

Research Article

Color Removal of Textile Wastewater Using Electrochemical Batch Recirculation Tubular Upflow Cell

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The progress in textile industrial technologies comes along with a massive increase in the discharge of dyes in the wastewater which considers a serious environmental problem. In this regard, a new electrochemical system has been developed for the treatment of simulated dye solutions of permanent methylene blue dye by an electrochemical cyclic ring reactor. An aluminum rod and a stainless steel mesh were used as the anode and cathode. The experiments on the artificial dye solutions have been carried out in a 6-liter electrochemical cell containing 50 ppm neutral dye solutions. The effects of various parameters such as electrolysis time applied current density (2, 3.32, 5.31, 6.64, and 7.46 mA cm⁻²), electrolyte concentrations (600, 900, 1200, 1500, and 1800 ppm), and flow rates (1, 1.5, 2, 2.5, and 3 Lh⁻¹) on the process removal efficiency were examined. The results demonstrated that the removal efficiency reached 94–99% within 40–50 minutes of electrolysis time. The removal efficiency increased by increasing the flow rates until it reaches a maximum value at a flow rate of 2 Lh⁻¹; thereafter, it declined with the farther augment of recirculation speed. It is indicated that raising the applied current resulted in increasing the removal efficiency. However, the power consumption builds up to the maximum value by increasing the applied current, where the power consumption rose from 8.51 to 30.3 kWh kg⁻¹ with an increase in the current density from 2 to 7.46 mA cm⁻², and a removal efficiency increased from 94% to 99%, accordingly. The results also showed that by increasing the electrolyte concentration, the power consumption can be reduced to its minimum value and the removal efficiency increased remarkably.

1. Introduction

Wastewater from many industries contains various pollutants, such as active bacteria, dissolved salts, suspended solids, organics, and dyes [1–4]. Among various types of industrial effluents, wastewater of dyes produced from industries such as paper, printing, textile painting, and cosmetics industries causes a series of health and environmental problems [5, 6]. In addition, around 700,000 tons of pigments and dyes are produced annually around the world [7]. The major drawbacks associated with the presence of dyes in the water resources are that it prevents the penetration of

sunlight, impairs oxygen replenishment, causing chronic and acute toxicity, aesthetic pollution, and serious health risks [8]. Usually, these synthetic dyes have complex molecular structures that are chemically and biologically stable, and it is difficult to be prone to degradation [9]. The synthetic structure of the dye may include azo, diazo, base, acid, sulfone, anthraquinone, a metal complex, azine, or a combination of all these structures [1].

Therefore, it is vital to the industries using dyes to find adequate wastewater treatment methods to satisfy the health and environmental concerns of the dyes in industrial effluents [2]. Consequently, various techniques have been

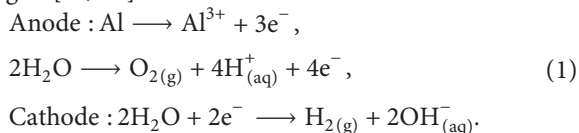
utilized for removing dyes from wastewater which include biodegradation, oxidation, adsorption of Fenton, membrane separation, ozone, and coagulation [10–13]. Enzymatic or biological processing is less effective because most of the dye synthesis is biodegradable [1, 14].

Electrochemical (EC) processes are commonly known as green progress oxidation processes for wastewater treatment [6]. This treatment method has been successfully employed for gaining consideration as a promising technology for the treatment of different wastewater. The electrochemical method offers incomparable features in the treatment of dye wastewater such as high removal and energy efficiency, versatility, and less space requirement [15–19]. Furthermore, in electrochemical technology (electrocoagulation-electroflotation), it is not necessary to add external chemicals during the treatment, and the process prevents the release of other pollutants into the environment [20, 21]. During the electrochemical process, the toxic organic substances are demolished by the action of active coagulant precursors into the solution which produces by oxidation of the anode material [22–25].

However, in this process, the sacrificial anode is replaced regularly. Another possible drawback of this method is the potential effect of extra doses of the electrode metal on the biomass if the process is used in harvesting biomass [26–31].

In the electrocoagulation process in which metals, such as iron and aluminum, are dissolved by electricity and metal hydroxides are formed in wastewater, which causes coagulation/agglomeration of pollutants [32–38]. The flocs generated can be removed from the solution either sedimentation or flotation by the action of the hydrogen and oxygen gas bubbles produced on the anode and cathode electrodes. In practice, this treatment method is suitable for many types of wastewater: protein and food waste, drinking water, nonferrous wastewater and textiles, petroleum waste, industrial wastewater, heavy metals, fluorides, and polyvinyl alcohol [39, 40].

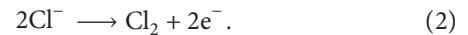
The cathodic reduction of water solution and the metal anode (mostly Al or Fe) oxidation are the primary reactions that may happen, a competitive reaction of water oxidation to oxygen [41, 42]:



The anode that emits metal cations may move by various equilibrium reactions; yet, depending on the solution, the most distinguish include metallic hydroxides formation which can adsorb various kinds of poisons and precipitate them. The water solution reduction reaction in the cathode leads to hydrogen bubbles creation, which changes the aqueous solution density preferring the detachment of the flocs created. However, in closed systems, it is probable to gather the generated gas and reuse it as a reagent or fuel [43, 44].

It is important to spotlight that in the presence of chlorides, a situ-oxidizing agent is generated during the electrocoagulation process. Active chlorine species such as

chlorine, hypochlorous acid, and/or hypochlorite could be produced by the anodic oxidation of chlorine according to the following reactions [45, 46]:



The released chlorine produces hypochlorous acid:

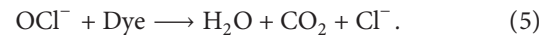


Chloride separates more to produce hypochlorite ions, which at a higher pH, $\text{pH} > \sim 6$, dissociate to produce hypochlorite ions:



That extremely vigorous oxidizing agent (HOCl, OCl, ClO^{-2} , and Cl_2) could deteriorate the surface of the organic substance that outcomes in the destruction of the pollutants [42, 43].

The role of hypochlorite in the wastewater electrochemical treatment with dyes in the production of chlorine is given as follows:



The goal of the current research is to verify the optimal working conditions for the elimination of methylene blue dyes in aquatic solution using a recirculation batch reactor. Aluminum and stainless steel, which are inexpensive materials for the manufacture of electrodes, were utilized as the anode and cathode. The experiment was carried out using a single upflow reactor consisting of a middle aluminum anode ringed by a stainless steel cathode mesh. The effect of the key operating parameters, such as salt concentration, flow rate, and the density of current on removal efficiency, was on the current efficiency and operation cost investigation.

2. Experimental Work

2.1. Materials. Methylene blue dye (Thomas Baker, India) was used as a model to study the efficiency of the electrocoagulation treatment process. The chemical formula of this dye is $\text{C}_{16}\text{H}_{18}\text{ClN}_3\text{S}$, and the molecular weight is 319.85 g/mol. The chemical structure of methylene blue dye appears in Figure 1. Distilled water was used to dilute the methylene blue dye to the concentration of 50 ppm. The initial solution pH was 7.2.

NaCl was added to the solution to improve the electrolyte conductivity provides higher removal rates compared to other salts. Since Cl^{-} is useful for scattering electrodes surrounding a metal oxide layer. An electrolyte of NaCl is used to higher the dissolution rate of the anode and to ensure the good release of aluminum hydroxide. This electrochemical method provides an alternative method of indirect oxidation to discolor waste staining with reactive chlorine forms on the anode [39].

2.2. Experimental Procedure. The experiments were conducted by using the laboratory setup as shown in Figure 2. A tubular electrochemical reactor is made of an acrylic glass tube

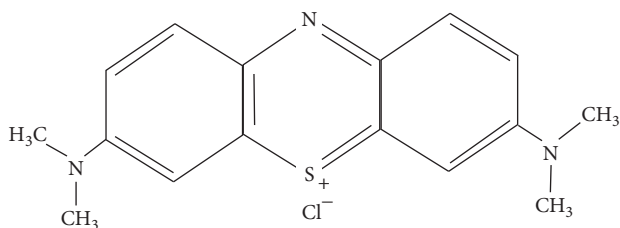


FIGURE 1: Chemical structure of methylene blue dye.

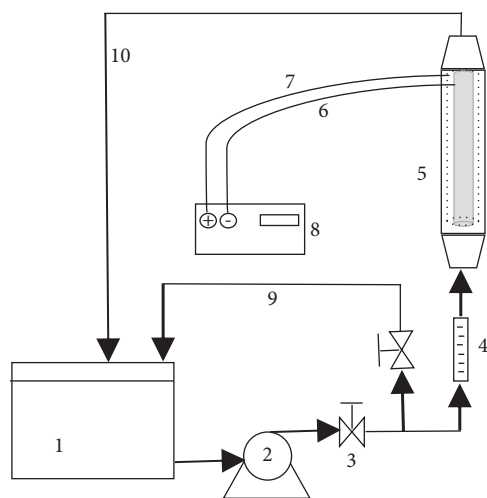


FIGURE 2: Schematic diagram of the laboratory-scale setup: (1) sample tank, (2) pump, (3) valve, (4) rotameter, (5) tubular reactor, (6) anode wire, (7) cathode wire, (8) DC power supply, (9) recycle, and (10) treated effluent flow.

with a 7 cm diameter and 30 cm height. A perforated circular plate was fixed in the bottom of the reactor to maintain a uniform distribution of flow and hold the axial anode.

The anode was fabricated from a middle aluminum bar of 5 cm diameter and 24.5 cm height, suspended by a perforated plate fixed at the top of the tubular reactor. The cathode which was made from cylindrical stainless steel mesh no. 10 and was fixed around the anode of 1 cm distance. A controlled DC power supply was utilized to provide various anodic current densities of 2, 3.32, 5.31, 6.64, and 7.46 mA cm⁻². The current and voltage were measured using an ammeter and voltmeter. The other elements of the setup are a reservoir of 7 L volume, a rotameter and a pump, rubber tubes for joining, and valves.

2.3. Electrochemical Method. The synthesised dye solution consists of methylene blue with a concentration of 50 ppm of dye in distilled water. Various concentrations of the electrolyte (NaCl) i.e., 600, 900, 1200, 1500, and 1800 ppm were used in the experiments. In each run, the reservoir of the recirculation batch mode contained 6 liters of simulated dye aqueous solution at room temperature of 25°C (±1). The electrolyte was introduced to the cell by an electrical pump at different volumetric flow rates of 1, 1.5, 2, 2.5, and 3 Lh⁻¹. During 90 minutes of the experiment proceeding, samples

were collected every 10 min from the reservoir to examine the concentration of the dye. The solutions' pH is maintained by adding 0.01 M solutions of HCl and NaOH.

After the accomplishing the experiments, the anode and cathode were washed with distilled water and propanone for 5 min to remove the organic pollutants.

The dye removal efficiency R.E.%, which indicates the color removal degree from the dye solution, is estimated by the following equation [47]:

$$\text{R.E.}\% = \frac{A^{\circ} - A}{A^{\circ}} * 100, \quad (6)$$

where A_o and A are the light absorbance of methylene blue dye before and after electrocoagulation, respectively.

The concentration of methylene blue dye in the solution was examined by using a spectrophotometer (Jenway 6300) at a wavelength of $\lambda_{\text{max}} = 662$ nm.

3. Results and Discussion

3.1. Removal Efficiency as a Function of Time Effect. The influence of electrolysis time on the efficiency of removal was investigated for 90 minutes. In this experiment, the current density was maintained at about 5.31 mA cm⁻², 50 ppm dye concentration, the flow rate of 2 Lh⁻¹, 1500 ppm electrolyte concentration, and initial pH was kept around 7 ± 0.1. Figure 3 demonstrated that the raising in the electrolysis time from 10 to 90 minutes leads to an increase in the color removal by 37%. In this first state, the dye removal efficiency was 62% at electrolysis time of 10 min and it increased to 99% at electrolysis time of 90 min at the final state. Also, it was observed that the removal efficiency increased sharply to around 96% during the first 40 minutes of treatment time, thereafter, it is increased gradually to about 99% for the rest 50 minutes of operation time.

The augment of removal efficiency with the electrolysis time could be attributed to the increase of the hydroxide concentration with the rising of electrolysis time. That agrees with the results obtained by most of the electrocoagulation process [48, 49].

3.2. Recirculation Speed Effect on Removal Efficiency. The dynamic response of dye removal performance of the system was studied at various flow rates of 1, 1.5, 2, 2.5, and 3 Lh⁻¹, at specific conditions of dye concentration (50 ppm), electrolyte concentration (1500 ppm), and current density of 5.31 mA cm⁻². The results are presented in Figure 4.

The organic contaminant destruction rate is significantly high at higher rates of flow because of the increased oxidants production during the process. Cl⁻ ion transport from the bulk to the surfaces of the electrode accelerates when the rate of electrolyte circulation increases. This would facilitate the Cl₂ dissolution to create OCl⁻ for organic contaminant reaction as well as Cl₂ generation [50].

The recirculation flow of an electrochemical device performs two features in the upflow reactor. Firstly, increasing the flowrate enhances the mixing of solution leads to promoting the mass transfer of a solute from the solution

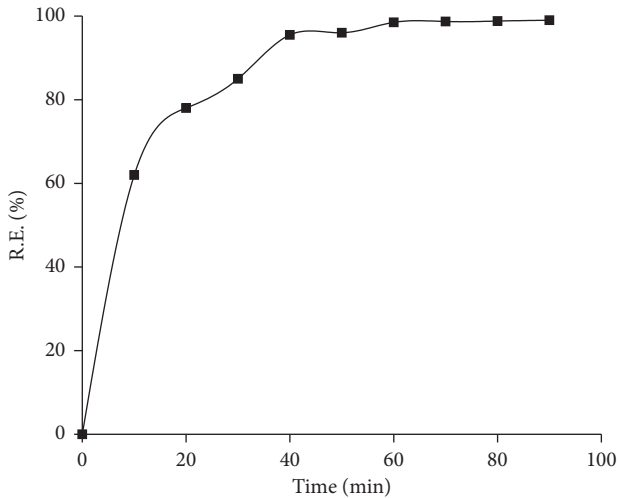


FIGURE 3: Color removal efficiency with time.

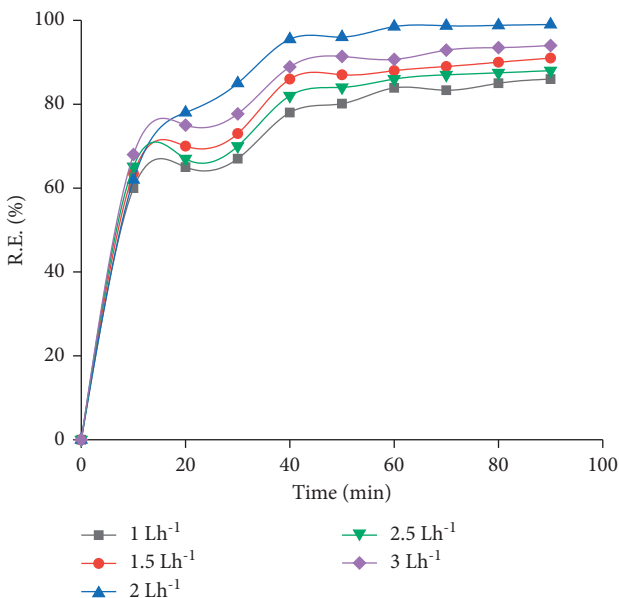


FIGURE 4: Influence of the flow rate on removal efficiency with electrolysis time.

to the stable thin liquid film on the electrode surface and accelerates the adsorption and desorption of organic substances with flug the electrode surface. The other function is to reduce the liquid film that covered the surface of the electrode [23].

This is verified in Figure 4, that the elimination efficiency of the contaminates enhanced significantly with the increase of the upflow rate. It can be noticed that at an electrolysis time of 40 minutes, the removal efficiency increased from 78% at a flow rate of 1 Lh⁻¹ to around 96% with a liquid flow rate of 2 Lh⁻¹. The improvement in the process performance after 50 min of operation was marginal.

However, when the recirculation flow rate exceeds 2 Lh⁻¹, the dye removal efficiency was decreased. This can be because the flocs generated in the suspension might be degenerated by the action of high mixing corresponding to a

high flow rate [51]. The residence time is not sufficient to complete the electrooxidation of dye on the surface of the electrode by high recirculation speed. An increase in the recirculation flow rate might not positively affect the mass transfer of organic substance and oxygen so that the dye removal does not enhance significantly [52]. Therefore, it is necessary to maintain an appropriate recirculation velocity (2 Lh⁻¹) throughout the remediation. That result comes in line with [53, 54].

3.3. Applied Current Density Effect. It is well known that the applied current is one of the essential parameters in the electrochemical process. Different current densities of 2, 3.32, 5.31, 6.64, and 7.46 mA cm⁻² were utilized to study the density of current influence on the methylene blue dye removal efficiency. The dye concentration was maintained at 50 ppm, the NaCl concentration was 1500 ppm, 2 Lh⁻¹ flow rate, and pH of 7 ± 1.

The results demonstrated that the removal efficiency is highly influenced by the current density and is positively related as illustrated in Figure 5. It can be noted that at 20 minutes of electrolysis time, the removal efficiency increased from 65% at a density of current of 2 mA cm⁻² to around 81% at a current density of 7.4 mA cm⁻². After 30 minutes of electrolysis time, the increase in applied current from 2 to 7.4 mA cm⁻² achieved around 7% increment in removal efficiency, and that difference in removal efficiency was persisted for the rest of the electrolysis time.

The dye removal efficiencies are enhanced by elevating the current densities due to the enhancement in the dissolving rate of aluminum according to the law of Faraday. That increases the formation of Al(OH)₃ and Al_n(OH)_{3n} and also augment the hypochlorite/chlorine generated in the suspension. Furthermore, the increasing of the current density leads to an increase in the rate of H₂ and O₂ microbubble formation of the surface of the electrodes that enhance the upper force and prompt the flotation process [54].

After a certain time of operation, around 30 minutes, the aluminum oxides that formed in the bulk reaches a level enough to consume most of the pollutants in the solution. Therefore, after that time, the increase in the applied current attained scant progress in removal efficiency [55]. The results of wastewater dyes removal obtained by other research demonstrated that the increasing of applied current caused a dramatic increase in dyes removal efficiency [54, 56].

3.4. Supporting Electrolyte Concentration Influence on Dye Removal Efficiency. The influence of salt electrolyte (NaCl) concentration on the rate of removal of contaminants was studied for five various values of 600, 900, 1200, 1500, and 1800 ppm with a methylene blue concentration of 50 ppm, the flowrate of 2 Lh⁻¹, and 5.31 mA cm⁻² current density as illustrated in Figure 6.

The removal efficiency of dye was greatly improved by the increase of the NaCl concentration. The removal efficiency increased from 62% to around 80% when the NaCl

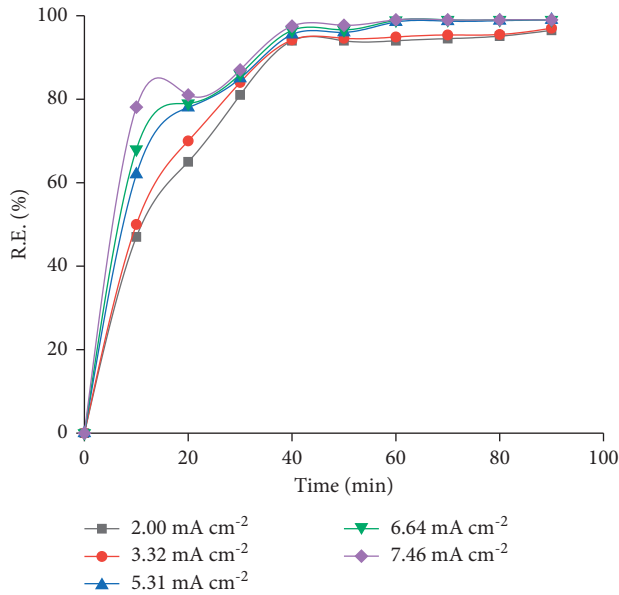


FIGURE 5: Current density's influence on the efficiency of removal.

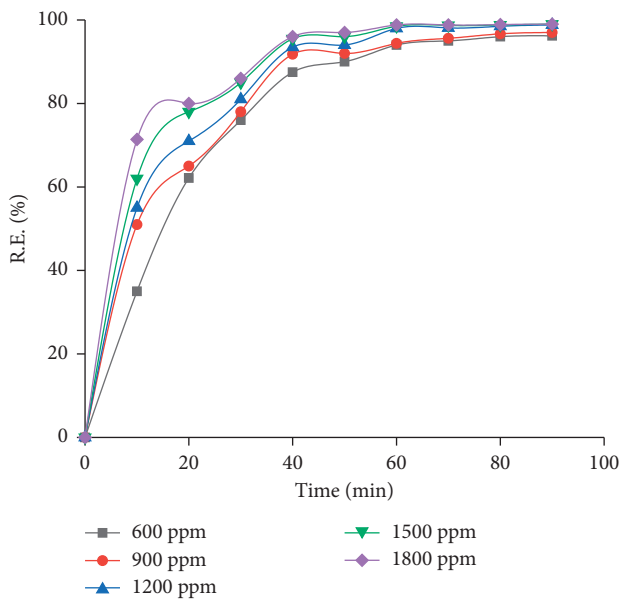


FIGURE 6: Influence of salt electrolyte on the efficiency of removal.

concentration increased from 600 ppm to 1800 ppm at the same electrolysis time of 20 minutes.

The advantage of the addition of sodium chloride was demonstrated by the role of chloride ion indirect electrochemical oxidation. The color could be successfully decomposed through the strong oxidizing of active chlorine species (chlorine, hypochlorous acid and/or hypochlorite, and hypochlorous acid) which were created throughout the reactions (2)–(5).

In addition, the presence of Cl ions is useful for scattering electrodes surrounding the metal oxide layer. Also, NaCl was applied as an electrolyte to increase the anode's dissolution rate, subsequently increasing the rate of

aluminum hydroxide released into the solution [28]. After 50 minutes of remediation, the enlarge in the efficiency of the dye removal operation was marginal. As the electrolyte concentration increases, electrolyte conductivity was consequently increased, and the reaction rate progressed simultaneously. The addition of NaCl to the electrolyte has improved the performance of the electrocoagulation process of different applications [28, 41]. Furthermore, it has a comparable impact on the removal of dyes from wastewater [54, 55].

3.5. Energy Consumption. Energy consumption is clarified in equation (7) as the electrical energy in kilowatt-hours (kWh) is needed to eliminate 1 kg dye pollutant in the contaminated water [44].

$$E.C. = \frac{Pt}{C_o(0.01 R\%)V_l}, \quad (7)$$

where E.C., P , t , C_o , $R\%$, and V_l are energy consumption for dye removal (kWh kg^{-1} dye removal), power (kWatt), reaction time (h), initial dye concentration (mg/L), dye removal percent, and liquid electrolyte volume (L), respectively.

Figure 7 shows the energy consumption with different electrolyte concentrations at an electrolysis time of 60 minutes, and operating conditions were 2 Lh^{-1} flow rate, 5.31 mA cm^{-2} current density, and dye concentration of 50 ppm.

The consumption of energy lowered drastically with higher NaCl concentrations; however, with increased NaCl concentration past 1500 ppm, the curve becomes flat which means to be no significant decrease in energy consumption. At the point when the NaCl concentration was 600 ppm, the energy consumption used to be as excessive as $39.72 \text{ kWh kg}^{-1}$, and when NaCl concentration expanded to 1500 ppm, it decreased to $16.24 \text{ kWh kg}^{-1}$. This behavior could be clarified through the reduction of voltage with the increase of NaCl concentration due to the improvement of the electrolyte conductivity. The voltage was 7 V at NaCl concentration of 600 ppm, and it lowered to 3 V for 1800 ppm. Consequently, the power consumption is reduced to its lower value by adding a certain salt quantity.

The current density influence on the energy consumption is demonstrated in Figure 8. The current densities were changed from 2 to 7.5 mA cm^{-2} , and operating conditions were flow rate of 2 Lh^{-1} , 1500 ppm concentration of NaCl, 50 ppm concentration of dye, and the values were calculated at electrolysis time of 60 minutes. The current density has an important effect on dye energy consumption. When current densities were changed from 2 to 7.5 mA cm^{-2} , the removal efficiencies of dye from 94 to 99% and the energy consumption increased dramatically from 8.5 to 30 kWh kg^{-1} .

These results confirm that at a low current density the overall performance would be economically effective; however, it require a long period of treatment, whilst at an increased current density, it used to be greatly proficient but costly. Therefore, as indicated by the elimination efficiency and current density efficiency, the current

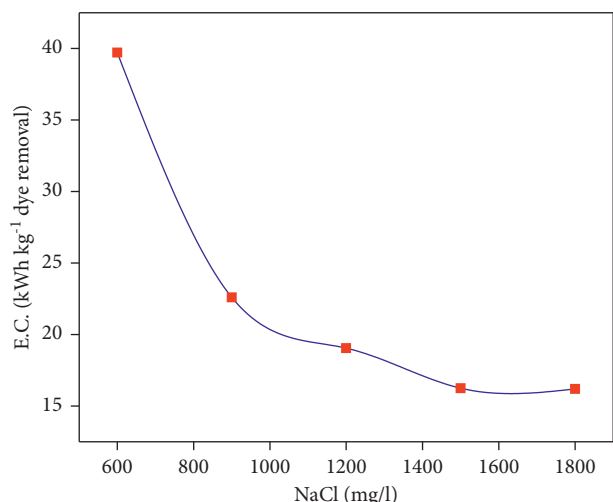


FIGURE 7: Effect of electrolytic concentration on energy consumption (CD 5.31 mA cm⁻² and EC time 60 min).

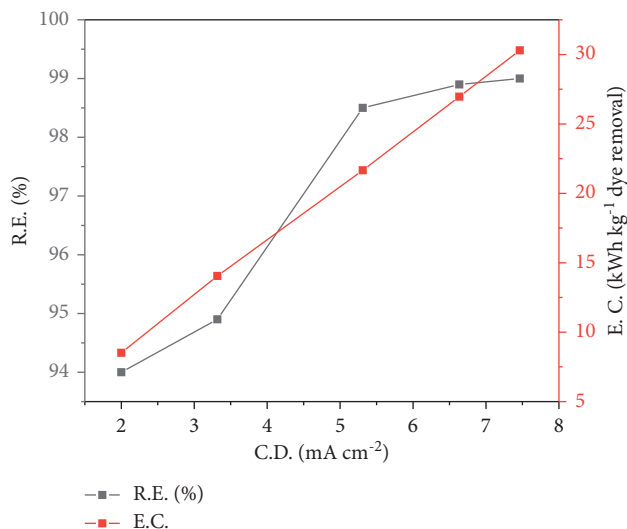


FIGURE 8: Current density effect on energy consumption and dye removal efficiency (NaCl concentration 1500 ppm and EC time 60 min).

density of 2 to 3.32 mA cm⁻² would be a suitable option, where the dye removal effective and energy consumption would be moderate. It was found that the energy consumption of 4.3 kWh m⁻³ was required to achieve 98% removal of methylene blue by the electrocoagulation method with aluminum anode [56]. The results of Assémian et al. [57] showed that at optimum conditions, a current density of 9.66 mA cm⁻² and pH of 8, the specific energy consumption was 7.451 kWh m⁻³ achieved at removal efficiency of 93.2%.

4. Conclusion

Changing the color using applying electrocoagulation technology to treat the effluent of dyes industries seems very promising for independent development studies.

The results show that the electrocoagulation process using aluminum electrodes is a quick and effective way to remove the color of a methylene blue solution during electrolysis. According to removal efficiency, the best color removal from 94% to 98% was achieved shortly after 50 minutes of electrolysis time. The removal efficiency is improved by increasing the current applied by reducing the time required to reach the optimum removal efficiency. However, a dramatic increase in energy consumed has been observed when the current density increased. Despite that the augment of the flow rate shows a favorable effect on removal efficiency, but increasing the flow farther than 2 Lh⁻¹ has a negative influence on the efficiency of removal. Moreover, the increase of salinity level plays an essential key role in increasing the efficiency of removal and decreasing energy consumption.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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