



ORIGINAL ARTICLE

Removal of dye from industrial wastewater with an emphasis on improving economic efficiency and degradation mechanism



Azam Pirkarami, Mohammad Ebrahim Olya *

Department of Environmental Research, Institute for Color Science and Technology, P.O. Box 16765-654, Tehran, Iran

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Abstract This paper is a report on a research into the impact of different parameters of current density, anode type, temperature, pH, and electrolyte concentration on the removal of Reactive Red 120 in synthesized wastewater through electrocoagulation using solar energy for the purpose of improving economic efficiency of the process. Current density of 45 Am^{-2} proved to be optimum level for dye removal. Other optimum alternatives were iron anode, a temperature level of $25 \text{ }^\circ\text{C}$, a pH of 7, and an electrolyte concentration of 15 mg L^{-1} . The characterization of the post-treatment product using GC–MS studies revealed intermediate compounds. Cost analysis was also performed for the treatment process. Further, the obtained optimum conditions were applied to the treatment of six samples of real textile effluent. Electrocoagulation was satisfactory in only four of the cases. Lastly, efficiency of treating the real samples was evaluated by subjecting the experimental electrodes to the SEM technique.

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1. Introduction

The dye stuff lost in the textile industry poses a major problem to wastewater sources [14,21]. Indeed, textile industry produces high levels of dye and floating solid materials [17]. It is esti-

mated that 5000 tons of dyeing materials are discharged into the environment every year. These poisonous materials absorb the oxygen of the water [15,20]. This has raised much as it threatens human life and the environment. Industrial wastewaters contain various kinds of toxic substances such as cyanides, alkaline cleaning agents, degreasing solvents, oil, fat, and metals [3]. Common ways of wastewater treatment include adsorption, sedimentation, chemical analysis, chemicoagulation, biological methods, and advanced oxidation procedures [3–5]. However, these approaches are not without their disadvantages. Biological methods, for example, take much time and cannot degrade complicated dyes [5]. In addition, some commercial dyes are harmful to some microorganisms [16]. Furthermore, absorbents are not reusable in general [5].

* Corresponding author. Tel.: +98 2122944184.

E-mail addresses: me.olya2@gmail.com, olya-me@icrc.ac.ir (M.E. Olya).

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Chemicoagulation even causes pollution due to the production of colloids in wastewater [3]. Common chemical oxidation like using chlorine is slow and needs reactive materials which are dangerous to transport and store [6]. Another problem is that these methods are not efficient enough because there are various materials in wastewater [4]. Advanced oxidation methods such as ozonation, photocatalyst, and photo fenton are costly and uneconomical [5]. Therefore, there is a need for more effective and cheaper ways of treating textile wastewater which consume the smallest amounts of chemicals and energy.

Research into wastewater treatment has focused on electrochemical coagulation over the past few years [1,4,9]. Electrocoagulation provides some significant advantages such as simple equipment, easy operation, small retention time, high velocities, reduced amount of sludge, and no chemical additives [11]. Electrocoagulation is an electrochemical technique in which particles generated by the anode combine to free active coagulation factors in the solution. Metal ions are generated in anodes, and hydrogen is produced in cathodes [8]. Hydrogen can help mass particles float and come out of water [22]. The resulting metal ions, *i.e.* $\text{Al}^{3+}(\text{aq})$ and $\text{Fe}^{3+}(\text{aq})$, undergo some reactions to produce hydroxide or polyhydroxide, which strongly absorb dispersed compound molecules and result in coagulation [10].

In the present research, synthesized wastewater and real textile effluents were subjected to the process of electrocoagulation in order to explore the impact of a number of operating parameters on the efficiency of removing a reactive dye. The parameters were current density, anode type, temperature, pH, and electrolyte concentration. It should be noted at this point that we used solar energy for the purpose of improving the economic efficiency of the process. Indeed, our review of the previous research showed us no instance of solar energy being used in electrocoagulation. Cost analysis was also performed for the treatment process. Further, in order to evaluate the efficiency of the electrocoagulation process, the experimental electrodes were characterized using the SEM technique. The post-treatment product was characterized using GC-MS techniques.

2. Experiment

2.1. Materials and equipment

The solution was prepared by pouring Reactive Red 120 obtained from the Iranian company of Alvan Sabet into doubly distilled water. To test the proposed electrocoagulation treatment, we used a sample of real textile wastewater obtained

from a local textile dyeing factory. This wastewater contained a mixture of dyes, iron, and zinc. The properties of the real wastewater are presented in Table 1.

NaOH and HCl (used to adjust the pH of the wastewater) and NaCl were purchased from the German company of Merck. NaCl is minimally poisonous, reasonably priced, and highly conductive and soluble, and it has very little impact on pH. The solar cell was purchased from PTL Solar (Germany). In addition, the solar cell battery was obtained from Faran Electronic Industries (a manufacturer in Tehran, Iran). A rheostat controlled the electrical current provided by the battery. The electrochemical characteristics of the reactor used in the experiment can be seen in Table 2.

2.2. Procedure

The synthetic wastewater was prepared by dissolving 150 mg L^{-1} of dye into distilled water. This particular choice was made because it was the optimum concentration at which the reactor could maximally degrade the dye with regard to the operating conditions applied.

Five different levels of current density were applied ($15, 25, 35, 45, \text{ and } 75 \text{ Am}^{-2}$) in order to determine which level leads to the highest efficiency of color removal. Four anode types were compared in terms of their effect on the efficiency of dye removal and energy consumption: iron, aluminum, a combination of iron and aluminum, and titanium. To determine the effect of temperature on the removal of dye from the experimental wastewater, five degrees of temperature were considered: $15, 25, 35, 45, \text{ and } 55 \text{ }^\circ\text{C}$. To accomplish this, the reactor was placed in a water bath. The effect of pH was studied by setting the pH of the wastewater at ten values: acidic (pH 2–6), neutral (pH 7), and alkaline (pH 9–11). The pH was adjusted with NaOH and HCl. The influence of salinity was studied using NaCl at four concentrations: $5, 1, \text{ and } 15 \text{ mg L}^{-1}$. Before each run of the experiment, the electrodes were first washed thoroughly with tap water, dipped in HCl solution for at least 15 min, and then washed with pure water. One liter of dye solution was poured into the reactor. The electrodes were immersed in the solution and connected to the DC power supply. The treatment time was for 15 min. To ensure uniformity, the solution was magnetically stirred at 200 rpm during electrocoagulation. Samples were taken from the solution every two minutes and were put inside a UV-Vis spectrophotometer cell in order to measure the maximum absorption of wavelength for the dye. An absorbance value at 530 nm was used to measure dye concentrations. The dye removal efficiency, R%, was expressed as a percentage as follows (Eq. (1)):

$$(\%) R = \frac{C - C_0}{C_0} \times 100 \quad (1)$$

where C_0 and C are the initial and final dye concentration, respectively. The amount of energy consumed by the process of electrocoagulation, denoted by EEC and expressed in kWh/m^3 wastewater, was calculated through Eq. (2) [18]:

$$\text{EEC} = \frac{(U \cdot I \cdot t)}{v} \quad (2)$$

where U is the operating voltage (volt), I is the operating current (in amperes), t is the length of reaction (in seconds), and v

Table 1 Properties of the real wastewater used.

Property	Range
pH	8.27–9.71
Current density Am^{-2}	10–75
COD (mg dm^{-3})	267–714
BOD ₅ (mg dm^{-3})	143
TSS (mg dm^{-3})	73–328
TS (mg dm^{-3})	1797–5709
Fe (mg dm^{-3})	0.31–1.43
Zn (mg dm^{-3})	0.01–0.16

Table 2 Properties of the electrochemical reactor used.

Property	Description
Dimensions (mm)	70 × 80 × 290
Volume (L)	1
Material	Glass
Anode	Iron (Fe), aluminum (Al), titanium (Ti)
Cathode	Titanium (Ti)
Electrode thickness (mm)	2
Electrode size (mm)	30 × 180
Electrode arrangement	Parallel
Electrode gap (mm)	10
Current range (A)	0–18
Voltage range (V)	0–75

is the volume of wastewater (m^3). The process of electrocoagulation was also performed on samples of real textile effluent.

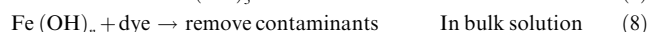
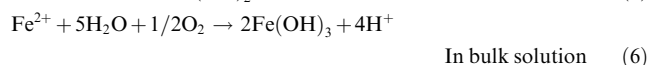
3. Results and discussion

3.1. The effect of current density on the removal of Reactive Red 120

The strong ability of iron and aluminum as anodes to remove contaminants may be explained as follows: during electrocoagulation, metal hydroxides are formed, Fig. 1. These flocs have a large surface area and as a result can rapidly adsorb dye polymers and trap colloidal particles. These flocs can easily be removed from the aqueous solution by means of sedimentation or flotation. The monomeric and polymeric hydroxyl complexes can remove contaminants thanks to their high oxidative potentials [13]. In addition, if the iron and aluminum potential is sufficiently high, other reactions such as direct oxidation of organic compounds may take place at the anode [7].

The polynuclear hydrolytic species of Al and Fe, when produced in large numbers, are very good coagulants for forming flocs. Hydrolysis of Al and Fe ions leads to the formation of large networks (e.g. Al–O–Al–OH) capable of chemically absorbing contaminants [10].

It is also worthy of note at this point that the formation of metal hydroxides results in electrode mass loss [2]. The following mechanism [23] has been proposed for the production of metal hydroxides when iron electrode is used as anodes:



A similar mechanism [12] has been proposed for the case of aluminum anode:

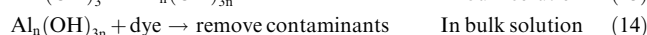
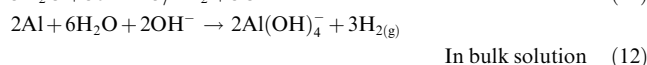


Fig. 1a depicts the efficiency of removing Reactive Red 120 using the electrochemical reactor with iron and titanium as anode and cathode, respectively. At the current densities of 35, 45, and 75 A m^{-2} , a high efficiency of dye removal was obtained after 3 min of electrocoagulation. Dye removal reached higher than 97% after 5 min. With the current density of 25 A m^{-2} , this amount of dye removal was achieved at a longer time (13 min). At 15 A m^{-2} current density, dye removal remained below 75% at all electrocoagulation times. The experiment was rerun with aluminum used as anode. As Fig. 1b shows, aluminum resulted in a similar, but to some extent lower, dye removal. This was especially so at an electrocoagulation time of less than one minute.

3.1.2. The effect of anode type on the removal of Reactive Red 120

Fig. 2a compares the four anode types of iron, aluminum, iron-aluminum combination, and titanium in terms of the magnitude of dye removal. In this run of the experiment, a

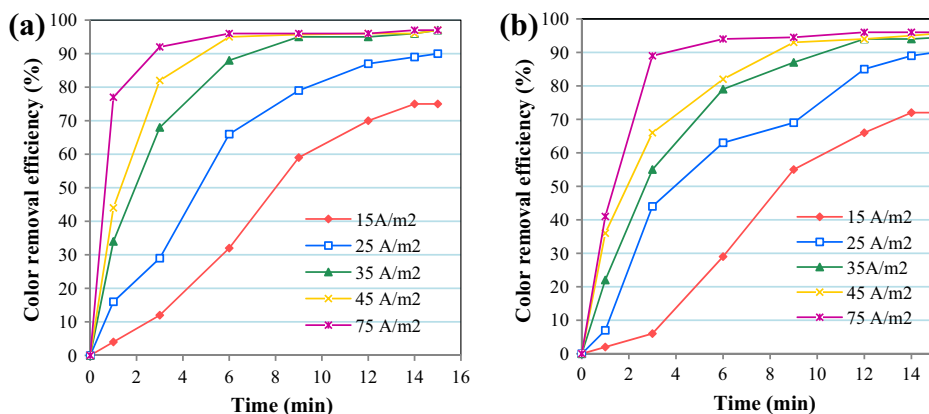


Figure 1 The removal efficiency of Reactive Red 120 as a function of current density and electrocoagulation time: (a) iron anode and (b) aluminum anode (distance between electrodes: 10 mm, electrode surface area: 30 × 180 mm, temperature: 25 °C, pH: 7).

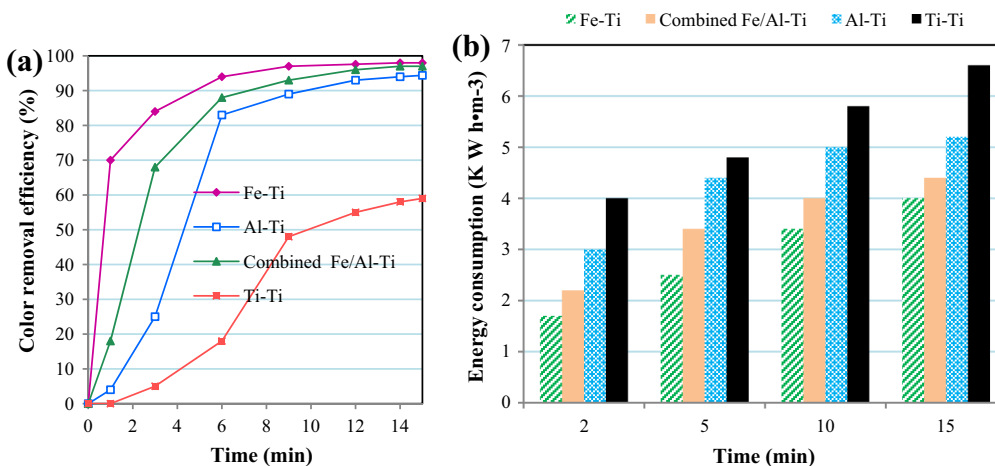


Figure 2 (a) The removal efficiency of Reactive Red 120 as a function of anode type and electrocoagulation time, (b) Energy consumption for the treatment of dye as a function of anode type and electrocoagulation time (current density: 75 Am^{-2} , distance between electrodes: 10 mm, electrode surface area: $30 \times 180 \text{ mm}$, temperature: $25 \text{ }^\circ\text{C}$, pH: 7).

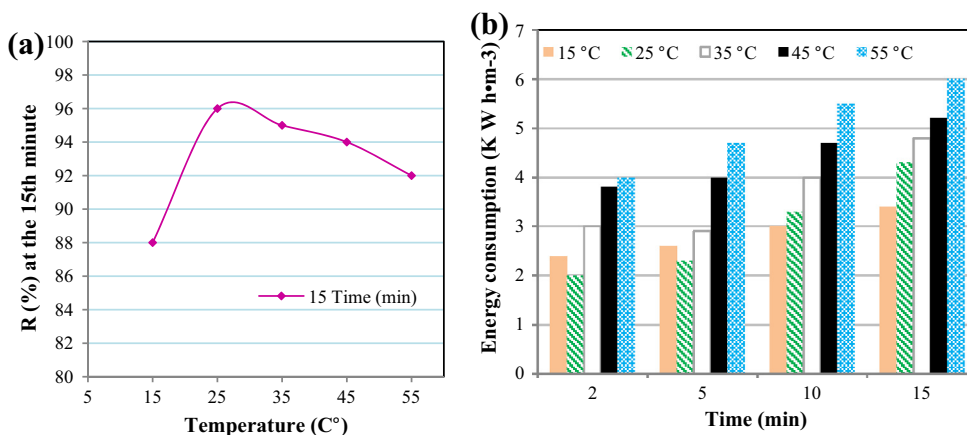


Figure 3 (a) The removal efficiency of reactive Red 120 as a function of temperature at a current density 75 Am^{-2} with an anode iron, (b) Energy consumption, (current density: 75 Am^{-2} , anode type: iron, distance between electrodes: 10 mm, electrode surface area: $30 \times 180 \text{ mm}$ temperature: $25 \text{ }^\circ\text{C}$, pH: 7).

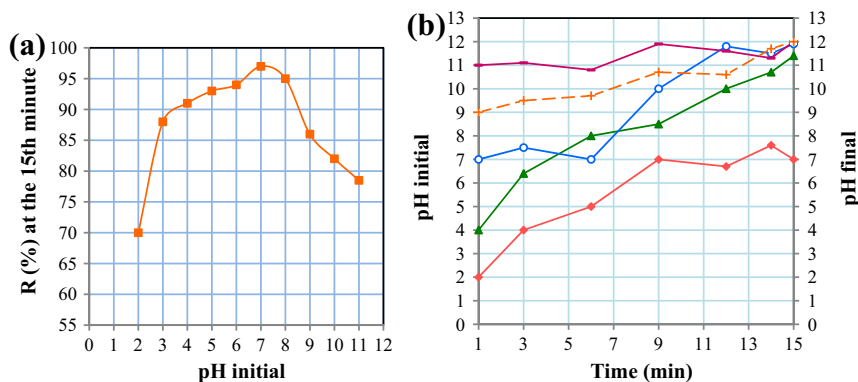


Figure 4 (a) The influence of initial pH on the removal efficiency of Reactive Red 120 by electrocoagulation, (b) The evolution of pH with time in removing Reactive Red 120 by electrocoagulation (current density: 75 Am^{-2} , anode type: iron, distance between electrodes: 10 mm, electrode surface area: $30 \times 180 \text{ mm}$, temperature: $25 \text{ }^\circ\text{C}$).

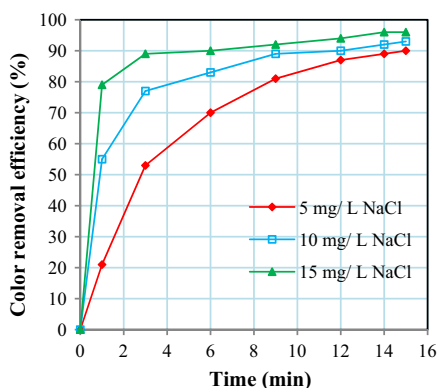


Figure 5 The effect of electrolyte concentration on the removal efficiency of Reactive Red 120 by electrocoagulation (current density: 75 Am^{-2} , anode type: iron, distance between electrodes: 10 mm, electrode surface area: $30 \times 180 \text{ mm}$, temperature: 25°C , pH: 7).

current density of 75 Am^{-2} was applied, and the cathode was titanium. The highest removal efficiency was obtained in the case of iron: around 90% after 5 min and 96% after 15 min of electrocoagulation. The lowest degree of dye removal was obtained when titanium anode was used: an efficiency of 59% after 15 min. This can be explained by the fact that metal hydroxide was not formed.

Fig. 2b presents a comparison of the four anode types in terms of the amount of energy they consumed in the process

of removing Reactive Red 120 from the aqueous solution. A current density of 75 Am^{-2} was applied. As can be observed, the iron anode required the lowest level of energy, ranging $1.7\text{--}4 \text{ kWh/m}^3$ of wastewater. Also, titanium consumed the largest amount of energy: $4\text{--}6.6 \text{ kWh/m}^3$ of wastewater. An explanation is that when titanium anode is used, metal hydroxide is not formed, and this increases electrical resistance, resulting in greater energy consumption.

3.1.3. The effect of temperature on the removal of Reactive Red 120

Fig. 3a displays the impact of five levels of temperature on the efficiency of dye removal (namely $15, 25, 35, 45,$ and 55°C) at a current density of 75 Am^{-2} with an anode iron. The best temperature for dye removal was 25°C . The difference between the temperature levels was minimal. As for energy consumption, 25°C proved to be the best level of temperature. Fig. 3b shows this. An explanation is that as the temperature increases, ion movement also increases, and this in turn reduces the possibility of metal hydroxide groups being formed. This fact increases energy consumption and reduces the efficiency of dye removal. The temperature level and removal efficiency were found to be inversely related. An explanation is that the rise in the temperature of the solution speeds up the chemical reactions. This leaves less time for the formation of metal hydroxides such as FeOH^{2+} , $\text{Fe}(\text{OH})_2^+$, $\text{Fe}(\text{OH})_2^{4+}$, $\text{Fe}(\text{OH})_4^-$, $\text{Al}(\text{OH})_2^+$, $\text{Al}(\text{OH})_4^-$, $\text{Al}_2(\text{OH})_4^{4+}$, and $\text{Al}_6(\text{OH})_3^{3+}$ [12,7]. This in turn reduces the potential of these flocs and decreases the removal of dye.

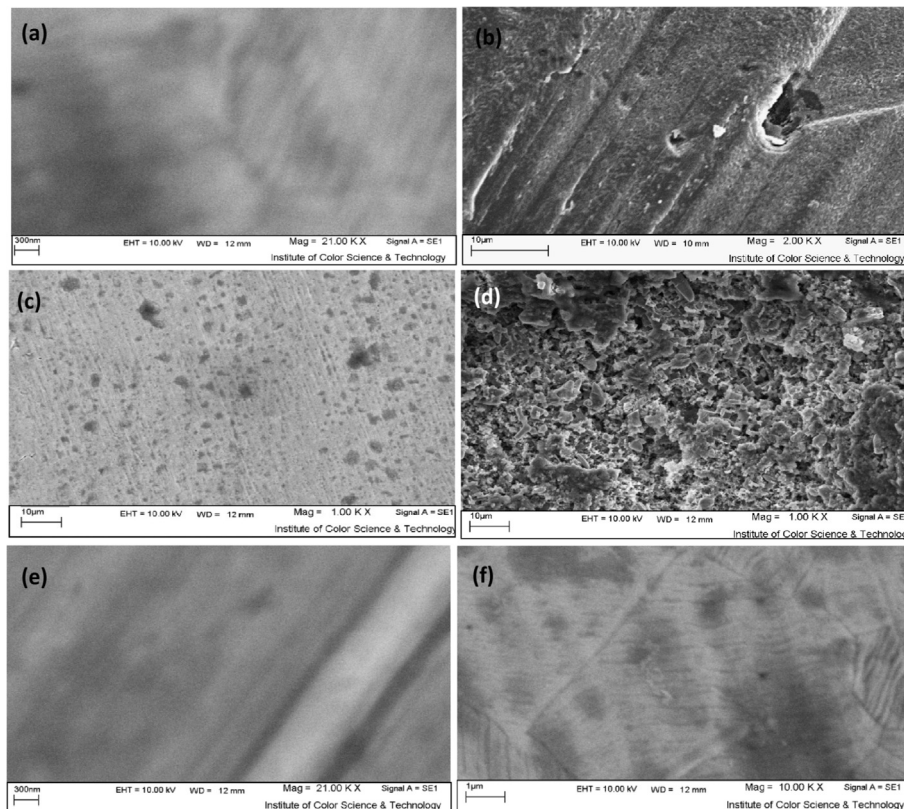


Figure 6 SEM images of experimental electrodes: (a) pre-treatment aluminum, (b) post-treatment aluminum, (c) pre-treatment iron, (d) post-treatment iron, (e) pre-treatment titanium, (f) post-treatment titanium.

3.1.4. The effect of pH on the removal of Reactive Red 120

Maximum dye removal was obtained when the solution had a pH of 7 (Fig. 4a). A possible explanation is that in acidic and neutral conditions, Fe(OH)^{+2} is formed, but in alkaline conditions, Fe(OH)^{+4} occurs [19]. Indeed, Fe(OH)^{+2} is more capable than Fe(OH)^{+4} of absorbing dyes. An increase in the pH of the aqueous solution was also observed here. Fig. 4b gives a schematic representation. The initial pH of 2 rose to 4 after 2 min and continued to increase, ultimately reaching 7 after 15 min. Interestingly, although the samples had different initial pH levels, they became similarly alkaline (a pH of 7–12) after 15 min of electrocoagulation.

3.1.5. The effect of salinity on the removal of Reactive Red 120

Fig. 5 portrays the effect of different concentrations of NaCl as electrolyte (5, 10, and 1.5 mg L^{-1}) on the removal of Reactive Red 120 from the synthesized wastewater through the process of electrocoagulation. The removal efficiency im-

proves as NaCl concentration increases. The concentration level of 15 mg L^{-1} again proved optimum. The explanation given above for the effect of salinity holds true in this case as well.

3.2. Electrode characterization

The efficiency of the treatment was evaluated by studying the surface characteristics of the experimental electrodes using a scanning electron microscope (SEM) unit (HITACHI-3000 SH Model, Japan). Fig. 6 shows the pre- and post-treatment surface characteristics of the three electrodes used in the experiment.

3.3. GC-MS analysis

To identify the intermediate products formed during the EC treatment of Reactive Red 120, GC-MS analysis was performed. From these results, a pathway can be proposed for the degrada-

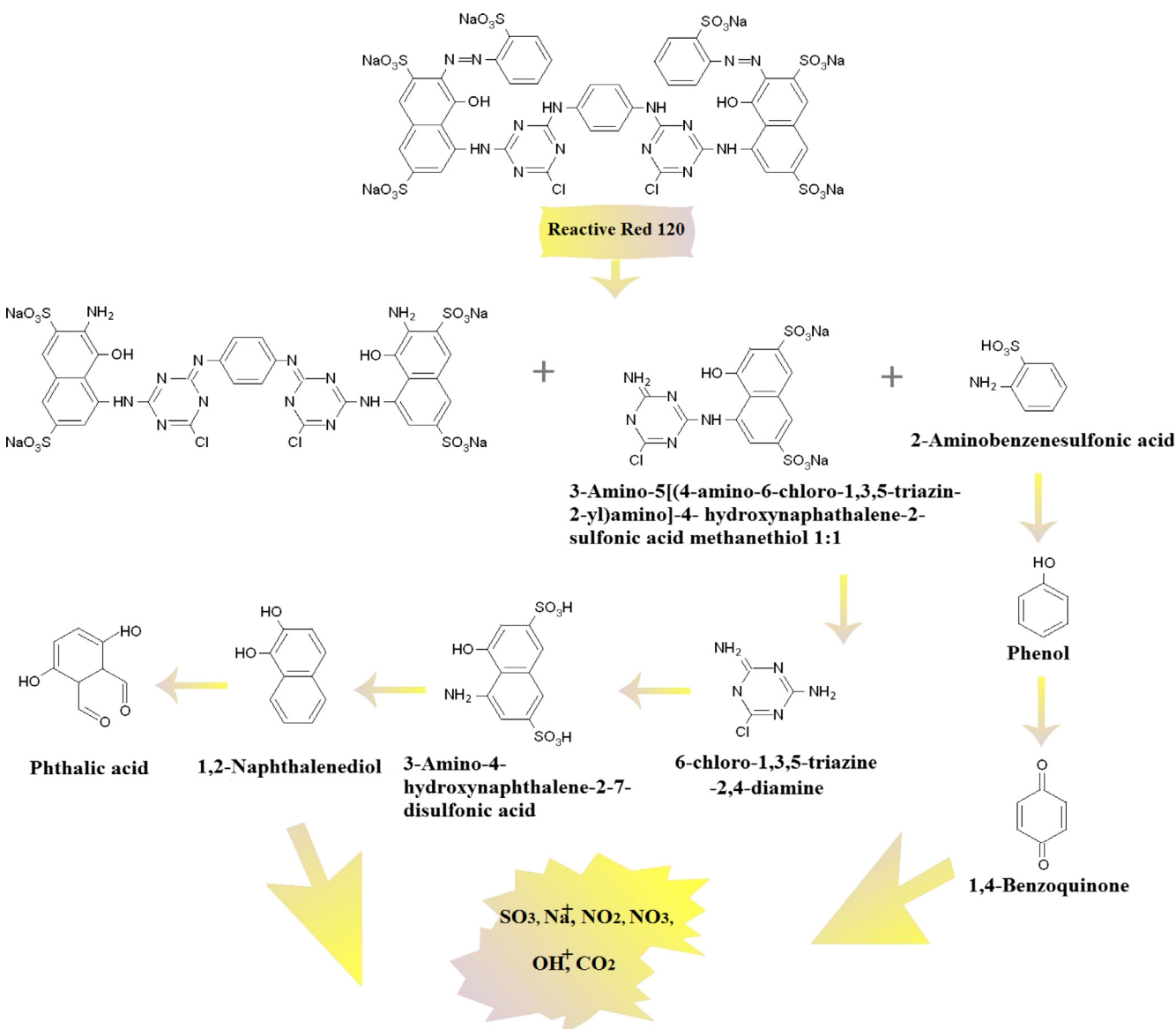


Figure 7 Proposed pathway of Reactive Red 120 degradation under EC.

Table 3 Properties of the real textile effluent before and after electrocoagulation.

Property	Expt. 1	Expt. 2	Expt. 3	Expt. 4	Expt. 5	Expt. 6
	Before/After	Before/After	Before/After	Before/After	Before/After	Before/After
Dye removal (%)	93.8	95.5	94.2	96.7	55.3	63.4
pH	9.5/9.6	8.4/9.3	8.3/9.7	9.1/8.5	9.3/9.5	8.9/9.5
Energy Consumption (kWhm ⁻³)	0.57	0.65	0.83	0.70	0.81	0.79
COD* (mg dm ⁻³)	428/70	439/55	389/222	683/132	766/377	440/89
TS** (mg dm ⁻³)	4054/891	4864/1032	5877/598	6695/462	2042/1261	1772/1823
TSS*** (mg dm ⁻³)	402/15	381/24	398/21	377/19	327/16	212/53
TSS*** (mg dm ⁻³)	1.10/1.02	0.69/0.32	1.66/0.29	1.73/0.17	0.66/1.14	1.71/0.97

* COD: chemical oxygen demand.

** TS: total solids.

*** TSS: total suspended solids.

tion of the dye (Fig. 7). To explain the process, the metal hydroxides attacked the carbon atom bearing the dye leakage, leading to cleavage of the C–N bond and the generation of 2-Aminobenzenesulfonic acid (m/z 27), and 3-Amino-5 [(4-amino-6-chloro-1,3,5-triazin-2-yl)amino]-4-hydroxynaphthalene-2-sulfonic acid methanethiol (m/z 465). The main transformation products were 3-amino-4-hydroxynaphthalene-2-7-disulfonic acid (m/z 320), 6-chloro-1,3,5-triazine-2,4-diamine (m/z 146), and 2-naphthalenol (m/z 144). If the EC process continues, these organic compounds will change into CO₂ and H₂O.

3.4. Economic analysis

Cost analysis was done for the treatment process. To this end, the costs of energy and electrolyte material were taken into consideration as major cost items. The operating cost was calculated using Eq. (3) below [12].

$$\text{Operating cost (USD/m}^3\text{)} = a\text{ENC} + b\text{CC} \quad (3)$$

where ENC is the energy consumption (kWh/m³) and CC is the chemical consumption (kg/m³). The letters *a* and *b* are unit prices for the Iranian market in December 2012, as follows: (a) electrical energy: 0.042 USD/kWh and (b) electrolyte (NaCl): 5.70 USD/kg. The cost of electrical energy was calculated using Eq. (2). In this study, treating 1 m³ of the synthesized aqueous solution containing both dyes under the optimum conditions obtained from the earlier runs of the experiment consumed 3.48 kWh/m³ of energy and 15 mg L⁻¹ of NaCl. Given these data, the operating cost was calculated to be 1.86 USD/m³. However, it should be noted that we paid no money for the electrical energy consumed during the treatment as this energy was supplied by solar cell. This means 15% off the total costs.

3.5. Electrocoagulation of real textile effluent

The process of electrocoagulation was also applied to six samples of real textile effluent. The experiment was carried out at a current density of 75 Am⁻². The surface area of each electrode was 30 × 180 mm. The electrodes were placed at a distance of 10 mm from each other. The temperature was set to 25 °C. Iron and titanium were used as the anode and cathode, respectively. NaCl was used as the electrolyte.

Table 3 gives the features of each sample before and after the process of electrocoagulation. The dye removal efficiency

was satisfactory in only four of the cases. This inconsistency shows that the experiment parameters were not sufficiently conducive to the degradation of dye molecules in the real textile wastewater. Another reason may be the presence of particles which interfere with the electrocoagulation process.

4. Conclusion

This study was an investigation into the impact of current density, anode type, temperature, pH, and salinity on the removal of Reactive Red 120 from synthesized wastewater. Regarding current density, 45 Am⁻² turned out to be optimum level. The levels of 25 and 35 Am⁻² were also effective, but they resulted in a longer electrocoagulation time. The other optimum alternatives were iron anode, a temperature level of 25 °C, a pH of 7, and an electrolyte concentration of 15 mg L⁻¹. Cost analysis was also done for the treatment process. Additionally, the application of the obtained optimum parameters to the treatment of samples of real textile effluent did not give consistent results. The post-treatment product was characterized using GC–MS studies. This indicates that dye removal is more complicated in the case of real effluent than with synthesized wastewater. On the whole, the findings of this work can be applied to treating industrial wastewater although further research should increase our understanding of various aspects of the problem.

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