



Electrochemical Treatment of a Real Textile Wastewater Using Cheap Electrodes and Improvement in Cod Removal

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Abstract Various types of dyes and other hazardous pollutants used in the textile industry are one of the leading pollutants of surface water. In this study, real textile wastewater was electrochemically treated with two different electrode combinations (graphite/graphite and iron/graphite) by changing the anode electrode type. Comparison studies were carried out by adding Fe^{2+} or H_2O_2 to these combinations to improve Chemical Oxygen Demand (COD) removal. Current density (5, 7.5, 10 mA cm^{-2}), initial pH (2.5–8), and electrochemical (EC) oxidation time (0–120 min) were investigated to determine the optimum electrooxidation conditions. The results showed that in the electro-oxidation (EO) process, 100% colour removal, and 75.39% COD degradation efficiencies were achieved at pH 5.5, current density (I) 7.5 mA cm^{-2} , and electrolysis time (t) 40 min. In the peroxi-coagulation (PC) process, 89.41% colour removal, and 74.28% COD degradation efficiencies were achieved at pH 3, current density 7.5 mA cm^{-2} , and electrolysis time 120 min. In the $\text{EO} + \text{Fe}^{2+}$ and $\text{PC} + \text{H}_2\text{O}_2$ processes, 99.9% colour removal efficiencies, 96.38 and 90.63% COD degradation efficiencies were reached at pH 3, current density 7.5 mA cm^{-2} , and electrolysis time 40 min., respectively. In systems

using EO, PC, $\text{EO} + \text{Fe}^{2+}$ and $\text{PC} + \text{H}_2\text{O}_2$, energy consumption, and operating cost were estimated as 2.85, 2.34, 0.54, 0.62 kWh m^{-3} , and 0.304, 0.249, 0.199, 3.466 $\text{US\$ m}^{-3}$, respectively. Among all processes applied in the study, the most efficient one in terms of COD removal performance, energy, and cost is the ($\text{EO} + \text{Fe}^{2+}$) system.

Keywords Textile wastewater · Electrochemical treatment · Graphite electrode · Iron electrode · COD improvement · Energy consumption

1 Introduction

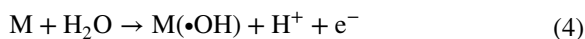
Environmental damage caused by the rapidly changing fashion industry ("fast fashion" trend) and textile industries has recently become one of the most criticized issues. According to the OECD report in 2017 (Organization for Economic Cooperation and Development), the textile industry is one of the industries that consume the most water. According to the statistics, the fashion industry is responsible for 20% of global wastewater (OECD, 2018; Shamsuzzaman et al., 2021; Bilińska and Gmurek 2021). The characteristics of textile wastewater are significantly affected by the types of fabric produced, the types of chemicals consumed, the equipment used in the processes, and the weight of the fabric, and they contain various chemicals as dyestuffs and additives that have dangerous effects on the environment and human beings.

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Textile wastewater is not environmentally acceptable due to its persistent, toxic, non-biodegradable, mutagenic, carcinogenic, and coloured contents (Yehuala et al., 2022). Textile wastewaters from different dyeing stages are resistant to light, hydrolysis, and solubility, and most of them are difficult to decolour due to their synthetic origin (Rosa et al., 2020). Although various traditional methods are applied in the textile industries, it is known that the required standards are not met with these treatment techniques. Therefore, it is necessary to use advanced techniques in the treatment of this wastewater.

Technologies based on Electrochemical Advanced oxidation processes (EAOPs) have attracted a lot of attention in recent years as promising and high-efficiency technologies for textile wastewater treatment (TWWT) (Zou et al., 2020). The main advantages of these technologies are the safety of the process, no sludge formation, simple equipment design, ease of automation, progressive reaction with electrons, moderate or low construction cost, and effective mineralization of pollutants (Turan et al., 2020). In addition, electrochemical techniques are clean and green technologies with the advantages of using electrons instead of chemicals in reactions, short reaction times, and widespread use of renewable energy sources for energy needs, which is one of the main cost items. EAOPs, using non-sacrificial electrodes, involve two oxidation procedures, direct and indirect. In the first oxidation process, the pollutants adsorbed on the surface of the anode material are decomposed by the electron transfer between pollutants and the surface of the anode material. The most popular direct EAOP is anodic oxidation (AO), which works based on hydroxyl radical production at the anode surface. The advantage of anodic oxidation is that chemicals are not needed, and fewer secondary contaminants are produced. In the second oxidation process, the pollutants are decomposed in the bulk solution through an oxidation reaction with electrochemically produced strong oxidative species such as chlorine hypochlorite and hydrogen peroxide (Liu et al., 2019). EAOPs are based on the generation of highly reactive chemical species (hydroxyl radicals [OH radical]) capable of degrading organic matter. These radicals oxidize organic compounds and degrade them into simpler compounds, up to their total mineralization in carbon dioxide (CO₂) and water. Electrochemical processes based on Fenton's reaction such as Electro-Fenton

(EF) process, Peroxi-coagulation (PC), Fered-Fenton and Anodic Fenton (AF) processes and Photo-electro-Fenton process and photocatalytic methods are among indirect AOPs (Nidheesh et al., 2018; Ramirez-Pereda et al., 2020). Fenton-based EAOPs represent a combination of the Fenton oxidation and electrochemical process. The major mechanisms for pollutant degradation are the origin of Fenton's reaction to generate hydroxyl radicals (Eqs. (1–4)). Externally added or electrochemically produced Fe²⁺ and H₂O₂ supply continuously •OH to the solution (Titchou et al., 2021).



As seen in Eq. 2, the H₂O₂ formed by the reduction of oxygen on the cathode surface and Fe²⁺ added externally form the EF process. In the AF process, hydrogen peroxide is added externally while producing iron ions from the sacrificial anode as shown in Eq. (5).



With the continuous and controlled production of OH radicals, pollutants are mineralized. However, in electrochemical processes, as in Eq. 3, the regeneration of iron at the cathode (Fe²⁺) prevents the accumulation of iron (III) and thus prevents the formation of excess sludge. Instead of adding iron externally, the peroxi-coagulation (PC) process (PC) can be performed by producing the iron in situ

Table 1 The properties of the textile wastewater used in the study

COD (mg/L)	280 ± 10
pH	7.78 ± 0.22
EC (mS/cm)	5.06 ± 0.1
Absorbance (at 615 nm)	2.450
Sulfate (SO ₄) (mg/L)	180 ± 4
Ammonia (NH ₄ -N)	62 ± 5
Phosphates(PO ₄)	5.85 ± 3
Chloride (g/L)	2.2 ± 0.05

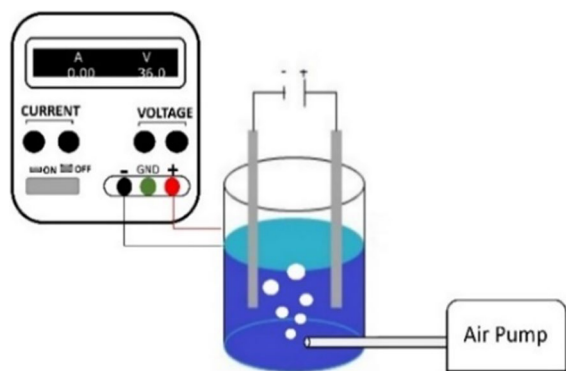
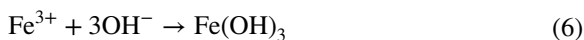


Fig. 1 The experimental apparatus

using a sacrificial iron electrode. Thus, pollutants are removed by the combined effect of coagulation with $\text{Fe}(\text{OH})_3$ (Eq. 6) and oxidation in the same process (Suhan et al., 2020).



In electrochemical techniques, the used electrode materials are a decisive factor in the elimination of pollutants, as the properties of the electrode define the type of process and thus the electrochemical and chemical mechanisms involved. Until today, various electrode materials have been used by researchers in the treatment of textile wastewater and electrochemical techniques have been successfully applied (Kuchtová et al. 2020). However, expensive electrode materials have limited industrial-scale applications. Carbon-based electrodes such as graphite have attracted the attention of researchers due to their low cost, good conductivity, and porosity, which encourage the production of hydrogen peroxide by absorbing oxygen gas and have been used in the treatment of textile wastewater (Nidheesh et al., 2018; Rodríguez-Narváez et al., 2021). Comparative studies of real textile wastewater using graphite and iron anode materials are scarcely reported. Applications that are obtained without the need to consume pure raw materials such as scrap iron and that allow the reuse of raw materials are gaining in importance (De Maman et al., 2022). In addition, in most studies of dyed wastewater treatment, the average value of COD removal is around 70%. Hence, it may remain

below the discharge limits. In order to treat it by desired standards, its current efficiency needs to be increased even more. In this study, commercially available cylindrical graphite and scrap iron (SI) were used as electrode materials for real textile wastewater treatment. The high cost of electrode materials limits the industrial-scale use of electrochemical processes. Therefore, the use of scrap iron and inexpensive graphite will increase the applicability of this process on a large scale. Two different anode materials were tested under the same experimental conditions. The treatment efficiencies were evaluated with colour removal and COD removal, and the energy requirements were calculated. Different H_2O_2 and $\text{Fe}(\text{II})$ combinations were studied for further mineralization. This work includes various AEOP processes such as electro-oxidation (EO), peroxi-coagulation (PC), anodic Fenton (AF), and electro-Fenton (EF). The aim of this study is to evaluate the factors affecting COD and colour removals, such as applied current, wastewater pH, and electrolysis time, in real textile wastewater and to compare the efficiency and costs of electrochemical processes.

2 Materials and Methods

2.1 Wastewater Sample and Materials

The real wastewater used in the experimental studies was obtained from a textile factory in Aydın (Turkey) where various reactive textile dyes were used. The content of wastewater is illustrated in Table 1. All chemicals used for the analysis were purchased from Merck. Graphite was commercially available. SI was used as the iron electrode. Before experimental studies, the raw wastewater was filtered through 30 μm membrane filters to simulate sand filtration to remove any suspended particles (Ganzoury et al., 2022) and then stored in a dark environment.

2.2 Experimental Setup

Batch electrochemical oxidation experiments were conducted in a 250 mL cylindrical glass reactor containing 200 mL wastewater volume. Commercially available cylindrical graphite

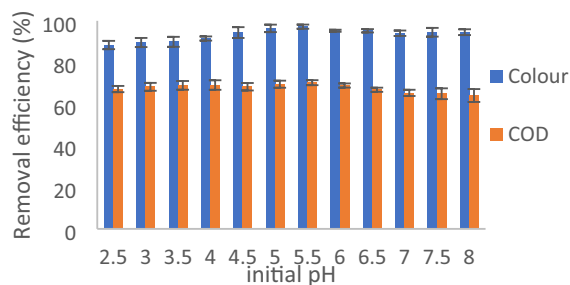


Fig. 2 Effect of the initial pH on colour and COD removal with graphite/graphite electrode; current density: 5 mA/cm²; electrolyse time: 60 min

pencil tip and SI electrodes (99.9%) with a diameter of 7 mm and a height of 9 cm were used as electrode material in the EO (graphite anode and graphite cathode) and PC (SI anode and graphite cathode) processes, respectively. For AF, the SI anode and graphite cathode electrodes mentioned in the PC process were used and H₂O₂ was added to the system externally. For EF, as mentioned in the EO process, graphite electrodes were used as anode and cathode materials and Fe²⁺ was added externally to the system. The total effective surface areas of electrodes were approximately 20 cm². The size and shape of the electrodes were similar to keep the surface areas same. The experimental apparatus was illustrated in Fig. 1. The anode and cathode electrodes were placed vertically and parallel to each other. The distance between the electrodes was 2.5 cm. The studies were carried out using a digital power supply (Gw instek GPS-3303, in the interval 0–30 V, 0–3 A). In the experiments, the pH value of the wastewater was reduced to the desired value by using 0.1 N H₂SO₄. Since the natural conductivity of the textile wastewater is high, no electrolyte was added. A commercially available air pump was used to support hydrogen peroxide

production at the cathode and provide the oxygen required for the system. The samples were taken at the specified time. The pH of the taken sample was increased to about 9.0 and the samples were kept for 15 min to settle the sludge formed in the experiments where an iron electrode was used as the anode. Then, the samples were filtered through 0.45 µm disc filters (Sartorius). Colour and COD were monitored in the samples taken and these parameters were expressed as percent removal. The % removal efficiency of the samples for COD and colour parameters was calculated as follows.

$$\text{Removal}(\%) = \frac{(C_o - C_t)}{C_o} \quad (7)$$

C_o and C_t (mg L⁻¹) show COD and colour concentrations in wastewater before and after electrochemical treatment, respectively.

Chemical oxygen demand (COD) and the colour of the wastewater samples were analysed using HACH LANGE DR3900 UV–vis spectrophotometer. The pH of the solutions was measured using a HANNA HI 8424 pH meter. The conductivity value was measured using a conductivity meter HANNA instruments HI8819. Spectrophotometric method using cuvette test (Hach Lange GmbH, Berlin, Germany) LCK 314 was used for the determination of COD in the range of 15 mg/L to 150 mg/L O₂.

2.3 Statistical Evaluation and Quality Control

The experiment studies were performed twice, and the average values were used. In figures, error bars (at a confidence level of 95%) show the standard error of the mean. All chemical procedures and analyses were performed in accordance with the “Standard Methods for Examination of Water and Wastewater (Baird & Bridgewater, 2017), followed by QA/QC

Table 2 Energy consumption of the electrochemical processes for optimum conditions

Process	I (A)	Voltaj	t (h)	E(Wh)	Energy Consumption (kWh/m ³)	ΔCOD	kWh/kgCOD
EO	0.15	5.7	0.67	0.57	2.85	211.09	13.50
PC	0.15	3.9	2	1.17	2.34	207.98	11.25
EO + Fe ²⁺	0.15	2.7	0.67	0.27	0.54	269.86	2.001
PC + H ₂ O ₂	0.15	3.1	0.67	0.31	0.62	253.76	2.44

(Quality Assurance/Quality Control), through the same procedure”.

2.4 Energy Consumption and Cost Analysis

Energy consumption in the electrochemical processes is calculated as follows (8) and (9).

$$\text{kWh/kgCOD} = \frac{I * \text{voltage} * t}{\Delta\text{COD} * V} \quad (8)$$

$$\text{kWh/m}^3 = \frac{I * \text{voltage} * t}{V} \quad (9)$$

where I (A) is the operating current intensity, V is the volume of the solution (m^3), t is the electrolysis time (h) and ΔCOD is the COD degradation in solution (mg L^{-1}).

3 Results and Discussion

3.1 Effect of Initial pH

The effect of initial pH on EC treatment of real textile wastewater was studied with graphite/graphite and iron/graphite electrodes. The wastewater pH is one of the main operational parameters influencing the performance of the EC process.

3.2 Graphite/Graphite Electrode (EO)

The effect of wastewater pH within the range of 2.5 and 8.0 on colour and COD removal was shown in Fig. 2. The maximum removal efficiency of the EO process was obtained at pH 5.5, where the colour removal was 97.2%, COD removal was 70.28% at 60 min. The lowest removal efficiencies were at pH 8.

4 Results and Discussion

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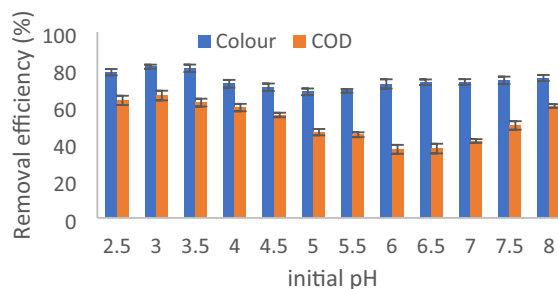
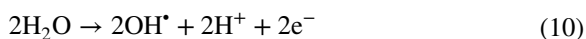


Fig. 3 Effect of the initial pH on colour and COD removal with iron/graphite electrode; current density: 5 mA/cm^2 ; electrolyse time: 60 min

4.2 Graphite/Graphite Electrode (EO)

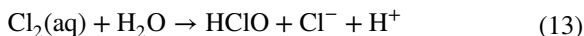
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In electrochemical processes using graphite electrodes, oxidation of pollutants occurs in a combination, both by direct oxidation at the electrode surface and by indirect oxidation by in situ-produced oxidants (Raju et al., 2009). During the electrochemical process, oxidation occurs with the help of various oxidants (oxygen, hydrogen peroxide, free chlorine) and free radicals ($\text{OH}^\bullet$, $\text{ClO}^\bullet$ and $\text{Cl}^\bullet$). The formation of hydroxyl radicals on the anode surface occurs according to Eqs. 10–11 (Bhatnagar et al., 2014):



The presence of chloride/chlorine in the wastewater increases the degradation efficiency by oxidizing the organics in the solution or at the anode. As illustrated in Table 2, the wastewater contains chloride at a concentration of 2.2 g L^{-1} . While chloride in textile wastewater increases the conductivity of the medium, it also increases the concentration of hypochlorite ions, which are known to be strong oxidants and oxidize organic compounds in wastewater during the electrochemical oxidation process. Indirect oxidation of organic compounds in wastewater occurs when the electro-generated chlorine

or hypochlorite produced is reduced to chlorite ions (Eqs. 12–14). The oxidative power of this process is pH-dependent. In an acidic pH environment, faster oxidation of organics mediated by the active species of chlorine is achieved (Garcia-Segura et al. 2018).



It is reported that chlorine content and indirect oxidation in wastewater are effective in pollutant removal (Rajkumar et al., 2005). Therefore, it can be said that the hydroxyl radical and active chlorine produced in the electrolytic environment are the main oxidants behind the process's colour and COD removal (Chanikya et al., 2021). In alkaline conditions, the ionization of chlorine/hypochlorite ions is low. This may explain the fact that the lowest values for colour and COD removal are at pH 8, as a factor reducing the efficiency of the electrolytic system. However, increased O_2 discharge at the expense of Cl_2 evolution can affect efficiency (El-Ashtouky et al., 2012). The optimum pH 5.5 was chosen. This finding is consistent with previous studies where the system was used to treat textile wastewater (Saleh et al., 2021).

Also, other electrolytes in the wastewater, such as phosphate and sulphate, are susceptible to oxidation and can react as mediators for the following oxidation of organic pollutants. From the electro-oxidation of these species, peroxodiphosphate (Eq. 15) and peroxodisulphate (Eq. 16) are formed de (de Araújo et al., 2018). However, compared to the main oxidants,

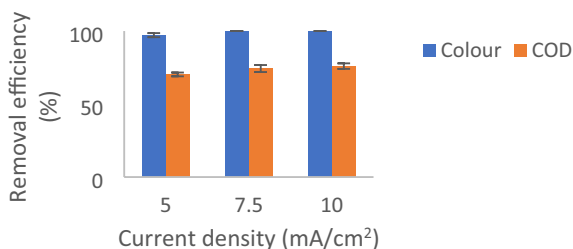


Fig. 4 Effect of the current density on colour and COD removal with graphite/graphite electrode; pH:5.5; electrolyse time: 60 min

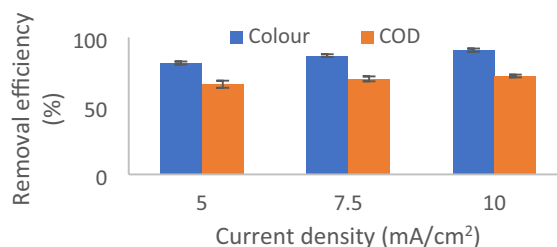
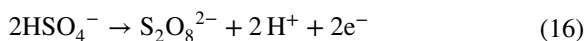


Fig. 5 Effect of the current density on colour and COD removal with iron/graphite electrode; pH:3; electrolyse time: 60 min

these species are weaker oxidants and do not have the ability to completely mineralize organic pollutants.



4.3 Iron/Graphite Electrode (PC)

In this system called PC, pollutants are expected to be removed by two mechanisms, electrooxidation, and electrocoagulation. In electro-oxidation, the removal of pollutants is provided by the attack of hydroxyl radicals. In electrocoagulation, the removal of pollutants is provided by coagulation of iron precipitates with $\text{Fe}(\text{OH})_3$.

The effect of initial pH on colour and COD removal was shown in Fig. 3. The colour removal efficiency decreased from 81%, to 68% at electrolysis time of 60 min with increase in initial pH from 3.0 to 5.0. COD removal efficiencies were 65.8% and 36.9% with increase in initial pH from 3.0 to 6.0., respectively. When the pH increased from 6 to 8, the COD removal rates increased again. As shown in Fig. 3, the highest COD and colour removal ratios of the process were reached at initial pH 3.0. In this process, Fe^{2+} at the iron anode and H_2O_2 at the graphite cathode are produced at the same time, quite likely that the two react at low pH to produce Fenton reagents (reaction 1–4). “Fenton reactions have maximum catalytic activity in the pH range of 2.8–3. Free radical production occurs in acidic conditions” (Gümüş & Akbal, 2016). Therefore, COD and colour removal efficiencies increased in acidic conditions. “At pH higher than 3, Fe^{3+} starts precipitating as $\text{Fe}(\text{OH})_3$

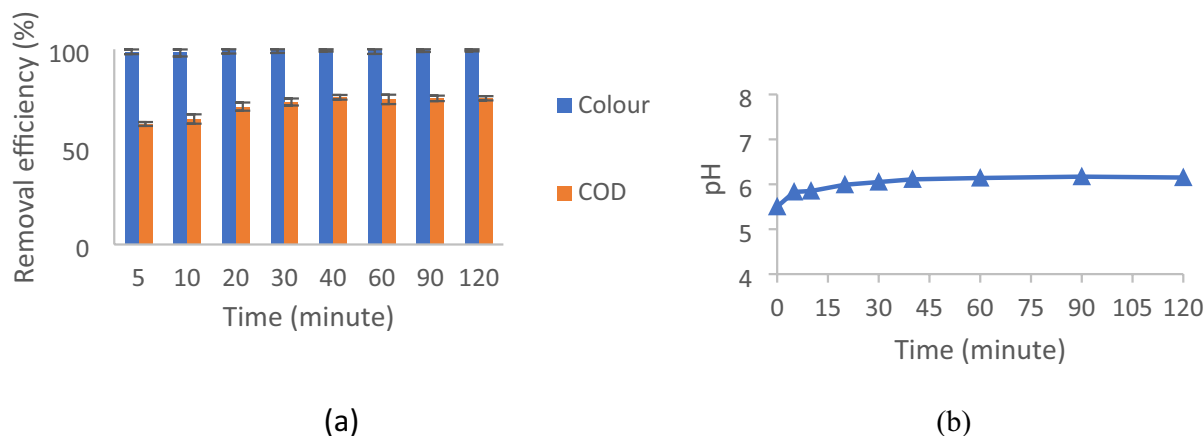


Fig. 6 (a) Effect of the electrolysis time on colour and COD removal with graphite/graphite electrode; pH:5.5; current density: 7.5 mA cm^{-2} (b) the change in pH during the electrolysis time

and breaks down the H_2O_2 which is unstable at neutral pH, into O_2 and H_2O . Besides, the formation of Fe^{2+} complexes at high pH values leads to a drop of the Fe^{2+} ion concentration” (Ghanbari & Moradi, 2015). This indicates that the oxidative reaction does not occur at high pH and at high pH the main removal mechanism is via coagulation from the formation of $\text{Fe}(\text{OH})_n$ complexes.

4.4 Effect of Current Density

The effect of current density on electrochemical treatment of textile wastewater was studied in the EO and PC processes. Current density is an important factor

affecting wastewater treatment efficiency and reaction rate in all electrochemical processes. The optimum current density should be selected considering operating cost and electricity consumption.

4.5 Graphite/Graphite Electrode (EO)

As seen in Fig. 4, the colour and COD removal efficiency of the electrochemical oxidation system increased as the applied current density increased. The increase in colour and COD removal efficiency with the increase of applied current density is related to the increase in strong oxidant production. Colour removal efficiencies were 97.2%, 100%, and 100%

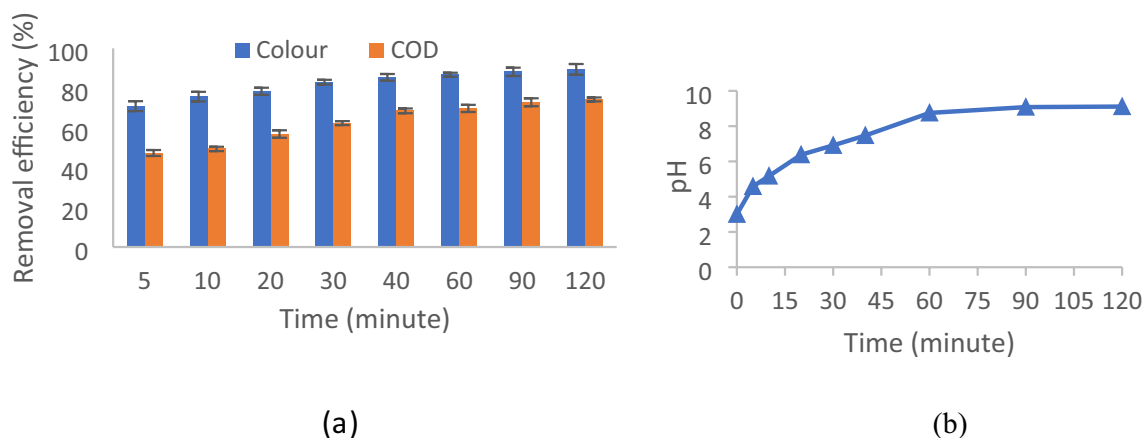


Fig. 7 (a) Effect of the electrolysis time on colour and COD removal with iron/graphite electrode; pH:3; current density: 7.5 mA cm^{-2} (b) the change in pH during the electrolysis time

when current densities were 5, 7.5, and 10 mA cm⁻², respectively. Almost completely colourless water was obtained at all current densities. Under the same operating conditions, the COD removal efficiencies were 70.28%, 74.32% and 75.98%, respectively. However, when the current density was increased from 7.5 mA cm⁻² to 10 mA cm⁻², the system performance did not increase at the same rate. The balance between energy costs and desired efficiency is required to be finely adjusted. Therefore, 7.5 mA cm⁻² was selected as the optimal current density, where the system performance is efficient, and the energy consumption is economical. Other researchers have reported similar findings in the literature. Periyasamy and Muthuchamy (2018) reported that they achieved higher removal efficiency at higher current density in a study they conducted with graphite electrodes.

5 Iron/Graphite Electrode (PC)

5.1 Iron/Graphite Electrode (PC)

Electrochemical treatment of textile wastewater with iron/graphite electrodes was applied at 5, 7.5 and 10 mA cm⁻² current densities, keeping other parameters constant. The rate of decay increased with the increase of current density. It can be seen in Fig. 5 that the current exceeding a certain value does not improve the performance of the system at the same rate. Since a higher current density requires more energy in the system, like the graphite anode, the optimum current density of 7.5 was chosen for the subsequent experiments. The final COD removal efficiencies were 65.82%, 69.76%, and 71.85%, respectively, under the current densities of 5, 7.5 and 10 mA cm⁻². The final colour removal efficiencies were 81.34%, 86.73%, and 90.7%, respectively for 5, 7.5, and 10 mA cm⁻². This is because an increase in current density accelerates the COD and colour removal kinetics due to the formation of larger hydroxyl radicals and Fe (OH)₃ flocks. It has also been confirmed in previous studies that at larger current densities, the amounts of H₂O₂ and iron (II) in the bulk solution are higher and the excess iron (II) ions react with hydroxyl radicals to produce excess iron (III) radical points (Eq. 1) (do Vale-Junior et al., 2018; Ghanbari & Moradi, 2015; Zarei et al., 2009).

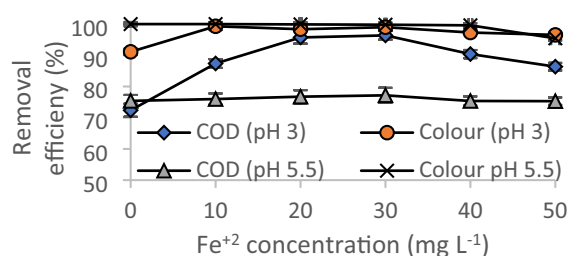


Fig. 8 Effect of the Fe²⁺ concentration on colour and COD removal with graphite/graphite electrode; pH:3; current density: 7.5 mA cm⁻², t:40 min

5.2 Effect of Electrolysis Time

In this section, the effect of electrolysis time on COD and colour removal in electrochemical treatment processes was investigated under a current density of 7.5 mA cm⁻² for each electrode. The initial pH values for graphite/graphite and iron/graphite electrodes were 5.5 and 3.0, respectively.

5.3 Graphite/Graphite Electrode (EO)

In order to determine the effect of electrolysis time on the performance of the EC process with graphite/graphite electrodes, experiments were performed at electrolysis times of 5, 10, 20, 30, 40, 60, 90, and 120 min. As can be seen from Fig. 6 (a), colour and COD removal increased with increasing electrolysis time. The colour concentration in the wastewater was reduced and almost completely colourless within 5 min. A rapid discoloration was observed. However, the COD degradation in textile wastewater gradually increased with increasing reaction time within 40 min. There was no significant change after 40 min.

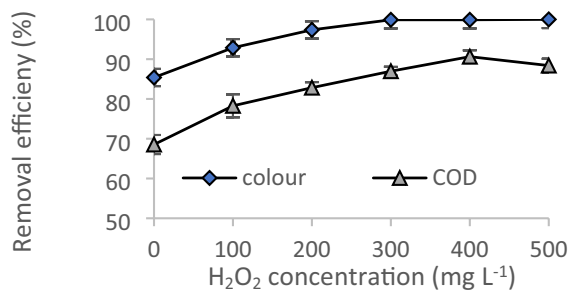


Fig. 9 Effect of the H₂O₂ concentration on colour and COD removal with iron/graphite electrode; pH:3; current density: 7.5 mA cm⁻², t:40 min

where the COD and colour degradation rates were 75.39 and 100%, respectively. With the increase of the electrolysis time, the radicals and oxidants formed in the solution were able to oxidize the organic compounds in the textile wastewater in a short time. Then, the removal efficiency gradually stabilized due to the limitation of the mass transport of the pollutant (Donaghue & Chaplin, 2013) by decreasing the pollutant concentration in the water over time (Zou et al., 2020). As seen in Fig. 6 (b), the initial pH increased slightly during the electrolysis period possibly due to H^+ consumption (Eq. 2), and OH^- accumulating in the solution during the process (Orkun & Kuleyin, 2012).

5.4 Iron/Graphite Electrode (PC)

The effect of electrolysis time within the range of 5 and 120 min on colour and COD removals are shown in Fig. 7 (a). The COD degradation rates increased from 47.32% to 74.28%, at initial pH 3.0 when reaction times increased from 5 to 120 min., respectively. Colour removal rates at the same reaction times reached from 70.91% to 89.91%, respectively. As seen in the Fig. 7 (a), the COD and colour removal rates in the wastewater gradually increased with increasing electrolysis time within 120 min.

Under acidic conditions (at pH 3), the oxidized Fe^{2+} at the anode and a certain amount of H_2O_2 , which is advantageously electrogenerated at the cathode led to the Fenton reaction that generates hydroxyl radicals. These radicals strongly oxidized organic compounds in wastewater in a short time. Afterward, the COD and colour removal efficiencies gradually increased. As the electrolysis time increased, the pH

increased and became neutral due to H^+ consumption (Eq. 2). At higher pH the Electro-Fenton reaction lost its effectiveness, and electrocoagulation based on $Fe(OH)_n$ formation played a dominant role. Some of the non-oxidizable and suspended organic pollutants were removed by precipitation (Casado, 2019; Sun et al., 2015). As the electrolysis time increases, this increase in pH is due to the OH^- ions formed by the reduction of water on the cathode surface (Eq. 9) (Nidheesh & Gandhimathi, 2014a, 2014b*) and the Fenton reaction (Eq. 1). Figure 7 (b) shows the pH change of the wastewater during the electrolysis time.

6 Improvement of COD Removal in Electrochemical Processes

6.1 Influence of Addition of Fe^{+2} (EO + Fe^{+2})

In the EO system, which was optimized with a graphite anode at pH 5.5, Fe^{2+} was added to the system in the range of 10 to 50 $mg\ L^{-1}$ to further improve the COD removal. The COD degradation efficiencies at pH 3 and pH 5.5 in this system called Electro Fenton (EF) are presented in Fig. 8. When Fe^{2+} is added to the EO system, Fenton reactions are expected to occur at pH 3. In the EF process, the COD removal rate is higher than in the EO process for the two pH conditions. The wastewater was mineralized at 96.38% and 77.17% for, respectively, pH 3 and 5.5, after 40 min of electrolysis at optimum Fe^{+2} value of 30 $mg\ L^{-1}$. COD removal increased in acidic pH, however, the addition of Fe^{+2} didn't significantly contribute to the higher removal of COD at pH 5.5. At pH 3, high degradation is due to the high oxidizing power of Fenton reactions and the continuous regeneration of Fe^{2+} at the cathode (Eq. 3). Under lower acidic pH conditions, the radical point kinetics of the conversion of Fe^{2+} to Fe^{3+} is preserved and Fe precipitation is prevented (Vasudevan, 2014).

6.2 Influence of Addition of H_2O_2 (PC + H_2O_2)

The anodic Fenton (AF) process takes place when H_2O_2 is added to the electrochemical system in which a graphite cathode and an iron anode are used. H_2O_2 is the main factor as the oxidizing agent in the Fenton reaction. To investigate the effect of H_2O_2 in the AO system, varying concentrations of H_2O_2 from 100 to

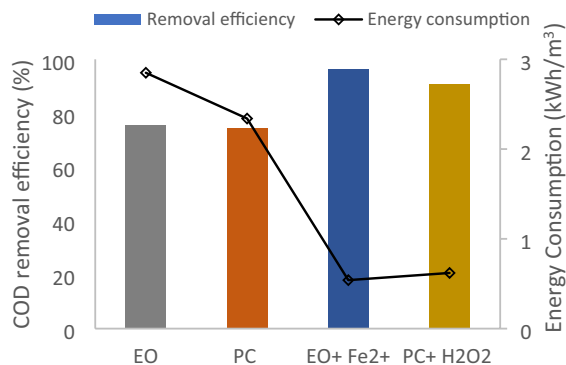


Fig. 10 Comparison of the electrochemical processes

Table 3 Cost analysis of the electrochemical processes for optimum conditions

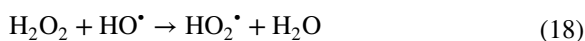
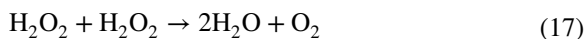
Process	Energy Cost		US\$/m ³		Total operation US\$/m
	US\$/kg-COD	US\$/kWh/m ³	³ H ₂ O ₂ cost	Fe ²⁺ cost	
EO	1.44	0.304	-	-	0.304
PC	1.20	0.249	-	-	0.249
EO + Fe ²⁺	0.21	0.058	-	0.141	0.199
PC + H ₂ O ₂	0.26	0.066	3.4	-	3.466

*unite price of H2O2 is 3.4 \$/Kg

*unite price of iron is 0.945 \$/Kg

*unite price of energy is 0.071\$/kWh

500 mg L⁻¹ were studied and the results are presented in Fig. 9. Figure 9 shows the effect of H₂O₂ concentration on COD and colour removal efficiency. The COD and colour removal efficiencies increased when the H₂O₂ concentration increased. The COD removal efficiency was 90.63% at H₂O₂ concentration of 400 mg L⁻¹. Hence, 400 mg L⁻¹ was selected as the optimum concentration of H₂O₂. A completely colourless water was obtained at H₂O₂ concentration of 300 mg/L. It is clear that with the increase in H₂O₂ concentration in the electrochemical cell, more radicals are produced (Eq. (1)), which causes the degradation of organic compounds and removal of colour. Nevertheless, the degradation rate decreased in 500 mg/L H₂O₂. This can be attributed to the self-decomposition of high concentrations of H₂O₂ based on Eq. 17 (Nidheesh et al., 2013). Moreover, the excessive H₂O₂ can react with HO• and produces the HO₂• (hydroperoxyl) which is a less reactive radical (Eq. 18) (Moradi & Ghanbari, 2014).



6.3 Comparison of Electrochemical Processes and Cost Analysis

Each system was optimized with the criteria of improving COD removal and minimizing energy consumption, and the results were compared with each other. Figure 10(a) and (b) presented a comparison of the electrochemical processes at COD removal (%) and energy consumption (kWh/m³),

respectively for their optimal conditions. As can be seen from the figures, process efficiencies in COD removal performance and energy consumption (kWh/m³) are (EO + Fe²⁺) > (PC + H₂O₂) > EO > PC for optimum conditions. As can be seen in Fig. 10 (a), over 70% of degradation occurred in all processes. However, in the case of the EO + Fe²⁺, known as an Electro-Fenton (EF), almost all COD (96.4%) has been degraded at 40 min. EO and EF, both using a graphite/graphite electrode, can lead to COD and colour removal from wastewater by the attack of OH radicals. In the EF system, the addition of Fe²⁺ at acidic pH induced the Fenton reaction (Eq. 1), improving COD removal from 75.4% to 96.4%. PC process using iron anode and graphite cathode, in the chain reactions, H₂O₂ can be consumed more rapidly than they are produced. For this reason, the reactions were supported by adding H₂O₂ externally to the system. Thus, COD removal performance increased from 74.3% to 90.6%. No sludge formation was observed in the EO system. Only a negligible amount of dark particles settled to the bottom. No excessive sludge formation was observed in the EF system to which Fe²⁺ was added. The conversion of Fe³⁺ to Fe²⁺ at the cathode (Eq. 3) prevented sludge formation and precipitation to a large extent. On the other hand, sludge formation in the PC and AF processes was much higher than in the others. Especially in the PC system, sludge formation at the bottom and floating flocs and foam formation at the top of the reactor made the system disadvantageous. Sludge formation and floc formation slightly improved when hydrogen peroxide was added, but still lagged behind the

Table 4 Summary and comparison of literature review for textile and dyed wastewater

Conclusion	Conclusion	Conclusion	Conclusion	Conclusion	Conclusion
Graphite/Graphite	Real textile waste-water (COD: 84–114 mg L ⁻¹)	pH:3, I: 0.55 A (10 mA cm ⁻²), Fe ²⁺ : 2 mM, t:30 min	%89 colour, %93 COD, %58 TOC	16 kWh m ⁻³	(Kuleyin et al., 2021)
Graphite/Graphite	Fabric-dyeing wastewater (COD:833 mgL ⁻¹)	pH:5, I: 150 A/m ² , t:120 min	96.6% colour, 77.69% COD	34.9 kWh m ⁻³ /2.58 US\$	(Saleh et al., 2021)
Iron/Graphite	Rhodamine B	pH:3, t:180 min	95% dye	-	(Nidheesh & Ghandimathi, 2014a)
Ti-RuO ₂ / Al	Real textile waste-water	pH:5.54, I: 1.37 A, t:157.6 min	94.74% colour, 86.22% COD	0.012 kWh	(Kaur et al., 2018)
Iron/Carbone fiber	Real textile waste-water	I: 100 mA	69.8% TOC	-	(Yıldız et al., 2019)
Stainless steel (EF)	Remazol black B	pH:3, I: 10 mA cm ⁻² , t:50 min	94% colour, 76% COD,	-	(Suhan et al., 2020)
Stainless steel (EC)			91% colour, 19.8% COD		
pt-Ti / Iron plate	Real textile waste-water (1024 mg L ⁻¹)	pH:3, 3 V, t:120 min	(EAOP + EC) 93.5% COD, (EC + EAOP) 74.8% COD	-	(Chanikya et al. 2021)
Graphite/Graphite (EF)	Real textile wastewater	t:60 min	97% colour 64% COD	331.04 US\$ kg ⁻¹	(Nidheesh & Ghandimathi, 2014b)
Iron/Graphite (PC)	(COD:1555 mg L ⁻¹)		97% colour 70% COD	301.28 US\$ kg ⁻¹	
Iron/Graphite (PC-PH)			%82 colour 75% COD	409.65US\$ kg ⁻¹	
Platin/Graphite (EF)	Real textile waste-water (COD: 1310 mg L ⁻¹)	pH:3, I: 300 mA, t:160 min	77.2% colour, 64% COD	63.64 kWh kg COD ⁻¹	(Ghanbari & Moradi, 2015)
Iron/Iron (EC)		pH:6.5, I:200 mA t:40 min	92.1% colour, 78.6% COD	2.73 kWh kg COD ⁻¹	
Iron/Iron + H ₂ O ₂ (ECF)		pH:3, I: 200 mA, t:40 dk	94% colour, 82.1% COD	3.38 kWh kg COD ⁻¹	
Iron/Iron (PC)		pH:3, I:300 mA, t:120 min	79.9% colour, 71.1% COD	23.19 kWh kg COD ⁻¹	
Nb-BDD/Carbone PTFE (EO) with H ₂ O ₂	Azo dye (MO)	pH:3, I:33.3 mA/cm ² C ₀ :100 mg L ⁻¹ (MO) 0.050 M Na ₂ SO ₄	13% COD	0.06 kWh g COD ⁻¹	(do Vale-Júnior et al., 2018)
Nb-BDD/Carbone PTFE (EF)			84% COD	0.09 kWh g COD ⁻¹	
Fe plate /Fe plate (EC)			29% COD	0.7 kWh g COD ⁻¹	
Fe plate/carbone PTFE (PC)			70% COD	0.1 kWh g COD ⁻¹	
Graphite/Graphite (EO)	Real textile waste-water -	pH:5, t:120 min. I: 0.28A/cm ²	93.89% colour, 89.71% COD,	-	(Subramaniam et al., 2016)

Table 4 (continued)

Conclusion	Conclusion	Conclusion	Conclusion	Conclusion	Conclusion
Iron/Iron (EC)	Indigo Carmine	5 mA cm ⁻²	100% colour	0.511 kWh m ⁻³	(Stergiopoulos et al., 2014)
Ti-Pt / graphite (EO)	Textile Dye (Co:100 mg L ⁻¹)	5 mA cm ⁻²	100% colour	0.825 kWh m ⁻³	
Iron/Iron + H ₂ O ₂ (EF)		0.33 and 0.66 mA cm ⁻²	100% colour	4.75 × 10 ⁻³ and 5.23 × 10 ⁻³ kWh m ⁻³	
Graphite/Graphite (EO)	Real textile wastewater (COD:347 mg L ⁻¹)	pH: 4, I=27.78 A m ⁻² , t=110 min	96.27% colour, 90.78% COD	23.58 kWh/kg COD	(Bhatnagar et al., 2014)
Graphite/Graphite (EO)	Real textile wastewater (COD:280 mg L ⁻¹)	pH: 5.5, I:7.5 mA cm ⁻² , t: 40 min	100% colour, 75.39% COD	2.85 kWh m ⁻³ /0.304 US\$ m ⁻³	This study
Iron/Graphite (PC)		pH:3, I:7.5 mA cm ⁻² , t:120 min	89.41% colour, 74.28% COD	2.34 kWh m ⁻³ /0.249 US\$ m ⁻³	
Graphite/Graphite (EO + Fe ²⁺)		pH:3, I:7.5 mA cm ⁻² , t: 40 min. Fe ²⁺ : 30 mg L ⁻¹	99.9% colour, 96.38% COD	0.54 kWh m ⁻³ /0.199 US\$ m ⁻³	
Iron/Graphite (PC + H ₂ O ₂)		pH:3, I:7.5 mA cm ⁻² , t: 40 min. H ₂ O ₂ :400 mg L ⁻¹	99.9% colour, 90.63% COD	0.62 kWh m ⁻³ /3.466 US\$ m ⁻³	

EF system in terms of COD removal, sludge formation and energy consumption performance.

The cost of the wastewater treatment process is one of the most important parameters. In this study, the operating cost was defined as the overall cost of electrical energy and the cost of chemicals (H₂O₂ and Fe²⁺) per m³ of treated textile wastewater. The cost of 1 kWh of energy consumption was approximately 0.1057 US\$ (according to the Turkish Electricity Distribution Company). Tables 2 and 3 illustrate energy consumption and cost analysis of these processes for the optimum conditions, respectively. The energy consumption per 1 m³ of textile wastewater, EO, PC, PC + H₂O₂, and EO + Fe²⁺ were calculated as 2.85, 2.34, 0.62, and 0.54 kWh for optimum conditions, respectively. Among all the processes applied, the most efficient one in terms of COD removal performance, energy, and cost is the (EO + Fe²⁺) system. As can be seen in Table 3, the additional cost of externally added H₂O₂ has made the PC + H₂O₂ system a very expensive process even though the energy cost is low. A detailed comparison of the electrochemical

studies available in the literature on real textile wastewater and dyed wastewater is presented in Table 4.

7 Conclusion

In the study, the real wastewater, resulting from the textile industry, was treated using graphite/graphite and iron/graphite electrodes for EO and PC, respectively. In addition, EO + Fe²⁺ and PC + H₂O₂ processes were evaluated as an alternative to improve COD degradation and all processes (EO, PC, EO + Fe²⁺ and PC + H₂O₂) were compared with each other. At the determined optimum operational conditions, the electrochemical processes achieved total discoloration of 100, 89.41, 99.9 and 99.9%, and COD degradation of 75.39, 74.28, 96.38, and 90.63%, using EO, PC, EO + Fe²⁺ and PC + H₂O₂, respectively. At the same time, the electrochemical processes calculated energy consumption of 2.85, 2.34, 0.54, and 0.62 kWh m⁻³ using EO, PC, EO + Fe²⁺ and PC + H₂O₂, respectively. From all these findings, it can be concluded

that the $\text{EO} + \text{Fe}^{+2}$ process is preferable to other processes in terms of COD, colour removal, and energy consumption in textile industry wastewater. It was concluded that the $\text{EO} + \text{Fe}^{2+}$ process with graphite/graphite electrodes is an economical and efficient alternative for the treatment of wastewater from the real textile industry, with almost no sludge production.

8 Conflict of Interest

The authors declare that they have no conflict of interest.

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Data availability The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

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