FACTORS AFFECTING REMOVAL EFFICIENCY OF MERCUROCHROME FROM AQUEOUS SOLUTION VIA ELECTROCOAGULATION USING ZINC ELECTRODES

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Abstract: *The aim of using electrocoagulation process to remove Mercurochrome dye from wastewater was studied. Zinc electrodes were used in an electrolysis cell with a capacity of 300 ml and the effect of the following factors was studied: initial dye concentration (10-25 ppm), voltage (10-30 V), distance (1 cm), coagulation time (1-100 min), surface area (24.5-28 cm^2), effect of adding NaCl in Na_2SO_4 at concentrations of (10-30 ppm) and temperature (303-333 K). The results of the experiments showed that the removal efficiency increases with increasing temperature, initial concentration, applied voltage, electrode surface area, coagulation time, and addition of NaCI. The efficiency decreases with increasing electrode spacing and addition of Na_2SO_4. The maximum removal (96%) was achieved at 25 ppm, 333 K, and 30 V and with an area of 28 cm^2 and a spacing of 1cm with a time of 100 min. The thermodynamic functions (ΔS°, ΔH°, ΔG°) were calculated. The values of these functions indicate that the process is spontaneous, endothermic, and random. The kinetics of the reaction at the best removal ratio were studied and found to follow first-order kinetics. The thermodynamic functions of the activated complex were studied by the negative activation entropy value $\Delta S^{\#}$, indicating that the activated complex is more ordered than the reactants.*

Keywords: *electrocoagulation, zinc electrode, mercurochrome dye, wastewater treatment*

Introduction

Pollution is one of the major problems facing the world today, so finding ways to get rid of pollution is essential [1]. Dyes are one of the leading causes of wastewater pollution, as industrial progress has caused environmental damage due to the draining of toxic compounds from factories into river water [2]. Electrocoagulation is an electrochemical technique where a zinc electrode (anode) is corroded to release active coagulants into the solution. Dyes can have serious and severe effects on living systems [3]. About 110,000 tons, or more than 100,000 types of commercially available dyes, are lost annually during the dyeing process as liquid waste [4]. Most of these dyes are carcinogenic, mutagenic, and toxic to aquatic organisms [5, 6].

Mercurochrome is a compound of organic disodium salts and fluorescein that is available in many countries. However, the drug was withdrawn from the market due to the possibility of mercury poisoning. It is the trade name for merbromin, commonly used as a topical antiseptic agent for first aid [7].

In [8], the color removal rate from wastewater using the electrocoagulation process was studied. This was done by studying some parameters such as current at 0.03, 0.06, and 0.09 Amp., pH at 3, 6, and 9, and reaction duration at 20, 40, and 60 min. When the electrode was incorporated in the form of Al-Al (anode-cathode)(cathode-anode) and Fe-Fe (anode-cathode)(cathode-anode), the removal rate was 95.50% and 97.24%, respectively. About 98.03 and 91.95% of removal changes when the connection changes. Fe-Al (anode-cathode) and Al-Fe (anode-cathode) were incorporated at 9 and 60 min of sintering duration, on the edge.

D.R. Hadi et al. in [9] studied the comparison of the ability of the electrocoagulation process to remove reactive blue dye (RBD) from industrial wastewater in bipolar contact using planar perforated aluminum electrodes. The reaction kinetics and thermodynamic functions were studied. Two methods were used in this study, constant voltage mode (CVM) (200V) and constant current mode (CCM) at 3 A during sintering time 70min and pH=8. By examining the results, it was found that the removal rate of RBD was better using the CCM technique, where the removal rate was 98.82% using the CCM and 97.74% using the CVM under the same conditions at a concentration of 100 ppm and pH=8.

R. Muttaqin et al. [10] used an electrocoagulation process to remove pharmaceutical contaminants such as ciprofloxacin dye and methylene blue dyes using aluminum electrodes and stainless steel 316. At a voltage of 50 V and a pH of 5, 7, and 10 for 1, 2, 3, and 4 hours. The results showed that the optimal removal rate was obtained at a pH of 10 with a time of 4 hours at a rate of 84.60% and 68.19% for methylene blue and ciprofloxacin, respectively.

The efficiency of electrocoagulation (EC) in removing two reactive dyes was investigated by M. M. Ramadan et al. [11]. The compositional difference between reactive red 49 (RR49) and reactive yellow 15 (RY15) was investigated by the EC process. The optimum conditions were 80.60 mg/L of dye concentration and 5.9 and 4 pH for RR49 and RY15, respectively. The best removal rate of 98.5% was obtained for both dyes at 20 and 30°C for RR49 and RY15, respectively.

In research [12], the EC process was used to remove total dissolved solids using different electrodes: Al, Fe, and Zn. It was observed that Al is slightly better than iron and zinc electrodes, and that perforated aluminum electrodes are better than non-perforated aluminum electrodes. Within a distance of 2cm, voltage 25 V and current density 1.5 A/cm^2 during sintering time 150 min.

The aim of this study is to investigate the efficiency of the electrocoagulation process in removing the mercurochrome dye from wastewater. Furthermore, study the effect of some variables, such as dye concentration, voltage effect, distance change effect, electrolyte addition effect, and temperature, on the efficiency of the flocculation process and determine the optimum conditions for it. Finally, to investigate the thermodynamic functions and kinetics of the removal process.

Experimental part

Methods, apparatus and materials. The apparatuses and materials utilized in this study are shown in Table 1.

Table 1. Apparatuses used

Device used	Manufacturer	Origin
Spectrophotometer	HIGHTOP	Chine
D.C Power supply	Ps-303-2	Germany
Hotplate stirrer	DAIHAN Lab. tch	Taiwan
Sensitive Balance	Sartorius-210S	Germany
PH-meter	Starter2000 Ohaus	Taiwan

The zinc electrode consists of a high percentage of zinc and a small percentage of other elements, such as Al (4.5-5.0), cadmium (0.005), copper (0.2-0.3), iron (1), and lead (0.007). Furthermore, Mercurochrome is a neutral red dye made by combining dibromofluorescein with mercury acetate and sodium hydroxide, and its characteristics are shown in Table 2. Mercurochrome contains a high percentage of mercury, which constitutes about 27% of its weight. The high percentage of mercury gives it disinfectant properties. At the same time, it makes it a toxic substance if used incorrectly.

Table 2. The characteristics of the used Mercurochrome

Properties		Properties	
Common Name	Mercurochrome	Solubility in water	20 mg/ml
Scientific Name	Merbromin	Solubility in ethanol	10 mg/ml
CAS-Number	129-16-8	Molecular Weight	750.16 gm/mol
λmax	505 nm	Boiling point	264 °C
pH	6.9	Molecular formula	$C_{20}H_8Br_2HgNa_2O_6$

Color	Red	Density	0.89-0.95 gm/cm^3

Samples preparation. A standard dye solution with a concentration of 1000 ppm was prepared by dissolving 1 g of the dye in 1000 ml of distilled water. Different concentrations (10, 15, 20, and 25 ppm) of the dye were prepared from it using the dilution law. In the same way, the electrolytic solution Na_2SO_4 was prepared by dissolving 1 g of salt in 1000 ml of distilled water. Another electrolytic solution, NaCl, was prepared in the same way by dissolving 1 g of the salt in 1000 ml of distilled water. Using the dilution law ($M_1.V_1=M_2.V_2$), different concentrations of the salt were prepared, respectively (10, 15, 20, 30 ppm).

For electrolytic cell preparation, an electrolytic cell made of glass with dimensions of 10 cm in length, 8 cm in width, and 6 cm in height and a capacity of 300 cm^3 and using zinc electrodes with dimensions of 8 cm in length, 7 cm in width, and 0.1 cm in thickness and a distance between the electrodes of 1, 2, 3, or 4 cm.

Results and discussion

Effect of changing initial concentration of mercurochrome dye. The mercurochrome dye calibration curve was prepared by measuring the maximum absorbance of different dye concentrations at 505 nm. This curve is used to calculate the concentration of the dye over time (Fig. 1).

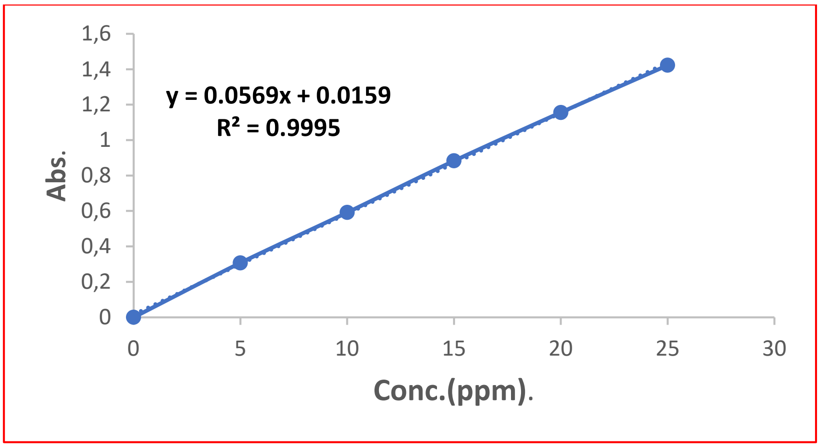

Fig. 1. The calibration curve for the mercurochrome dye at a wavelength of 505 nm

Four initial concentrations of the dye were used in the study (10, 15, 20, and 25 ppm), and the absorbance of the remaining concentration was measured at different times and for a period of 100 minutes with voltage 30 V, the distance between the electrodes 1 cm, and temperature 313 K using zinc electrodes, as shown in Figure 2. The percentage of removal was calculated using the equation (1).

$$\%R = (Ao - At) / Ao * 100\% \qquad (1)$$

where R denotes the removal percentage rate. Ao, and At denote the absorbance at the initial and absorbance at any time, respectively.

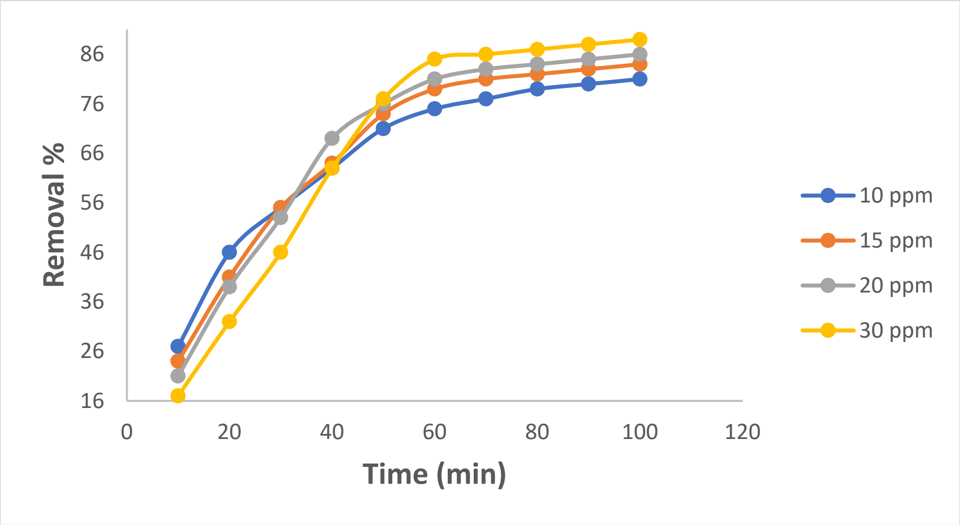

Fig. 2. A graph of the effect of changing the initial concentration on the dye removal rate

We note from Figure 2 that with increasing concentration, the removal rate increases. This is attributed to the fact that increasing the initial concentration of the dye leads to an increase in the reactions that occur, including dye oxidation, as the amount of dye that has been removed has increased with the increase in the initial concentration of the dye [13, 14].

Effect of Changing Voltage. Five different voltages were used (10, 15, 20, 25, and 30 V) over a time of 100 min at a distance of 1 cm and a temperature of 313 K, using zinc electrodes as shown in Fig. 3.

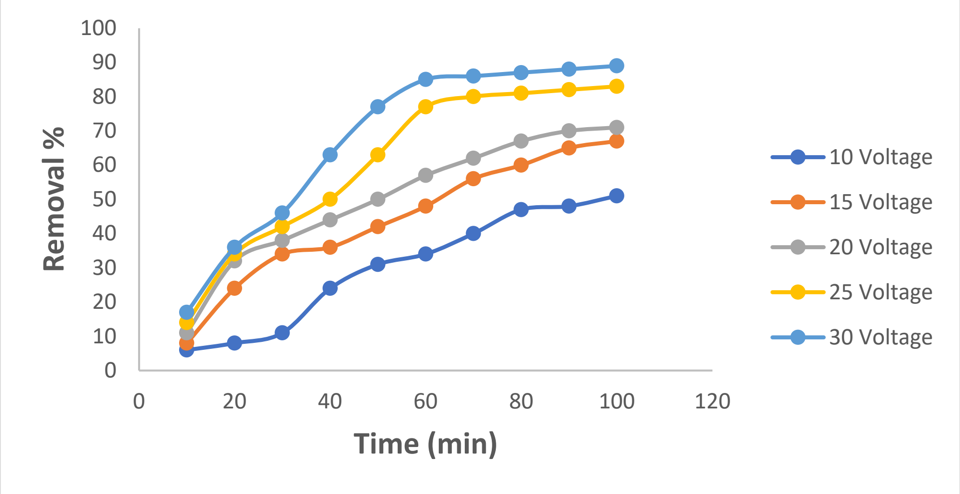

Fig. 3. Graph of the effect of voltage change on dye removal rate

From Fig. 3, it becomes clear that the removal rate increases with increasing voltage. This is attributed to the increase in ions released from the electrodes, which is directly proportional to the amount of current to form the metal hydroxide, which is primarily responsible for the removal process through the oscillation of the remaining pollutant with increasing current [15]. From the results obtained, it appears that increasing the voltage leads to obtaining a high removal efficiency. When comparing the values we obtained for the removal ratios at different voltages and under the same conditions, it is noted that the highest removal ratio is obtained at 30V, and the opposite was the case at low voltage [16]. This can be attributed to the direct relationship between voltage and electric current. The higher the voltage, the higher the electric current passing through the electrocoagulation cell, which supports increasing the number of ions released from the black electrode (zinc) according to Faraday's law (the weight of the precipitated or released material at the electrodes is directly proportional to the amount of electricity passing through). Consequently, the percentage of $Zn(OH)_2$ formed in the solution will increase, which works to adsorb the dye and form a clot on the surface of $Zn(OH)_2$, which increases the removal efficiency [17].

Effect Of Electrode Surface Area On Removal Efficiency. This study was conducted using zinc electrodes in the electrocoagulation cell, and the surface area of the electrode (A) was one of the factors studied. To determine the effect of this factor on the removal efficiency, the electrocoagulation process was carried out to remove the mercury-chromium dye using electrodes within two different areas. The values of the selected areas were 24.5 and 28 cm^2. The experiments were conducted at a temperature of 323 K and an applied voltage of 30 V with a distance between the electrodes of 1cm and a concentration of 25 ppm during time 100 min.

Fig. 4 shows the effect of changing the surface area of the electrode exposed to the sintering process with two areas, 24.5 and 28 cm^2 at a distance of 1 cm, a constant voltage 30 V, a concentration of 25 ppm and a temperature of 323 K during a time of 100 min.

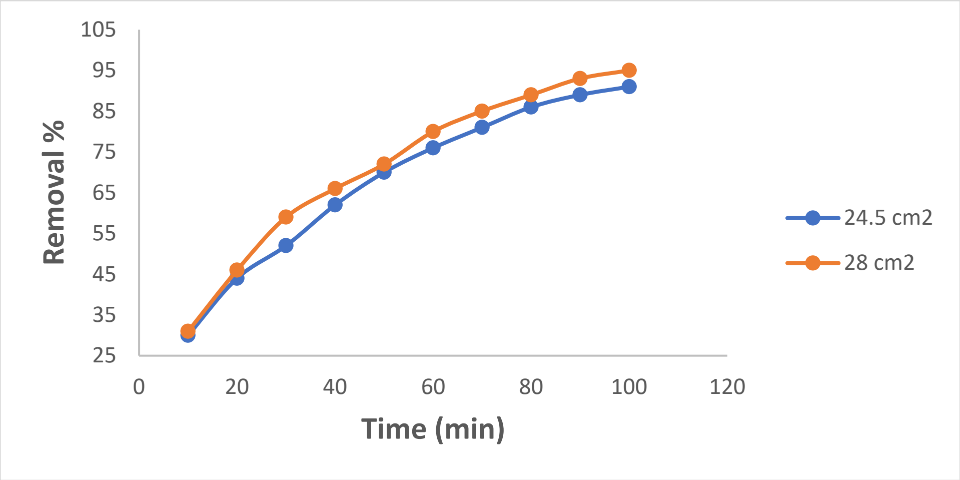

Fig. 4. Graph of the effect of changing the surface area of the electrode on the dye removal rate

From Fig. 4, it is noted that the removal ratio values are higher at the surface area of the electrode A = 28 cm^2 than at A = 24 cm^2.We conclude from this that the removal efficiency increases with the increase in the surface area of the electrode. This is attributed to the increase in the ionic flux of Zn^{+2} ions from the metal surface, which increases the number of aluminum hydroxide molecules needed to adsorb the dye from the solutions and thus increases the removal efficiency [18].

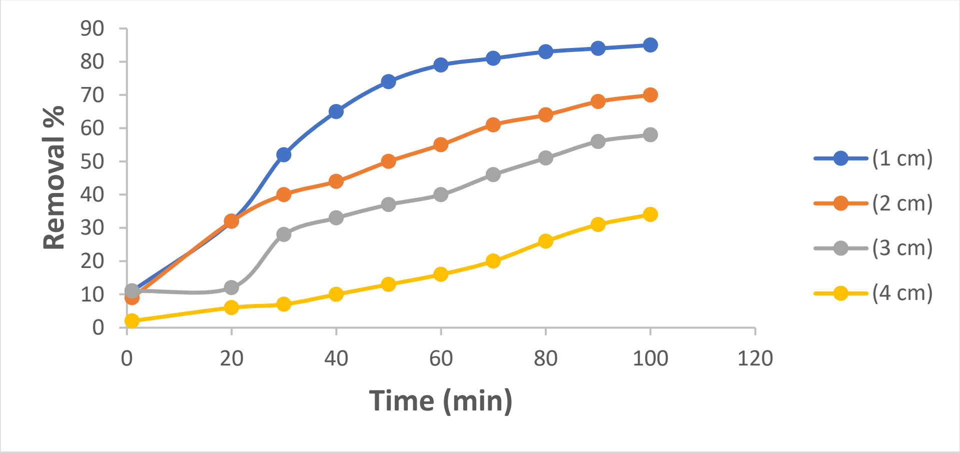

Fig. 5. A graph of the effect of changing the distance between the electrodes on the dye removal rate

Effect Of Distance Between Electrodes. The removal process was studied using four different distances (1, 2, 3, and 4 cm) and at a constant concentration of 25 ppm, a constant voltage of 30 V, and a constant temperature of 323 K during a time of 100 min, as shown in Fig. 5.

From Fig. 5, it was noticed that the removal efficiency decreases with increasing the distance between the electrodes. This is due to the decrease in the resistance of the solution with decreasing distance between the electrodes due to electrical dispersion. The smaller the distance between the poles, the lower the electrical energy consumption [19]. Reducing electrode distance decreases energy consumption during electrocoagulation Larger distances increase resistance due to the formation of metal hydroxides, which reduces efficiency [20]. Therefore, the smaller the distance between the electrodes, the less energy is consumed, and the removal efficiency increases, which leads to increased conductivity, which increases the coagulation process and the formation of clots. The larger the distance between the electrodes, the slower the ions move, which increases energy consumption and reduces removal efficiency [21].

Effect Of Electrolyte. Two types of electrolytes, namely Na_2SO_4 solution and NaCl electrolyte, were used. Na_2SO_4 solution was used with different concentrations (10, 15, 20, and 30 ppm) at a constant dye concentration of 25 ppm, with a voltage of 30 V and a distance of 1cm between the electrode and a temperature of 303 K during a time of 100 min using zinc electrodes. The results are shown in Fig. 6.

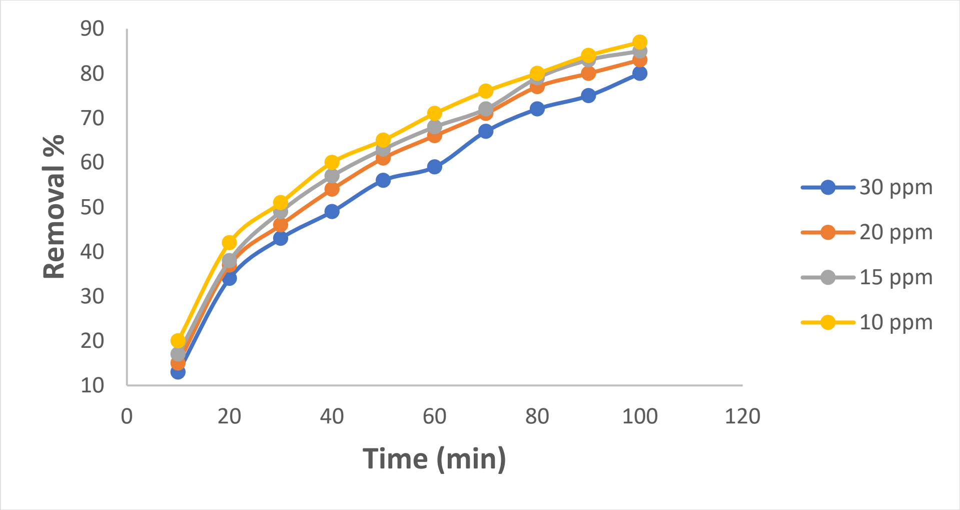

Fig. 6. A graph of the effect of changing the concentration of the electrolyte Na_2SO_4 on the removal rate of the dye

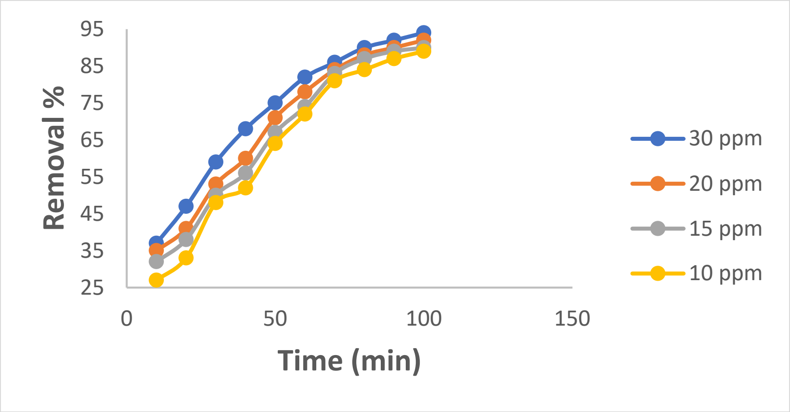

Fig. 7. A graph of the effect of changing the concentration of the electrolyte NaCl on the removal rate of the dye

NaCl electrolyte was used with different concentrations (10, 15, 20, and 30 ppm) at a constant dye concentration of 25 ppm, a voltage of 30 V, a distance of 1 cm, and a temperature of 303 K for a time of 100 min using aluminum electrodes, as shown in Fig. 7.

Fig. 7 shows that with increasing the concentration of the electrolyte (NaCl), 6 the concentration of the remaining dye increases (the removal rate decreases) as a result of increasing the electrical conductivity to the highest value. However, by increasing the concentration of the added electrolyte, the removal efficiency will decrease. This is due to the fact that it occurs between OH^- and SO_4^{-2} for Zn^{+2} ions, so the amount of metal hydroxide formed, $Zn(OH)_2$, decreases, and thus the removal rate will decrease [22].

$$H_2O \rightarrow H^+ + OH^- \quad (2)$$

$$Na_2SO_4 \rightarrow 2Na^+ + SO_4{}^{-2} \quad (3)$$

When adding salt Na_2SO_4, it will dissociate, and water will ionize in the cell because there are two electrodes of the same metal. There will be competition between OH^- ions and $SO_4{}^{-2}$ ions for Zn ions, so some of them will form a complex $ZnSO_4$, which reduces the amount of metal ions released and reduces the formation of the complex $Zn(OH)_2$, which is the basis for the electrocoagulation process. The presence of the electrolyte causes competition between Zn^{+2} ions and the dye, part of which is shared with the electrolytic solution and remains dissolved in the solution, thus reducing the amount of dye that is removed using Zinc electrodes [23].

From fig. 7, the experimental result shows that by increasing the concentration of the electrolyte NaCl, the removal efficiency increases [24]. This is attributed to the increase in the concentration of ions in the solution, thus reducing the resistance between the electrodes. Increasing the electrolyte concentration leads to a decrease in the cell voltage at a constant current density and a decrease in energy consumption in electrolytic cells [25]. Due to the presence of chloride ions that form with the aluminum ion $ZnCl_2$, which forms materials that increase the sintering process, the removal efficiency of the mercury dye will increase.

$$NaCl \rightarrow Na^+ + Cl^- \quad (4)$$

Table 3. Comparison of the effect of NaCl and Na_2SO_4 electrolytes on the dye removal rate

Electrolyte type	**Electrolyte Conc. 10 ppm**	**Electrolyte Conc. 15 ppm**	**Electrolyte Conc. 20 ppm**	**Electrolyte Conc. 30 ppm**
NaCl	89%	90%	92%	94%
Na_2SO_4	87%	85%	83%	80%

These two salts were used as electrolytes due to their ease of obtaining and low toxicity. It is observed that when the concentration of the electrolyte Na_2SO_4 increases, the removal rate of Mercurochrome dye decreases. This is attributed to the presence of SO_4^{-2} ions in the solution, which combine with the aluminum ion to form aluminum sulfate, a protective layer on the surface of the zinc electrode, thus reducing corrosion and releasing the metal ion. When using NaCl as an electrolyte, we observe an increase in the removal rate with increasing electrolyte concentration. This is attributed to the presence of chloride ions (Cl^-), which work to remove the passivation layer on the surface of the electrodes, which leads to an increase in the release of zinc ions and thus an increase in the generation of zinc hydroxide [26].

Effect Of Temperature. The effect of temperature on the removing Mercurochrome dye was studied using a zinc electrode at a fixed concentration of 25 ppm of the dye, a distance between electrodes of 1cm, and a voltage of 30 V, during a time of 100 min and at variable temperatures (303, 313, 323, and 333 K), as Figure 8, where the removal percentage for each temperature was (82, 85, 88, and 91 %), respectively.

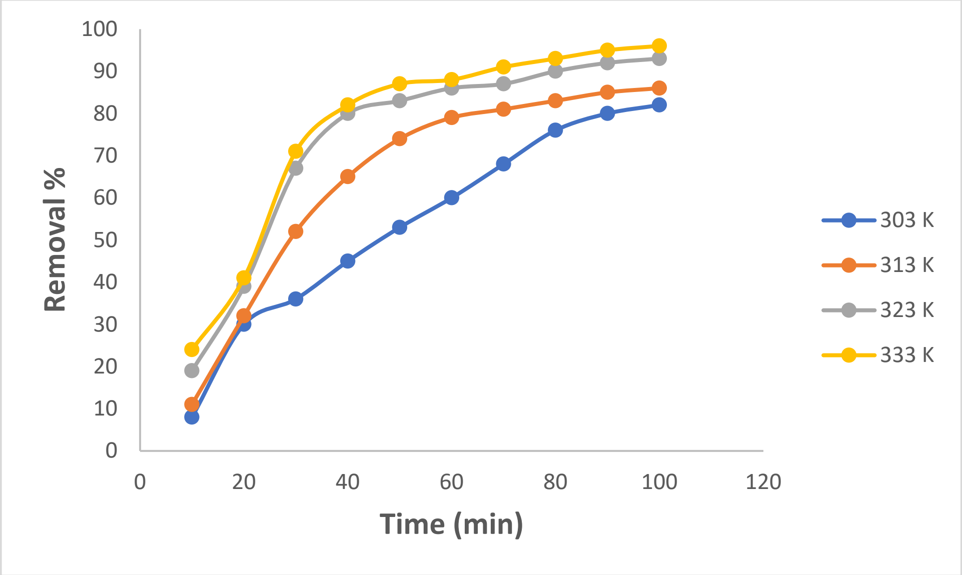

Fig. 8. Graph of the effect of temperature change on dye removal rate

It is noted from Fig. 8 that with increasing temperature, the removal rate increases. This is attributed to the fact that increasing temperature increases the speed of movement of the released ions and increases collisions, and thus $Al(OH)_3$ is in a higher proportion, which increases the electrocoagulation process by following the electrodes and observing the electrode surface, it is noted that there is a deposition of dye on the electrode surface because the process is possible to be coagulation with adsorption and that the dye molecules adsorbed on the electrode surface with increasing temperature the adsorbed molecules separate which helps to separate them from the electrode surface and thus the electrode is released and its conductivity increases and thus it is oxidized more and releases more ions. As a result, the amount of $Al(OH)_3$ becomes larger, which is the basis of the electrocoagulation process, so the removal rate will increase [17, 27].

Calculation of thermodynamic functions. Thermodynamic functions provide a distinctive explanation when studying the electrocoagulation process of Mercurochrome dye and are considered essential variables that clarify the nature of the system and the type of forces controlling it [28]. In addition, they give an idea about the type of molecular interactions that can occur during the sintering process, which has a significant role in determining its efficiency, depending on the change that occurs to remove the dye at a time of 100 min as a value for the equilibrium constant for the dye concentration 25 ppm by studying the effect of temperature on the values of the equilibrium constant. ΔG° the Gibbs free energy change (J/mol) can be calculated using the following equations [29].

$$\Delta G^{\circ} = -RT \ln K_{eq} \tag{5}$$

Where R is the gas constant (8.314 J/(mol·K)), T is the absolute temperature (K), K_{eq} is the equilibrium constant of the adsorption process, and can be calculated from knowing the initial concentration C_o (mg/L) and the concentration at equilibrium C_e (mg/L) using the equation below:

$$K_{eq} = \frac{C_O - Ce}{Ce} \tag{6}$$

ΔH° the enthalpy change (J/mol), ΔS° the entropy change (J/mol K) can be determined from the rearranged equation below:

$$\ln K_{eq} = \frac{\Delta S^{\circ}}{R} - \frac{\Delta H^{\circ}}{R} \tag{7}$$

ΔS° and ΔH° values can be calculated from the intercept and slope of the straight line of the plot between ln K_{eq} versus 1/T, respectively as in the Fig. 9. The thermodynamic functions are found in Table 4.

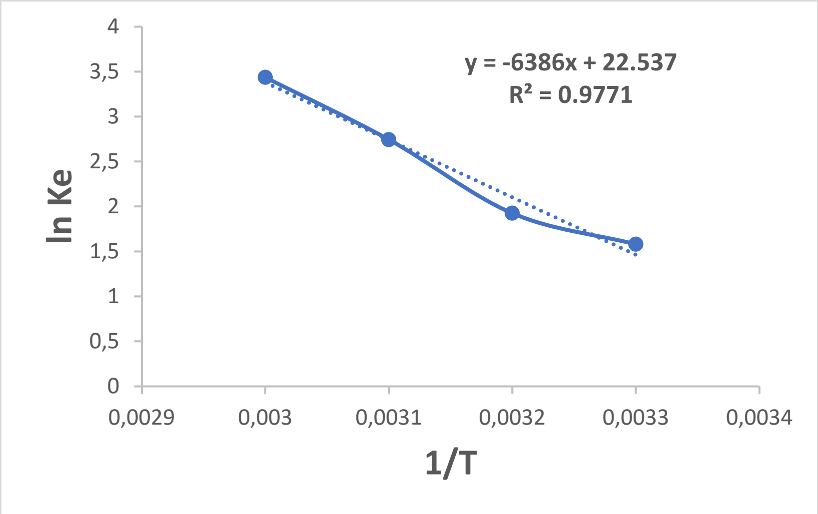

Fig. 9. Graph of lnK_{eq} and reciprocal of temperature (in Kelvin) 1/T

Table 4. The values of the equilibrium constant and thermodynamic functions at different temperatures.

T (K)	$\Delta G^{\circ}(KJ.mol^{-1})$	$\Delta H^{\circ}(KJ.mol^{-1})$	$\Delta S^{\circ}(KJ.K^{-1}.mol^{-1})$
303	-3.980	53.093	0.187
313	-5.012		
323	-7.369		
333	-9.513		

Kinetic Studies. The kinetics of dye removal by the electrocoagulation process were studied using the pseudo first-order equation [30].

$$ln\frac{a}{(a-x)} = k \cdot t \tag{8}$$

This is done by drawing a graph of ln(a-x) versus time in minutes. At a concentration of 25 ppm of dye, at a voltage of 30V, at a distance of 1 cm, with different temperatures. The value of the rate constant is calculated from the value of the slope, where slope = -k. We notice that with the increase in temperature, the rate constant *k* increases (Table 5).

Table 5. The effect of temperature on the rate constant of the reaction.

Temperature (K)	**k (min^{-1})**
303	**0.0187**
313	**0.0214**
323	**0.0282**
333	**0.0343**

Calculation of Thermodynamic Variables For The Activated Complex. A chemical reaction usually proceeds in a path that includes several steps, each of which requires activation energy. The reaction rate is also greatly affected by changes in temperature, as the reaction rate can increase approximately to double its original value when the temperature increases by ten degrees. This is expressed by the Arrhenius equation:

$$k_r = A\, e^{-\frac{Ea}{RT}} \tag{9}$$

In general, the temperature dependence feature of the reaction is expressed mathematically by entering two coefficients, one of which represents the intercept (ln A) and the other is the slope (-Ea/R) of the straight line called an Arrhenius plot between (Ink) and (1/T), where the equation can be written in the following:

$$\ln k_r = \ln(A) - Ea/RT \tag{10}$$

The activation entropy $\Delta S^{\#}$ was calculated from the value of lnA using the following relation:

$$\Delta S^{\#} = R ln \frac{A}{B} \tag{11}$$

$$B = \frac{KT}{h} \tag{12}$$

where: K is Boltzmann constant ($1.38*10^{-23}$ J/K), h is Planck's constant ($6.62*10^{-34}$ J·S), R is gas constant,

The activation coefficient $\Delta H^{\#}$ was calculated from the relation below:

$$\Delta H^{\#} = Ea - nRT \tag{13}$$

The free energy of activation $\Delta G^{\#}$ is calculated from the following relationship:

$$\Delta G^{\#} = \Delta H^{\#} - T\Delta S^{\#} \tag{14}$$

From equations (11-14), the values of the thermodynamic functions of the activated complex and the activation energy were found as in Table 6.

The activation energy value was found by drawing a relationship between lnk and 1/T, as shown in Fig. 10.

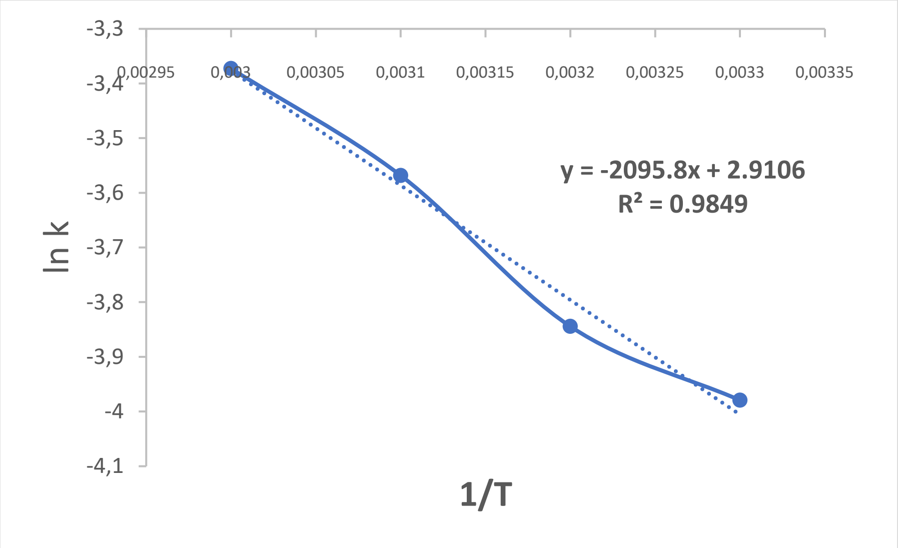

Fig. 10. The graph of *lnk* versus the reciprocal of temperature *1/T*

Table 6. Values of thermodynamic functions and activation energy of the activated complex

Temperature (K)	**Ea KJ.mol^{-1}**	$\Delta S^{\#}$ **KJ.mol^{-1}.K^{-1}**	$\Delta H^{\#}$ **KJ.mol^{-1}**	$\Delta G^{\#}$ **KJ.mol^{-1}**
303	17.424	-0.2361	12.386	**83.924**
313		-0.2364	12.220	**86.213**
323		-0.2367	12.054	**88.508**
333		-0.2370	11.887	**90.808**

The negative activation entropy value indicates that the activated complex is more ordered than the reactants, and this is often the case in combination reactions.

Conclusions

The removal rate is affected by the dye concentration, i.e., the removal rate increases with the increase in the initial dye concentration. The removal rate is also increased by increasing applied voltage and increasing temperature, and efficiency decreases by increasing the distance between electrodes and is affected by the electrolytic solution added to the cell. Furthermore, the best removal rate conditions of Mercurochrome dye were 95% at a concentration of 25 ppm and an electrolyte NaCl concentration of 30 ppm, a distance of 1 cm, a voltage of 30 V, and a temperature of 333 K during a time of 100 min. The lowest removal rate was estimated at 34% at a concentration of 10 ppm, a voltage of 30 V, and a distance of 4 cm. Moreover, the removal rate is affected by the addition of electrolyte, and the removal rate decreases with increasing electrolyte Na_2SO_4 and using a zinc electrode, where the lowest removal rate at a concentration of 30 ppm of electrolyte reached 80%, and the highest removal rate at a concentration of 10 ppm of electrolyte reached 87%. As for adding another electrolyte, NaCl, the removal rate increases with increasing electrolyte concentration, where the highest removal rate at a concentration of 30 ppm of electrolyte reached 94%, and the lowest removal rate at a concentration of 10 ppm of electrolyte reached 89%. Finally, by calculating the thermodynamic functions, it was found that the process is spontaneous, endothermic, and more random. From studying the kinetics of the reaction, it was found that it behaves in an imaginary second order due to the high value of R^2. From studying the thermodynamic functions of the activated complex, it was found that the activated complex is more regular than the reactants, and this occurs in combination reactions.

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