Original Article

Electrochemical Treatment of Textile Dye Bath Wastewater Using Activated Carbon Cloth Electrodes

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Abstract
The performance of electrooxidation (EO) treatment using activated carbon cloth (ACC) electrodes on textile dye bath wastewater was investigated. ACC electrode pairs were used as anode/cathode for EO experiments. The effect of current density (50–150 A/m^2), operating time (0–90 minutes), and solution pH (6-11) were tested for removal of chemical oxygen demand (COD), color, and chloride, as well as the changes in conductivity. 95.5% COD and color removal efficiencies were obtained at current density (CD) of 100 A/m^2 at solution pH of 10 for 90 minutes. Moreover, the chloride concentration decreased from 4254 to 35.5 mg/L and solution conductivity decreased from 160 to 131 mS/cm at the same conditions. Operating cost of the EO process was calculated to be 3.13 US$/m^3 for 36 kWh/m^3 energy consumption. The results indicated that the EO process with ACC electrodes achieved high pollutant removal from textile dye bath wastewater.
Keywords*: Textile dye bath wastewater, Electrooxidation, Activated carbon cloth electrodes

1. Introduction

A large amount of wastewater is produced daily by the textile industry that contains various types of unreacted dyes, inorganic and common organic compounds of complex pollutants, and surfactants (1). Especially, the effluent from the dyeing process contains strong color and high chemical oxygen demand (COD) levels that require satisfactory treatment for sufficient removal efficiency (2). Otherwise, if the wastewater is not treated sufficiently during the dyeing process, dye contamination and high COD levels reduce the water quality (3). Moreover, high dye and COD levels in wastewater may cause aesthetic pollution, as well as mutagenic and carcinogenic effects if the wastewater is discharged directly into the receiving environment without any treatment (4).

Several treatment technologies have been used for the treatment of textile wastewater so far. Among these methods, membrane filtration (5), photocatalytic process (6), adsorption (7), coagulation (8), advanced oxidation (9), biological processes (10), and coupled processes (11-13) are the most commonly used treatment processes. Although they have a satisfactory dye removal efficiency, some disadvantages such as an expensive investment and operating cost, formation of sludge and toxic intermediates, and membrane fouling limit the use of these technologies (4). Moreover, conventional biological

processes cannot offer sufficient treatment efficiency, especially for dye removal due to the resistance of dyes to biodegradation (14). Therefore, studies on electrochemical treatment methods such as electrooxidation (EO) and electrocoagulation have increased recently due to their high dye removal efficiency in textile wastewater (2).

Electrochemical treatment processes are generally based on the indirect or mediated oxidation of the contaminants (15). The formation of hydroxyl radicals with high reactivity facilitates the degradation of the contaminants in textile wastewater (16,17). Many parameters such as solution conductivity, current density, solution pH, and electrode materials can affect electrochemical treatment processes (18). Recent studies in the literature have focused on innovative and environmentally friendly electrodes in which activated carbon cloth (ACC) electrodes have become prominent due to their high mechanical strength, easy handling, regeneration properties, and direct usage potential (19). While there are quite a lot of studies on the adsorption and electrosorption using ACC, studies on the use of electrochemical processes have just begun (20,21). Gineys et al (21) investigated the effect of polarization on the nanotexture of the ACC which can be used as an electrode in the electrochemical processes. It was observed that the oxidation of the pristine carbon material was enhanced by

anodic polarization while the surface chemistry and the properties of the nanotexture were not affected too much by cathodic polarization. Moreover, Wang et al (2) studied the removal of COD from real dye wastewater by the electro-Fenton process in which hydrogen peroxide was generated by polyacrylonitrile-based activated carbon fiber cloth cathode. They observed that COD removal efficiency was over 70% for 240 minutes of treatment. Additionally, they reported that the higher current densities deteriorated the COD removal efficiency due to the generation of side reactions.

Chloride rich cotton textile dye bath dump wastewater was treated using electrocoagulation process (22). Four electrode combinations (stainless steel-stainless steel, iron-iron, aluminium-aluminium, and iron-aluminium) were tested on the removal of color and COD concentration. Iron electrodes enhanced efficient treatment and 98.8% color removal efficiency was observed at 12 V and 15 minutes treatment time with a sludge generation rate of 22.7 g/L. Moreover, 80.3% COD removal efficiency was obtained at 5.8 V and 5.7 minutes treatment time with a sludge generation rate of 5.7 g/L (22). In another study, textile dye wastewater was treated with electrochemical oxidation in a batch reactor. A maximum COD removal efficiency of 97.17% was obtained at a reactor volume of 300 mL, electrolysis time of 6 hours, and current density of 4.0 A/dm^2 (23).

This study focused on investigating the performance and feasibility of treatment of textile dye bath wastewater by EO process using ACC electrodes. The effect of current density (50–150 A/m^2), operating time (0–90 minutes), and pH of the initial wastewater (6-11) were systematically investigated for the removal of COD, color, and chloride, as well as the changes in conductivity. Operating cost and energy consumption of the EO process were also examined.

2. Materials and Methods
2.1. Characterization of Textile Dye Bath Wastewater
Textile dye bath wastewater was kindly provided by a large-scale textile industry (Seckin Textile, Gaziantep, Turkey). The produced dye bath wastewater quantity was about 50 m^3/d. Samples were collected weekly from January to February 2020. Wastewater was used as received and it was not diluted during the experiments. The characterization of textile dye bath wastewater is given in Table 1.

2.2. Experimental Setup
The schematic illustration of the batch reactor setup is shown in Fig. 1. It was composed of a borosilicate glass reactor, electrodes, a digital DC power system, connecting wires, a magnetic stirrer, and a water bath. EO experiments were performed in a 500 mL reactor with a working volume of 250 mL. The reactor was placed in a temperature-controlled water bath to supply a constant reaction temperature (25±1°C). A magnetic stirrer (Wisd -Wisestir MSH-20A) and a Teflon-covered magnetic stirring bar were used to mix the wastewater at 300 rpm. ACC was used as anode/cathode electrode pairs. The ACC electrodes were kindly provided by Norm Technologies, Turkey. The dimensions of anode/cathode electrode pairs were arranged as 5 cm width × 8 cm high × 1 mm thickness with the total effective area of 40 cm^2 and distance of 2 cm between the electrodes. DC power source (AATech ADC-3303D, the maximum voltage of 30 V) was used to set anode and cathode connected to the positive and negative outlets.

2.3. Analysis
Four different current densities (50, 75, 100, and 150 A/m^2), five different EO times (15, 30, 45, 60, and 90 minutes), and four different wastewater pH values (6, 8, 10, and 11) were tested for EO treatment of textile dye bath wastewater. At appropriate time intervals, samples were taken from the reactor, centrifuged at 6000 rpm for 5 minutes, and analyzed to measure pH, conductivity, COD, color, and chloride. The conductivity and pH were measured using a pH/Cond 340i Handheld Multimeters, WTW. COD was measured according to Standard Method No. 5220C (24). The color was measured by Platinum–Cobalt (Pt–Co) method in accordance with Standard Method No. 2120 (24). The chloride concentration was measured by Argentometric method in accordance with Standard Method No. 4500B (24). All EO experiments were performed in duplicates.

Removal efficiencies of color and COD were calculated

Table 1. Characterization of Textile Dye Bath Wastewater

Sampling Date	pH	Conductivity (mS/cm)	Color (Pt-Co)	Chloride (mg/L)	COD (mg/L)
05.01.2020	11.1	160	3320	4254	5312
15.01.2020	11.0	155	3300	4500	5300
30.01.2020	11.3	162	3330	4375	5305
05.02.2020	11.5	158	3325	4450	5325

Fig. 1. Experimental Set-up for Electrooxidation (1. Magnetic Stirrer, 2. Magnetic Bar, 3. Glass Reactor, 4. Anode ACC Electrode, 5. Cathode ACC Electrode, 6. DC Power Source).

using Equation (1):

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

where C_i was the initial concentration and C_f was the concentration after defined reaction time t (min).

3. Results and Discussion

EO was used as an electrochemical treatment for textile dye bath wastewater treatment using ACC electrode pairs as the anode/cathode electrodes. The effect of current density (CD) and solution pH were investigated on COD and color removal efficiencies as well as variations of the solution conductivity and chloride concentration. Energy and cost analyses were also presented. The detailed explanation is given as follows.

3.1. The Effect of Current Density on Removal Efficiencies

The electrochemical reactor was operated under different current density (50, 75, 100, 150 A/m²) conditions. COD and color removal efficiencies as well as variations of the chloride concentration and solution conductivity were affected when the current density was increased. The COD and color removal efficiencies changed in the range of 73.6%–79.1% (Fig. 2A) and 21.7%–80.6% (Fig. 2B) for 50–150 A/m² current density, respectively. Chloride concentration and solution conductivity changed in the range of 70.4–212.7 mg/L
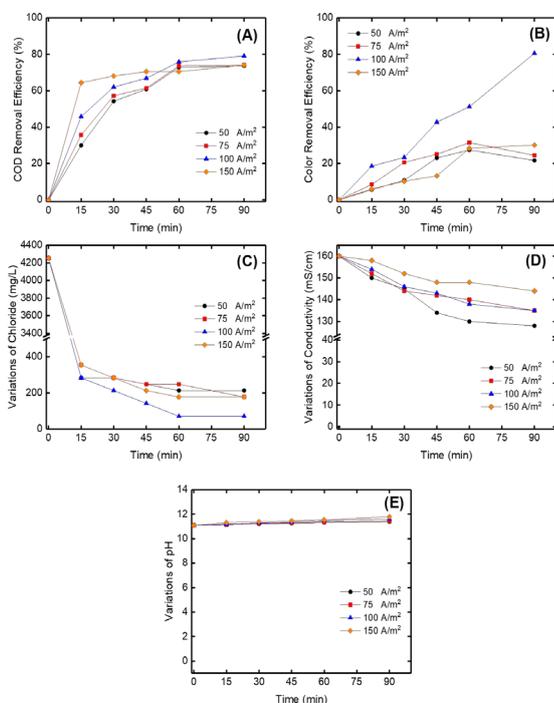
Fig. 2. The Effect of Current Density on **(A)** COD Removal Efficiency, **(B)** Color Removal Efficiency, **(C)** Variations of Chloride Concentration, **(D)** Variations of Conductivity, and **(E)** Variations of pH During the Reaction.

(Fig. 2C) and 128–144 mS/cm (Fig. 2D) for 50–150 A/m² current density, respectively. pH increased from 11.4 to 11.8 when the applied current density was increased from 50 to 150 A/m² (Fig. 2E). The results showed that the COD and color removal efficiencies decreased when the applied current density reached up to 150 A/m², denoting that the electrogeneration rate of hypochlorite decreased. It is well known that chlorides (Cl⁻) are the most widespread species for the mediated oxidation and textile dye bath wastewater includes chlorides which can be easily converted into chlorine (Cl₂) and hypochlorite according to the reaction of Eq. (2) at the anode (25). Additionally, the main reaction at the cathode is given in Equation (3) (26).

$$2Cl^- \rightarrow Cl_2 + 2e^- \qquad (2)$$

$$2H_2O + 2e^- \rightarrow H_2 + 2OH^- \qquad (3)$$
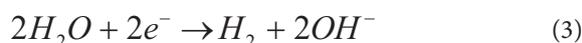
The discharged Cl₂ gas is hydrolyzed and ionized as the reactions given in Eq. (4) and Eq. (5);

$$Cl_2 + H_2O \rightarrow HOCl + H^+ + Cl^- \qquad (4)$$

$$HOCl \rightarrow H^+ + OCl^- \qquad (5)$$

In addition, the side reactions occurred in anode via the formed O₂ which caused the formation of chlorate (ClO₃⁻) according to Eq. (6) and Eq. (7);

$$2H_2O \rightarrow 4H^+ + O_2 + 4e^- \qquad (6)$$

$$6OCl^- + 3H_2O \rightarrow 3/2O_2 + 6H^+ + 4Cl^- + 2ClO_3^- + 6e^- \qquad (7)$$

At higher pH (11.1 in our case), OCl⁻ species controls the oxidation reaction and enhances the color and COD removal efficiency (22, 27). Although hypochlorite (ClO⁻) is the key product, some intermediate reactions such as chlorine (Cl₂) and hypochlorous acid (HOCl) are also formed (28). These reactive species tend to react quickly with many organic compounds and promote their mineralization (29).

At the end of the reaction, we did not observe any sludge in the reaction medium and any electrode solubility in this study.

3.2. The Effect of Solution pH on Removal Efficiencies

The electrochemical reactor was operated under different pH (6, 8, 10, and 11) conditions. COD and color removal efficiencies as well as variations of the chloride concentration and solution conductivity were affected by changing the solution pH. The COD and color removal efficiencies increased in the range of 79.1%–95.5% (Fig. 3A) and 80.6%–95.5% (Fig. 3B) for solution pH of 6–11, respectively. Chloride concentration and solution conductivity changed in the range of 106.3–35.4 mg/L
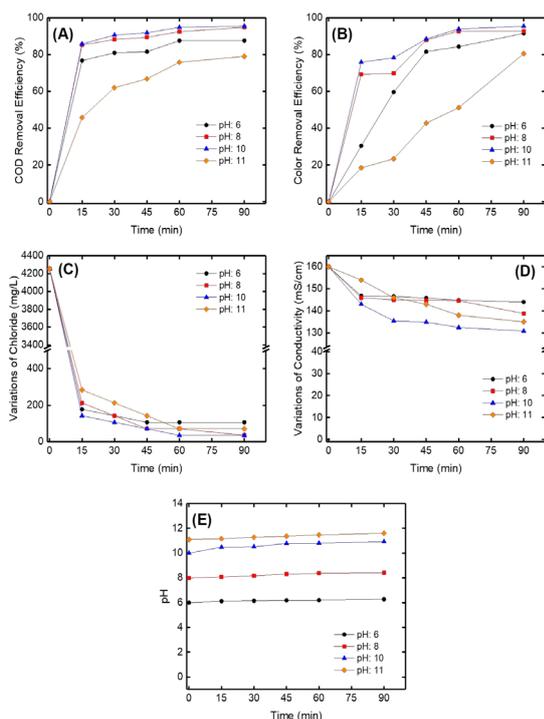
Fig. 3. The Effect of Solution pH on **(A)** COD Removal Efficiency, **(B)** Color Removal Efficiency, **(C)** Variations of Chloride Concentration, **(D)** Variations of Conductivity, and **(E)** Variations of pH During the Reaction.

(Fig. 3C) and 144–131 mS/cm (Fig. 3D) for solution pH of 6–11, respectively. However, pH increased from 6.3 to 11.6 when the solution pH was increased from 6 to 11 (Fig. 3E).

HOCl supplies 84% of the ionic species at pH 6.8, but only 0.34% at pH 10. The primary species is the ClO^- ion at pH 10 (30). In our study, we obtained lower COD and color efficiencies at low pH value (pH=6) compared to higher pH values except for pH=11. It can be explained that ionization of HOCl species decreases at lower pH values, leading to a decrease in the oxidation efficiency. The reaction of Cl_2 in acidic and alkaline medium is given by Equation (8) and Eq. (9).

$$Cl_2 + H_2O \rightarrow HClO + H^+ + Cl^- \quad \text{(acidic medium)} \quad (8)$$

$$Cl_2 + 2OH^- \leftrightarrow ClO^- + Cl^- + H_2O \,\text{(alkaline medium)} \quad (9)$$
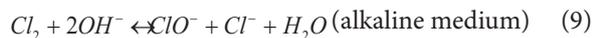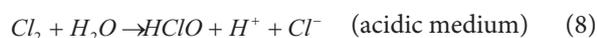
The reaction of $HClO \leftrightarrow H^+ + ClO^-$ is incomplete at pH 5.0-8.5 and both species are present to some degree (31). When the pH goes down from 8 to 6, it is obvious that H^+ ions become readily available again and the ClO^- ions return to HOCl, which is the active or killing form of chlorine. Therefore, this form of chlorine decreased the available oxidant and negatively affected COD and color removal efficiencies.

The presence of chloride ions in the wastewater causes an increase in contaminant removal efficiency because of

the formation of active chlorine. In an alkaline medium, the hydrolyzed Cl_2 gases produce the hypochlorite ions that can oxidize the organics in the anode and/or in the bulk of the solution (Eq. 10) (32, 33).

$$Organics + OCl^- \rightarrow Intermediates \rightarrow Cl^- + CO_2 + H_2O \quad (10)$$

3.3. Energy Consumption and Cost Analysis

The operating cost is one of the most important parameters for wastewater treatment plants.

Generally, the cheapest and most effective processes are preferred for wastewater treatment plants (34). The formula of energy consumption is given by Eq. (11):

$$E_{EO} = \frac{I \times V \times t}{V_R} \quad (11)$$

where E_{EO} (kWh/m³) is electrical energy consumption, V is voltage (V), I is current (A), t is time (h), and V_R is wastewater volume (m³) (1 kWh = 0.074 US$ according to Turkish Electricity Distribution Company for the date 05.06.2020). Energy consumptions for different current densities and solution pH values are given in Figs. 4A and 4C, respectively. Cost analyses for different current densities and solution pH values are shown in Figs. 4B and 4D, respectively. The optimum energy consumption and energy cost were calculated to be 36 kWh/m³ (Fig. 4C) and 2.66 US$/m³ (Fig. 4D), respectively, for current density of 100 A/m² and solution pH of 10.

The photographs of treated textile dye bath wastewater are shown in Fig. 5. Color density decreased dramatically after 90 minutes of electrooxidation.

Table 2 shows a performance comparison between the ACC electrode used in this study and the previously reported other electrodes for textile dye wastewater treatment using EO process. As shown, the use of ACC electrodes has considerable advantages over the
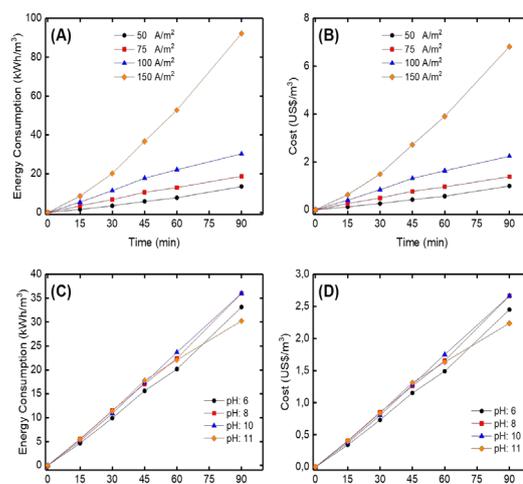
Fig. 4. (A) Energy Consumption and **(B)** Cost Analysis for Different Current Densities. **(C)** Energy Consumption and **(D)** Cost Analysis for Different Solution pH Values.

Table 2. The Performance Comparison of the Electrodes in the Electrooxidation Process for Textile Dye Bath Wastewater Treatment

Electrode	Experimental Conditions	Removal Efficiency (%)	Energy Consumption	Ref.
Anode: Ti/Pt Cathode: Stainless steel 304	EO time: 18 min CD: 0.89 A/cm^2	COD: 86% Color: 100%	21 kWh/kg COD	(35)
Anode: Pt Cathode: Pt	EO time: 60 min Voltage: 12 V	COD: 35.28%	0.054 kWh/m^3	(36)
Anode: Pt cage Cathode: Pt foil	EO time: 90 min Voltage: 40 V	COD: 60% Color: 100%	–	(37)
Anode: ACC Cathode: ACC	EO time: 90 min Voltage: 100 A/m^2	COD: 95.5% Color: 95.5%	36 kWh/m^3	This study
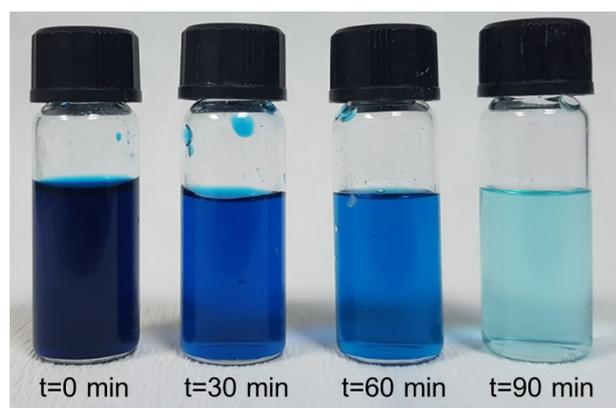
Fig. 5. The Photographs of Treated Textile Dye Bath Wastewater after Different Electrolysis Times.

reported methods considering color and COD removal efficiencies. Additionally, using ACC electrodes has other advantages such as simplicity, cost-effectiveness, no sludge production, and environmentally-friendliness.

4. Conclusion

Textile wastewater may contain some toxic dyes and must be treated efficiently before being released into the receiving environment. Additionally, certain dyes are assumed to have carcinogenic effects on humans and living organisms. In the present study, optimization of operational parameters in the electrochemical oxidation process for treatment of textile dye bath wastewater using ACC electrodes was performed successfully. The effects of operational parameters such as current density, solution pH, and electrolysis time were investigated on COD and color removal efficiencies, as well as variations of chloride concentration and solution conductivity. High COD and color removal efficiencies (95.5%) were achieved at the current density of 100 A/m^2 and solution pH of 10 for 90-minute electrolysis time. Energy consumptions and energy cost were calculated to be 36 kWh/m^3 and 2.66 US$/m^3, respectively. In the optimized experimental conditions, the treatment of textile dye bath wastewater by electrochemical oxidation method using ACC as an anode is suitable for the removal of color and COD.

Conflict of Interest Disclosures
The authors declare that they have no conflict of interests.

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