Optimization of Operating Parameters in the Removal of Synthetic Textile Dyestuff with the Electrocoagulation Process

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ABSTRACT: *Electrocoagulation (EC) is one of the most effective electrochemical wastewater treatment techniques for removing color and organic pollutants from wastewater, and reducing sludge formation. In this study, the removal of Remazol Ultra Red RGB (reactive red 239) dye, which is used for commercial purposes, by the EC process was investigated. For this purpose, an electrochemical reactor was designed using monopolar parallel connected aluminum and iron electrodes. The effect of operational parameters such as the initial pH of the solution, current density, and electrolysis time were investigated to achieve higher color, Chemical Oxygen Demand (COD), and turbidity removal efficiency. The optimum conditions of the EC process were determined by evaluating the data obtained as a result of the experimental studies. It was observed that the removal efficiency increased with the increase of the electrolysis time and stabilized after 20 min. The optimum experiment conditions for the aluminum electrode were pH:3, current density of 50 A/m^2, and conductivity of 250 µS/cm, for the iron electrode was pH: 5, current density of 75 A/m^2, and conductivity of 500 µS/cm was found as. In all studies, the mixing speed was chosen as 250 rpm. As a result of this study, 95.49-99.94% color, 89.34-66.83% COD, and 92.18%-83.15% turbidity removal efficiencies were obtained with aluminum and iron electrodes under optimal conditions. Under optimum conditions, electrical energy consumption was calculated as 11.48 for Al, 6.60 kWh/m^3 for Fe, and the energy consumption 0.56, 0.46 kg/m^3. As a result of the experimental studies, high removal efficiencies were obtained in color, COD and turbidity removal with the EC process. As a result, it was concluded that EC treatment is an effective method for the purification of synthetic textile dyestuffs.*

KEY WORDS: *Textile dye; Electrocoagulation; Decolorization; COD removal; Electrode material; Operating parameters.*

INTRODUCTION

Wastewater from textile industries has allergic, mutagenic, toxic or carcinogenic properties that pose a serious problem for environmental protection [1,2]. Textile wastewater is characterized by high color content,

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high fluctuating pH and temperature due to the presence of unfixed dyes [3]. In addition, textile wastewater is among the important pollutants for the environment because of its properties such as heavy metals, corrosive substances, inorganic salts, oils, total phosphate, surfactant, Absorbable Organic Halogens (AOX), suspended solids, high concentration chemical oxygen demand [4,5]. In particular, the coloring matter is the most important pollutant in textile wastes and should be removed before being discharged to the receiving environment by applying appropriate treatment technologies to prevent its negative effects on the environment. Textile industry processes have different steps such as sizing, washing, bleaching, dyeing, printing, mercerizing, and finishing in pretreatment, dyeing, and final processes. These steps consume large amounts of pure water and produce large amounts of wastewater [6,7]. In Table 1, the characteristics of wastewater produced from different types of textile industry and wastewater discharge standards stipulated by legal authorities are given.

Depending on the resolution and chemical properties, dyes, which are the main pollutant class, can be widely classified as anionic (acidic, reactive, and direct according to painting characteristics), cationic (basic dyes), and nonionic (distributed) [10,11]. Reactive dye groups have increased their use in the textile industry in recent years because they have a very high solubility in water [12]. Reactive dyes are not toxic, but under anaerobic conditions, they can degrade into aromatic amines that are potentially carcinogenic and toxic. Reactive dyes are stable and anionic and resist light [13,14]. Previous research has shown that conventional methods for textile wastewater treatment are inadequate and not economically viable due to the toxic substances they contain. Some products are not biodegradable and can also produce large amounts of sludge and toxic materials. Due to the wide variety of components of textile waste, these methods are often inadequate to meet receiving ambient discharge standards [15,16]. Therefore, there is a need for different treatment applications and the development of additional methods. Adsorption [17,18], biological treatment [19,20], electrochemical oxidation [21,22], coagulation and flocculation [23,24], nanofiltration-ultrafiltration [25,26], membrane distillation [27,28], reverse osmosis [29,30], enzymatic color removal [31] and advanced oxidation [32,33] are the main methods used for the removal of dyes from colored wastewater. The choice of applied technology depends on the type and characterization of wastewater. Electrooxidation (EO), Electroflotation (EF), and Electrocoagulation (EC) processes are commonly used in electrochemical wastewater treatment. These processes can be in a system together or separately and pollutants in the wastewater, the oxidation, adsorption, or separation by accumulation on the surface is provided. Among the electrochemical wastewater treatment processes, it is EC that has more applications. EC is an emerging technology in water and wastewater treatment as it combines the benefits of coagulation, flotation, and electrochemistry [34, 35]. It removes particles from wastewater by destabilizing/neutralizing the repulsive forces that keep the particles suspended in water. When the repulsive forces are neutralized, the suspended particles will form larger particles that can settle to separate more easily from the water [36]**.** Since no chemicals are added, and there is no possibility of secondary pollution due to the high concentration of chemicals. The gas bubbles produced from EC float the contaminants above the solution, facilitating their removal so that they can be easily collected. Even the smallest colloidal particles are removed by the EC as the applied electric current accelerates disintegration and facilitates coagulation. Waste water treated by EC yields clear, colorless and odorless water. The flocs formed by EC are much larger and more stable, so they are easily separated during filtration. In EC, coagulants are produced in situ by electrolytic oxidation of a suitable anode material, resulting in much less sludge formation and the sludge formed is more stable and non-toxic [37-40]. In addition, the compact treatment system offers many advantages such as no need for chemical coagulant addition, simple design, short electrolysis time and relatively low capital costs [41,42]. Studies on the treatment of textile wastewater have shown that color, COD, turbidity and dissolved solids are effectively removed by the electrocoagulation process [43-47]. The most important feature that distinguishes electrochemical wastewater treatment processes from each other is the shape and structure of the electrochemical process. Electrodes are classified into cathode and anode according to the type of reaction occurring on the surface. When a potential from an external power source is applied, the anode material is oxidized, while reduction of elemental metals occurs at the cathode.

Table 1: Characteristics of textile industry wastewater [8,9].

Characterictics	Scouring	Bleaching	Mercerizing	Dyeing	Composite	Discharge limit into inland water (WPCR-Turkey)
pH	10-12	8.5-11	8.0-10	9.0-11	8.0-10	6.0-9.0
TDS (mg/L)	12,000-30,000	2500-11,000	2000-2600	1500-4000	5000-10,000	-
TSS (mg/L)	1000-2000	200-	100-400	50-350	100-700	100-300
COD (mg/L)	10,000-20,000	1200-1600	250-400	400-1400	250-8000	200-300
Color	-	-	Highly colored	Strongly colored	Strongly colored	Pt-Co 260

Oxidation and reduction reactions in the EC process take place at both electrodes. The presence of oxygen and neutral pH is necessary to obtain an appropriate reaction rate [48-51]. Depending on the environmental conditions, metal ions dissolved from the electrodes produce metal-polymer complexes that coagulate. Particles coagulating attract and adsorb micro colloidal particles and ions contained in wastewater. In the EC process, oxidation, coagulation and flotation events occur together in the removal of pollutants, unlike conventional chemical coagulation [52].

With the EC process, wastewater treatment can be carried out in intermittent and continuous operation mode. Optimizing the EC operation in start-up mode is much easier than in continuous-run mode. But with continuous flow, the electrolysis time and energy consumption are reduced, and, accordingly, operating costs and sludge production. In addition, as the flow rate increases, the turbulence density and therefore the mass transfer between the organic matter and the electrodes increase, which has a positive effect on the COD removal efficiency, which is an important pollutant indicator in wastewater [53,54]. Various factors such as potential difference (IR) drop between electrodes, accumulation of gas bubbles and mass transfer must be considered when designing the EC reactor. These aspects will affect the COD removal efficiency, which is an important pollutant indicator in wastewater. The continuous mode of operation supports an efficient flow distribution within EC reactors, mass transfer during electrolysis, current and potential distributions, and electrolytic energy consumption [55].

Electrode configuration is also an important element in the EC process. The selection of the appropriate electrode and electrode connection affects the treatment efficiency and cost. The arrangement of the electrodes can be monopolar or bipolar (Table 2) [56,57]. The use of unipolar electrode EC cells in parallel or series configurations improves performance. In the MonoPolar-Serial (MP-S) connection, each electrode pair (anode-cathode) is connected internally where it is not connected to the external electrodes, whereas in the monopolar-parallel (MP-P) connection, the dedicated sacrificial anode is directly connected to with other anode [58,59]. In parallel configurations, a relatively low potential difference (same voltage) is provided as the current density is divided between the electrodes. In contrast, in series systems, each electrode pair (anode-cathode) is connected to each other, resulting in the same current density and an additive voltage. Therefore, removing pollutants (e.g. COD) from wastewater using the EC technique with bipolar configuration requires more energy than monopolar configuration. Sometimes, however, monopolar configurations exhibit negligibly lower removal efficiency than that of the bipolar configuration, but a significant reduction in power consumption. The bipolar electrode arrangement is always in serial connection [60]. The high removal efficiency shown in the bipolar configuration is attributed to the higher current densities (for the same amount of current density) found in the bipolar electrodes. As interpreted, high current density values make it easier to remove organic substances in wastewater. This latter results in a more anodic electrodissolution compared to that in the monopolar junction. EC reactors using parallel bipolar electrodes show better potential and current distribution (more uniform); then, parasitic reactions decrease in bipolar configurations and their energetic use increases [55].

It has been determined that the electrode connections (MP-P, BP-S, MP-S, BP-P) have different effects on the EC process depending on the targeted pollutant [38,69]. The results showed that the monopolar connection gives much higher current efficiency with lower operating costs compared to the bipolar connection. In a study examining the effect of different electrode junctions on the color and turbidity removal and total treatment cost of EC in textile

Table 2: Applications of EC in the treatment of various industrial wastewater.

Category	Pollutants removed	Reactor operation mode	Electrode material	Electrode configuration	Optimum removal efficiency	Ref.
Textile industry	Color COD	Batch	Anode (Iron) Cathode (Steel)	-	99 % 75 %	[61]
Textile industry	Color COD TOC	Batch	Anode (Iron) Cathode (Iron)	-	99 % 54 % 62 %	[59]
Printing press facilities	COD Color TSS	Continuous	Anode (Aluminum) Cathode (Aluminum)	-	82 % 98 % 85 %	[62]
Cardboard factory	Color COD	Batch	Anode (Iron) Cathode (Steel)	Monopolar	74.7 % 97.5 %	[63]
Textile industry	Color COD TOC	Batch	Anode (Aluminum) Cathode (Iron)	Monopolar	EC(Fe)→O_3 99 % EC(Fe)→$_{O3}$ 44.5 % EC(Fe)→O_3 51 % EC(Fe)→O_3 99,51 % EC(Fe)→O_3 62 % EC(Fe)→O_3 18.6 %	[64]
Textile industry	Color	Batch	Anode (Iron) Cathode (Iron)		96.11%	[65]
Leachate	KOİ NH_3 TSS	Batch	Anode (Iron) Cathode (Steel)	-	SBR + EC 98 % SBR + EC 98 % SBR + EC 99 %	[66]
Textile industry	Color COD	Batch	Anode (Iron) Cathode (Iron)	Monopolar	97 % 89 %	[43]
Rainwater	COD Turbidity	Batch	Anode (Iron) Cathode (Iron)	Monopolar	Sup. elect(sea salt) 100 % 100 %	[67]
Greywater	KOİ BOİ NO_3 PO_4	Batch	Anode (Aluminum) Cathode (Aluminum)	Monopolar	70 % 87.5 % 82.7 % 84.7 %	[68]

wastewater treatment, the efficiencies of removal efficiencies were similar for all three junctions. However, the MP-P configuration was evaluated as the most cost-effective connection model [70]. In another study, various electrode designs were investigated to treat palm oil mill wastes using high current density application in the EC process. It has been emphasized that the vertical electrode orientation was carried out with the anode at the bottom, while the horizontal electrode orientation was such that the anode was on the top. The highest removal efficiency was obtained in the MP-S arrangement with vertical orientation, with 74% COD, 70% BOD, and 66% SS removal efficiencies. Although the MP-S arrangement has a higher removal efficiency, it has been highlighted in the economic evaluation to have higher operating costs than the MP-P and BP arrangement [71].

The choice of electrode material in the EC process depends on the type of pollutants and the chemical properties of the electrolyte. If the wastewater contains significant amounts of calcium or magnesium ions, it is recommended to use inert cathode materials such as metal oxide coated titanium and graphite. Various electrode materials such as Al, Fe, Ca, Mg, Ba, Na, Zn, Ag, As, Cd, Cr, Cs, Si and Sr have been used in EC studies [72-79]. These electrodes are multivalent metal electrodes that form metal hydroxide coagulant during EC. Due to their potential to produce iron (Fe) and aluminum (Al) ions in the EC process, low price, non-toxic, generally soluble and coagulation properties, they are widely used electrodes for various applications. In the literature, very successful results have been reported in different pollutant removal studies [80,81]. The use of aluminum and iron electrodes was preferred in our study because it produces colorless wastewater, lower energy consumption, and less metal consumption than when steel electrodes are used. Aluminum is oxidized only in the Al(III) form. Iron, on the other hand, Fe can be oxidized to Fe(II) and Fe(III) forms under suitable conditions, depending on the ambient pH and the electrical potential exerted by atmospheric oxygen or anode oxidation during coagulation (Fig.1) [69,82-83].

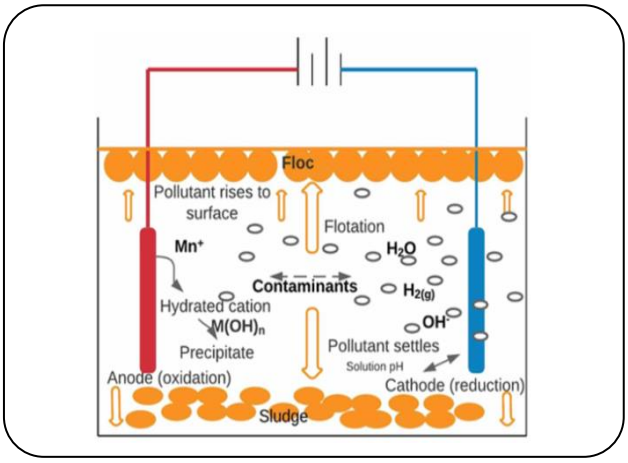

Fig. 1: Bench-scale two-electrode electrocoagulation cell schematic representation [84].

In a study with EC for the treatment of palm oil mill effluent, a new steel wool was used as the electrode and found to be effective in removing contaminants quickly and cost-effectively. In the steel wool application, it was found that the COD, BOD, and SS removal efficiencies (74%, 70%, 66%) were higher than those of iron and aluminum [71]. In another study, in which aluminum and iron electrodes were used, the percentage of COD removal with aluminum electrodes was 93%, and the percentage of removal with iron electrodes was 85%. However, with respect to fats, oils, and grease (FOG) removal, it was emphasized that the removal efficiency of iron electrodes (98%) is higher than that of aluminum electrodes (90%) [85]. The poor performance of iron electrodes on COD removal compared to aluminum electrodes is due to the poor precipitation of the Fe^{2+} ion. Fe^{2+} is the common ion produced in the electrolysis field of iron electrodes. It has high solubility at acidic or neutral pH and can be easily oxidized to Fe^{3+} by dissolved oxygen in water. Although aluminum electrodes show higher COD removal efficiency compared to Fe electrodes in many studies, Al produces a significant amount of sludge [86]. In another study, the removal of Acid red 182 (AR182) azo dye by EC method was investigated in an electrochemical batch reactor. A Box-Behnken Design (BBD) has been used to optimize process-influencing factors such as current density, electrolysis time, and initial pH on dye removal efficiency. Graphical counterplots and response surfaces were used to determine the optimum conditions. Analysis of Variance (ANOVA) has provided a high coefficient of determination (R^2=0.9817, R^2_{adj}=0.9487, and R^2_{pred}=0.7070) and a satisfactory predictive second-order regression model.The optimum current density, electrolysis time and initial pH of the dye solution have found to be 16 mA/cm^2, 14.2 min, and 8, respectively. It has been reported that dye removal efficiency is very low at acidic or very alkaline pH. Under optimum conditions, AR182 and COD removal efficiency were determined as 98.7% and 88.3%, respectively [87]. In another study carried out to remove acid orange 5 from synthetic wastewater by EC method, the effect of operational variables such as current density, initial pH, and electrolysis time, and the initial concentration of the dye was investigated. The results showed that optimum conditions were obtained at initial pH 7, current density 2 mA/cm^2, 60 mg/L Acid orange 5, and reaction time of 60 minutes. At optimum conditions, the removal efficiency of acid orange 5 and COD were 99.3% and 85.5%, respectively. The kinetic study has shown that the removal reaction is first order and the rate constant and half-life of the reaction are obtained [88].

The reactions that take place for iron and aluminum in the electrocoagulation process are as follows [89,90]. Al^{3+} and OH^- ions produced by equation (1) and (2) form various $Al(OH)^{2+}$, $Al(OH)^{2+}$ monomeric types in the pH 4-5 range; if the pH is between 5-6, it forms various polymeric types such as $Al_6(OH)_{15}{}^{3}$+, $Al_2(OH)_{17}{}^{4+}$, $Al_8(OH)_{20}{}^{4+}$. It eventually transforms into $Al(OH)_3$(s) according to complex precipitation kinetics [91,92].

$$\text{Anode}: Al_{(s)} \rightarrow Al^{+3}{}_{(aq)} + 3e^- \quad (1)$$

$$\text{Cathode}: 2H_2O + 2e^- \rightarrow H_{2(g)} + 2OH^-{}_{(aq)} \quad (2)$$

$$\text{Precipation}: Al^{+3}{}_{(aq)} + 3HO^-{}_{(aq)} \rightarrow Al(OH)_{3(s)} \downarrow \quad (3)$$

In acidic condition the following reactions take place [1,3,15,38,40].

$$\text{Anode}: Al_{(s)} \rightarrow Al^{+3}{}_{(aq)} + 3e^- \quad (4)$$

$$\text{Precipation}: Al^{+3}{}_{(aq)} + 3H_2O \text{®} Al(OH)_{3(s)} \downarrow \quad (5)$$

$$\text{Cathode}: 3H^+{}_{(aq)} + 3e^- \rightarrow 3/2\ H_{2(g)} \quad (6)$$

Iron ions produced by the electrochemical oxidation of the iron electrode can form polymeric hydroxyl complexes depending on the pH (3.5-7) of the aqueous medium. In the pH range of 7-10, monomeric $Fe(OH)_3$

is formed [93,94]. These hydroxide/polyhydroxide/ polydroxymetal compounds have a strong affinity to cause clotting/adsorption with dispersed/dissolved molecules [61,95]. $Al(OH)_3$ and $Fe(OH)_3$ components display enormous surface areas beneficial to rapid adsorption of soluble organic compounds and capture of colloidal particles and they can be easily removed from the aqueous environment via precipitation or flotation [96].

$$\text{Anode}: Fe_{(s)} \rightarrow Fe^{+3}_{(aq)} + 3e \quad (7)$$

$$\text{Cathode}: 2H_2O + 2e^- \rightarrow H_{2(g)} + 2OH^-_{(aq)} \quad (8)$$

$$\text{Precipation}: Fe^{+2}_{(aq)} + 2HO^-_{(aq)} \rightarrow Fe(OH)_{2(s)} \downarrow \quad (9)$$

In the acidic state, the following reactions occur [44,54,61,69,81,82, 97]

$$\text{Anode}: 4Fe_{(s)} \rightarrow 4Fe^{+2}_{(aq)} + 8e^- \quad (10)$$

$$\text{Cathode}: 8H^+_{(aq)} + 8e^-_{(aq)} \rightarrow 4H_{2(g)} \quad (11)$$

$$\text{Precipation}: 4Fe^{+2}_{(s)} + 10H_2O + O_2 \rightarrow \quad (12)$$

$$4Fe(OH)_{3(s)} \downarrow + 8H^+_{(aq)}$$

Color, COD and Turbidity parameters are the parameters that require meeting the receiving environment discharge standards determined for textile wastewater in Turkey due to the reuse of treated water. In our study, it was aimed to remove the reactive dye solution (reactive red 239) with the EC method, which is thought to be effective in reducing these parameters. For this purpose, for Color, COD and Turbidity removal with EC process; the effects of operational parameters such as pH, current density and electrolysis time, which play an important role in EC process efficiency, have been determined. Reactive dye, which is the subject of the research, is toxic, water-soluble, mutagenic and even more resistant to biological treatment. In addition, these dyes reduce the rate of photosynthesis and the concentration of dissolved oxygen in the recipient's body, are carcinogenic to humans and cause various allergic reactions and anaphylactic shock in exposed persons. These paints are extremely durable and resistant to light and washing, they do not decompose easily [98,99]. As mentioned earlier, it is essential that such dyestuffs are treatment with appropriate methods before they are discharged into the environment.

EXPERIMENTAL SECTION

Material

Reactive dyestuff used in the study; Remazol Ultra Red RGB (Reactive Red 239-RR239) was obtained from the distributor company of DyStar in Turkey. The chemical structure of this dye is presented in Fig. 2 (CI: Reactive Red 239 (RR-239); MF: $C_{31}H_{19}ClN_7Na_5O_{19}S_6$; MW:1136.32 (g /mol)).

Experimental Setup

The electrocoagulation unit consists of a cylindrical glass cell in which the anode and cathode electrode are fixed at certain intervals, a direct current power source, a magnetic stirrer and a synthetic wastewater (Fig.3). Monopolar parallel connected 2 anode and 2 cathode electrodes were used in the reactor in the solution immersed condition. Aluminum and iron plates were used as electrode material. Aluminum plates 99.53% Al; iron plates have 99.32% Fe content. After the electrodes were placed in the electrocoagulation reactor, 750 mL of electrolyte (dye solution) has been put into the reactor. Samples have filtered through double-layer coarse filter paper and subsequently analyzed. Current and voltage control were provided by a digital power supply (GPC 6030D DC power supply). At the end of each experiment, the electrodes and electrochemical cells were held in dilute HCl solution for 1-5 minutes in order to prevent the passivation that may occur in the next study, and then passed through ultra-pure water. 100 mL of HCl of 35% and 200 mL of hexametylenetetramine $(CH_2)_6N_4$) solution of 2.8% were used as cleaning solution. In each set of experiments, electrodes were weighed and noted to determine electrode wear amount (mass loss). For pH adjustment, 0.1 M NaOH or 0.1 M HCl solutions were used. Conductivity adjustment was made with Na_2SO_4.

The maximum absorbance for RR 239 dyestuff was determined to be 516 nm as a result of scanning with a UV-VIS spectrophotometer between 190 nm and 800 nm at 1 nm intervals. It has been observed that there is no change in the maximum wavelength of the dye solution at different pHs. A calibration curve was created using solutions containing 0-100 mg/L RR239 and the relationship between dyestuff concentration and absorbance was determined mathematically. COD measurements were made according to the "Open Reflux" colorimetric method. COD absorbance measurement has been

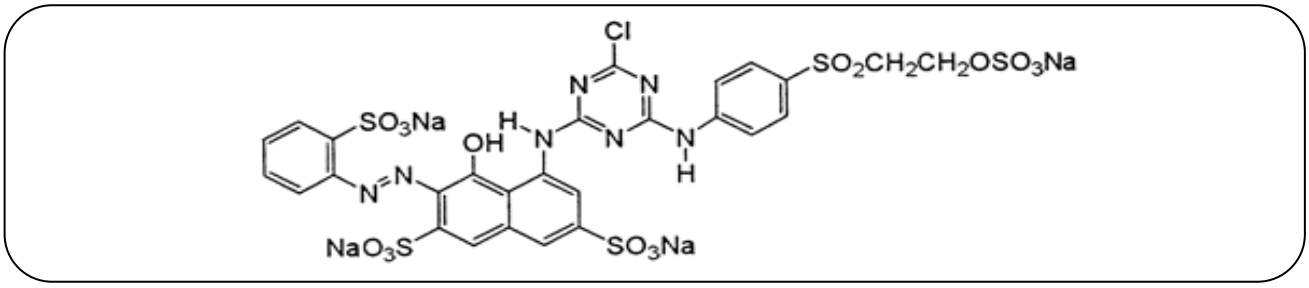

Fig. 2: The chemical structure of the reactive textile dye used.

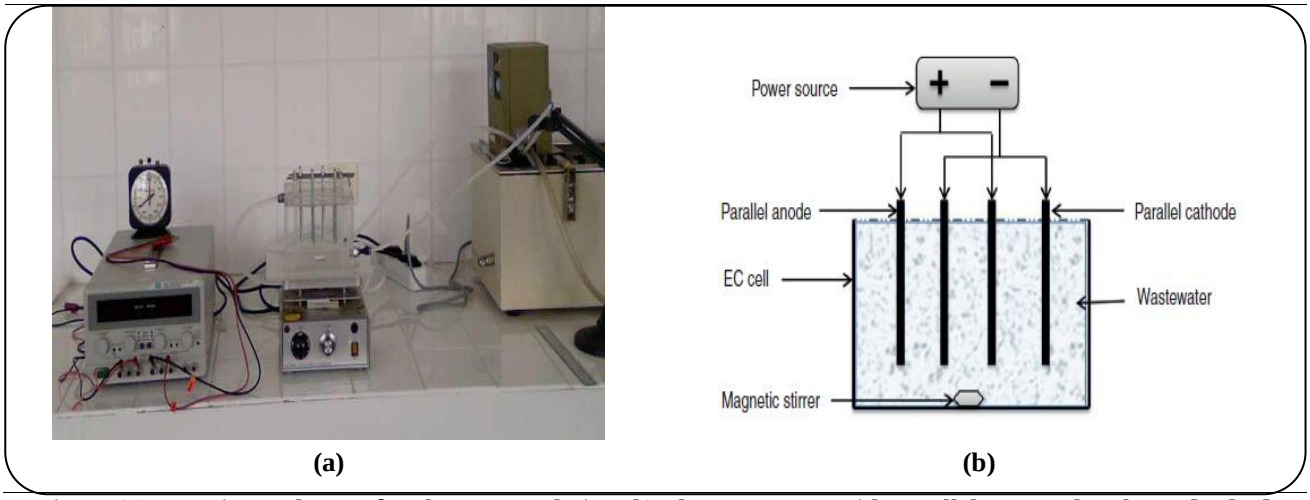

Fig. 3: (a) Experimental setup for electrocoagulation (b) The EC reactor with parallel monopolar electrodes [51].

performed an optimum-one UV-VIS spectrophotometer. The calibration solution has been prepared from potassium hydrogen phthalate standard solution (20-1000 mg O_2/L COD). pH, COD, turbidity and conductivity measurements were made according to the analysis methods specified in the standard methods [100].

Analysis

The percentage of color and COD removal was calculated by the Eqs. (13) and (14): [101,102].

$$RE = \frac{C_0 - C_e}{C_0} \times 100 \quad (13)$$

In this equation, C_0 and C_t (mg/L) represent the dye solution concentration at the beginning and at any time t, respectively.

$$\text{COD removal efficiency}(\%) = \frac{COD_0 - COD_t}{COD_0} \times 100 \quad (14)$$

In this equation; COD_0 is the initial, COD_t is the COD value at time t.

Electric energy (kWh/m^3) and electrode (kgAl-Fe/m^3) costs are calculated by the Eqs. (15) (16) and (17) [103,104].

$$C_{energy} = \frac{U.t.I}{V} \quad (15)$$

$$C_{electrode} = \frac{I.t.MW}{z.F.V} \quad (16)$$

$$\text{Operating cost} = aC_{energy} + bC_{electrode} \quad (17)$$

C_{energy} and $C_{electrode}$ show the consumption amounts per m^3 of wastewater treated. U: applied voltage (V), t: time (s), I: applied current (A), V: solution volume (m^3), MW: molecular weight of the electrode used (g/Mol), z: number of moles of electrons involved in the reaction (Al=3, Fe=2) and F: Faraday's constant (F =96500C/ Mol) which is used to calculate the amount of metal cations dissolved during reactions at the anode.

RESULTS AND DISCUSSION

Effect of Initial pH

Solution pH is a very important factor in determining the solubility of the soluble electrode and metal speciation

in aqueous solution in the electrocoagulation process, thus affecting the dye removal efficiency [38]. In general, two main mechanisms are thought to exist. In the low (pH< 4) pH range, precipitation occurs by flocculation, and in pH > 6.5, color removal occurs by adsorption [51]. In order to determine the effect of pH in the experimental study, the COD, color, and turbidity removal efficiencies were investigated at the initial conditions of current density, 100 A/m2 dye concentration 300 mg/L, and conductivity 1000 µS/cm (Fig.4). As a result of preliminary experimental studies, electrolysis time was kept constant at 20 minutes for both electrode pairs and 250 rpm in all other studies. In this study, the electrocoagulation process was performed by connecting the electrodes in monopolar parallel to the system. In the circumstances using both iron and aluminum electrodes in the EC process, due to the hydrogen gas output formed at the cathode, the flocs collected in the upper part of the reactor and a clear solid-liquid separation was observed.

As a result of the studies carried out under these conditions, as can be viewed in Fig.4a best decolorization efficiency was obtained at pH 3 (55.46%) for the aluminum electrode. In the case of using Fe electrode, it was determined that the color removal efficiency was too high (99%) at all initial pH values, that is, the pH did not affect the color removal efficiency much. It was observed that the pH values increased in both electrodes as a result of the experimental procedures. In the electrocoagulation process, the pH value increases due to the hydrogen gas output and the accumulation of hydroxide ions caused by the breakdown of water in the cathode during the electrolysis time. In acidic conditions, the removal and degradation efficiencies of pollutants are higher compared to neutral and alkaline solutions.

In addition, the increase in H^+ concentration accelerates the corrosion of iron. The rise in H^+ concentration increases the speed of the corrosion of iron. At high pH values, hydroxide formation increases, and excess hydroxide precipitates on the electrode surface [105,106]. When evaluated in terms of removal efficiency and power consumption for all three parameters, the initial pH value for the Al electrode was selected as 3, and for Fe electrode, it was 5. Thus, it can be deduced that higher removal efficiencies are obtained with both electrodes in an acidic medium. Made works show that the treatment efficiency decreases at low and high pH values in electrocoagulation. We can explain the main reason for this as the presence of ionic species that gain weight in the environment depending on the pH. The high removal efficiencies witnessed under acidic and neutral pH conditions may be due to the efficient conversion of Fe(II) to Fe(III), which forms positively charged precipitates due to its low solubility.

Fe^{3+} and Fe^{2+} ions hydrolyze both $Fe(OH)_2$ and $Fe(OH)_3$ hydroxyls. The main type of Fe^{m+} formed in solution at pH 6 is $Fe(OH)_2$, which can neutralize organic materials and suspended substances and take them to the aggregation process. At higher or lower pH, species such as $Fe(OH)_3$ or $Fe(OH)_4^-$ dominate instead of $Fe(OH)_2$, but these species are not very effective coagulants [107]. The pH value causes different coagulation mechanisms by affecting the conversion process of Al^{3+} and therefore coagulant species.

In studies using aluminum electrodes, monomeric and polymeric aluminum hydroxide complex types are formed depending on the pH, and the dominant chemical species are also different according to the initial pH [108,109]. Monomeric hydrolysis types such as Al^{3+}, $Al(OH)^{2+}$ dominate at pH 2-3 and monomeric species can transform into polymeric products. In the pH range of 4-9, polymeric species such as $Al_{13}O_4(OH)_{24}^{7+}$ occur and precipitated as $Al(OH)_{3(s)}$. For example, hydrolysis of Al^{3+} ions forms the aqueous $Al(H_2O)_6^{3+}$ complex, which is common at pH>4.5 [110,111]. At pH>9, the soluble species $[Al(OH)_4]^-$ is formed, but amorphous aluminum hydroxide is still present in lesser quantity [112].

In EC experiments using Al electrode, the highest COD removal efficiency was obtained at pH 3 with 41.60 % (Fig.4b). While the highest turbidity removal efficiencies were obtained in the pH 3-5 range (86-87%), it was observed that the turbidity removal efficiency decreased in other pH ranges (Fig.4c). When the COD removal efficiency is examined as a result of the use of iron electrodes, it is seen that removal efficiency of 63%-65% is obtained in the pH 3-5 range, while the efficiency decreases at other pH values. This tendency was attributed to the fact that during electrolysis at constant time and current, the amount of ions dissolved from the anodic reaction is fixed according to Faraday's law. The turbidity removal efficiency was above 85% at all pH values, and the highest removal efficiency was found at pH:5 with 89.26%. In the same pH range, the color removal

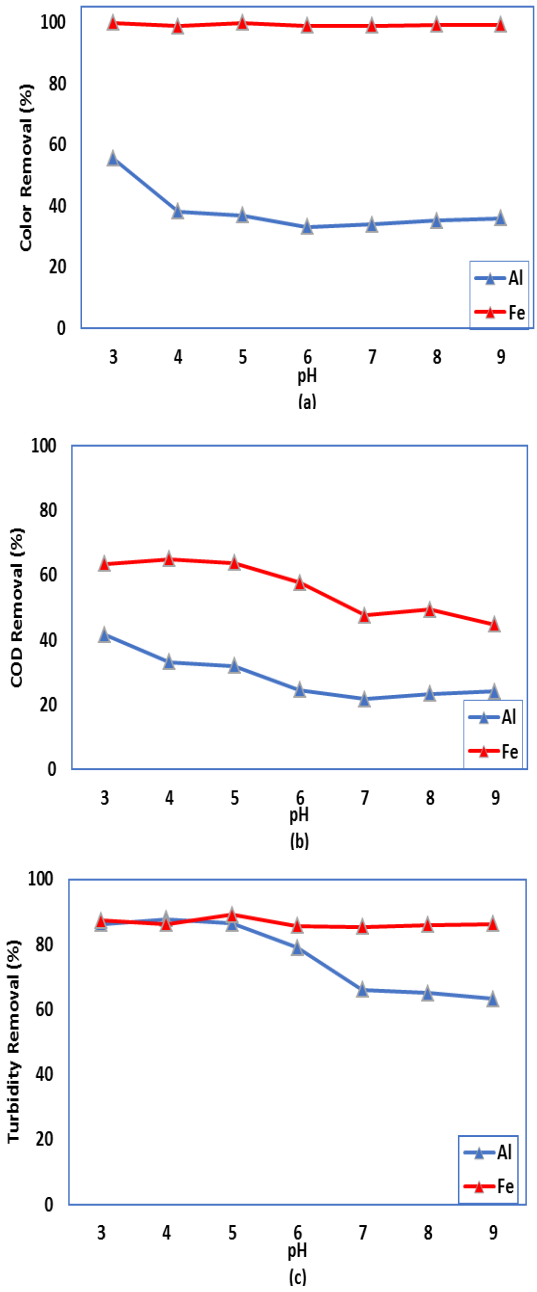

Fig. 4: Effect of initial pH value on (a) color, (b) COD and (c) turbidity removal efficiencies (current density 100 A/m^2, dye concentration, 300 mg/L, conductivity 1000 µS/cm, 20 min electrolysis time, stirring speed 250 rpm).

efficiency was 99.64% and the COD removal efficiency was 63.8%. COD removal was always lower than decolorization as shown in Figure 4b. Increasing the pH value reduces the precipitate formation and consequently the COD removal efficiencies. Experimental results showed that the onset of instability occurs when the reactive phase is reached, and the instability is promoted by amorphous iron hydroxide precipitate and polymeric cations [113-115].

Effect of current density

Since ion movements are important as a result of the reactions taking place in the EC process, the current and therefore the current density is an important parameter that should be considered in the operation of the system. The amount of aluminum and iron separated from the electrodes, the size of the gas bubbles, the rate of formation, and the development of flocs are determined by the current density. The current density not only affects the response time of the system but also strongly affects the dominant pollutant separation mode [116, 117]. With the intention of investigating the effect of current density on the COD, color, and turbidity in the electrocoagulation process, it was studied at the initial pH ranges (Al-3, Fe-5) where the highest removal efficiency was obtained. Experiments were carried out by keeping the 300 mg/l dye solution and 1000 µS/cm conductivity, 20 min electrolysis time and 250 rpm mixing speed constant, changing the current density in the range of 25-150 A/m^2. Fig.5, shows the effect of current density on color, COD and turbidity removal efficiencies in the electrocoagulation process. We detected that COD, color and turbidity removal efficiencies increased with the rise of current density from 25 A/m^2 to 100 A/m^2. Color, COD and turbidity values for the Al electrode were 17.87-64.42%, 24.92-52.78, 60, 24-90.12%, respectively; for the Fe electrode, it was obtained 76.74%-99.07, 29.38-41.85, 75.46-93.19, respectively. It is seen that there is a rapid increase in the removal efficiency of all pollutants up to a current density of 100 A/m^2, and this increase is stabilized between 100-150 A/m^2 by slowing down. The increase in current density increases the energy and electrode consumption and thus the cost. Figure 5 shows the effect of current density on color, COD, and turbidity removal efficiencies in the electrocoagulation process. Increasing the current density from 25 A/m^2 to 100 A/m^2 resulted in an increase in COD, color, and turbidity removal efficiencies. As the current density increased from 25 to 150 A/m^2, the potential difference between the electrodes increased from 6.03 to 27.13 for the Al electrode, and the energy consumption 0.86 to 23.27 kWh/m^3.

Table 3: Al/Fe energy and electrode consumption depending on current density

Current Density (A/m^2)	25		50		75		100		125		150	
Current (A)	0.32		0.64		0.96		1.29		1.61		1.93	
Al/Fe												
Voltage (V)	6.03	7.07	11.14	12.72	15.49	19.17	20.02	21.40	23.44	24.01	27.13	30.17
Energy Consumption (kWh/m^3)	0.86	1.01	3.17	3.62	6.60	8.18	11.48	12.12	16.77	17.18	23.27	25.88
Electrode consumption (kg/m^3)	0.19	0.16	0.36	0.31	0.53	0.46	0.56	0.61	0.74	0.77	0.91	0.91
Energy consumption (kg paint/kWh)	11.94	3.26	42.88	9.13	64.13	20.63	69.55	30.79	73.05	43.01	122.59	65.22
Energy consumption (kgCOD/kWh)	10.06	10.0	31.39	29.81	48.70	56.82	63.90	86.11	107.80	123.44	160.26	181.67

For the Fe electrodes, the potential difference between the electrodes increased from 7.07 to 30.17 for the current densities in the same range, and the energy consumption increased from 1.01 to 25.88 kWh/m^3 (Table 3). As a result, current density directly affects energy consumption as it affects energy consumption and also the potential difference between electrodes. Therefore, considering the optimum current density as a result of local discharge limits, energy and electrode consumptions, local energy unit prices, and some other limiting factors, the optimum current density was taken as 100 A/m^2 for Al and 75 A/m^2 for Fe. All subsequent experiments have been carried out at this current density.

Electrode cost is calculated according to the amount of electrodes that break off. The two most important factors that cause a break of the electrodes are the current density and the electrocoagulation time, and the increase in both increases the amount of spent electrodes and therefore the cost. As can be seen from Table 3, both the energy and the electrode cost, that is, the total cost, increased simultaneously with the rise in the current density, but after a certain point, a decrease was recorded in the removal efficiency. This situation is also supported by studies in the literature [118,119]. The current density must not be excessive in order to avoid the formation of excessive amounts of oxygen and also to avoid negative effects such as heat generation. At an increase above the optimum current density, the degree of anodic metal dissolution increases, causing more coagulants to be produced. As there are enough metal hydroxide flocs present to precipitate the pollutant, it does not give rise to a further rise in pollutant removal efficiency [120,121]. With increasing current density, hydrogen bubbles can be adsorbed more easily on the particles and accordingly, the precipitation rate decreases. The increase of H_2 bubbles ensures that the contaminants are transported into the solution and removed from the solution [122,123]. In other words, high current density will increase the flock, which will increase the efficiency of COD, color, and turbidity removal.

The obtained results reveal that the removal efficiency of RR 239 increases up to 93.87% by increasing the current density for the iron electrodes. This is because, as explained above, the coagulant and bubble generation rates depend on the density available.

The increase in current density did not cause a significant increase in COD removal efficiency. It is seen that the organic removal rate increases with the increase of the current density up to a certain limit. The low COD removal efficiency may be due to the presence of by-products formed by direct oxidation on the anode surface and may be directly proportional to the rate of electrochemical reactions occurring on the electrode surface (Fig.5b). Although a rapid removal efficiency was acquired at the very beginning of the reaction in trials with high current densities, the removal efficiency remained at low levels in the following minutes. The reason for this may be that the electrodes are not sufficiently dissolved due to the large amount of sludge formed in the reactor in the trials where high current density is applied. If the sludge formed in the system at high current densities during the reaction is removed from the environment, the treatment efficiencies obtained at low current densities can be obtained in a much shorter time.

For Fe electrode, current densities of 25, 50, 75, 100, 125, and 150 A/m^2 and at 20 min electrolysis time the turbidity removal efficiency were obtained 75.5%, 82.2%, 91.9%, 93.2%, and 94%, respectively. For the Al electrode,

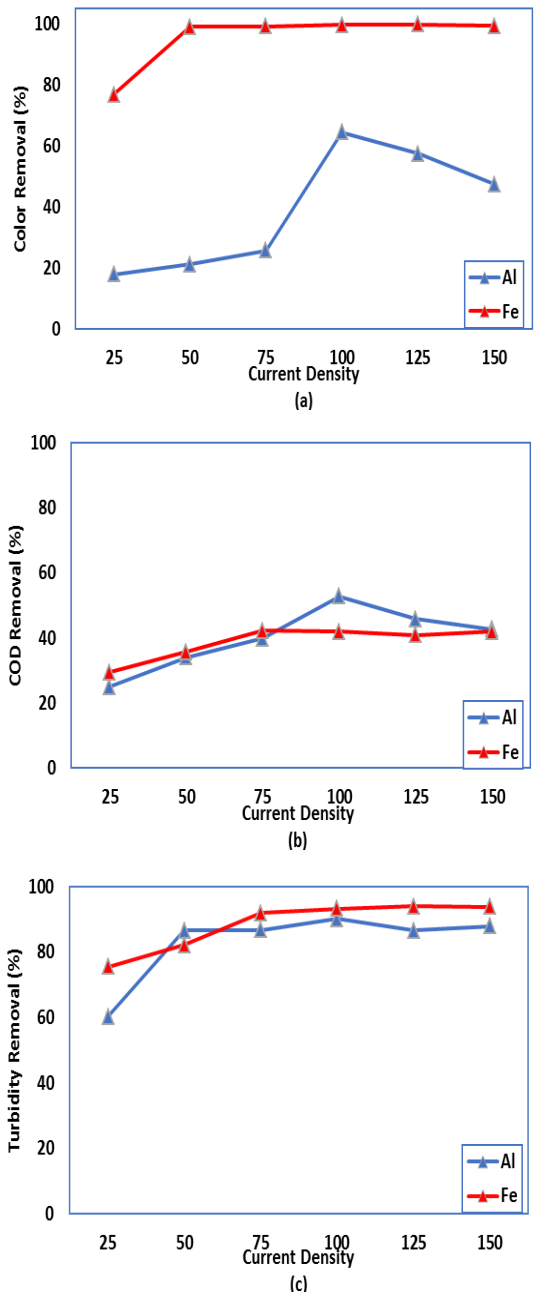

Fig. 5: The effect of current density on (a) color, (b) COD, and (c) turbidity removal efficiency in the electrocoagulation process (for Al electrode pH:3, for Fe electrode pH:5, dye concentration 300 mg/l, conductivity 1000 µS/cm, electrolysis time 20 min, stirring speed 250 rpm).

at the same current densities and electrolysis time, the turbidity removal efficiencies were determined as 86.7%, 86.8%, 90.1%, 86.7%, and 87.9%, respectively (Fig.5c). These results were attributed to the increased degree of anodic dissolution of the electrodes at higher current densities, giving rise to more $Fe(OH)_{3(s)}$ or $Al(OH)_{3(s)}$ particles for pollutant removal. So it is clear that the turbidity removal efficiency shows the same trend for both materials. Considering the research, we can say that Al^{3+} is the main species present when the pH value is lower than 3.5, and $Al(OH)_{3(S)}$ is dominant for pH values between 4 and 9.5. If the pH is greater than 10, $Al(OH_4^-$ is formed and $Al(OH)_4^-$ is known to be a very soluble weak coagulant because it carries a negative charge and cannot destabilize colloids. These results are quite plausible, as $Al(OH)_{3(S)}$ $Fe(OH)_{3(S)}$ precipitates, keeping the colloids/contaminants in the form of scavenging coagulation [38]. When the effects of voltage, electrode material, and their combination on pollutant removal were examined, as expected, the removal efficiency increased with increasing current intensity.

Effect of electrolysis time

To investigate the effect of electrolysis time on removal efficiency, experiments have been performed at intervals ranging from 5 to 70 minutes (Fig. 6). The treatment efficacy of the electrochemical process is also affected by the electrolysis time, which can increase or decrease with current density or pH [124]. Increasing the electrolysis time increases the energy consumption and the efficiency of color, turbidity, and COD removal. The electrolysis time (t) determines the rate of production of Fe^{2+} or Fe^{3+} ions from the iron electrodes. The same situation applies to Aluminum as well. The removal efficiency is directly dependent on the concentration of hydroxyl and metal ions produced at the electrodes [61]. The concentration of the ions and their hydroxide flocs increase with the increase in electrolysis time. The free ions released neutralize the particle charges and thereby initiate coagulation. In other words, the formation of appropriate and adequate flocs in the EC process takes place depending on time and also provides an increase in the bubble formation rate.

In an experimental study increasing the electrolysis time resulted in higher removal efficiency due to the formation of hydroxyl radicals, which means that more metal polymeric species are formed. The increased concentrations of dissolved metal ions, which improve color and COD removal, are responsible for the high coagulation efficiency. The results show that the dye removal efficiency is low in the first 5 minutes of the reaction, but the yield increases with the increasing

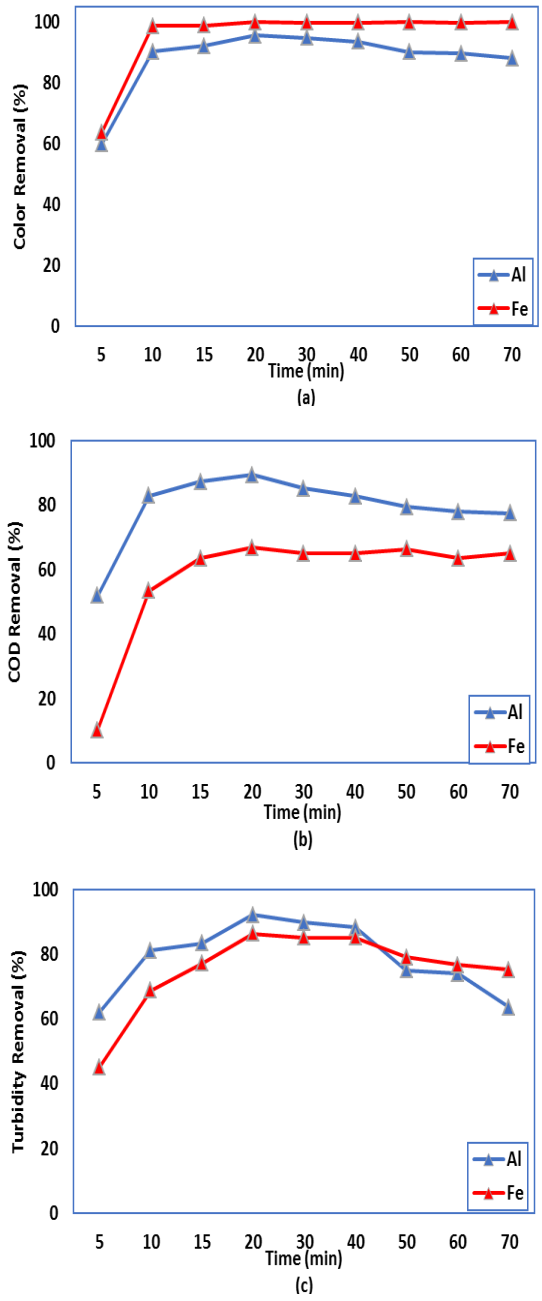

Fig. 6: Effect of electrolysis time on (a) color, (b) COD (c) turbidity removal efficiency in EC process (for Al electrode pH:3, current density 100 A/m^2, conductivity 250 µS/cm, for Fe electrode pH:5, current density 75 A/m^2, conductivity 500 µS/cm, 250 rpm stirring speed; dye concentration 300 mg/L).

reaction time. For all the three parameters, it was observed that while there was a rapid increase in the removal efficiency of pollutants in the electrolysis times of 5-20 minutes, this rate of increase decreased at higher electrolysis times and then the removal efficiency became stable. The very fast dye removal rate during the first 10 minutes of electrolysis can be attributed to the formation of OH- ions at the cathode. Therefore, according to the results shown in Fig.6, the optimum electrolysis time was found to be 20 minutes. During this electrolysis period, the COD, color and turbididy removal efficiencies for Fe electrodes were obtained 66.83%-99.2% and 86.38%, respectively, and 89.34-95.49% and 92.18% for Al electrodes.

CONCLUSIONS

In the electrocoagulation process using aluminum and iron electrodes, the effect of various operating parameters on the color removal efficiency was investigated and optimized for the removal of the commercially used Remazol Ultra Red RGB (reactive red 239) dye. The results were obtained as 66.83%-99.2% and 86.38%, respectively, for the COD, color and turbidity removal efficiencies for Fe electrodes and 89.34-95.49% and 92.18% for Al electrodes under optimum operating conditions (For Al electrode pH:3, current density 100 A/m^2, conductivity 250 µS/cm, for Fe electrode pH:5, current density 75 A/m^2, conductivity 500 µS/cm, 300 mg/l dye for both electrodes solution). For Al electrode, energy consumption at optimum current density is 11.48 (kWh/m^3), and electrode consumption is 0.56 (kg/m^3); at the optimum current density for the Fe electrode, the energy consumption is 8.18 (kWh/m^3) and the electrode consumption has been found 0.46 (kg/m^3). Experimental results show that the effects of current density on process efficiency have similar effects in terms of electrical energy and electrode consumption values.

Fe electrodes gave higher results in color and turbidity removal than Al electrodes. With the increase of the initial dye concentration, the COD removal efficiencies decreased in parallel with the decolorization. In the treatment of textile wastewater in Turkey, especially in terms of discharge standards to the receiving environment, COD, Color and pH are the parameters to be controlled. The data obtained as a result of the study show that the desired wastewater discharge standards are met. The results from Al and Fe electrodes look promising for further studies due to their cheapness and availability. This technology can be used in textile wastewater treatment due to significant improvements in the conventional mode and chemical coagulation process.

The following suggestions can be made regarding this study;

● More research is needed to study EC reactor, operating parameters, electrode dissolution events etc. However, the shape and configuration of the electrodes, the EC mechanisms, can be scaled for possible improvement of the pollutant removal efficiency. Efficiency can be investigated for electrocoagulation reactors (series-connected monopolar, parallel-connected bipolar). By adding chemical coagulants to the electrocoagulation process, the variation in treatment efficiency can be investigated.

● The effectiveness of combined systems (such as EC+O_3, EC+EF) in the treatment of textile industry wastewater can be investigated. It is believed that these systems are effective in strongly reducing the parameters that ensure the achievement of discharge standards for the purpose of reuse of treated water.

● Electrocoagulation efficiency can be investigated for different electrode types (nickel, platin, steel, copper, etc.) in electrocoagulation applications.

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