Research Article GU J Sci 32(1): 104-114 (2019)

Gazi University

Journal of Science

http://dergipark.gov.tr/gujs

Decolourisation of Disperse Brown Dye Solution by Electrocoagulation Process with Al and Fe Electrodes

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Article Info

Received: 24/04/2018
Accepted: 11/09/2018

Keywords

Electrocoagulation
Dye Removal
Fe and Al Electrodes

Abstract

Dyes used in the textile industry can easily contaminate water and are becoming a world-wide pollution problem. The efficiency of colour removal process from artificially prepared and actual textile wastewater using electrocoagulation (EC) was investigated in this study. A disperse dye was selected for the synthetic dye wastewater experiments. Different operating conditions such as dye concentration, pH, conductivity values and current density (CD) were investigated using synthetically prepared dye solution in a batch EC reactor with parallel-connected electrodes. EC optimum working conditions for decolourisation rate of 50 mg/L synthetic dye solution were found as current density of 30-37.5 mA/cm^2, conductivity value of 1 mS/cm, pH value of 7.5 at 25^{o}C for both Al and Fe electrodes in this experimental work. Additionally, a real textile wastewater was tested at similar experimental conditions achieved with synthetic dye solution using a batch EC reactor. Dye removal rate was achieved as %80 with real wastewater with energy consumption of 9.16 kWh/m^3.

1. INTRODUCTION

In many countries, the textile industry is one of the important sources for national economies and there are many different types of textile processes available around the world. Technological developments have introduced huge amount of various new materials and processes to the industries resulting into new chemical pollutants which adversely affect the environment. Large quantity of coloured wastewaters is generated in the textile industry as a result of large volumes of water usage [1, 2]. The high quantities of dyestuffs and auxiliaries such as enzymes, detergents, dyes, acids, sodas and salts, have also been used to meet the different customer demands in textile industry. These chemicals are usually resistance to biological treatment and are discharged in to the environment without treatment even if the discharge standards of organic matter in the effluents are not met [1, 2]. It is also known that environmental monitoring data for groundwater and soil showed many chemicals beyond the legal limit values. These chemicals can easily distribute and move through the water cycle and contaminate the food chain through irrigation, fish production, domestic and industrial water use, consequently becoming a world-wide pollution problem. Also, the secondary materials used the dyeing process are not immersed by the dyed materials and can cause extra pollution in the environment being, hence harmful to the natural ecosystems due to their genotoxic and mutagenic effects. Dye and auxiliary chemicals do not only cause health problems but also effect next generations due to transferrable changes in genetic materials and eco-toxicological effects. Another problem attributed to discharge of dye wastewater into the environment is the aesthetically unpleasant outlook, which blocks light penetration, and may be harmful to the receiving waterbodies and consequently becomes toxic to organisms and to aquatic life [1, 2]. Removal of synthetic dyes from industrial effluents and discharging to the environment is very important issue in as far as getting good quality water and healthy ecosystems are concerned. Therefore, finding proper treatment option for textile industry is a major ecological issue.

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Many wastewater treatment technologies such as chemical precipitation, membrane separation, coagulation/flocculation and ion exchange resins, have been developed for treatment of industrial wastewaters in recent years [3-6]. Among these methods, electrochemical treatment processes can effectively be applied to wastewater treatment systems to remove organic and inorganic pollutants and pathogens [7-10]. The electrochemical wastewater treatment has also many advantages such as high removal efficiency, relatively less environmental effect, easy operation and compacted design. In addition, higher removal rate for pollutants with better process conditions such as creating waste sludge and dissolved solids, more robust and easy to automation, less reaction and sedimentation time etc. are achieved by EC. The only concern in EC is to maintain sacrificial anodes and energy cost. However, EC is more compatible than other treatment methods, so it should commonly be used as easy, economical and better wastewater treatment system.

EC is a rising emerging and operative technology for the water and wastewater treatment purposes [11, 12]. EC uses similar mechanism found in the chemical coagulation. In an electrochemical reactor, electrodes are dipped in a solution, and then linked to a direct current (DC) power source. The electrical current passes through the electrodes and initiate electrochemical dissolution of metal electrode in situ. The released metal cations from dissolved electrodes are involved in multi chemical and physical process in EC. Therefore, EC needs no additional chemicals and can provide a cost effective and easy operable treatment process with short reaction times [13-15]. The released metal ions from electrode cause the creation of metal monomeric and polymeric complexes that aid in destabilization of colloidal particles and pollutants from solution. Also, less sludge is produced in EC compared to chemical treatments hence reducing final costs associated with disposal of sludge.

The electrocoagulation processes contain three stages: adsorption/charge neutralization, clarifying coagulation mechanisms and changing them to floatable or settable flocs. Different gases such as hydrogen and oxygen are formed during the EC process and help in the removal process through mixing and flocculation. The basic reactions that take place in an EC cell are shown in Eq. 1-5. As can be seen from these equations, metal ions are released from the sacrificial anode with simultaneous generation of gases at the anode and cathode due to water electrolysis reactions [11-15].

Anode Reactions

$$M \rightarrow M^{n+}(aq) + ne^- \tag{1}$$
$$2H_2O \rightarrow 4H^+ + O_2\,(g) + 4e^- \tag{2}$$

Cathode Reactions

$$M^{n+}(aq) + ne^- \rightarrow M(s) \tag{3}$$
$$H_2O + 2e^- \rightarrow H_2\,(g) + 2OH^- \tag{4}$$

Reactions in bulk of the solution

$$M^{n+} + nOH^- \rightarrow M(OH)_n \tag{5}$$
$$M(OH)_n + Dye\ Molecules \rightarrow flocs \tag{6}$$

Where, M = material of anode and n = Number of electrons occurred in the redox reactions.

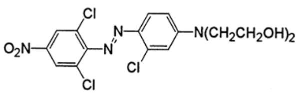
In EC, metal ions are created from dissolved anode whereas hydroxide ion (OH^-) and H_2 gas are created at the cathode as given to Equations 1-4. Metal ions (M^{n+}) and OH^- ions are produced by electrode reactions can form some monomeric compounds which then transform into solid $M(OH)_n$ according to precipitation kinetic reactions. This freshly formed shapeless unsolvable flocs of $M(OH)_n$ can react with dye molecules by surface interaction or electrostatic reaction causing coagulation and then precipitate on the bottom of the reactor (Equation 6). The produced gaseous hydrogen can aid in bringing flocs together on the liquid surface and enhance the pollution elimination process. Many investigations on removal of dyes using EC can be found in the literature [16-19].

The removal process of Disperse Brown from aqueous solutions using EC with different electrodes was investigated in this paper. The effects of concentration, pH, conductivity and current on the EC process were observed to find out the best working conditions. These optimum parameters were used to examine electrochemical treatment of real textile wastewater. The novelty of this experimental work is rooted on the use of optimal experimental values as base parameters to enhance actual textile industry wastewater treatment on a lab scale. The experimental and cost analysis of real textile wastewater could be of paramount importance to many researchers working to scale-up to an effective reliable, simple robust and low-cost removal process for textile industry.

2. MATERIAL AND METHODS

Commercially available disperse dye, Dianix Brown S-3R, from DyStar was used in the study (Table 1). 500 mg/L synthetic stock dye solution was prepared from dye powder using appropriate amount and used to prepare a diluted series of dye solutions (50, 250 and 500 mg/L). Dye concentration in aqueous solution was analysed using a UV–Vis spectrophotometer (Hach Lange, DR 2800). The pH values of the solutions were adjusted using H_2SO_4 or NaOH. No effect of pH variations was observed on the maximum absorbance wavelength of synthetic dyes used as shown in Figure 1. NaCl was added to get sufficient conductivity of the synthetic dye solution when necessary. Distilled water was used in all experiments. Analytical grade chemicals and reagents were used for this study without purification.

Table 2. *Some properties of dye used in the study*

Chemical Name	Chemical Structure	Colour Index Number	Molecular Formula	CAS Number	UV Maximum Absorption Wavelength (nm)	Molecular Weight (g/mol)
Dianix Brown S-3R		C.I. 11152	$C_{16}H_{15}Cl_3N_4O_4$	12236-00-9	456	433.17

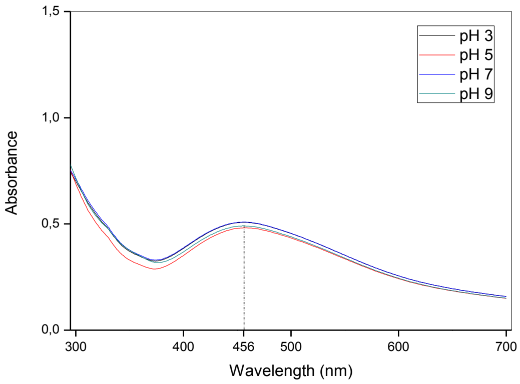


Figure 1. *UV-Vis spectrum of Dianix Brown S-3R at different pH values*

The batch electrochemical reactor (95x95x105 mm) shown in Figure 2 was used with two pairs of rectangular shape electrodes made of Al (area of 20cm^2) and Fe (area of 25 cm^2) in the experiments [20]. The reactor was filled with 600 cm^3 of the synthetic dye solution or real textile wastewater in each experiment. A set of electrodes made from the same material were used as cathode and anode for each trial. The electrodes used were cleaned with 35% HCl solution and dried after each experiment. The separation between two electrodes was measured as 38 mm.

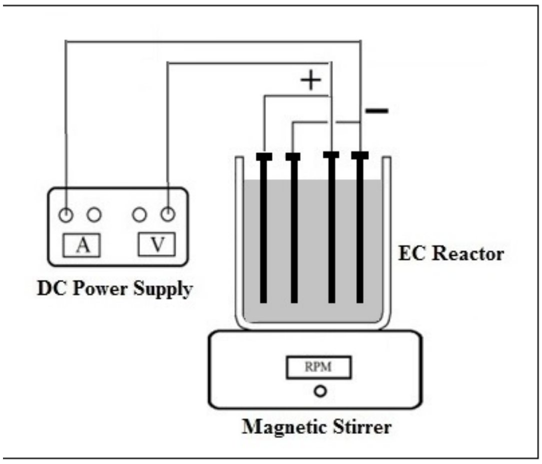


Figure 2. *EC process experimental set-up*

A digital DC power supply that gives current and voltage values up to 5 A and 30 V, respectively (NETES 3306D), was used in monopolar-parallel mode. Initial dye concentrations, pH, conductivity and current density ere explored as the process parameters. Samples were periodically taken from the EC process at certain intervals for 10 minutes reaction time to calculate the removal efficiency. The taken samples were centrifuged at 6000 rpm for 10 minutes to isolated from the flocs and then the concentration of dye analysed using the UV spectrophotometer for synthetic dye solution at 456 nm found in the UV-Vis spectrum. Previously prepared calibration graph was used to calculate dye concentrations, and the removal rates were subsequently calculated using initial and after dye concentrations measured at 456 nm.

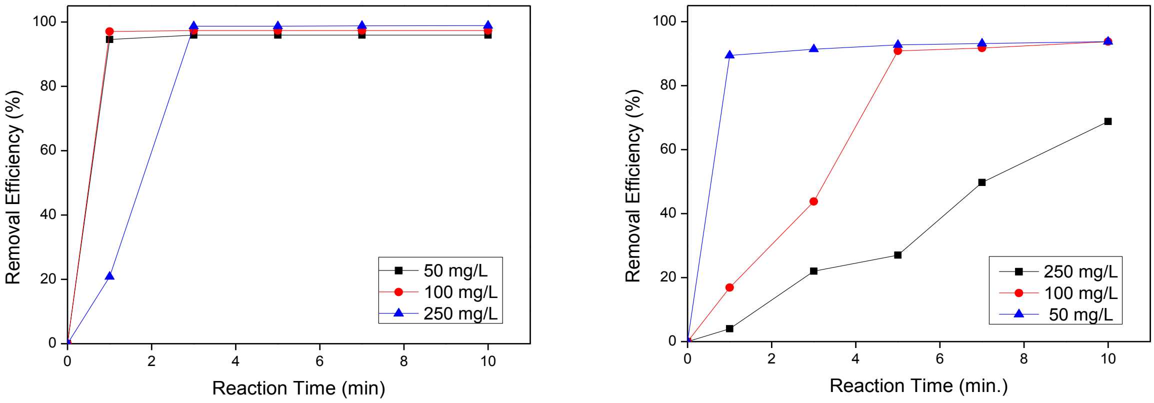
Textile industry wastewater was studied to examine optimum parameters found in the synthetic dye solutions trials. Chemical Oxygen Demand (COD) and colour were analysed by closed reflux method and Pt-Co units, respectively according to the Standard Methods [21]. The wastewater contained 850 mg O_2/dm^3 COD and 188 colour, 2 mS/cm conductivity and pH of 6.7. At various time intervals, samples were taken, centrifuged and analysed accordingly.

3. RESULTS AND DISCUSSION

3.1. Effect of Initial Dye Concentration On EC Process

Initial dye concentrations of 50,100 and 250 mg/L were used to examine the effect of the dye concentration on the colour removal. 37.5 mA/cm^2 and 30 mA/cm^2 current densities with 1 mS/cm solution conductivity were used in the both Al and Fe electrodes experiments, respectively. Initial pH value was 7.5 for both sets of the experiments. As seen from Figure 3a, with Al electrodes, the first 5 min of EC process give an efficient dye removal rate (95%) for all concentration used. Therefore, flocs formed by $Al(OH)_3$ were high enough to remove the higher number of dye molecules. Initial and final pH values in the reactor were measured to be around 7.5 and 9. It also is known that $Al(OH)_3$ is dominant in the solution since it is the main species in aqueous solutions at pH range between from 4 to 9.5 [22, 23] and this would help to achieve the high removal rate for higher dye concentrations.

When Fe electrodes were applied, it was seen that the highest colour removal rate was achieved with lower initial dye concentration (Figure 3b). The removal rate was around 70% for the highest initial concentration of 250 mg/L. This drop can be explained as numbers of iron ions dissolved from electrodes were constant at the fixed current value and time, hence the formed flocs from this amount of iron were not enough to remove dye at the increased concentration. However, the amount of iron dissolved from electrodes would increase for a longer reaction time and can be enough to form flocs to remove higher concentration of dye molecules. Many investigators have also reported similar phenomenon as an increase in dye concentration caused the decreased colour removal efficiency [19, 24, 25].

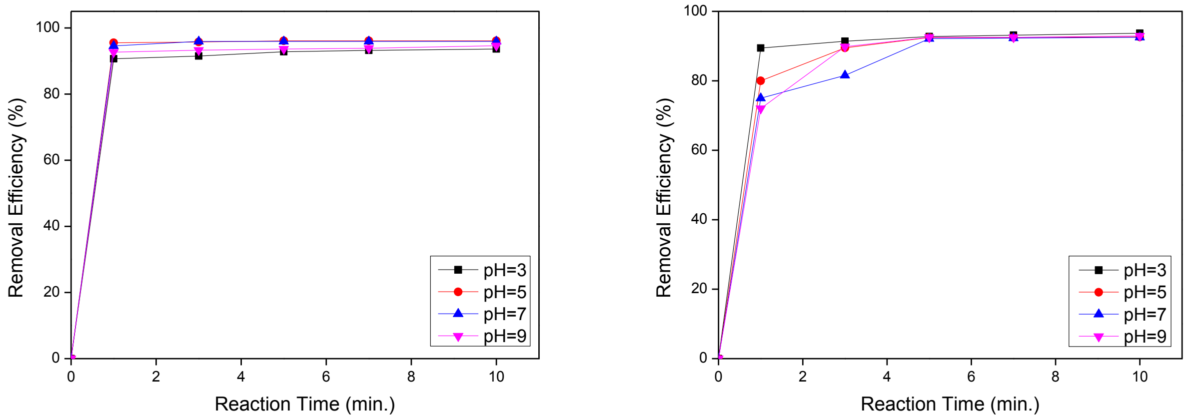


(a) (b)

***Figure 3.** The colour removal efficiency changing with initial dye concentration using; a) Al electrodes b) Fe electrodes [pH 7.5, $25^{o}C$, 1 mS/cm, CD_{Al}: 37.5 mA/cm^2, CD_{Fe}: 30 mA/cm^2]*

3.2. Initial pH Effect on EC Process Efficiency

The creation of undissolved metal hydroxides, which can aggregate pollutants from aqueous solution, depends on pH value of solution. As well known, the dominant chemical species for metal ions solutions can be changed by different pH values. Therefore, pH is a significant feature in electrocoagulation method and affects the performance of pollution removal process due to its influence on the formation of metal hydroxides in solution. It is known that at a pH below 3.5 the predominant specie is Al^{3+} ion. However, at pH values between 4 and 9.5 the principal aluminium type is $Al(OH)_{3(s)}$. Nevertheless, when pH value is higher than 10, soluble monomeric anions $Al(OH)_4^-$ occur and negatively affect pollutant removal [22, 23]. In case of iron electrode, at pH value lower than 7, the major species is Fe^{2+}, but between pH 3.5-7 and above 11 soluble monomeric iron ions such as $Fe(OH)+$ and $Fe(OH)_4^-$ may also respectively occur and lower the removal rate. At pH values higher than 7 the highly insoluble complex of $Fe(OH)_2$ is the major specie [22, 23] and increases the dye precipitation rate. Besides, iron can also be oxidized from Fe^{2+} to Fe^{3+} depending on pH and dissolved O_2, creating a mixture of soluble ferrous ions and insoluble $Fe(OH)_{3(S)}/FeOOH_{(S)}$ [14]. In all cases for Al or Fe electrodes, these freshly formed amorphous insoluble flocs of $M(OH)_n$ and other soluble ions can react with dye molecules and removal process occurs by mostly precipitation and/or adsorption, sweeping dyes molecules to the base of the reactor at suitable pH value. The effect of pH was studied using initial pH values in the range 3-9. As it is shown in the Figure 4, removal rates were almost the same for the low initial dye concentration (50 mg/L). This is mainly due to formation of enough amount of OH^- ion by the electrochemical reactions of the hydrolysis of water at the cathode according to Eq4. Because of this phenomenon, high final pH values (around 9) of the solutions were always measured at end of the experiments. Soluble flocs occur at wider pH range in case of Al electrode, removal rate was increased dramatically for the range pH studied. On the other hand, iron electrode required more time to attain suitable pH range to removal process. Therefore, it can be said that for low dye concentration, the dye removal process was not affected by pH for both Al and Fe electrodes. Comparable results were also reported in the treatment of Disperse Red dye by electrocoagulation process [26].

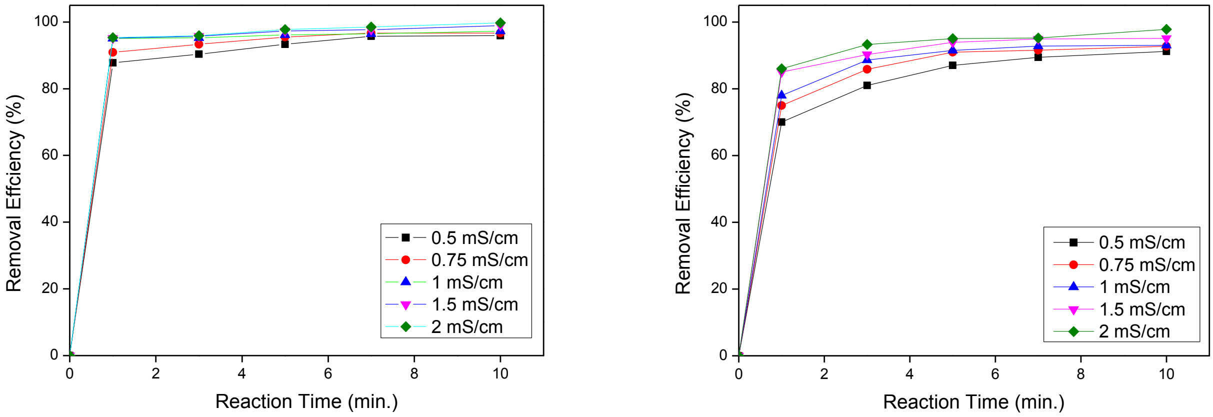


(a) (b)

Figure 4. *Different pH effect on the colour removal efficiency for; a) Al electrodes b) Fe electrodes [25^{o}C, 50 mg/L, 1 mS/cm, CD$_{Al}$: 37.5 mA/cm^2, CD$_{Fe}$: 30 mA/cm^2]*

3.3. Initial Conductivity Values on EC Process Effectiveness

Solution conductivity has a significant effect on the consumption of electrical energy used in the EC process [10]. In the present study, electrolyte solution conductivity was varied from 0.5 mS/cm to 2 mS/cm by using NaCl. Figure 5 shows that colour removal rates slightly increased with increasing conductivity of solution for both electrodes. These results suggest that increase in electrolyte concentrations do not significantly affect the removal efficiency, therefore the optimum conductivity value of 1 mS/cm was selected for the succeeding experiments. El-Ashtoukhy et al. (2016) also reported that increase in conductivity levels (0.5-2 g/L NaCl) can slightly increase dye removal efficiencies (88-95%) [27]. Nevertheless, middle value of the conductivity was selected as the optimal dye removal parameters due to cost-effectiveness of the process.



(a) (b)

Figure 5. *Effect of conductivity values on the colour removal efficiency; a) Al electrodes b) Fe electrodes [25^{o}C, 50 mg/L, pH 7.5, CD$_{Al}$: 37.5 mA/cm^2, CD$_{Fe}$: 30 mA/cm^2]*

3.4. Different Current Density Effect on the Dye Removal Efficiency

Current density is another vital parameter for EC process since it controls the quantity of metal ion coming from dissolution of electrodes that in turn increases the amount of the metal hydroxide flocs formed in the solution for pollutant removal. Therefore, various current densities were used to investigate their effect on colour removal efficiency in the EC reactor. In this study, due to different surface areas of Al and Fe electrodes, the current density parameters were given to ease comparison between the two types of electrodes. Figure 6 indicates the colour removal rate at different current densities used for both the electrodes in the EC reactor. It can be seen from the graphs, that increase in current density does not result in an increase in the dye removal rate due to the inadequate number of metal hydroxide flocs are existing for the removal of dye molecules in aqueous solutions even at low current density. From these results, it can be concluded that current density lower than 25 mA/cm^2 should have been studied to see effect of the lower current densities on the dye removal rate. Although, similar studies were reported 50 mg/L acid red removal by EC with iron anode using different current densities [28].

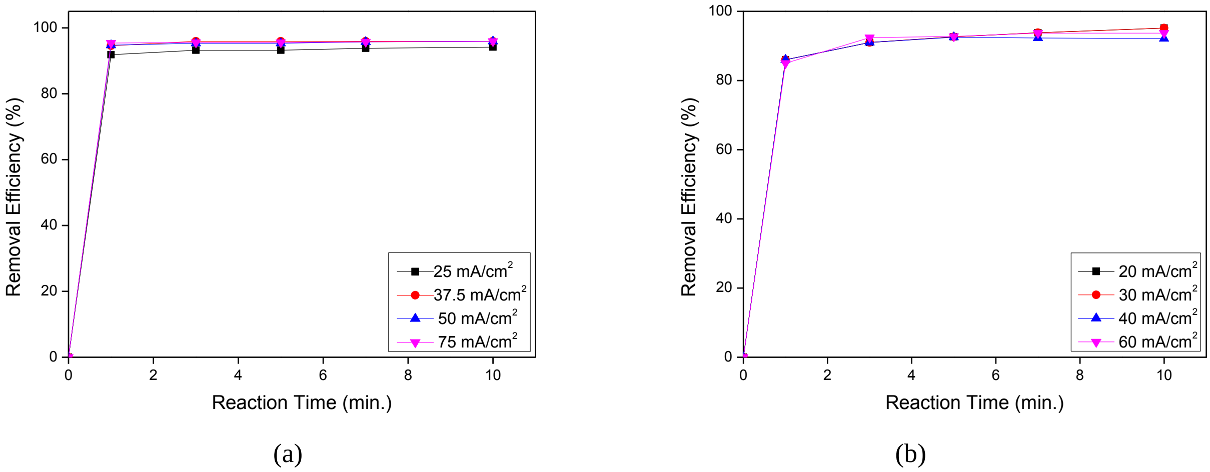


Figure 6. *Effect of current density on the colour removal rates with for; a) Al electrodes b) Fe electrodes [25^{o}C, 50 mg/L, 1 mS/cm, pH 7.5]*

3.5. Real Textile Wastewater Treatment with EC

Textile industry effluent was applied to the EC process in order to examine EC reactor optimum parameters found in the synthetic dye solutions. However, the first trial results in our laboratory showed that the optimum current density and reaction time for synthetic dye solution did not give satisfactory colour and COD removal rates for textile wastewater. Therefore, current density and reaction time were increased to 50 mA/cm^2 and 30 minutes, respectively to achieve higher removal efficiencies for real dye wastewater process. Although both Al and Fe electrodes successfully removed low synthetic dye concentration in the EC removal experiments, the literature results indicated Al electrode to be more efficient for removing pollutants at higher dye concentrations [29, 30]. This is because Al dissolves directly into Al^{+3} ions in all cases whereas Fe electrodes may dissolve in to Fe^{+2} and Fe^{+3}. Lower positive charge of Fe^{+2} may show weak coagulant properties [29,30]. Therefore, in this study only Al electrodes were used in the trail experiments for the treatment of real wastewater. Around % 80 and 68 removal rates were achieved for dye and COD parameters, respectively with real wastewater. This decrease in the dye removal can be explained as real textile wastewater may have many different chemicals such as salts, softener or mixed coloured agents, so the conditions for EC process would be different form synthetic dye solution e.g. required using higher current density and longer reaction time.

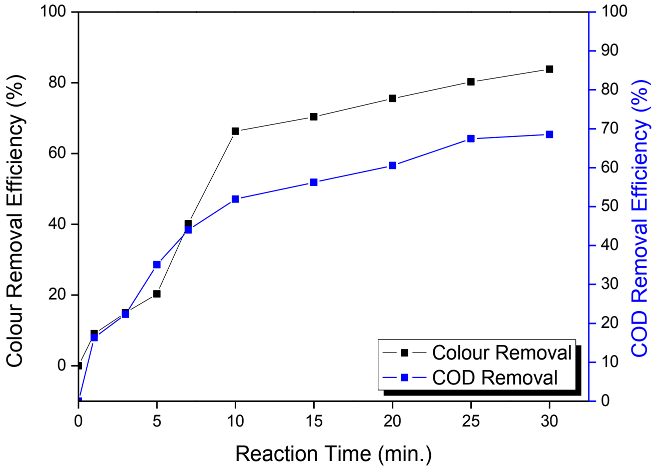


Figure 7. *Investigation of the colour and COD removal efficiencies using real textile wastewater with Al electrode [850 mg* O_2/dm^3 *COD and 188 Pt-Co units colour, 50 mA/*cm^2*, 2 mS/cm and pH 6.7]*

3.5.1. Consumptions of Electrode Material and Energy for Textile Wastewater Treatment

In this study, amount of electrode material and energy consumption were calculated for only real textile wastewater treatment experiments. The cost of electrochemical process is usually calculated from the usage of electrode material and spent electrical energy and are the most vital parameters affecting operational cost. The other necessary expenses such as sludge dewatering and disposal have not been considered in this study. The following well-known Faraday Law equation was used to calculate theoretical electrode consumption [13, 31]:

$$m_{anode} = \frac{ItM_{electrode}}{nFV} \tag{7}$$

where m_{anode} is dissolved electrode material amount (g/L), I is current intensity (A),t is time (sec.), $M_{electrode}$ is electrode molecular weight (g/mole), n is valence of metal ions (e^-/mole), F is Faraday constant (96500 Coulomb/mole) and V is treated solution volume (dm^3).

Experimental Energy usage of the EC process at the optimal conditions was calculated using the following equation [31];

$$E = \frac{IUt}{V} \tag{8}$$

where E is used energy (kWh/m^3), I is current intensity (A), U is voltage (V), t is time (h) and V is treated solution volume (m^3).

For real textile wastewater treatment, theoretically sacrificial Al electrodes consumption was calculated as 0.28 kg/m^3 from Faraday Law at experimental conditions of; 50 mA/cm^2, 850 mg O_2/dm^3 COD, 188 Pt-Co, 2mS/cm, pH:6.5, 30 min. Also, actual electrode loss was found as 0.35 kg/m^3 by weighing sacrificial Al electrodes before and after EC process. Under the same experimental conditions, energy consumption was achieved as 9.16 kWh per m^3 treated wastewater. Similar results [29, 32] and also little higher energy consumption [33] can be found in the literature for EC dye removal studies. As a result of this study, it can be said that EC appears to be an effective technique for the removal of dye wastewater.

4. CONCLUSIONS

The discharge of textile wastewater containing wide-ranging of organic pollutants in the environment is a big environmental threat due to contamination of watersheds and nearby areas with toxicological pollutants. These chemicals can eventually reach all living organisms and different ecosystems through the food chain. Electrocoagulation process with aluminium and iron electrodes was used to treat Dianix Brown S-3R synthetic dye solution. In this study, the effect of current density, initial dye concentration and pH on the performance of EC process were examined.

Dye removal rate was unchanged for Al electrode and decreased with increasing dye content for Fe electrode in different initial dye concentrations experiments. This was due to formation of adequate $Al(OH)_3$ flocs for removal of high dye concentration. The colour removal rates slightly increased with increasing conductivity of solution for both electrodes. Changes in initial pH value did not caused any remarkable effect on the treatment of synthetic dye solution by EC. Also, current density increase did not cause any change in dye removal due to sufficient amount of metal hydroxide flocs available for the removal of dye molecules in aqueous solutions for both electrodes.

Real textile industry effluent was applied to the EC process and maximum colour and COD removal of 83.8% and 68%, respectively were obtained after an electrolysis time of 30 min. Energy consumption of 9.16 kWh/m^3 was achieved under the process conditions (50 mA/cm^2, 2mS/cm conductivity and pH of 6.7). The electrode consumption was found as 0.28 and 0.35 kg of Al electrode per m^3 treated wastewater for theoretically calculated value and actual electrode loss, respectively. The results of this experimental work may help other researchers involved in wastewater treatment in finding effective, reliable, simple, robust and low-cost removal process for textile industry for keeping ecosystems healthy and less polluted water resources.

CONFLICTS OF INTEREST

No conflict of interest was declared by the authors.

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