $A_0$  and A are the absorbances of dye solution before and after the electrocoagulation at  $\lambda_{max} =$  512 nm, respectively.

#### 2.5. Experimental design

CCD is a statistical method based on the multivariate nonlinear model, which is widely used for the optimization of process variables, for the determination of the regression model equations and operating conditions from the appropriate experiments [36–38]. It is also useful to study the interactions of the various parameters affecting the process. A three-factor, five-level CCD was applied in the present study to determine the optimum condition for the alkali-clearing process (Table 2). The CCD was also used for fitting a second-order model which requires only a minimum number of experiments for modeling [39,40].

## 3. Results and discussion

## 3.1. Alkali clearing optimization

Basically the optimization process involves three major steps: (1) performing the statistically designed experiments, (2) estimating the coefficients in a mathematical model, and (3) predicting the response and checking the adequacy of the model [41,42]. The non-linear computer generating quadratic model given below (Eq. (9)) was used for the analysis of data.

$$y = b_0 + \sum_i b_i x_i + \sum_i b_{ii} x_i^2 + \sum_j \sum_{k(k>j)} b_{jk} x_j x_k$$
(9)

where y is the response function, b is the regression coefficient of the model, and x is the coded level of an independent variable. A total number of 20 dyed polyester samples were alkali cleared according to the conditions given in Table 3. The experimental and predicted optical densities of the alkali clearing solution (OD) are also presented in this Table.

The coefficients of the response function (Eq. (9)) and the estimated *P* values of parameters for OD are illustrated in Table 4. The *P* values for all independent parameters indicate that the three selected factors and  $X_1^2$  and  $X_2^2$  terms are significant ( $P \le 0.05$ ), but the interaction terms are insignificant to the response. Therefore, the response function for OD in terms of the coded factors is given by Eq. (10):

$$Y = 0.242398 - 0.029172x_1 + 0.225996x_2 + 0.026936x_3 - 0.02224x_1^2 - 0.84184x_2^2$$

Table 2
Independent variables in CCD and their values

	Level used							
Independent Variables	Lowest (-1.68)	Low (-1)	Medium (0)	High (+1)	Highest (+1.68)			
pH $(x_1)$	8.32	9	10	11	11.68			
Temperature (°C) ( $x_2$ )	56.8	70	80	90	96.8			
Time (min) $(x_3)$	6.6	10	15	20	23.4			

Table 3
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CCD matrix for alkali clearing of dyed polyester

	рH	Temperature	Time	OD	
Run Order	$(X_1)$	(X <sub>2</sub> , ℃)	(X <sub>3</sub> , min)	Exp.	Pred.
1	9	90	10	0.510	0.467
2	10	100	15	0.829	0.860
3	9	70	20	0.122	0.086
4	11	90	20	0.612	0.608
5	10	80	15	0.244	0.242
6	9	90	20	0.582	0.546
7	10	80	5	0.196	0.229
8	10	80	15	0.250	0.242
9	10	80	15	0.241	0.242
10	10	60	15	0.076	0.100
11	11	70	10	0.104	0.099
12	10	80	15	0.237	0.242
13	10	80	15	0.254	0.242
14	10	80	15	0.240	0.242
15	10	80	25	0.297	0.319
16	11	70	20	0.125	0.127
17	11	90	10	0.548	0.543
18	8	80	15	0.061	0.130
19	12	80	15	0.242	0.228
20	9	70	10	0.081	0.044

Table 4

Estimated regression coefficients for the obtained OD from alkali-clearing process

Terms	Coef.	SE Coef	T	Р
Constant	0.242398	0.015192	15.956	0
$X_1$	0.029172	0.01008	2.894	0.016
$X_2$	0.225996	0.01008	22.421	0
$X_3$	0.026936	0.01008	2.672	0.023
$X_{1}^{2}$	-0.02224	0.009812	-2.266	0.047
$X_2^{\frac{1}{2}}$	0.084184	0.009812	8.58	0
$X_3^{\overline{2}}$	0.011352	0.009812	1.157	0.274
$X_1 \cdot X_2$	0.00525	0.01317	0.399	0.699
$X_1 \cdot X_3$	-0.0035	0.01317	-0.266	0.796
$X_2 \cdot X_3$	0.00925	0.01317	0.702	0.498

According to Table 5, the *P* value ( $P \le 0.05$ ) justifies the reliability of the fitted polynomial model with confidence level of 95%. The coefficient of determination of the model and the adjusted coefficient of determination (adj.  $R^2$ ) appear to confirm the accuracy and variability of the model; so, the high  $R^2$  values also validate the statistical significance of the model for OD [43].

The ester groups of the disperse dye can be hydrolyzed to the corresponding acid by nucleophilic bimolecular substitution (S<sub>N</sub>2) mechanism under alkaline condition. Therefore, it is presumed that the azo disperse dyes containing a carboxylic acid ester group can be hydrolyzed under alkaline condition by S<sub>N</sub>2 mechanism shown in Fig. 1. This reaction involves the displacement of a leaving hydroxyl group, by a nucleophile, in which one bond is broken and another is formed synchronously. Since the addition of hydroxide ion to the electrophilic carboxylic acid ester group is considered to be the rate-determining step of the hydrolysis reaction, and the concentration of hydroxide ion is kept constant by the use of buffer solution, the reduction of dye concentration on fabric in the alkaline solution can be expressed by the absorption behavior of its wastewater [44]. Fig. 2 shows the hydrolysis behavior of the disperse dye at alkaline condition, in which the solubility of the synthesized disperse dye was calculated from the CCD experimental results. The resulted data from alkaliclearing process (Table 3) indicate that by increasing the solution temperature over 80°C, the obtained OD is increased significantly. The possible explanation for such a behavior can be related to the undesired hydrolysis of dye molecules in the fibers at elevated temperatures above glass transition, in which the polyester fibers become soft [45]. It can be seen from the Fig. 2 that the highest hydrolysis percentage was achieved for the bath number 2 having the pH of 10, time of 15 min, and temperature of 100°C.

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ANOVA for the obtained (	DD	from a	lkali	-cl	earin	g	process
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Fig. 1. Alkali-hydrolysis mechanism of the synthesized disperse dye.

Fig. 3 indicates the 3D plots of the optical density of the remaining solution obtained from the polyester fabrics alkali-clearing process. According to Fig. 3, with the increase of pH, the hydrolysis of ester groups existing in the structure of disperse dye increases and higher amount of adsorbed dye molecules will be removed from the fabric surface, which results in higher values of OD. Fig. 3 also illustrates that applying high temperatures in alkali-clearing process will increase the OD values, which can be explained by the fact that high temperatures increase the surface of the polyester fabric exposed to the alkali solution and favors the endothermic hydrolysis reaction of ester groups [46].

		EC						
Source	DF	Seq SS	Adj SS	Adj MS	<i>F</i> -value	Р		
Regression	9	0.836286	0.836286	0.092921	66.97	0		
Linear	3	0.719042	0.719042	0.239681	172.74	0		
Square	3	0.116241	0.116241	0.038747	27.93	0		
Interaction	3	0.001003	0.001003	0.000334	0.24	0.866		
Residual error	10	0.013875	0.013875	0.001387				
Lack-of-fit	5	0.013875	0.013875	0.002775	*	*		
Pure error	5	0	0	0				
Total	19	0.850161						

Note:  $R^2 = 98.37\%$ ,  $R^2$  (adj) = 96.90%.

\*No Lack-of-fit.



Fig. 2. Hydrolysis (%) of synthesized disperse dye at different conditions listed in Table 3.

#### 3.2. Effect of operating parameters on electrocoagulation

Several operating parameters such as conductivity, current density, initial dye concentration, and pH affect the EC process. To improve the removal performance of the newly synthesized dye, the effect of these parameters was investigated.

#### 3.2.1. Current density

Former studies have shown that the current density strongly affects the performance of EC [47,48]. The effect of current density on the color removal was investigated at various current densities  $(10-120 \text{ A/m}^2)$  (Fig. 4).

As it can be seen, when the current density raises up to  $40 \text{ A/m}^2$ , the color removal efficiency increases to 90%; on the other hand, by increasing the current density up to  $120 \text{ A/m}^2$  , no significant difference at dye removal can be seen. Therefore, the optimum current density was chosen as  $40 \text{ A/m}^2$ . It appeared that at lower current densities, the less iron ions were released from the anode and hence, the color removal efficiency was low. When the current density increases, the amount of iron ions species formed by the dissolution of the anode increases according to Faraday's law (which relates the current flow to the amount of iron ions generated within the electrolytic cell) and a significant amount of flocs are generated, which in turn enhances the color removal efficiency. Moreover, the higher hydrogen production rates by higher currents favors the flotation of the flocculated



Fig. 3. 3D plots for the OD of samples after alkali-clearing process.



Fig. 4. The effect of current density at different time intervals (pH 6, NaCl: 2 g/L, and initial dye concentration: 100 mg/L).

matter. However, when high current densities are applied, cell voltage increases and there is a high chance of wasting the electrical energy in heating up the water [26,49].

# 3.2.2. pH

The effect of pH on the dye removal by EC was investigated by varying the initial pH from 3 to 9. Fig. 5 shows the effect of pH on the dye removal.

The results showed that the dye removal at pH 6 is more than other pH values, because the majority of electrogenerated  $Fe^{3+}$  forms  $Fe(OH)_3$  flocs that can rapidly remove the dye molecules via complexation or electrostatic attraction, followed by coagulation. At pH 3, soluble  $Fe^{3+}$  is the dominant species and  $Fe(OH)_3$  flocs are quite poorly produced, whereas at pH > 8, a part of  $Fe(OH)_3$  is solubilized as  $Fe(OH)_4^-$  and lower amount of dye can be separated [35].

# 3.2.3. Electrolyte concentration (conductivity)

Sodium chloride is usually employed to increase the conductivity of the water or wastewater to be treated. It is expected that in the presence of NaCl, the insulating layer on the surface of the electrodes decreases. This insulating layer can sharply increase the potential between the electrodes to result in a significant decrease in the current efficiency. Increasing the electrolyte concentration increases the solution conductivity, which leads to the decrease in power consumption [33,50]. Fig. 6 shows the effect of electrolyte concentration on the solution conductivity and cell voltage.

Fig. 7 illustrates the evolution of removal efficiency as a function of the electrolyte concentration using the optimum conditions obtained previously for current and pH. Based on the experimental results, it could be



Fig. 5. The effect of initial pH at different time intervals (current density:  $40 \text{ A/m}^2$ , NaCl: 2 g/L, and initial dye concentration: 100 mg/L).

concluded that electrolyte concentration did not show a considerable effect on the color removal efficiency. An excessive amount of NaCl induces an overconsumption of the iron electrodes and Fe dissolution may become irregular due to the "corrosion pitting" [51]. This is the reason why NaCl addition has been limited to 2 g/L.

## 3.2.4. Initial dye concentration

Fig. 8 illustrates the influence of initial dye concentration on dye removal efficiency in the range of 100–300 mg/L. It shows that after 10 min of EC, the dye removal efficiencies are 93, 80, 53, 43, and 36% at the initial dye concentration of 100, 150, 200, 250, and 300 mg/L, respectively.

Here, according to Faraday's law, the constant amount of  $Fe^{2+}$  is passed to the solution at the same current density and time for all dye concentrations; thereupon, the same amount of flocs are produced in the solution. As a result, the flocs produced at high dye concentrations were insufficient to remove the



Fig. 6. The effect of electrolyte concentration on conductivity and cell voltage (pH 6, current density:  $40 \text{ A/m}^2$ , and initial dye concentration: 100 mg/L).



Fig. 7. The effect of electrolyte concentration at different time intervals (pH 6, current density:  $40 \text{ A/m}^2$ , and initial dye concentration: 100 mg/L).



Fig. 8. The effect of initial dye concentration (mg/L) at different time intervals (current density:  $40 \text{ A/m}^2$ , NaCl: 2 g/L, and pH 6).

dye molecules completely. In other words, with an increase in the dye concentration, the rate of color removal decreased considerably [34].

## 4. Conclusions

In the present study, the newly synthesized alkaliclearable disperse dye was investigated from two points of view: firstly, the optimization of the alkaliclearing process and secondly, its removal from the colored wastewater using electrocoagulation process with iron electrodes. The Response surface methodology was employed as a proper statistical tool to optimize the alkali clearing condition. The results indicated that the synthesized dye was highly hydrolyzed in the alkali-clearing process at the optimized condition (pH 10, time = 15 min, and the temperature of 80°C). In order to approach the second purpose of the current study, the electrocoagulation process was selected as the treatment method, and the influence of the key parameters such as pH, initial dye concentration, current density, and electrolyte concentration was surveyed. It was found that increasing the current

density up to  $40 \text{ A/m}^2$  has increased the color removal up to 90%. The data indicated that increasing the conductivity decreased the cell voltage, but did not have a significant effect on color removal efficiency. The results also showed that by increasing the initial dye concentration, the dye removal efficiency decreased, and pH 6 was the optimum value to work with. The investigation of dye removal process indicated that the electrocoagulation process can be a viable, successful, and promising technique with high removal efficiency as well as low expenditure for the removal of the new disperses dye.

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