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Diazo Red B Dye Removal by Electrocoagulation Method using Aluminum Electrode

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Abstract – The removal of *diazo red B* dye was studied in water using the electrocoagulation method with an aluminum electrode. The study aimed to reduce the presence of harmful *diazo red B* dyes in the environment and their impact on living organisms. This study was conducted by testing various parameters with specified values. The initial dye concentrations were set at 10, 20, 30, 40, and 50 mg/L, while the electrocoagulation times were varied at 30, 60, 90, 120, 150, and 180 minutes. The initial pH levels were adjusted to 5, 6, 7, and 8. The applied voltages were 2.5, 5, 7.5, 12.5 V, and the distances between electrodes varied at 1, 1.25, 1.5, 1.75, and 2 cm. The study also examined the effects of initial dye concentration on electrocoagulation time, its interaction with pH, and the influence of pH on electrocoagulation time. These parameters aim to determine the optimum conditions for *diazo red B* removal, as measured by a UV-Vis spectrophotometer at a wavelength of 420 nm. The optimum removal efficiency of *diazo red B* was achieved with 94.32% for an initial dye concentration of 30 mg/L, 94.13% for an electrocoagulation time of 180 minutes, 96.00% at a pH of 6, 95.32% for a voltage of 7.5 V, and 95,75% for an electrode distance of 1.5 cm. Additionally, the efficiencies were 99.5% for concentration relative to time was 99.5%, for concentration relative to pH was 97.85%, and for pH relative to time was 96.00%. Additionally, the coagulant analysis of the electrocoagulation results was carried out using FTIR, and the morphological analysis of the surface of the damaged aluminum anode was done using an optical microscope.

Keywords: Electrocoagulation, *diazo red B*, removal efficiency, electrode, aluminum.

Introduction

The growing number of textile industries, ranging from large to small, has led to the textile industry's rapid development in recent decades. Rapid industrial growth can positively impact the economy. However, this rapid development also has a negative consequence, which is the contamination of the aquatic environment (Oginawati et al., 2022). On a medium to small scale, waste management in the textile industry is generally simplistic, with many industries still disposing of waste directly into the river (Hevira et al., 2020).

Textile waste is highly polluted due to its high concentrations, slow degradation, high pH, and temperature (Secula et al., 2011). Textile waste contains many hazardous and toxic contaminants, one of which is a dye (Oginawati et al., 2022; Hevira et al., 2020). The azo functional groups found in synthetic dyes can adversely affect humans and aquatic organisms. This is due to the dyes' resistance to natural degradation, which allows them to block sunlight, impede the photosynthesis process, and create anaerobic conditions in aquatic environments. Additionally, these synthetic dyes can have detrimental effects on human health, including skin irritation, allergies, and dysfunction of the kidneys, liver, brain, and nervous system (Zein et al., 2019).

The negative impacts caused by textile industry effluents must be addressed. Researchers have developed several methods to mitigate the negative effects of synthetic dyes, such as membrane filtration, adsorption, ion exchange, oxidation, photoreduction, and photodegradation (Solís-Rodríguez et al., 2022; Abdulrazzaq et al., 2021). However, these methods are costly and have a high degree of difficulty (Valli Nachiyar et al., 2023). To overcome this, a method that is highly efficient is used, namely the electrocoagulation (EC) method. The electrocoagulation (EC) method is a simple and efficient water treatment method in which particles are deposed in water with the help of electrical energy (Herath et al., 2024).

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Research has been carried out on the electrocoagulation method for removing synthetic dyes, such as *malachite green, indigo carmine, and congo red*, among others (Hassaan et al., 2017; Akter et al., 2022; Zhang et al., 2023; Santiago et al., 2023). However, there are still synthetic dyes that have not been studied by the electrocoagulation method, one of which is *diazo red B*. *Diazo red B* is an azo group anionic dye with the chemical formula 379.5 g/mol. The maximum limit for using diazo salts and their derivatives is 30 mg/kg in textiles and 0.001 mg/kg in food. *Diazo red B* contamination can cause various symptoms such as skin irritation, allergies, and organ damage (Hassaan et al., 2017). Therefore, in this study, *diazo red B* dye removal was carried out using the electrocoagulation method with an aluminum electrode.

Materials and Methods

The synthetic effluent was prepared using *diazo red B* as a diazo dye with the chemical formula $C_{24}H_{21}N_5$ and a molecular weight of 379.5 g/mol. This textile dye was dissolved in distilled water (1000 mg/L). Sodium chloride was used as the electrolyte solution. Sodium hydroxide and sulfuric acid are used to adjust the pH of the solution.

The experimental setup (Fig. 1) consisted of a reactor measuring 10 × 5.5 × 4.5 cm with a capacity of 0.2 L. The anode and cathode were made of aluminum electrodes measuring 5 cm × 1.5 cm × 0.3 mm, positioned vertically and parallel to each other. The electrodes were connected to a DC power source (Gratten APS3003S-3D).

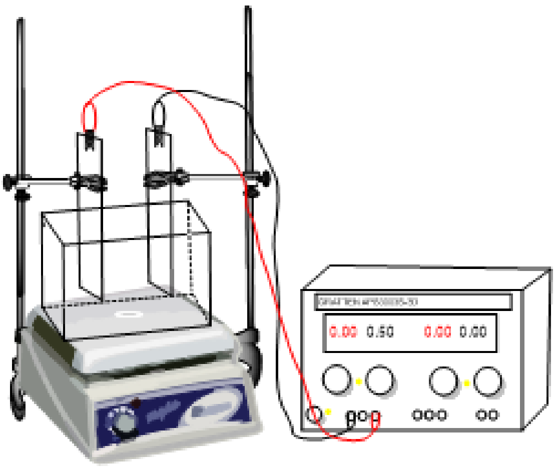

Figure 1. Electrocoagulation circuit

During the experiment, the samples were analyzed for several parameters. The parameters tested were initial dye concentration, electrocoagulation time, voltage, pH, distance between electrodes, initial dye concentration with time, pH with time, and initial dye concentration with pH. After the experiment, the solution was filtered using qualitative filter paper. The resulting filtrate was analyzed by a UV-Vis spectrophotometer (Shimadzu A120657, INA). Absorbance data obtained from UV-Vis spectrophotometer was utilized to calculate the removal efficiency of *Diazo Red B* dye using equation 1. The coagulant was dried and analyzed using FTIR to identify the functional groups contained in the coagulant. The dye removal efficiency was measured using the following equation:

$$Y = \frac{A_o - A_t}{A_o} \times 100\% \qquad (1)$$

Where Y is the dye removal efficiency, A_o is the initial absorbance, and A_{tt} is the absorbance at the time interval.

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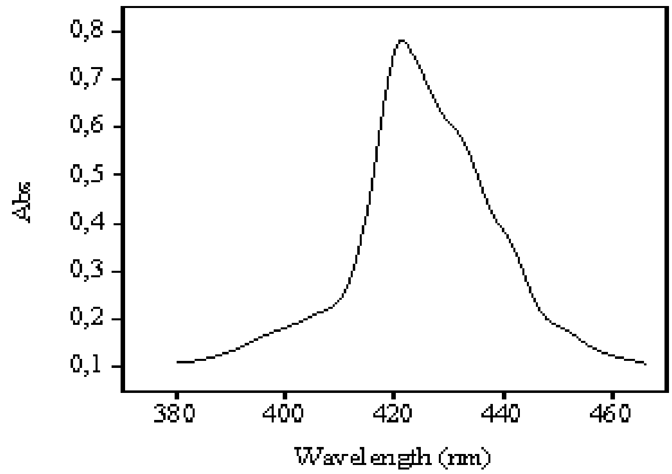

Figure 2. UV-Vis spectrum of *diazo red B*

Results

Fig. 2 shows the spectra at different wavelengths. The maximum wavelength was obtained at 420 nm with an absorbance of 0.808, which will be used to analyze the electrocoagulation results.

Effect of voltage

Based on Fig. 3, the percent removal efficiency of diazo red B increases from 86.90 to 95.49% when the voltage increases from 2.5 V to 7.5 V and is constant at 10 and 12.5 V, with efficiency percentages of 95.32 and 95.30%, respectively.

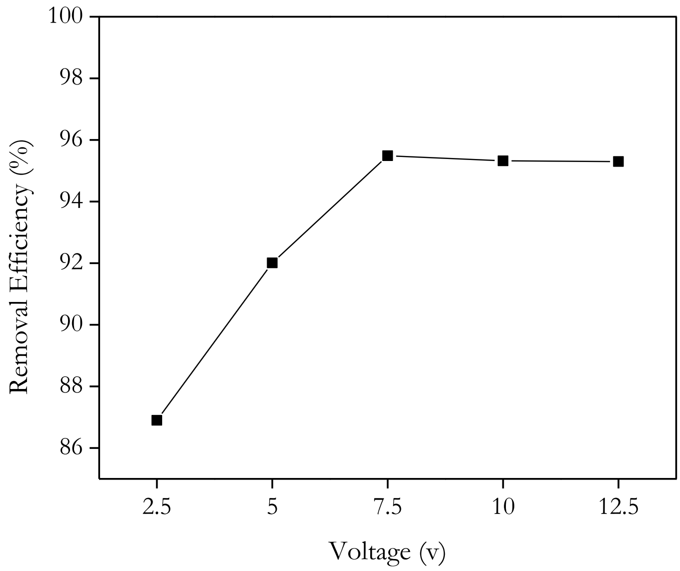

Figure 3. Effect of voltage on the removal efficiency of *diazo red B* (C_o = 30 mg/L, t = 180 min, initial pH = 6, distance between electrode = 1 cm)

Effect of distance between electrodes

The distance between electrodes varied from 1 cm to 2 cm. Fig. 4 illustrates that the removal efficiency of *diazo red B* increased from 1 cm to 1.5 cm, with a removal efficiency value of 93.45%, increasing to 95.75%. Then, efficiency decreased to 90.73% at 2 cm.

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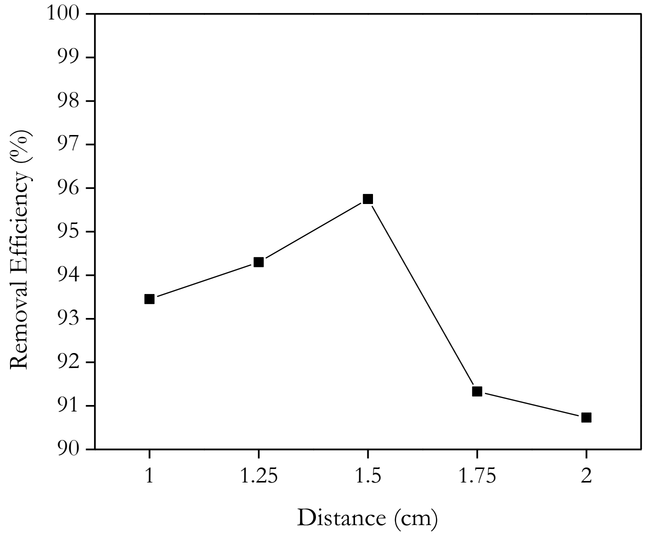

Figure 4. Effect of distance between electrodes on the removal efficiency of *diazo red B* (C_o = 30 mg/L, t = 180 min, initial pH = 6, voltage = 7.5 v)

Effect of concentration with time variation

The effect of concentration on the removal of diazo red B with time variation can be seen in Fig. 5. The optimum condition was obtained at a concentration of 10 mg/L with an electrocoagulation time of 180 min and a diazo red B removal efficiency of 99.57%. The efficiency increased with increasing time and decreased with increasing concentration.

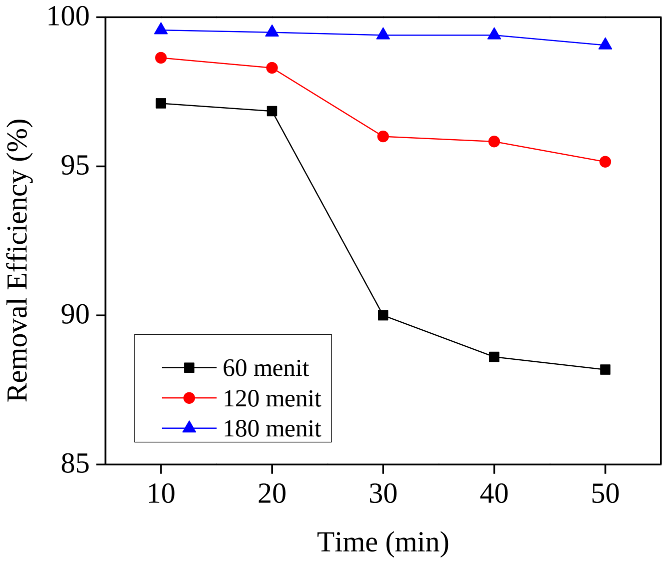

Figure 5. Effect of concentration on the removal efficiency of *diazo red B* with time variation (distance between electrodes = 1.5 cm, voltage = 7.5 v, initial pH= 6)

Effect of concentration with pH variation

Figure 6 shows that the removal efficiency of *diazo red B* decreased with increasing dye concentrations. However, there was an increase in *diazo red B* removal efficiency from pH 5 to pH 6, and the efficiency decreased at pH 7 and 8. The optimum condition was obtained at a concentration of 10 mg/L, and pH 6 with *diazo red B* was 97.85%.

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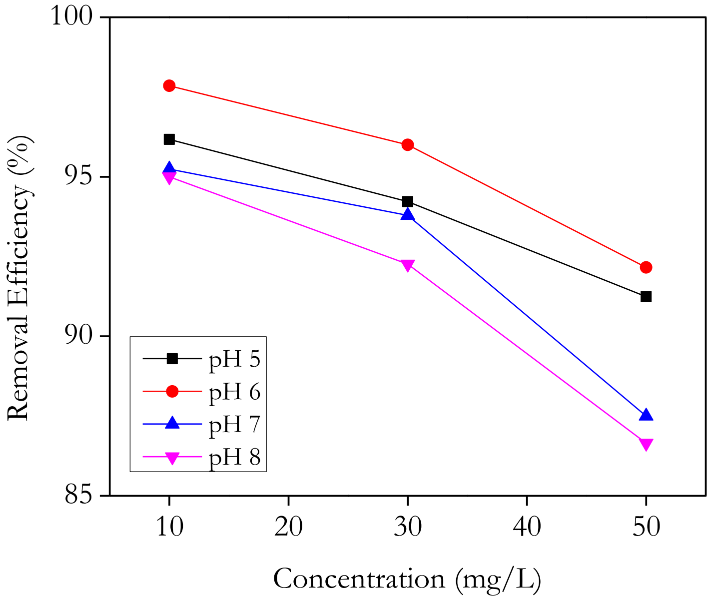

Figure 6. Effect of concentration on the removal efficiency of *diazo red B* with pH variation (distance between electrode = 1.5 cm, voltage = 7.5 v, t = 180 min)

Effect of pH with time variation

The effect of pH on the removal of *diazo red B* with time variation was carried out by varying pH 5, 6, 7, and 8 and time varied 60, 120, and 180 minutes. Figure. 7 demonstrates an increase in *diazo red B* removal efficiency with an extended electrocoagulation time. Then, at pH 6, the efficiency increased, and at pH 7 and 8, it decreased. The optimum condition was obtained at pH 6 and 180 minutes of electrocoagulation time, with a removal efficiency of *diazo red B* of 96.00%.

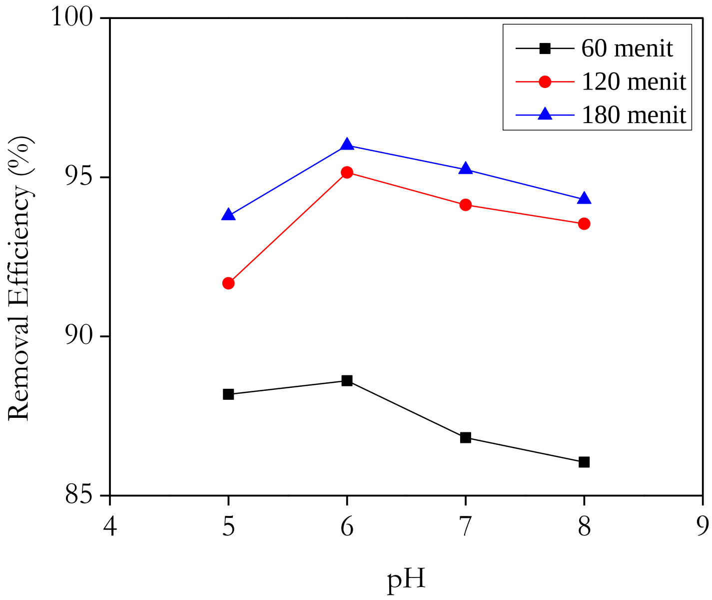

Figure 7. Effect of pH on *diazo red B* removal efficiency with time variation (distance between electrodes = 1.5 cm, voltage = 7.5 v, C_o = 30 mg/L)

FTIR Analysis

The FTIR spectra of *diazo red B* before the electrocoagulation process and the coagulant resulting from the electrocoagulation process can be seen in Figs. 8(a) and 8(b). In Fig. 8 (a), it is shown that there are N=N stretching bond vibrations at a wave number of 1487.90 cm^{-1}. Fig. 8 (b) shows the presence of C=C stretching bond vibrations at wave number 1649.20 cm^{-1}. In the coagulant, there is a stretching -OH group bond vibration at wave number 327.86 cm^{-1}.

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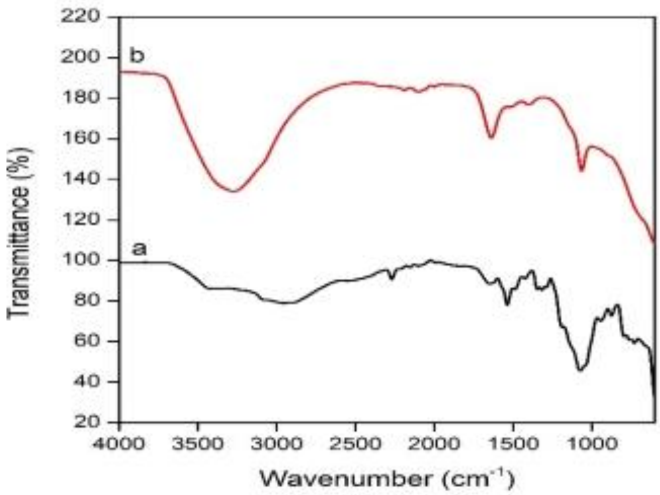

Figure 8. FTIR spectrum of (a) *diazo red B* (b) coagulant after electrocoagulation process

Optical microscope analysis

An optical microscope analysis was conducted to analyze the surface of the aluminum anode after the electrocoagulation process at a time variation of 30 to 180 minutes. Fig. 9 displays the results of the optical microscope analysis. The longer the electrocoagulation time, the more damage to the anode will occur.

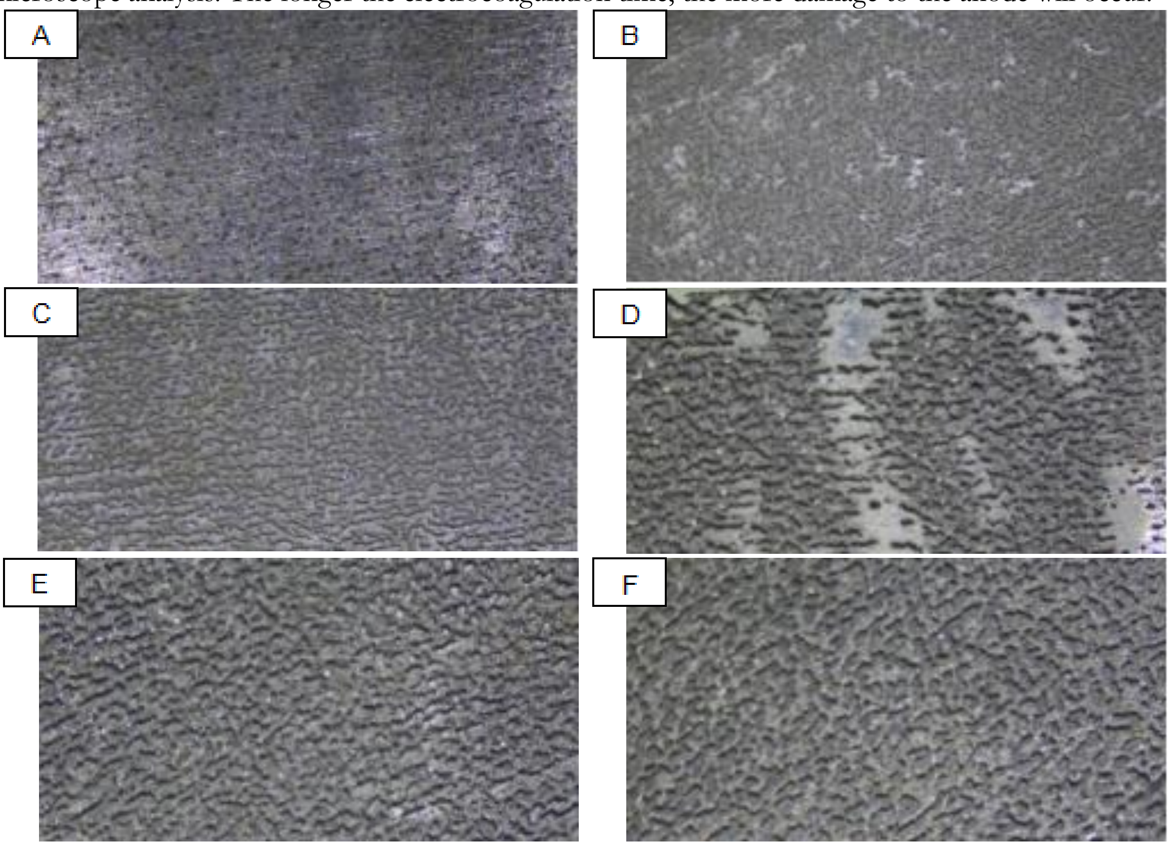

Figure 9. Surface morphology of aluminum anode after electrocoagulation with time variation (a) 30, (b) 60, (c) 90, (d) 120, (e) 150, and (f) 180 min at 10x magnification

Discussion

Effect of voltage

Figure. 3 shows that increasing the voltage can improve the removal efficiency of diazo red B (Zhang et al.,2023). The optimum removal efficiency was obtained at 7.5 V at 95.75%. The high voltage causes reactions to occur at the anode and cathode. At the anode, the greater the voltage, the more aluminum hydroxide is formed because more aluminum ions are released from the anode. These aluminum ions cause a neutralization reaction that reduces the repulsive force between particles, making the attraction force dominant. This force of attraction causes particles to coagulate, forming a coagulant that will absorb the dye.

The high voltage at the cathode produces many hydrogen gas bubbles, resulting in a larger surface area of hydrogen gas. The greater the surface area of hydrogen gas, the more flocs will form (Abdulrazzaq et al., 2021; Moneer et al., 2022). Excessive voltage cannot enhance the removal efficiency of *diazo red B*, as it absorbs the dye at optimal conditions (Khandegar et al., 2013).

Effect of distance between electrodes

Figure 4 shows that the removal efficiency of *diazo red B* at distances of 1 and 1.25 cm is lower than at 1.5 cm. This is because, at too small a distance, the coagulant formed is only around the electrode and not evenly distributed. As a result, the dye absorption process is not optimal (Trisnaawati et al., 2021). Furthermore, the small distance causes a short circuit between electrodes due to the large amount of coagulant formed around them (Setyawati et al., 2021).

The highest removal efficiency was obtained at 1.5 cm because, at this distance, there is a rapid transfer of electrons from the anode to the cathode, resulting in a reduction reaction at the cathode. The reduction reaction will produce hydroxyl ions, which will bind to Al^{3+} resulting from oxidation at the anode and produce an aluminum hydroxide coagulant. This coagulant will absorb the dye (Ramadhan et al., 2021). At 1.75 and 2 cm, there was a decrease in the efficiency of *diazo red B* removal. This decrease is caused by the increase in electron travel time from the anode to the cathode, causing weak intermolecular interactions and a decrease in dye coagulation power (Trisnaawati et al., 2021; Tahreen et al., 2020).

Effect of initial dye concentration with time variation

The removal efficiency of *diazo red B* decreases with increasing concentration. These drops happen because the coagulants and flocs that are made can't soak up all the dyes; there are too many dyes for them to handle (Green et al., 2020). Conversely, the smaller the concentration of *diazo red B*, the more coagulant and floc formed can absorb all the dyes, resulting in a higher removal efficiency (Akter et al., 2022; Zhang et al., 2023).

As electrocoagulation time increases, *diazo red B* removal efficiency increases (Fig. 5). This increase occurs because the number of Al^{3+} and OH^- ions generated with time increases. These ions will neutralize the particle charge and form an $Al(OH)_3$ coagulant. This means that the longer the electrocoagulation process, the more $Al(OH)_3$ is formed, so that dye absorption increases (Negash et al., 2023). In addition, the length of time electrocoagulation takes produces a lot of H_2 gas and causes the number of flocs to increase (Moneer et al., 2022). However, when exceeding the optimum time limit, the dye removal efficiency does not increase because the amount of coagulant and floc formed is small (Negash et al., 2023).

Effect of pH

As shown in Figure. 6 and 7, the *diazo red B* removal efficiency is optimal at pH 6. The efficiency at pH 5 is lower than that at pH 6 due to the less effective electrocoagulation process. At pH 5, the coagulant formed is $Al(OH)_4^+$ which is easily soluble in water (Aoudj et al., 2010). As a result, the dye absorption process is not optimal. The electrocoagulation process becomes effective when the initial pH is slightly acidic to neutral (generally >5.5). At that pH, a lot of $Al(OH)_3$ is formed with a large surface area, so the dye is easily adsorbed. The ionization state of dye molecules is highly dependent on pH, with the degree of ionization and the resulting charge significantly influencing their adsorption onto coagulant flocs. For diazo dyes, optimal adsorption occurs at a pH of 6, where the equilibrium between the positive charge of the coagulant and the negative charge of the dye facilitates strong electrostatic interactions, thereby enhancing the efficiency of dye removal (Zainudin, 2023). At pH levels below 6, increased acidity leads to the protonation of dye molecules, which may inhibit their interaction with the coagulant and consequently reduce removal efficiency (Dehghani, 2014). Therefore, at pH 6, the *diazo red B* removal efficiency is high. A decrease in efficiency occurs at pH 7 and 8 due to the formation of $Al(OH)_2^-$ coagulants that are easily dissolved in water, so the ability to adsorb dyes is minimal (Negash et al., 2023; Moneer et al., 2022; Titchou et al., 2020).

Analysis of FTIR

In Fig. 8 (a) and (b), there is a shift in adsorption bands from 1487.90 cm^{-1} and 1649.20 cm^{-1} to 1400.56 cm^{-1} and 1637.54 cm^{-1}, namely in N=N stretching and C=C stretching bond vibrations. In the coagulant resulting from electrocoagulation, the -OH stretching bond vibrations appear at wave number 327.86 cm^{-1}, which are not present in the *diazo red B* dye. The appearance of this adsorption band on the -OH group

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supports the adsorption mechanism in removing *diazo red B* by using Al electrodes and forming $Al(OH)_3$ (Bartošová et al., 2017).

Analysis of microscope optic

According to Fig. 9, the longer the electrocoagulation time, the greater the damage to the anode. This is characterized by an increasing number of holes formed on the anode's surface. Damage to the anode is caused by oxidation reactions on the anode, which cause the dissolution of aluminum on the anode and produce Al^{3+} ion. The dissolution of aluminum will be greater when the electrocoagulation process is carried out for a long time (Zhang et al., 2023).

Formation of aluminum hydroxide and its role in electrocoagulation

The removal of *diazo red B* by the electrocoagulation method begins with an oxide reaction at the anode that produces Al^{3+} ions (Nicolau et al., 2021).

$$Al_{(s)} \longrightarrow Al^{3+}_{(aq)} + 3e^- \quad (2)$$

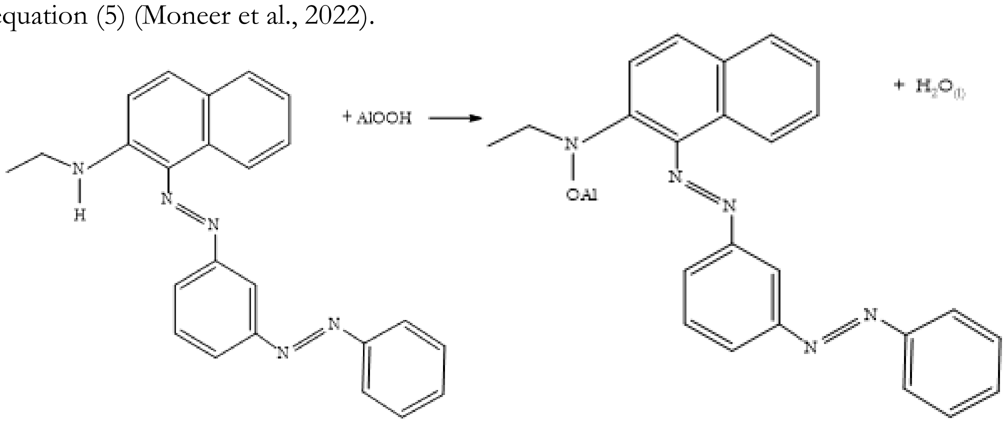
At the cathode, a reduction reaction occurs and produces hydroxyl ions and hydrogen gas. The hydrogen gas bubbles generated during the electrocoagulation process will carry pollutants to the water surface through the electroflotation process. Equation (3) shows the reduction reaction at the cathode (Gasmi et al., 2022; Zafar et al., 2024).

$$2H_2O_{(l)} + 2e^- \longrightarrow H_2 + 2OH^-_{(aq)} \quad (3)$$

The metal cations produced through the oxidation reaction will react with hydroxyl ions to form metal hydroxide, namely $Al(OH)_3$. This can be seen in equation (4) (Nicolau et al., 2021).

$$Al^{3+} + 3OH^- \rightleftharpoons Al(OH)_{3(s)} \quad (4)$$

The removal of *diazo red B* involves the adsorption of dye molecules by the coagulant, and flocs form, as seen in equation (5) (Moneer et al., 2022).



(5)

Conclusion

Under optimum conditions, the electrocoagulation method can effectively remove 99% of the *diazo red B* dye. To achieve optimum removal efficiency, an initial dye concentration of 10 mg/L was used, but the concentration used for the next parameter was 30 mg/L because 30 mg/L is the maximum limit for the use of diazo dyes in textiles. Then, the optimum electrocoagulation time was 180 minutes, pH 6, a voltage of 7.5 V, and a distance between electrodes of 1.5 cm. Adsorption of dyes by coagulants and flocs is seen from UV-Vis spectrophotometer analysis on sample filtrate, FTIR analysis on coagulants, and optical microscope analysis on aluminium electrodes after the electrocoagulation process. This study anticipated that the textile industry and other industries would be able to treat industrial waste so as not to pollute the aquatic environment. This expectation is supported by the high pollutant removal efficiency, versatility, environmental compatibility, and the potential for reusing treated water. Furthermore, it is anticipated that future studies will focus on the direct application of electrocoagulation technology to textile industry wastewater. This approach aims to identify specific challenges and evaluate potential solutions for the large-scale implementation of this method.

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