Application and Performance Evaluation of Chemical Coagulation, Electrocoagulation, Electro-Fenton and Anodic Oxidation Processes in the Treatment of Glass Fiber Manufacturing Wastewater

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ARTICLE INFO

ABSTRACT

This study investigated the oxidation of wastewater generated during the production of glass fiber manufacturing material, which contains high organic carbon (18.32 g/L) and has a pH of 8.8, by chemical coagulation, electrocoagulation and electro-advanced oxidation techniques. It was determined that the total organic content (TOC) of wastewater was reduced by 53% using the chemical coagulation method. After electrocoagulation with Al/Al electrode pair for 300 minutes at 500 mA, 73% TOC removal was achieved at pH 8.8. While 50% TOC removal was completed in 2 h at 400 mA in electrocoagulation with Fe/Fe electrode pair, 71% TOC removal was obtained in the combined electrocoagulation/electro-Fenton process by adding hydrogen peroxide to the medium under the same conditions. In addition, it was also observed that the success of the anodic oxidation methods alone was lower. The electro-Fenton application after electrocoagulation was effective and provided 78% TOC but required work at pH 3 and a longer treatment time.

1. Introduction

Glass fiber-based insulations are widely used in all areas of industry for thermal, acoustic and fire protection. In producing these products, phenol-formaldehyde resins are used as binders and add mechanical strength [1]. Depending on the application, the binder content of such insulation products is between 4.5 and 13.8% phenolic resin by weight of fiber [2]. The presence of phenol-based organic pollutants encountered in the production effluent created by such enterprises is a significant problem. Phenol is toxic to fish (1-2 ppm) and aquatic organisms (10-100 ppm) even at the lowest dosages [3]. The presence of phenol in the environment is also a cause of acute toxicity for humans [4].

Water is one of the most essential and fundamental resources for the sustainability of life on Earth. However, drinkable or usable water resources (in agriculture) are decreasing daily due to population growth, rapid industrialization, and urbanization. Water pollution affects the environment and poses a problem in reducing biological diversity. It causes severe diseases and even loss of life in humans and living things [5, 6]. Therefore, the protection of aquatic resources is one of the foremost priorities.

The reports published by the World Economic Forum (Weforum) in 2019 and 2020 stated that water scarcity would be the most significant global risk in the next decade. To overcome this problem, it is essential to convert industrial wastewater into reusable properties, at least in the process or irrigation [7]. Purification and reuse of wastewater in the world have been becoming more and more important every year. For example, only 860 Mm^3/year of treated wastewater was reused in California in 2010, whereas more than 80% (3440 Mm^3/year) of total

wastewater (4300 Mm^3/year) was discharged into the ocean. It is planned to reuse 2470 Mm^3/year in 2030 [8]. More than 500 Mm^3 of treated wastewater per year in Spain is currently reused and is expected to reach 1000 Mm^3/year [9]. More than 80% of treated wastewater effluent in Israel is reused mainly for agricultural irrigation. In Singapore, a significant part of the country's current water needs is provided by the water obtained from the national treatment project they created to process wastewater, and this value is expected to increase over 50% by 2060 [10]. In this context, it is important to design effective and low-cost treatment processes for the treatment of industrial wastewater containing persistent organic pollutants.

Chemical coagulation-flocculation processes, which include various chemical and natural coagulants, are frequently used in conventional water treatment processes. Natural (or biocoagulant), inorganic and organic polymers can be used as coagulants. The most effective inorganic coagulants are generally divalent and trivalent metallic ions (usually Fe(II)/(III) and Al(III)) [11]. Many developed electrochemical technologies have been used frequently in the last few decades to remove various pollutants from wastewater.

The high energy efficiency, cost-effectiveness, and application ease of these techniques make them remarkable in different wastewater applications [12]. Electrocoagulation and electro-oxidation have an important place in these electrochemical processes [13, 14]. In electrochemical coagulation, Al^{3+} and Fe^{2+}/Fe^{3+} ions are formed in situ by metal anode oxidation such as Fe or Al, while hydrogen gas and hydroxide ions are released at the cathode. Thanks to the coagulation agents formed by hydroxide ions and metal ions, the pollutants in the wastewater are effectively precipitated and removed [15].

Electrochemical advanced oxidation processes (EAOPs) are a method based on using radical oxidizing agents to oxidize pollutants. Among the strongest oxidizing radicals used is the hydroxyl radical ($^{\bullet}$OH, E°=2.80 V/SHE). Recently, studies for removing organic pollutants

from wastewater by sulfate radical ($SO_4^{\bullet-}$, E°=2.50-3.10 V/SHE) have increased remarkably [16]. Two of the methods that have an important place among EAOPs are the electro-Fenton (EF) and anodic oxidation (AO) processes used in this study. In the EF process, hydroxyl radicals are produced by the Fenton reaction between Fe^{2+} ions and hydrogen peroxide which is produced in situ by oxygen reduced on the cathode in an acidic environment [17]. In the AO process, hydroxyl radicals are produced on the surface (quasi-free) of the anode with a high O_2 evolution overpotential, such as Boron Doped Diamond (BDD) [18].

The aim of this study is to design treatment processes that will reduce the organic and inorganic pollution content of wastewater with high phenol content and evaluate them together with their performance. For this purpose the treatment of the wastewater was achieved by chemical coagulation (CC), electrocoagulation (EC) (using Al/Al and Fe/Fe electrode pairs), electrocoagulation/electro-Fenton (EC/EF) combined system, electro-Fenton (EF) and anodic oxidation (AO) processes. The effectiveness of the applied methods was determined by calculating the total organic carbon (TOC) and inorganic carbon (IC) removal.

2. Experimental

2.1. Material

Wastewater was obtained from a company that produces insulation materials in Mersin province. Hydrogen peroxide and iron(III) chloride were purchased from Merck.

2.2. Methods

2.2.1. Chemical coagulation process

Different volumes (2.5, 5.0, and 10 mL) of $FeCl_3$ solution (10 mg/mL) were added to the 100 mL wastewater sample and mixed rapidly for 2 min at a stirring speed of 150 rpm. Then it was kept for 25 min at 40 rpm for flocculation. The solution (mixture) was left to rest for 25 minutes and after the precipitation was complete, it was filtered with Whatman 54 filter paper.

2.2.2. Electrocoagulation processes

The electrocoagulation process was performed using Al/Al electrode pair (7 × 3 cm) or Fe/Fe electrode pair (10 × 5 cm) with a distance of 2 cm. The wastewater was kept at 8.8 which is its own pH value. If necessary, the pH value of the wastewater was adjusted with a 3 M H_2SO_4 solution. In the EC processes using Al/Al and Fe/Fe electrode pairs, 200 and 400 mL of wastewater were used, respectively. After EC methods were carried out using a DC power supply (MHC, China) at different currents, the precipitated part was separated by a filter and weighed, and the total organic carbon and inorganic carbon content of the filtrate were determined.

2.2.3. The simultaneous process: electrocoagulation and electro-Fenton

Electrocoagulation experiments with Fe/Fe electrode pair (10 × 5 cm) with a distance of 2 cm were performed in 400 mL wastewater. If necessary, the pH value of the wastewater was adjusted with a 3 M H_2SO_4 solution. The wastewater sample was filtered after 2 h of EC and then used in the EF process. For the combination of the electrocoagulation and the electro-Fenton process, H_2O_2 was added to the solution with a molar ratio of H_2O_2/Fe to 5 [19]. The total amount of hydrogen peroxide to be added, determined for each experiment, was added to the solution in parts every ten minutes. Before the total organic carbon analysis, the excess H_2O_2 remaining in the environment was removed by adding Na_2SO_3. Before each use of the electrodes, they were washed in 35% (v/v) HCl acid solution for 2 min and dried in an oven.

Fe ion mass passing into solution during EC was determined by Eq.(1) according to Faraday's law.

$$m_{Fe} = \frac{IM_At}{nF} \tag{1}$$

where I: current (A), t: time (s), M_A: Fe atomic mass (55.85 g/mol), n: number of electrons (2), F: Faraday constant (96487 C) [19].

2.2.4. The sequential process: electro-Fenton after electrocoagulation

The Electro-Fenton experiments were performed using the filtrate-obtained EC with Fe/Fe pairs at 400 mA. In EF process, edge plane pyrolytic graphite (EPPG, Momentive PG plate UEK, USA) was used as the cathode, while Pt (Aldrich, 9 cm^2) was used for the anode with a distance of 2 cm. The 400 mL solution pH was adjusted to 3.0 with 3 M H_2SO_4 solution and a constant current of 400 mA was applied for 4h. Starting 10 min before EF application, the wastewater was saturated with O_2 gas throughout the process.

2.2.5. Anodic oxidation method

In the AO method, 316L stainless steel cathode and BDD anode (Condias, 5×7 cm) electrodes were used. The electrodes were placed in the cell containing 400 mL of wastewater at a distance of 2 cm from each other. In addition, 3 different currents were used, 180, 350 and 530 mA, respectively, during the AO processes, which lasted 8 h. Energy consumption due to TOC removal in AO was calculated [20] using the following Eq. (2):

$$Energy\ consum.(kWh/L) = \frac{E_{cell}It}{1000V_{ww}} \tag{2}$$

In equation I, current (A); E_{cell} is cell potential (V); t is the electrolysis time (h); V_{ww} represents the volume (m^3) of wastewater.

2.3. Analyses

The organic and inorganic content of glass fiber manufacturing wastewater were analyzed and its physicochemical properties are given in Table 1.

Table 1. The physicochemical properties of the wastewater

Parameters	Value
pH	8.5-8.8
Temperature	25-35 °C
Total solid matter	24.75% (w/w)
Ash	2.11% (w/w)
Conductivity	18.83 mS/cm
Solved organic carbon	18.32 g/L
Solved inorganic carbon	8.19 g/L
Total phenol	0.3-0.5% (w/w)
Free phenol	0.02-0.08% (w/w)
Formaldehyde	0.04-0.12% (w/w)
Ammonia	0.07-0.20% (w/w)
Others (oil, etc)	0.6-0.8% (w/w)
Cl^-	13.94 mg/L
NO_3^-	0.74 mg/L
PO_4^{3-}	0.61 mg/L
SO_4^{2-}	25.05 mg/L
Na^+	50.96 mg/L
NH_4^+	22.39 mg/L
K^+	0.33 mg/L
Ca^{2+}	0.74 mg/L
Mg^{2+}	3.82 mg/L

Ion analysis in wastewater was determined by Metrohm Brand Ion chromatography device with a conductivity detector and Metrosep A Supp 5 (150/4.0) column. Na_2CO_3 (6 mM) / $NaHCO_3$ (12 mM) mobile phase was used for anion analysis at a flow rate of 0.70 mL/min, and the HNO_3 (1M) mobile phase at a flow rate of 0.90 mL/min for cation analysis.

Total phenol determination in wastewater was performed spectrophotometrically. In summary, 2.5 mL of NH_4OH (0.5 N) solution was added to 100 mL of phenol standard solutions and the sample, and the pH was adjusted to 7.9±0.1 with phosphate buffer. 1 mL of 4-amino antipyrine (2 g/100 mL) solution and 1.0 mL of $K_3Fe(CN)_6$ (8 g/100 mL) were added to the mixture, respectively. After waiting for 15 min, phenol content was determined at 500 nm (ASTM D1783-01).

A total organic carbon analyzer (Shimadzu, TOC-L) was used in the carbon analysis of wastewater and samples taken after the applied processes. Samples were taken at different times, and after filtering, the total organic carbon (TOC) and inorganic carbon (IC) contents were analyzed.

3. Results and Discussion

3.1. Chemical coagulation results

The decrease in TOC and IC values in chemical coagulation experiments performed by adding different volumes of $FeCl_3$ solution with a concentration of 10 mg/mL to the wastewater is given in Figure 1. In this method, the removal of organic and inorganic species due to adsorption is carried out on $Fe(OH)_3$ flocs, which are formed depending on the pH value of the wastewater with Eq. (3) [21].

$$FeCl_3 + 3OH^- \rightarrow Fe(OH)_3 + 3Cl^- \qquad (3)$$
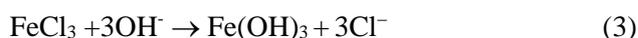
After 2.5, 5, and 10 mL of coagulant were added in the chemical coagulation process, 0.9765, 1.3542, and 1.7265 g of solid waste were obtained for 100 mL of wastewater, respectively.
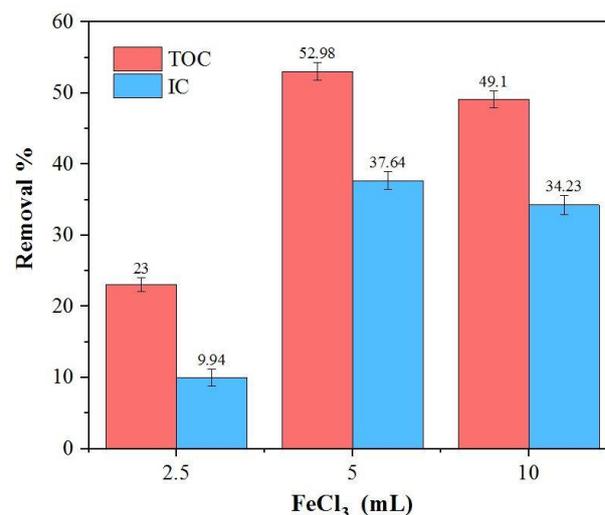
Figure 1. TOC and IC removal by chemical coagulation method (pH= 8.8, V=100 mL)

When 5 mL of $FeCl_3$ is used as a coagulation agent, it is seen that 53% and 38% of dissolved TOC and IC content in wastewater can be removed. However, while 2.5 mL of coagulation agent is not sufficient, the addition of 10 mL of coagulant does not seem to have a positive effect on TOC removal.

When Gasmi et al. (2022) compared textile wastewater treatment with EC and CC processes, they stated that although the EC operation cost is high, the treated water is more in line with Tunisian environmental discharge standards. EC technology can also be integrated into different

wastewater management systems for the complete mineralization of wastewater [21].

3.2. Electrocoagulation and the simultaneous process: electrocoagulation and electro-Fenton results

In the EC method, Al and Fe electrodes were used as sacrificial electrodes. Since the conductivity of the wastewater was at the appropriate value, it was carried out without adding any electrolyte. TOC removals in wastewater after EC with Al/Al electrode pair were obtained as 55, 67, 73, and 76% at 200, 300, 500, and 800 mA, respectively (Figure 2).
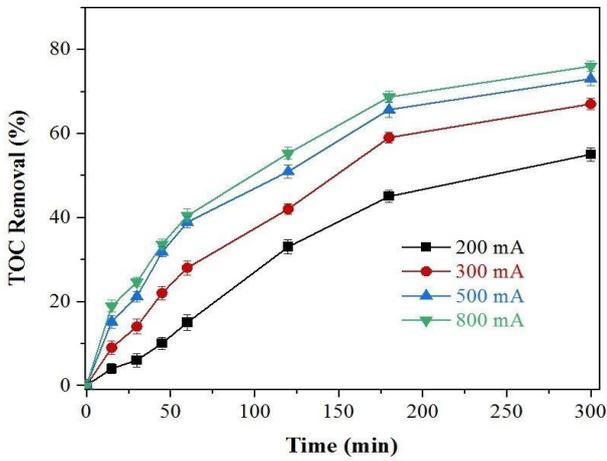

Figure 2. TOC removal results by electrocoagulation method with Al/Al electrode pair (pH=8.8, V= 200 mL)

As a result of oxidation at the anode due to the applied current, Al^{3+} ions pass into the solution. Since the pH value of the wastewater is basic, the $Al(OH)_3$ structures formed cause coagulation/flocculation and then collapse (Eq. (4,5)). In this way, pollutants including suspended solids in the wastewater are separated by precipitation. TOC removal was determined as 73% and 76% for 500 and 800 mA, respectively.

$$Al \rightarrow Al^{3+} + 3e^- \qquad (4)$$

$$Al^{3+} + OH^- \rightarrow Al(OH)_3(s) \qquad (5)$$
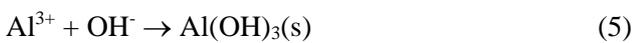
The reaction at the cathode (in alkaline solution) is given as follows [22]:

$$2H_2O + 2e^- \rightarrow 2OH^- + H_2 \qquad (6)$$
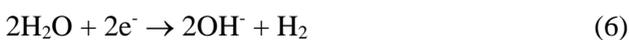
The amount of solid that precipitates out of the wastewater as a result of the applied EC method and the amount of Al that is oxidized on the Al anode surface and passed into the solution are shown in Table 2.

Table 2. Solid amount and energy consumption values depending on the method applied

| Method	Solid (g)	Energy Consumption (kWh/m^3)
EC-Al/Al, 5h pH 8.8, 200 mA	1.542	0.0120
EC-Al/Al, 5 h pH 8.8, 300 mA	2.673	0.0225
EC-Al/Al, 5 h pH 8.8, 500 mA	5.228	0.0438
EC-Fe/Fe, 2 h pH 8.8, 400 mA	5.280	0.0054
EC/EF-Fe/Fe, 2 h pH 8.8, 400 mA	4.681	0.0050
EC-Fe/Fe, 2 h pH 5.0, 400 mA	5.182	0.0055
EC/EF-Fe/Fe, 2 h pH 5.0, 400 mA	6.700	0.0054

As the amount of applied current increased, the amounts of solid precipitates and Al increased. Similarly, 70-77% TOC and complete color removal were achieved in the previous studies performed EC with Al/Al pair for olive mill wastewaters. This method allowed lower energy consumption with lower sludge formation than chemical coagulation [22, 23].

TOC removals after EC and combined EC/EF methods using Fe/Fe electrode pair as electrode pair are shown in Figure 3. While iron ions are formed at the anode, $Fe(OH)_3$ flocs are formed depending on the basic property of the wastewater with Eqs. (7) and (3) [24]. TOC removal in wastewater after 2 h of EC at pH 5.0 and 8.8 were determined as 18.8% and 49.8%, respectively.

$$Fe \rightarrow Fe^{3+} + 3e^- \qquad (7)$$
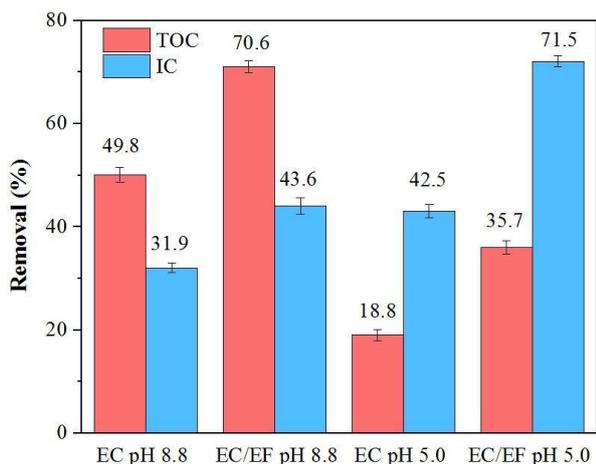
Figure 3. The efficiencies of electrocoagulation and electrocoagulation/electro-Fenton methods using Fe/Fe electrode pair (I= 400 mA, t= 2 h)

Recently, the general view of using the EC process is to employ it as an integrated step combined with other methods [14, 19 ,25]. In the combined EC/EF method with the addition of hydrogen peroxide, the TOC removal efficiencies at the same pH values increased to 35.7 and 70.6, respectively. In the EF method, as a result of Fenton reaction (Eq. (8)), hydroxyl radicals were formed and increased mineralization.

$$Fe^{2+} + H_2O_2 \rightarrow {}^\bullet OH + Fe^{3+} + OH^- \qquad (8)$$
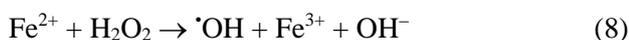
In the EC process, the types of iron hydroxocomplexes show different distributions depending on the pH of the solution [12, 14]. When the pH > 5.5, $Fe(OH)_2$ and $Fe(OH)_3$ begin to precipitate, and as the pH increases, their formation rate increases. Therefore, more effective TOC removal was achieved as a result of EC at pH 8.8. On the other hand, as the EC efficiency decreases at pH 5.5 and the possibility of Fenton and Fenton-like reactions between Fe^{2+}/Fe^{3+} ions and H_2O_2 increases. TOC removal occurs mostly through radical species (${}^\bullet OH/HO_2{}^\bullet$), but the TOC removal efficiency was obtained lower. Also, the higher IC removal percent was observed at more acidic pH values, as the decomposition of inorganic species in wastewater occurred more easily.

It is seen that the energy consumption values are close to each other in EC and EC/EF combined methods (Table 2).

3.3. The sequential process: electro-Fenton after electrocoagulation results

After EC treatment at 400 mA for 2 h, the obtained wastewater was filtered. The filtrate pH value was adjusted to 3.0 and oxidation was carried out by EF method at 400 mA using Pt/EPPG ano de/cathode pair (Figure 4). The hydroxyl radicals are produced as a result of the Fenton reaction between hydrogen peroxide (formed from the reduction of O_2 at the cathode) and Fe ions (formed by EC methods) due to Eqs. (8,9) [26].

$$O_2 + 2H^+ + 2e^- \rightarrow H_2O_2 \qquad (9)$$

While 49.8% TOC removal was achieved as a result of the EC process, a total TOC removal of 78.2% was achieved after 4 h of oxidation with the EF method. With the EC/EF combined system with external H_2O_2 added, 70.8% TOC removal was achieved at pH 8.8 and after 2 h of electrolysis. In addition to the higher TOC removal, the energy consumption increased in the EF application following the EC method. In addition to 0.0054 kWh/m^3 energy consumption in the two-hour EC application, 0.0170, 0.0341, 0.0510, and 0.0680 kWh/m^3 energy consumption in the EF method after 1, 2, 3, and 4 h of electrolysis, respectively, was calculated.
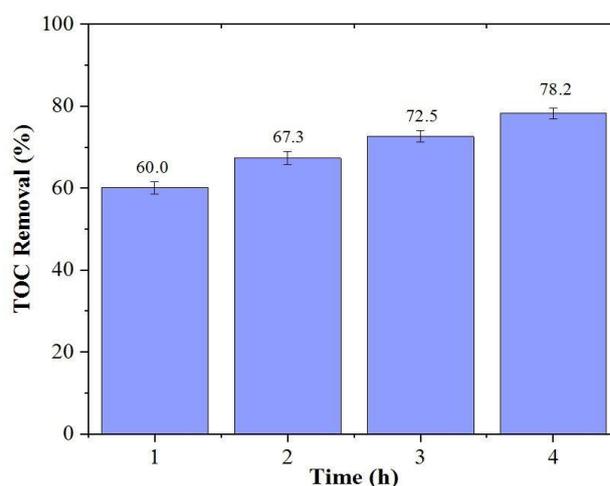
Figure 4. TOC removal by electro-Fenton method after electrocoagulation (I=400 mA, pH 8.8, pH 3)

The effectiveness of the electro-Fenton method for different pollutant groups containing phenol has been demonstrated by many previous studies. These studies have shown that effective oxidation of phenol and phenol-derived organic

pollutant groups can be achieved in low-concentration synthetic solutions with the EF process under appropriate operating parameters, but the time and energy requirement is high for the full mineralization of aliphatic products formed as a result of aromatic ring decomposition [26-28]. Similarly, in this study, after reducing the organic carbon load and suspended solids amount of wastewater with EC, 78% TOC removal was achieved with the EF process, but more time was needed for 100% efficiency.

3.4. Anodic oxidation results

The AO method was directly performed with the BDD anode and 316L cathode as a final method. Figure 5a depicted a rapid decrease in TOC value in the first two hours but then slowed down.

It is seen that TOC removal does not change much when the currents were applied as 180, 350, and 530 mA. For these applied currents, 48%, 51%, and 55% TOC removal were achieved, respectively (Figure 5b).
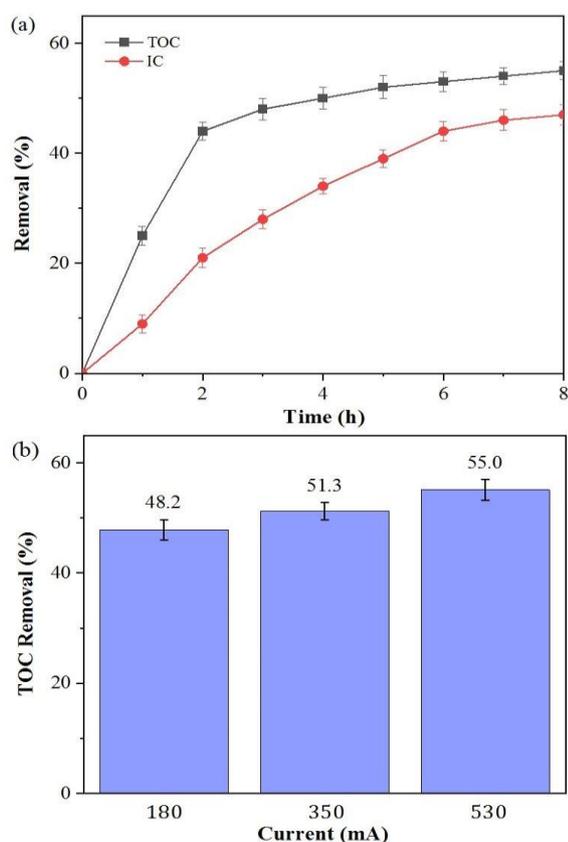
Figure 5. (a) Time-dependent TOC removal in anodic oxidation (I= 530 mA, pH 8.8), (b) the effect of current

As in all electrochemical methods, the selection of the suitable current amount or density is an important parameter for effective organic pollutant removal and low energy consumption in the AO process. As the applied current value increases, TOC removal by the AO process is significantly accelerated. However, the reaction rate decreased at a high current values, due to the rapid consumption of reactants and the side reaction of oxygen evolution [29].

In addition, it should be noted that the high performance results to be obtained in the AO process are too complex to be explained through a single parameter. It should not be forgotten that the synergistic effect of many parameters such as the type of pollutants, the preferred electrode material, the type of electrolyte, and the applied current or potential will affect the efficiency obtained as a result of the AO process. Therefore, increasing the applied current alone may not be sufficient to dramatically increase the degradation performance of pollutants [16].

While quasi-adsorbed hydroxyl radicals are formed on the BDD electrode surface (Eq. (10)), chlorine ions and sulfate ions in wastewater can be converted into oxidants such as persulfate and chlorine gas by oxidation (Eqs. (11,12)) [30, 31]. In this study, anodic oxidation did not provide sufficient mineralisation although the wastewater contained sulphate and chloride ions. This may be because the anode surface becomes passive due to different species in the waste.

$$BDD + H_2O \rightarrow BDD(^{\bullet}OH) + H^+ + e^- \qquad (10)$$

$$2SO_4^{2-} \rightarrow S_2O_8^{2-} + 2e^- \qquad (11)$$

$$2Cl^- \rightarrow Cl_2 + 2e^- \qquad (12)$$

4. Conclusions

This study investigated chemical coagulation and various electrooxidation methods to treat a real wastewater sample with high total organic carbon and suspended solids content. It was observed that chemical and electrochemical coagulation methods reduced the total suspended solids content and a significant amount of TOC content in the wastewater. In addition, the electrocoagulation method was kept shorter and

tested together and sequentially with the electro-Fenton process. Without changing the wastewater's alkaline pH value (8.8), 71% TOC removal efficiency was achieved in a short period (2 h) when H_2O_2 was added from the outside for the simultaneous implementation of the Fe/Fe electrode pair and the EC/EF method. The same energy consumption was spent with the EC method alone, while more organic pollution was removed. When the EF method was applied in the filtrate after the EC process at pH 3 to produce in situ H_2O_2, 4 h of processing time and an extra 0.0680 kWh/m^3 energy consumption were required for 78% TOC removal. The anodic oxidation method did not give an effective result in wastewater treatment, and it revealed the necessity of EC as a pre-treatment. When the data obtained as a result of these studies are evaluated, it is seen that the simultaneous or sequential use of different electrochemical method such as EC and electro-Fenton process will increase the yield of real wastewater with high organic pollution load.

Article Information Form

Funding
The authors has no received any financial support for the research, authorship or publication of this study.

Authors' Contribution
The authors contributed equally to the study.

The Declaration of Conflict of Interest/ Common Interest
No conflict of interest or common interest has been declared by the authors.

The Declaration of Ethics Committee Approval
This study does not require ethics committee permission or any special permission.

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