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Removal of Reactive Red 24 Dye by Clean Electrocoagulation Process Using Iron and Aluminum Electrodes

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Abstract

In this research, the efficiency of electrocoagulation treatment process using iron and aluminum electrodes to treat synthetic wastewater containing Reactive Red 24 (RR 24) was studied. The effects of parameters such as current density, pH, type of electrolyte, initial dye concentration, electrolyte concentration, temperature, and inter electrode distance on dye removal efficiency were investigated. The results showed that dye and chemical oxygen demand removals were 99.6% and 91.5% by using iron and were 97.9% and 83.8%, by using aluminum electrodes. The removal of dye exhibited pseudo first order with good correlation coefficients (0.955 and 0.990 for Fe and Al electrodes respectively). It can be concluded that electrocoagulation process by Iron electrode is very efficient and clean process for reactive dye removal from colored wastewater.

Keywords: Electrocoagulation; Iron; Aluminum; Electrode; Textile wastewater

Introduction

Wastewater from textile dyeing and finishing factories is a significant source of environmental pollution [1]. Reactive dyes are extensively used in textile industry, fundamentally due to the ability of their reactive groups to bind to textile fibers by covalent bonds formation [2]. These characteristics facilitate the interaction with the fiber and reduce energy consumption [3]. The major environmental problem associated with the use of the reactive dyes is their loss in the dyeing process. The fixation efficiency is in the range 60-90% [3]. This wastewater containing dyes causes aesthetic problems due to the color. Also these dyes damage the quality of the receiving water because many of dyes released and their breakdown products are toxic, carcinogenic or mutagenic to life forms [4]. Azo dyes are the largest group of dyes used in the textile industry [5] constituting 20-40% of the dyes used for dyeing cotton, rayon, silk, wool, nylon and leather [6,7]. They have one or more azo groups (R1-N=N-R2) having aromatic rings mostly substituted by sulfonate groups. These complex aromatic conjugated structures are responsible for their intense color, high water solubility and resistance to degradation under normal conditions [8,9].

Decolorization efficiencies of C.I. Reactive Red 24 (RR 24) were 98.9% with initial concentration of 2400 mg/L after 24 h incubation period at its natural pH (6.95) [10]. The decolorization of (RR 24) in ultrasound, ultrasound/H₂O₂, exfoliated graphite, ultrasound/exfoliated graphite, exfoliated graphite/H₂O₂ and ultrasound/exfoliated graphite/H₂O₂ systems were compared [11]. Different parameters were investigated to evaluate their effect on the process removal efficiency of reactive dye from simulated spent reactive dye bath, by solar/TiO₂/H₂O₂, including H₂O₂ concentration, TiO₂ loading and pH. As a result 99% of reactive dye can be removed at a TiO₂ loading of 400 mg/L, H₂O₂ concentration of 150 mg L⁻¹ and of pH: 5.2 [12]. The effects of operating parameters, such as initial pH, catalyst, initial dye concentration and hydrogen peroxide were studied for degradation of C.I. Reactive Red 45 (RR45) dye in aqueous solution using advanced oxidation processes (AOPs), UV/TiO₂, UV/ZnO and photo-Fenton [13].

Dyes are extensively used in many fields such as textile, leather, paper, food industries. These extensive applications of synthetic dyes create environmental pollution and their toxic properties cause

health risk [14]. The most widely used methods to remove dyes are adsorption [15], ultrafiltration [16] and nanofiltration [17], oxidation [18], biological methods [19], etc. Electrocoagulation (EC) process has been mostly applied to treat dye-containing wastewaters because of the simplicity and high efficiency of the decolorization. This process was based on the formation of coagulants via electrolytic reactions at electrode surfaces. The production of destabilization agents brings about neutralization of electric charge for removing pollutant [20]. Electrocoagulation process also facilitates flocculation by turbulence generated by oxygen and hydrogen evolution at the cathode and anode electrodes. With the gas evolution, particles are destabilized and they start attracting each other and generate bigger particles. EC process depends on pH, particle size, concentrations, and the conductivity of the continuous phase. These experimental factors affect the results in different ways. Electrocoagulation has some advantages including simple equipment requirement, easy automation of the process, environmental compatibility, versatility, rapid reactions and smaller systems [21,22]. The maximum decolorisation efficiency in removing color from real textile wastewater by using both iron and aluminum electrodes reached 96% and 90% respectively for 90 minutes of treatment at optimal initial pH was 10 [23].

The purpose of this study is to conduct an experimental investigation on the removal of a reactive textile dye and COD C.I. Reactive Red 24 (RR 24) from the wastewater using the electrocoagulation method. Several fundamental aspects regarding the effects of type of electrolyte, initial pH, current density, electrolyte concentration, dye concentration, and inter electrode distance on the dye and COD removal efficiency are explored.

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Materials and Methods

Chemicals

The dye used in the present work was Reactive Red 24 (RR24), a textile dye also known as brilliant Red K-4BC, Reactive Red K-2BP, its IUPAC name is Trisodium (3Z)-5-[[4-chloro-6-(N-methylanilino)-1,3,5-triazin-2-yl]amino]-4-oxo-3-[(2-sulfonatophenyl)hydrazinylidene] naphthalene-2,7-disulfonate. The properties of the dye are given in Table 1. Sodium chloride, sodium fluoride, sodium carbonate, sodium phosphate, calcium chloride, potassium chloride, sodium hydroxide, sulfuric acid, potassium dichromate, silver sulfate were of analytical grade and purchased from Merck. Distilled water was used for the preparation of solutions. Standard solutions of potassium dichromate ($K_2Cr_2O_7$), sulfuric acid (H_2SO_4) reagent with silver sulfate (Ag_2SO_4) and potassium hydrogen phthalate (KHP) were prepared to measure the COD. A stock solution of dye (1000 mg/L) was prepared by dissolving an accurate quantity of the dye in distilled water and suitably diluted to the required initial concentrations. Different standard solutions of RR24 with concentration from 20-300 mg L^{-1} were prepared to measure its removal at different conditions. The pH of the working solution was adjusted to the desired values with 0.1N HCl or 0.1N NaOH.

Equipments and procedures

The electrocoagulation unit consisted of a 100 ml electrochemical reactor with anode and cathode with an effective surface area of 4 cm^2 . The electrodes were 20 mm \times 10 mm and inter electrodes distance was 0.8 cm. The electrodes were positioned vertically and parallel to each other. The current density was maintained constant by means of a precision DC power supply; model (DZ040019) EZ Digital CO. Ltd. (Korea). The dye concentration was determined using a double - beam UV-Vis spectrophotometer, model UV 1601 is from Shimadzu (Japan) at 515 nm. Hot Plate, model (HB502), BIBBY STERILIN LTD. (U.K.). A pH meter model AC28, TOA electronics Ltd., (Japan). Water bath model SB-650, Tokyo Kikakai CO. Ltd., (Japan). A closed reflux titrimetric unit was used for the COD determination. Chemical Oxygen Demand (COD), HANNA instruments, Thermo reactor, model C9800 Reactor in Hungary - Europe.

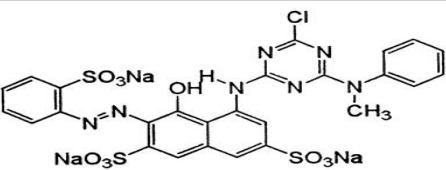
Dye	Reactive Red 24 (RR 24)
Chemical structure	
Chemical formula	$C_{26}H_{17}ClN_7Na_3O_{10}S_3$
IUPAC name	trisodium(3Z)-5-[[4-chloro-6-(N-methylanilino)-1,3,5-triazin-2-yl]amino]-4-oxo-3-[(2-sulfonatophenyl)hydrazinylidene] naphthalene-2,7-disulfonate
Molecular weight (g/mol)	788.07
λ_{max}	515 nm

Table 1: Properties of Reactive Red 24.

Analysis

Two main parameters were measured to evaluate the electrochemical treatment efficiency, the remaining pollutant concentration and the COD. Remaining pollutants (RR24) concentration was measured with the double-beam UV-visible spectrophotometer at λ_{max} = 515 nm using calibration curve with standard error $\pm 0.5\%$. The COD was determined using a closed reflux titrimetric method [24]. The equation used to calculate the color removal efficiency in the treatment experiments was:

$$\%E = [(A_0 - A)/A_0] \times 100 \quad (1)$$

Where A^* and A are absorbance values of dyes solutions before and after treatment with respect to their λ_{max} [25].

The calculations of COD removal efficiencies after electrocoagulation treatment were performed using the following formula [26].

$$C_R (\%) = [(C_0 - C)/C_0] \times 100 \quad (2)$$

Where C_0 and C are concentrations of wastewater before and after electrocoagulation.

Results and Discussion

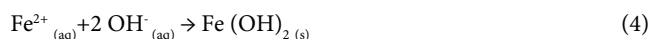
In the electrocoagulation process, coagulant is generated as a result of oxidation of the anode material by passing the electrical current. Electrocoagulation of dye solution using iron and aluminum electrodes takes place according to the following mechanisms [27-30] for the production of $Fe(OH)_n$ and $Al(OH)_n$, where $n=2$ or 3.

Mechanism 1:

Anode:



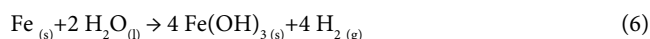
Bulk of solution:



Cathode:



Overall:

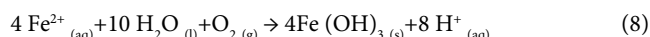


Mechanism 2:

Anode:



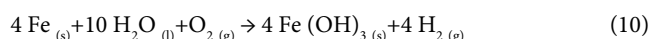
Bulk of solution:



Cathode:



Overall:

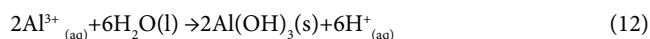


Mechanism 3:

Anode:



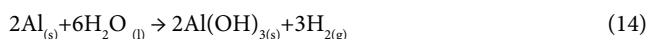
Bulk of solution:



Cathode:



Overall:



The generation of metal hydroxides $[\text{Fe}(\text{OH})_n]$ and $[\text{Al}(\text{OH})_n]$ are followed by an increase in the concentration of colloids (usually negatively charged) in the region close to the anode [31]. The produced ferrous ions hydrolyze to form monomeric hydroxide ions and polymeric hydroxide complexes that depend on the pH of the solution. The polymeric hydroxides, which are highly charged cations, destabilize the negatively charged colloidal particles allowing the formation of flocs. When the amount of iron in the solution exceeds the solubility of the metal hydroxide, the amorphous metal hydroxide precipitates is formed, which causes sweep-floc coagulation [32].

Effect of current density

The effect of current density on the removal of RR24 dye and COD was studied at four different current densities, i.e., 5, 10, 15 and 20 mA cm⁻². The reactions were carried out for 14 min and 35 min using Fe and Al electrodes respectively under the following conditions: the initial concentration of 100 mg L⁻¹, pH of 7.2, inter electrode distance of 0.8 cm, a temperature of 20 °C and NaCl concentration of 2.5 g L⁻¹. Figure 1 and Table 2 show the effect of current density for the removal of dye and COD from aqueous solutions. The removal efficiency of RR 24 and COD increased up to [99.85% and 90.5% at Fe and 98.7% and 83.8% at Al respectively] by increasing the current density. The current density determines the coagulant production rate and the size of the bubble production and hence affects their growth [33,34]. Upon increasing current density, the amount of oxidized iron increased and amounts of metal hydroxide compounds for precipitation and adsorption of the matrix were also increased [35].

Effect of initial pH

It has been established in previous studies that initial pH has a considerable effect on the efficiency of the electrocoagulation process for removal of organic pollutants [36,37]. Figure 2 and Table 2 show the effect of pH on dye removal and COD efficiency at 14 min and 35 min using Fe and Al electrodes respectively at initial concentration of 100 mg L⁻¹, a current density of 10 mA cm⁻², inter electrode distance of 0.8 cm, a temperature of 20°C and NaCl concentration of 2.5 g L⁻¹. From Figure 2(a) and Table 2(a), the removal efficiency of the dye and COD is low in acidic electrolyte, meanwhile, in neutral and alkaline medium; the removal efficiency is much higher using iron electrodes. Also, as pH increases the dissolved iron weight during the electrocoagulation process increases due to the formation of iron hydroxide species which absorb the dye molecules and causes the increase of the removal efficiency. The effect of pH on the process performance is explained as follows: the dominant iron species are different according to the solution pH; at basic pH, dye removal efficiency is increased as concentration of OH⁻ ion increases which help in formation of different iron hydroxide complexes [38-40].

Figure 2(b) and Table 2(b) display the removal efficiency of the dye and COD is high in acidic electrolyte using aluminum electrodes. At lower initial pH, OH⁻ ions are produced at the cathode, causing increase of pH of solutions. Al (OH)₃(s) and aluminum hydroxyl complexes formed in aqueous solutions can remove dyes by precipitation. The adsorption rate of dyes by those species is lower than precipitation at lower pH because of the protonated functional groups of dye molecules. At higher initial pH, deprotonated functional groups of dyes enable

adsorption of dyes on Al (OH)₃(s) and aluminum hydroxyl complexes. Hence dye removal by precipitation is easier at lower pH while both adsorption and precipitation occur at higher pH [41].

Effect of type of electrolyte

Figure 3 and Table 2 explain the effect of electrolyte type on the removal efficiency of RR24 dye and COD at 14 min and 35 min using Fe and Al electrodes respectively in the presence of different supporting electrolytes including NaCl, KCl, CaCl₂, NaF, Na₂CO₃, Na₃PO₄ were studied at initial concentration of 100 mg L⁻¹, time of 14 min, a current density of 10 mA cm⁻², inter electrode distance of 0.8 cm, a temperature of 20 °C, pH of 7.2 and at electrolyte concentration of 2.5 g L⁻¹. It can be seen from Figure 3(a) and Table 2(a) that in the presence of chloride ions of NaCl, KCl, CaCl₂ electrolytes the removal efficiency of RR24 dye [99.66, 99.6 and 98.5%] and COD [90.5, 84.2, 80.2%] were at the time of 14 min using Fe electrode respectively. But other electrolytes which not contain chloride ions as NaF, Na₂CO₃, Na₃PO₄ the removal efficiency reached to lower value at the same time. Also Figure 3(b) and

Fe (a)						
Current density (mA/cm ²)	-	5	10	15	20	-
COD (%)	-	67.5	90.5	85.1	83.3	-
pH	1.8	4.5	7.2	8	10	-
COD (%)	90.2	83.4	90.5	77.9	63.2	-
Electrolyte	KCl	CaCl ₂	NaCl	Na ₂ CO ₃	Na ₃ PO ₄	NaF
COD (%)	84.2	68.2	90.5	58	84.6	77.5
[NaCl] (g/L)	0.05	0.15	0.25	0.35	0.45	-
COD (%)	67.6	88.8	90.5	64.5	61.6	-
[Dye] (mg/L)	20	50	100	200	300	-
COD (%)	88.4	82.8	90.5	70.5	56.4	-
Inter electrode distance (cm)	0.4	0.6	0.8	1	1.5	2
COD (%)	75.6	72.2	90.5	72.3	60.3	48.3
Temperature (°C)	5	10	20	30	40	50
COD (%)	67.1	74.4	90.5	66.6	76.7	68.4
Al (b)						
Current density (mA/cm ²)	-	5	10	15	20	-
COD (%)	-	60.5	83.8	78	76.2	-
pH	1.8	4.5	7.2	8	10	-
COD (%)	82.2	77.4	83.8	70.9	56.4	-
Electrolyte	KCl	CaCl ₂	NaCl	Na ₂ CO ₃	Na ₃ PO ₄	NaF
COD (%)	77.4	61.2	83.8	50	75.8	77.5
[NaCl] (g/L)	0.05	0.15	0.25	0.35	0.45	-
COD (%)	59.6	82.8	83.8	58	54.8	-
[Dye] (mg/L)	20	50	100	200	300	-
COD (%)	80.6	75.8	83.8	64.5	48.3	-
Inter electrode distance (cm)	0.4	0.6	0.8	1	1.5	2
COD (%)	67.7	65	83.8	66.1	53.2	48.3
Temperature (°C)	5	10	20	30	40	50
COD (%)	58.5	67.7	83.8	59.6	70.9	61.2

Table 2: Effect of current density, pH, Type of electrolyte, Concentration electrolyte, dye concentration, inter electrode distance and temperature on the efficiency of COD removal for reactive red 24 dye using Fe (a) and Al (b) electrodes

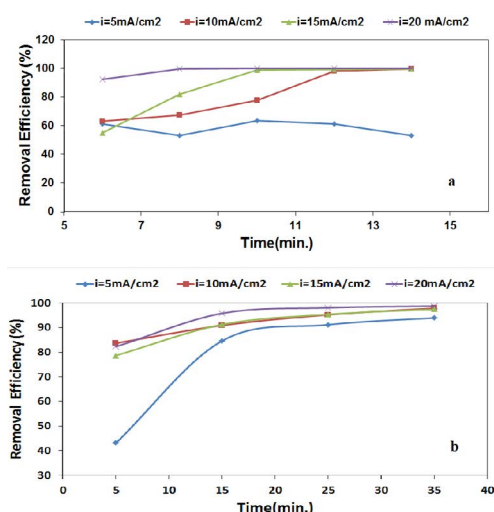


Figure 1: Effect of current density on the efficiency of reactive red 24 dye removal using Fe (a) and Al (b) electrodes. Initial concentration of the dye=100 mg L⁻¹, [NaCl] concentration=2.5 g L⁻¹, pH=7.2, inter electrode distance=0.8 cm and temperature=20°C.

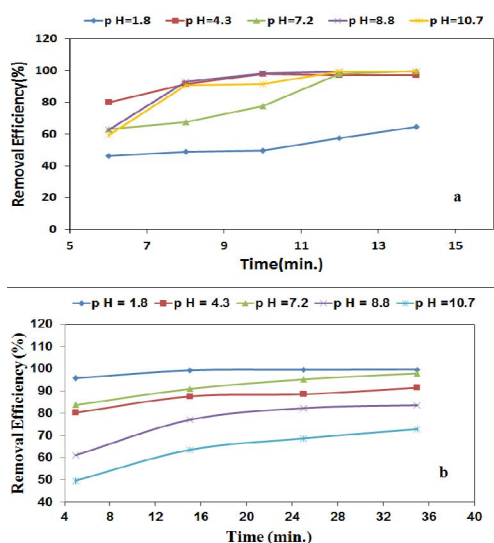


Figure 2: Effect of pH on the efficiency of reactive red 24 dye removal using Fe (a) and Al (b) electrodes. Initial concentration of the dye=100 mg/L, [NaCl] concentration=2.5 g L⁻¹, current density=10 mA cm⁻², inter electrode distance=0.8 cm and temperature=20°C.

Table 2(b) explain that in the presence of chloride ions of NaCl, KCl, CaCl₂ electrolytes the removal efficiency of RR24 dye [97.9, 96.4 and 95.9%] and COD [83.8, 77.4, 72.2%] were at the time of 35 min using Al electrode respectively. Later experiments were done using NaCl because it is cheap and the solution contains it has high conductivity thus it need low voltage for electrocoagulation and so it is economical in industrial scale.

Effect of electrolyte (NaCl) concentration

It is important to investigate the effect of electrolyte concentration since actual wastewater usually contains certain amount of salts. Figure 4 and Table 2 represent the effect of electrolyte concentrations on dye removal and COD at 14 min and 35 min using Fe and Al electrodes respectively at initial concentration of 100 mg L⁻¹, a current density of

10 mA cm⁻², inter electrode distance of 0.8 cm, a temperature of 20°C and at pH of 7.2. Figure 4 and Table 2 show that, as the electrolyte concentration increased, the removal efficiency increased due to the increment of the electrical conductivity reaching the maximum value at 2.5 g L⁻¹ NaCl. However, with the increase in NaCl concentration >2.5 g L⁻¹ the removal efficiency decreased. It can be attributed that at a constant voltage with increasing of electrolyte concentration, conductivity of dye solution increases and resistance decreases, so the passed current increases and the produced amount of metallic hydroxide and dye removal increases [42].

Effect of initial dye concentration

To determine influence of initial dye concentration on dye and COD removals efficiencies during electrocoagulation, different initial concentrations in the range of 20-300 mg L⁻¹ were treated at 14 min and 35 min using Fe and Al electrodes respectively in the optimum condition (a current density of 10 mA cm⁻², NaCl concentration of 2.5 g L⁻¹, a temperature of 20°C, pH of 7.2, and inter electrode distance of 0.8 cm). Results showed that when dye concentration increased from 20 to 300 mg L⁻¹, removals efficiencies decreased (Figure 5 and Table 2). One of the most important pathways of dye removal by electrocoagulation is adsorption of dye molecules on metallic hydroxide flocs. The adsorption capacity of flocs is limited and specific amount of flocs is able to adsorb specific amount of dye molecules. So, with increasing of dye concentration, amount of produced flocs is insufficient to adsorb all dye molecules, therefore dye and COD removal decreases.

Effect of inter electrode distance

Figure 6 and Table 2 display the effect of inter electrode distance on RR24 dye removal and COD at 14 min and 35 min using Fe and Al electrodes respectively at initial concentration of 100 mg L⁻¹, a current density of 10 mA cm⁻², a temperature of 20°C, pH of 7.2 and at NaCl concentration of 2.5 g L⁻¹. Figure and Table show that, as the distance between the two electrodes decreased, the removal efficiency increased due to the increment of the electrical conductivity reaching

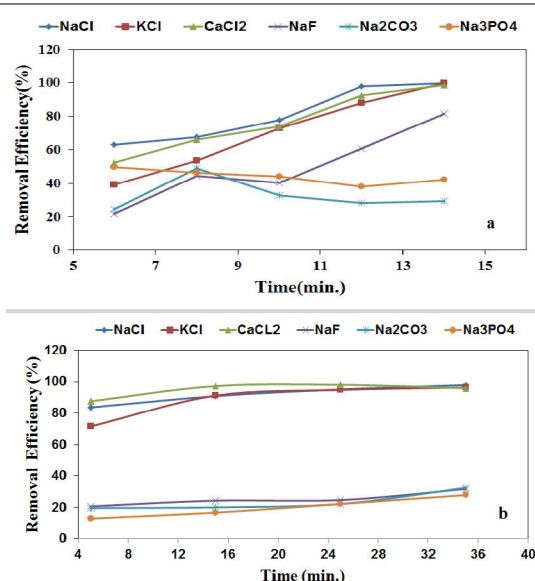


Figure 3: Effect of type of electrolyte on the efficiency of reactive red 24 dye removal using Fe (a) and Al (b) electrodes. Initial concentration of the dye=100 mg L⁻¹, current density=10 mA cm⁻², pH=7.2, inter electrode distance=0.8 cm and temperature=20°C.

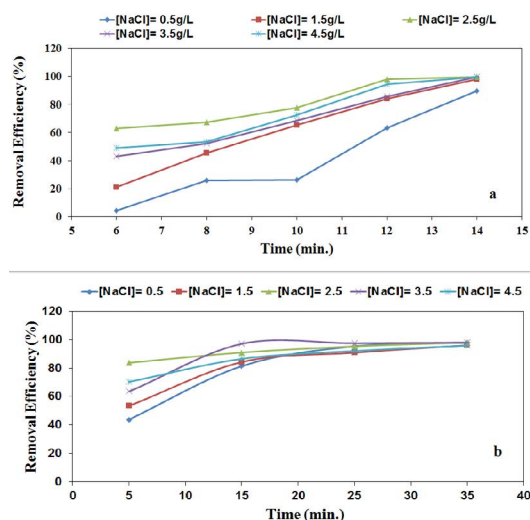


Figure 4: Effect of electrolyte concentration on the efficiency of reactive red 24 dye removal using Fe (a) and Al (b) electrodes. Initial concentration of the dye=100 mg L⁻¹, current density=10 mA cm⁻², pH=7.2, inter electrode distance=0.8 cm and temperature=20°C.

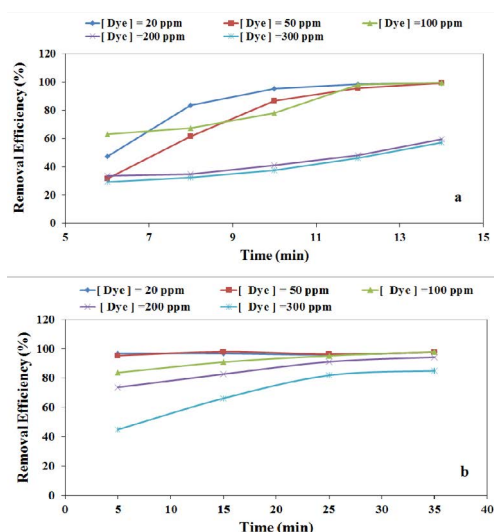


Figure 5: Effect of initial dye concentration on the efficiency of reactive red 24 dye removal using Fe (a) and Al (b) electrodes. [NaCl] concentration=2.5 g L⁻¹, current density=10 mA cm⁻², pH=7.2, inter electrode distance=0.8 cm and temperature=20°C.

the maximum value at 0.8 cm. However, with increasing the distance 0.8 to 2 cm the removal efficiency decreased. It can be attributed that with increasing of distance between electrodes at constant voltage, electrical resistance between electrodes increases and current passed through electrodes decreases. The decreasing of current, lead to lower production of hydroxyl ions and dye removal efficiency decreases [42].

Effect of temperature

Figure 7 and Table 2 show the effect of temperature on RR24 dye removal and COD at 14 min and 35 min using Fe and Al electrodes respectively at initial concentration of 100 mg L⁻¹, a current density of 10 mA cm⁻², inter electrode distance of 0.8 cm, pH of 7.2 and at NaCl concentration of 2.5 g L⁻¹. The results from Figure and Table indicate that increasing temperature has a negative effect on removal

efficiency of dye and COD, where at 20°C the dye removal and COD % reached to 99.67 and 90.5 using Fe electrode and reach to 97.9 and 83.8 using Al electrode respectively. While at higher temperature value (50°C) the dye removal and COD % dropped to 97.6 and 68.4 using Fe electrode and dropped to 95.1 and 61.2 using Al electrode respectively. However, it should be noted that the operation of electrocoagulation process at higher temperature significantly reduced electrical energy consumption and fluid conductivity increases. So, the production of hydroxide species increases rapidly then enhances pH value. The increase in pH may be affecting on iron and aluminum species to be in the oxide form and did not exist in the hydroxide form which is the coagulant species that should remove the dye.

Kinetic studies

Kinetics studies have important role in determining the rate constant and the order of reaction of this treatment removal. So, rate constant is very significant in the design of wastewater treatment units. It is very essential to know the type of reaction rates for design a wastewater treatment unit. Rate of reaction describes the rates of change in concentration of reactant per unit time. Figure 8 represents the removal of dye exhibited pseudo first order with good correlation coefficients (0.955 and 0.990 for Fe and Al electrodes respectively) according to following equation:

$$\ln A_0/A_t = -k t \quad (15)$$

where

A_0 , A_t , t , and k are the dye absorbance before reaction, dye absorbance after reaction, time of reaction (min), and reaction rate constant (min⁻¹), respectively. The values of rate constants at optimum condition and reaction time were 0.207 and 0.070 min⁻¹ using Fe and Al electrodes respectively. Results show that the removal rate using Fe electrode was higher than the removal rate using Al.

Conclusion

The removal efficiency of Reactive Red 24 (RR 24) dye and COD from aqueous solution was examined by electrocoagulation using iron

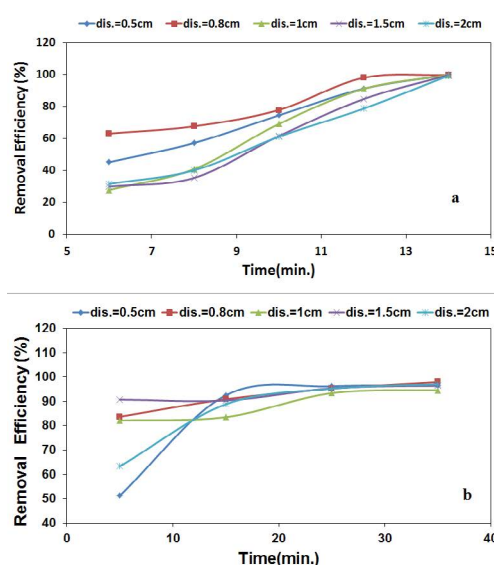


Figure 6: Effect of inter electrode distance on the efficiency of reactive red 24 dye removal using Fe (a) and Al (b) electrodes. Initial concentration of the dye=100 mg L⁻¹, current density=10 mA cm⁻², pH=7.2, [NaCl] concentration=2.5 g L⁻¹ and temperature=20°C.

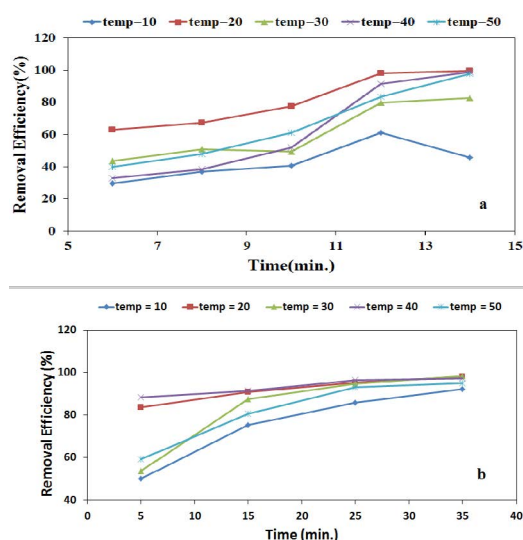


Figure 7: Effect of temperature on the efficiency of reactive red 24 dye removal using Fe (a) and Al (b) electrodes. Initial concentration of the dye=100 mg/L, current density=10 mA cm², pH=7.2, [NaCl] concentration=2.5 g L⁻¹ and inter electrode distance=0.8 cm.

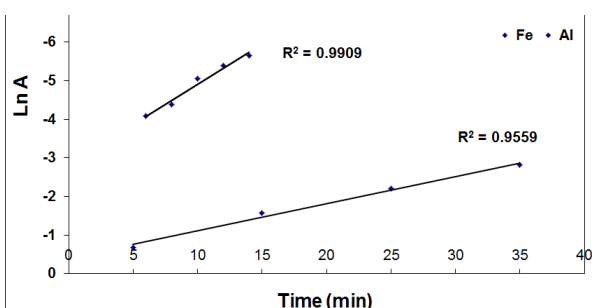


Figure 8: Relation between Ln A against the time for reactive red 24 dye removal using Fe (a) and Al (b) electrodes. Initial concentration of the dye=100 mg L⁻¹, current density=10 mA cm², pH=7.2, [NaCl] concentration=2.5 g L⁻¹, inter electrode distance=0.8 cm and temperature=20°C.

(Fe) and aluminium (Al) electrodes. The effects of initial pH, initial dye concentration, current density, type electrolyte, salt concentration, inter electrode distance and temperature was investigated on removal efficiency of dye and COD.

It was observed that these variables significantly affected the RR 24 dye removal efficiency. The optimum RR 24 dye removal was obtained with typical operating conditions: initial concentration of 100 mg L⁻¹, pH of 7.2, inter electrode distance of 0.8 cm, a temperature of 20°C and NaCl concentration of 2.5 g L⁻¹. The results showed that dye and chemical oxygen demand removals were 99.6% and 91.5% by using iron and were 97.9% and 83.8%, by using aluminum electrodes.

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