Santandei



Evaluación de los parámetros operativos de una celda de electrocoagulación a escala laboratorio en la eliminación del colorante azo Rojo 40 en solución acuosa usando electrodos de aluminio

Candelaria Tejada-Tovar^{1a}, Ángel Villabona-Ortíz^{1b}, Diego Navarro-Romero^{1c}

¹ Process Design, and Biomass Utilization Research Group (IDAB), Chemical Engineering Department, Universidad de Cartagena, Colombia. Orcid: 0000-0002-2323-1544 ^a, 0000-0001-8488-1076 ^b, 0009-0007-6601-317X ^c. Email: ctejadat@unicartagena.edu.co ^a, avillabonao@unicartagena.edu.co ^b, dnavarror@unicartagena.edu.co ^c

Received: 15 March 2024. Accepted: 26 July 2024. Final version: 20 November 2024.

Abstract

The objective of the present study was to analyze the removal efficiency of red dye 40 by electrocoagulation process using aluminum electrodes. The methodology used includes a first stage taking into account the operating parameters of the electrolyte concentration (0-0.5 g/L NaCl), pH of the solution (4.5-9.1), number of electrodes (2-4), distance between electrodes (1-2 cm), with the best results of this stage, a multilevel factorial experimental design with two variables was performed: (10, 15 and 20) V voltages and electrocoagulation duration of (3, 6, 10, 20, 40 and 60) min. The results show the main role of several parameters to maximize the removal efficiency and minimize the energy consumption. According to the experimental results, the maximum removal efficiency obtained is 85.29 % with an energy consumption of 14.08 kJ.

Keywords: Aluminum; Azo dye; Concentration; Electrolyte concentration; Energy consumption; Efficiency; Electrode; Operating parameters; Water treatment; Electrochemical treatment; Voltage.

Resumen

El objetivo del presente estudio fue analizar la eficiencia de la eliminación del colorante rojo 40 mediante el proceso de electrocoagulación utilizando electrodos de aluminio. La metodología utilizada, incluye una primera etapa teniendo en cuanta los parámetros operativos de la concentración de electrolito (0-0.5 g/L de NaCl) el pH de la solución (4,5-9,1), numero de electrodos (2-4), distancia entre electrodo (1-2 cm) con los mejores resultados de esta etapa se realizó un diseño experimental factorial multinivel con dos variables: los voltajes (10, 15 y 20) V y la duración de la electrocoagulación de (3, 6, 10, 20, 40 y 60) min. Los resultados muestran el papel principal de varios parámetros para

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ISSN Online: 2145 - 8456

maximizar la eficiencia de eliminación y minimizar el consumo de energía. Según los resultados experimentales, la máxima eficiencia de remoción obtenida es de 85.29 % con un consumo de energía de 14,08 kJ.

Palabras clave: Aluminio; Colorante azoico; Concentración; Concentración de electrolito; Consumo energético; Eficiencia; Electrodo; Parámetros operativos; Tratamiento de aguas; Tratamiento electroquímico; Voltaje.

1. Introduction

Nowadays, the freshwater available for humans is less than 1%, while saltwater in the oceans and seas represents more than 97% of the total water on earth [1]. That 1% is decreasing due to its contaminant due to industrial, urban, and agricultural activities, where industrial development represents 20% of this worldwide decrease [2]. Concern about this contamination has resulted in increased research and development of water treatment technologies to prevent the discharge of contaminants into the environment, which could cause problems for human health and environmental impact [3]. The textile industry is one of the largest contaminants regarding the volume and characteristics of its discharges, presenting a high concentration of dyes. When discharged into the environment, they cause interference in the biological processes that occur in water bodies since they reduce dissolved oxygen, generate eutrophication, formation of recalcitrant compounds that are toxic to cells, and hinder the passage of light into water bodies [3], [4], [5].

Different technologies have been implemented in dye removal, such as adsorption, precipitation, chemical degradation, photodegradation, biodegradation, chemical coagulation, and electrocoagulation [6]. While the first two are time-consuming, costly, and of low efficiency, degradation processes can generate by-products that can become toxic [7]. Thus, chemical coagulation and electrocoagulation are more viable alternatives, with electrocoagulation having several advantages over chemical coagulation, which include simple operation, less sludge production, and easy automation [8]. In addition, it combines coagulation, flotation, and electrochemical processes in one cell, where electrooxidation of electrodes (commonly made of Aluminum and Iron) generates the coagulating agent, and similarly by electrolysis hydrogen bubbles are generated which can entrain the clots forming a froth on the surface and since electricity is the driving force for the generation of metal coagulants, the need for adding chemicals is reduced [9], [10].

In the treatment of water with electrocoagulation, its use is reported for the removal of heavy metals such as chromium, cadmium, and nickel, among others, as well as other inorganic contaminants [1], [11], such as fluorides, phosphates and nitrates [12], [13], [14]. It has also been applied for the treatment of turbid water [15], as well as the removal of organic contaminants such as surfactants like linear alkylbenzene sulfate [3], phenolic substances [16] or distillery effluents [17]. It has also been implemented in the removal of dyes, especially those of Azo type, such as Reactive Red 2 or Acid Black 1, where the work is done with aluminum electrodes and the influence of operational parameters such as electrolysis time, initial pH, current density, the distance between electrodes, dye concentration, and conductivity are evaluated [18], [19]. It was reported the use of artificial neural networks to model the decolorization process of a solution of C I Basic Yellow 28, to evaluate those parameters [20], as well as the response surface methodology (RSM) to find the optimal values of the operating parameters (initial dye concentration, pH and distance between electrodes), for the removal of Congo Red using iron electrodes [2]. In the treatment of dyes as well as other organic contaminants, the use of aluminum and iron electrodes have presented similar removal capacities [21], although aluminum electrodes present several advantages such as faster floc formation. due to the lower solubility of aluminum hydroxide compared to iron, causing part of the released iron to remain in the solution [14], in addition, aluminum hydroxide has amphoteric characteristics, maintaining the pH close to 9, while "that treatments with iron electrodes lead the treated solution to pH conditions of more than 11 [3].

Works such as this aim to contribute to the SDGs, which call for the contribution to the eradication of poverty and the protection of the planet, as well as to ensure peace and prosperity in communities [22]. Thus, the objective of the present study was to design and evaluate the performance of a laboratory-scale electrocoagulation cell in the treatment of a synthetic solution of azo dye Red 40 using aluminum electrodes, optimizing its operation, evaluating the parameters of conductivity, initial pH, number of electrodes, electrode distance, voltage and operation time.

2. Methodology

2.1. Materials

The red 40 dye was removed using an electrocoagulation cell with external support and aluminum electrodes, using a 1 L beaker as an electrocoagulation cell, where 1 L solutions of red 40 at 40 ppm were prepared. For the electrolyte concentration and conductivity tests, different concentrations of NaCl were prepared in water used as electrolyte; a 0.1 M hydrochloric acid solution and a 0.1 M sodium hydroxide solution were prepared for the pH tests.

2.2. Design of experiments

A multilevel factorial experimental design was carried out with two variables: voltages (10, 15, and 20) V and electrocoagulation duration of (3, 6, 10, 20, 40, and 60) min, resulting in several tests of (3x6), with a total of 18 experiments. The tests carried out in this degree work did not have replicates. Table 1 shows the different variables considered, and Table 2 shows the variation levels.

Table 2. Experiment matrix

Electrode material	Voltage (V)	Time (min)		
Al	10	3, 6, 10, 20, 40, 60		
	15	3, 6, 10, 20, 40, 60		
	20	3, 6, 10, 20, 40, 60		

2.3. Experimental methods

A 1 L beaker was used as an electrocoagulation cell, with a RockSeed power supply and clamps providing electrical energy with two multimeters to measure voltage and electrical intensity, and aluminum foils were used as sacrificial electrodes. The best operating conditions were obtained taking into account the following parameters: electrolyte concentration/conductivity, pH, number of electrodes, and distance between electrodes, using the experimental design shown in Table 2. The above was followed by the design of a calibration curve, constructed with 8 solutions of diluted red 40 at concentrations of 1, 2, 5, 10, 20, 40, 80, and 160 ppm, then analyzed using the UV-VIS with a wavelength of 502 nm to obtain the absorbance data and with the concentration data, the calibration curve was made, and an equation was obtained to calculate the concentration of the dye removed from the absorbance.

Subsequently, electrolyte concentration and conductivity tests were performed with 3 tests of red 40 at 40 ppm each one [7], concentrations of 0, 0.2, and 0.5 g/L of NaCl were used as an electrolyte to vary the conductivity [19], then the conductivity, temperature and pH data were taken, the electrocoagulation setup was assembled with aluminum electrodes and a constant voltage of 15 V for 20 min; then the behavior of the current intensity was observed, taking amperage data every minute. The concentration values and the percentage of removal were obtained using the data of temperature, pH, absorbance, and the calibration curve.

Туре	Name	Description	Unit
	Number of electrodes	Number of anodes and cathodes performing electrocoagulation	Dimensionless
T. J	Distance between electrodes	The distance separating the electrodes	ст
Independent	Electrical voltage	Effort exerted by the electric field per unit of charge	Volts (V)
	pH	Level of acidity and alkalinity	-log[H+]
	Conductivity	The ease with which electrons move in the solution	μS, μS/cm
	Time	The time that electrocoagulation is applied	min
Dependent	Dye removal	Amount of dyes removed compared to the initial value	
	Solution volume	Amount of gray water solution inside the electrocoagulation cell	<i>cm</i> ³
	Exposed area	Area of the electrodes where the oxidation and reduction processes occur	cm^2
Intervener	Current intensity	Velocity at which electric charges flow	Amps (A)
	Type of current	Loads behavior in relation to their direction over time	AC/DC
	Electrode configuration	Type of connection between electrodes and electrical source	Monopolar /bipolar
	Resistance	Obstruction of the flow of electric current through a conductor	Ω

Table 1. Variables

Tests were also carried out at pH (acid, basic, and neutral), and for each, 1 L of solution of red 40 at 40 ppm was prepared with distilled water, HCl, and NaOH at 0.1 M were used to alter the pH of the solutions. In order to know how it influences the number and distance separating the electrodes, a multilevel experimental design was performed with 3 values for 2 variables, which are distance between electrodes (1, 1.5 and 2) cm and the number of electrodes (2, 3 and 4), for that 9 solutions of 1 L of red 40 at 40 ppm are prepared, using the electrolyte concentration and pH value with better results in the 2 previous tests [23], amperage data were taken every minute to observe the behavior of the current intensity and samples were taken at 10 and 20 min, acquiring the data of conductivity, temperature, pH and absorbance, also using the calibration curve the concentration values and the percentage of removal were obtained.

Subsequently, the electrocoagulation cell was evaluated to remove the dye according to the experimental design in Table 1. The concentration results of 18 samples were determined, with 1 L solutions of red 40 at 40 ppm, using NaCl as an electrolyte to control the conductivity at the concentrations with the best results in the preliminary test; the pH was left in its natural state, and the sacrificial electrodes were adjusted in the amount and distance with the best results in the preliminary test.

Further on, 18 samples were taken using the aluminum electrodes, 6 samples at 10 V, 6 at 15 V, and the other 6 at 20 V, for 60 min; the initial values of temperature, pH and conductivity were taken; samples were taken after 3, 6, 10, 20, 40 and 60 min, each one was taken its temperature, pH and conductivity value and the absorbance value was found using the UV-VIS plus the calibration curve the concentration was found, which, compared with its initial value, gave the percentage of removal.

Finally, for the quantification of sludge, the resulting solution was filtered through a filter paper with a vacuum pump, which was stored and kept in Petri dishes in a dark space to avoid possible degradation, then dried in an oven (T =84 °C; 357 °K) and analyzed by FTIR for functional groups, in order to confirm whether or not there was a chemical reaction in the process, which could alter the red 40 and produce another substance, confirm the presence of the coagulant in them [2].

3. Results

3.1. Design of the Electrocoagulation Cell Used

This design was inspired by external support-type cells because, in comparison to internal support-type cells, these do not involve the introduction of an apparatus inside the electrocoagulation reactor [3], [24], [25], this design showed promise; therefore, it was taken as the final design, as electrocoagulation cell it was decided to use a laboratory beaker with volume of 1 L [26], [27] the dimensions of this are shown in Figure 1.



Figure 1. (a) Beaker Side View, (b) Beaker Top View.

3.2. Electrodes

A total of 4 electrodes were fabricated from a 1 mm thick aluminum plate each; the electrode dimensions are shown in Figure 2 [3]. The electrodes are submerged at 8 cm, giving a contact area of 48 cm².



Figure 2. Electrode Dimensions.

3.3. Electrode holder

As in other studies [3], 2 supports were made for the electrodes, for the first one, 2 PVC pipes were taken as shown in Figure 3, 6 slots of 1 mm wide and 2.5 mm depth separated by 1.5 cm each one, then at the ends

behind the slots, perforations were made where a stainless steel screw was placed with a nut at the end to adjust the support and ensure that the sheets do not move as shown in Figure 3.



Figure 3. (a) Side View Support, (b) Top View Support.

For the second one, it was also used 2 PVC pipes, but this time of 1" diameter and 25 cm long, and were made several slots 1 mm wide and 2.5 mm depth, separated by 1 cm each one; then, at the ends behind the slots were drilled perforations through which a steel screw was placed with a nut at the end to adjust the support.

3.4. Electrical system

It used a method that would allow the control of the voltage as a variable and also to obtain the Amperage data when performing the electrocoagulation; for this reason, 2 multimeters were connected to the system, one as a Voltmeter and the other as an Ammeter. Since tests were made varying the number of electrodes, the connection of electrodes varied according to the number of electrodes, as shown in Figure 4.

3.5. Assembly of the Electrocoagulation Cell

With the parts designed and built, the assembly was assembled; first, the electrodes were sanded by washing them with water and drying them; after that, they were inserted into the slots of the holder, and the holder was tightened with nuts to keep the electrodes fixed, and



finally, it was placed inside the cell to connect the

Figure 4. (a) Connection with 2 Electrodes, (b) Connection with 3 Electrodes, (c) Connection with 4 Electrodes.



Figure 5. Laboratory scale Electrocoagulation System.

3.6. Assembly of the Electrocoagulation Cell

The results of the preliminary tests for the analysis of the operating factors are presented below.

3.6.1. Calibration Curve

Figure 6 shows the red calibration curve 40.

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3.6.2. Electrolyte Concentration Tests

As can be seen in Table 3, the electrolyte concentration shows a trend of higher percentage removal with higher electrolyte concentration, which can be attributed to the increase in the initial conductivity, which in turn is accompanied by an increase in the average intensity of the process, also as expected from a process that involves the release of an electric current in a liquid, an increase in the temperature of the solution during the process can be noted at the same time as an increase in pH and a slight decrease in the conductivity of the solution during the process which can be attributed to the release of aluminum ions followed by the formation of hydroxides during the process and its consequent precipitation. An extra test was carried out using an electrolyte concentration of 1 g/L NaCl. Results were obtained of 80.7% removal with 1838 μ S, which is attributed to the increase in bubbling or the massive release of metal ions that hindered the formation of flocs; with these results, it was decided that the best electrolyte concentration to use would be 0.5 g/L NaCl.



Figure 6. Calibration Curve.

 Table 3. Electrolyte Concentration Test Results

Electrode material	Aluminum										
Initial	40 ppm	40 ppm									
concentration	to ppm										
Distance between	1.5 cm	1.5 om									
electrodes	1.5 CIII	1.5 cm									
# Electrodes	4										
Voltage	15 V										
Initial pH	Normal s	olution pH									
NaCl concentration (g/L)	Time (min)	Conductivity (µS)	Intensity (A)	рН	T (°C)	Concentration (ppm)	% Removal				
0.00	0	27	0.035	8.40	25.50	40.00	0.00				
	10	22		8.80	27.00	27.50	31.25				
	20	17		8.80	29.90	15.39	61.52				
	0	410	0.743	8.40	25.00	40.00	0.00				
0.20	10	370		9.10	26.00	20.11	49.72				
	20	360		9.10	27.00	7.87	80.30				
	0	926		7,70	25.00	40.00	0.00				
0.50	10	912	1.93	8.50	26.00	7.31	81.72				
	20	889		8.9	29.00	3.42	91.45				

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3.6.3. pH tests

For this test, 3 pH were established: one neutral, one acidic, and one basic. As can be seen in Table 4, the results of the pH tests, similar to the electrolyte concentration test, show a tendency to increase the temperature and pH of the solution during electrocoagulation as well as a decrease in conductivity, in terms of removal of the neutral pH showed better removal, this because, at low and high pH values, the soluble species with positive/negative charge dominate the solution [28]. One effect of decreasing the pH appears to be the slight increase in conductivity and average intensity due to the anionic character of the dye. Because the pH of the red 40 at 40 ppm solution is naturally close to 7 it was decided to carry out all the following tests without altering the natural pH of the solution in order not to spend neither reagents nor time adjusting the pH of each prepared solution. The data obtained can be seen in Table 4.

3.6.4. Electrode Number and Distance Tests

As can be seen below in Table 5, the results of the tests of the number and distance of electrodes show that the increase of electrodes and decrease of the distance of separation lead to an increase in the current due to an increase of the electrolysis zones and the decrease of the resistance respectively [29], on the other hand, the trend of increasing temperature and pH and decreasing conductivity during the process continues, showing the highest rate of decrease in conductivity in the 1.5 cm and 3 electrodes arrangement and the lowest in the 1 cm/3 electrodes and 2 cm/4 electrodes arrangements, while a higher rate of increase in pH is observed in the 1 cm/4 electrodes and 2 cm/3 electrodes arrangements and the lowest rate of pH change in the 1 cm/2 electrodes arrangement.

Regarding the removal of the dye, the arrangements 1 cm/3 electrodes, 1.5 cm/4 electrodes, and 2 cm/4 electrodes stand out, which have a current intensity range between 1.6 and 2.34 A, while 1.5 cm/2 electrodes and 2 cm/2 electrodes are in the 0. 6 A being the only ones with final removal percentages higher than 90%, this difference in intensity indicates two zones and coagulation mechanisms, where the first one occurs a coagulation by sweeping due to the high release of aluminum that form large amount of flocs that drag the dye, while in the low current zone coagulation occurs by neutralization of charges between the anionic dye and positively charged aluminum [30], at intermediate currents there is a decrease in efficiency due to the stabilization of the flocs in the solution and at intensities of 2.86 A (1cm/4 electrodes) there is a decrease due to the increase of bubbles generated at the cathode that prevent the correct formation of flocs [3], [31], for all these reasons the 2cm/2 electrode arrangement was chosen for the real tests since it presents the lowest average intensity and therefore has the lowest energy consumption of arrangements with a removal percentage greater than 90%.

l'able 4. pH Test Result

Electrode n	naterial	rial Aluminum							
Initial concentration 40 ppm									
Distance be electrodes	etween	1.5 cm							
# Electrode	s	4							
Voltage		15 V							
NaCl conce (g/L)	entration	0.5							
Testitical en II	Time	Conductivity	Intensity	11	Т	Concentration	%		
Initial pH	()			рн	$(0,\mathbf{O})$				
-	(min)	(μS)	(A)	-	$(^{\circ}\mathbf{C})$	(ppm)	Removal		
	(min) 0	(µS) 1105	(A)	4.50	24.90	(ppm) 40	Removal0		
4.5	0 10	(µS) 1105 1048	(A) 2.19	4.50 8.30	(°C) 24.90 26.70	(ppm) 40 7.55	Removal 0 81.12		
4.5	0 10 20	(µS) 1105 1048 1038	(A) 2.19	4.50 8.30 8.60	(°C) 24.90 26.70 29.10	(ppm) 40 7.55 4.11	Removal 0 81.12 89.70		
4.5	0 10 20 0	(µS) 1105 1048 1038 928	(A) 2.19	4.50 8.30 8.60 7.00	(°C) 24.90 26.70 29.10 25.50	(ppm) 40 7.55 4.11 40.00	Removal 0 81.12 89.70 0.00		
4.5	0 10 20 0 10	(µS) 1105 1048 1038 928 912	(A) 2.19 1.81	4.50 8.30 8.60 7.00 8.40	(°C) 24.90 26.70 29.10 25.50	(ppm) 40 7.55 4.11 40.00 4.23	Removal 0 81.12 89.70 0.00 89.41		
4.5	0 10 20 0 10 20	(µS) 1105 1048 1038 928 912 895	(A) 2.19 1.81	4.50 8.30 8.60 7.00 8.40 8.50	(°C) 24.90 26.70 29.10 25.50 25.50 27.90	(ppm) 40 7.55 4.11 40.00 4.23 2.50	Removal 0 81.12 89.70 0.00 89.41 93.75		
4.5	0 10 20 0 10 20 0 0	(µS) 1105 1048 1038 928 912 895 906	(A) 2.19 1.81	4.50 8.30 8.60 7.00 8.40 8.50 9.10	(°C) 24.90 26.70 29.10 25.50 25.50 27.90 24.70	(ppm) 40 7.55 4.11 40.00 4.23 2.50 40.00	Removal 0 81.12 89.70 0.00 89.41 93.75 0.00		
4.5 7 9.1	$\begin{array}{c} (\mathbf{mn}) \\ 0 \\ 10 \\ 20 \\ 0 \\ 10 \\ 20 \\ 0 \\ 10 \\ 1$	(µS) 1105 1048 1038 928 912 895 906 871	(A) 2.19 1.81 1.83	4.50 8.30 8.60 7.00 8.40 8.50 9.10 9.60	(°C) 24.90 26.70 29.10 25.50 25.50 27.90 24.70 26.00	(ppm) 40 7.55 4.11 40.00 4.23 2.50 40.00 5.60	Removal 0 81.12 89.70 0.00 89.41 93.75 0.00 86.00		

Electrode ma	terial	Alumir	um						
Initial concentration		40 ppm							
Voltage		15 V							
Initial pH		Neutral	[
NaCl concentration (g/L)		0.5							
Distance between electrodes	# Electrodes	Time (min)	Conductivity (µS)	Intensity* (A)	pН	T (°C)	Concentration (ppm)	% Removal	
		0	924		6.05	28.70	40.00	0	
	2	10	896	1.26	6.82	29.60	6.14	84.65	
		20	884		6.96	30.50	4.79	88.02	
		0	956		5.95	28.60	40.00	0.00	
1 cm	3	10	932	2.34	8.64	28.70	3.84	90.40	
		20	930		8.86	30.70	1.90	95.25	
		0	934		6.00	25.90	40.00	0.00	
	4	10	880	2.86	9.20	27.70	6.0	84.00	
		20	870		9.24	31.10	4.25	89.35	
	2	0	926	0.63	6.80	25.60	40.00	0.00	
		10	902		7,54	26.50	9.18	77.05	
		20	885		8.33	27.90	2.48	93.80	
	3	0	928	1.20	6.80	24.00	40.00	0.00	
1.5 cm		10	873		8.26	25.00	5,35	86.62	
		20	811		9.07	26.00	4.84	87.90	
	4	0	949	1.80	6.80	24.00	40.00	0.00	
		10	900		8.30	25.00	6,75	83.12	
		20	873		8.40	28.40	2.32	94.20	
	2	0	950	0.60	6.20	26.50	40.00	0.00	
		10	937		7.25	26.70	7.17	82,.7	
		20	919		8.49	26.90	2.64	93.40	
		0	917		5.95	27.30	40.00	0.00	
2 cm	3	10	908	1.19	9.04	27.50	8.21	79.47	
		20	887		9.26	28.20	6.21	84.75	
		0	928		6.02	29.40	40.00	0.00	
	4	10	906	1.62	8.59	29.70	2.55	93.62	
		20	902		9.02	29.90	1.83	95.40	

Table 5. Electrode # and Distance Test Results

3.6.5. Test results of the electrocoagulation system

The following are the test results. The effect of the variation of the voltage difference and the operating time was studied. From the results obtained, it can be noticed in Figure 7 the conductivity that in comparison with the results of the preliminary tests where a tendency to decrease the conductivity was noticed, this time we observed an increase in the conductivity, being the greatest change in the first 10 minutes.

In the aspect of pH, from Fig. 7, it is observed that the trend of the preliminary tests to an increase of pH during the operation was maintained, reaching pH values close.

To 9 during the first 10 minutes regardless of the applied voltage, this because, at pH close to 9, the generation of aluminum and hydroxide ions reaches equilibrium with the formation of aluminum hydroxide [19].

During the rest of the operation, the pH shows slight variation, reaching values of 9.39, 9.47, and 9.26 with 10, 15, and 20 voltages, respectively.

In the temperature, the tendency to increase the temperature of the solution is maintained, and, as expected, the final temperatures are obtained.

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Figure 7. (a) Change of Conductivity with Respect to Time; (b) Change of pH with Respect to Time.



Figure 8. (a) Change of Removal of Red 40 Respect to Time; (b) Change of Removal of Red 40 Respect to energy consumed.

Finally for the removal, a discoloration was noted with the passage of time, although an increase in the percentage of removal is maintained with respect to time and there are results with final removal percentages greater than 80%, it should be noted that this was after 60 min of operation compared to the 20 min of the preliminary tests, since it can be seen that at 20 min the aluminum and 15 V arrangement which is the same that gave the highest results at the end of the preliminary tests barely reaches 80% of removal compared to 93.4 % of removal that was seen in the preliminary tests, in the case of higher voltage they correspond to a higher removal rate, in less time as can be noted with the values of 15 and 20 V where it reaches removals of more than 80% at 20 min while with 10 V it passes that threshold after 40 min reaching values of 85.29% at 60 min, reaching values close to the treatments with 15 and 20 V in that same time, seeming to reach maximum removals close to 90% as can be seen in Fig. 8. Now, if we take into account the energy consumption, as expected, the higher the voltage, the higher the energy consumption, with the consumption at 15 and 20 V double and quadruple, respectively, and the consumption at 10 V; this evaluates the removal concerning energy expenditure, we note that with 10 V and 60 minutes a removal of 85.29% is achieved, close to those obtained at 40 min with 15 volts (85.17%) and 20 minutes with 20 V (86.51) but with a lower energy consumption of 14.08 kJ (Figure 9).



Figure 9. Final Red 40 removal percentages according to the electrode material and voltage.

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3.7. Results of sludge

During electrocoagulation, sludge formation was observed as a result of the generation of flocs from the process; the behavior and appearance of this sludge showed differences with the variation of the applied voltage. The higher the voltage, the greater the amount of sludge generated; on the other hand, the sludge tended to possess a red color of red 40, with slight traces of white substance that were concluded to be aluminum hydroxides. In addition, the aluminum sludge tended to float to the surface due to bubbling. The results of the FTIR analysis of the sludge samples are presented below, along with an FTIR analysis of red 40 to confirm the coagulation process with forming aluminum hydroxides.

In Fig. 10, peaks of functional groups present in red 40 are noted, the peak at 3432.44 cm⁻¹ can be attributed to the hydroxyl group, the peaks between 1620.71 and 1410.01 cm⁻¹ can be attributed to the azo group, the peaks at 1270.59 and 1251.42 cm⁻¹ can be attributed to the methyl group, the peaks between 1182.19 and 1024.41 cm⁻¹ can be attributed to the sulfonic and methoxyl salt group, the rest corresponds to the benzene rings [2], [32].

In Figure 11 (a), a substantial variation in the peaks is noted, indicating the presence of coagulant species that are generated from the aluminum-based electrocoagulation leading to the formation of AlOOH, the intensity of all peaks decreased due to the formation of a complex dye-metal, the presence of peaks at 2323.99 and 2161.6 cm⁻¹ is observed which is not present in the following 2 analyses of the aluminum sludge which can be attributed to the formation of a thiocyanate or isothiocyanate compound [2], [33]. Figure 11 (b) shows evidence of the formation of coagulating agents along with the decrease in the % transmittance of all peaks, a decrease in % transmittance from 88% to 54% of the hydroxyl group, from 83% to 75% of the azo group and from 85% to 75% of the sulfonic salt group [2], [33]. Figure 11 (c) once again shows evidence of the formation of aluminum coagulating agents and how all the peaks decrease their % transmittance, with a decrease in % transmittance from 88% to 53% for the hydroxyl group, from 83% to 72% for the azo group and from 85% to 80% for the sulfonic salt group [2], [33].

4. Conclusions

In this study, the efficiency of electrocoagulation using aluminum electrodes and the effect of the operating parameters were investigated for their use in the treatment of Red 40 dye solutions. The variables electrolyte concentration and pH influence the removal rate. of the electrocoagulation process, a higher electrolyte concentration is equivalent to a higher conductivity, which in turn leads to an increase in the removal rate of more than 90% at concentrations of 0.5 g/L of NaCl with conductivities that range between 800 and 1000.



Wavenumber (cm⁻¹) Figure 10. FTIR analysis of Red 40.

Evaluation of the operating parameters of a laboratory-scale electrocoagulation cell in the elimination of the azo dye Red 40 in aqueous solution using aluminum electrodes

From that point somewhere between the concentrations of 0.5 g/L and 1 g/L of NaCl with conductivities between 1100 and 1800 the removal rate begins to decrease; meanwhile acidic and basic pH negatively affect the rate removal, while neutral pHs give the best removal results, at the same time it was noted that the electrocoagulation process causes changes in both conductivity and pH, decreasing and increasing it respectively.

The maximum number of electrodes and distance between electrodes are determined by the volume and design of the reactor and are determining the current that flows in the reactor, and therefore affecting the removal rate, with removals greater than 90% being obtained with only two electrodes. and 2 cm apart current intensity of 0.6 amperes. Likewise, it was observed that the variables voltage and residence time affect the dye removal rates, with the voltage being proportional to the intensity of the electric current, which in turn is proportional to the amount of metal ions released in water and the intensity of the bubbling, which leads to better removal, and the longer the operating time, the better the removal results. Finally, although at 20 V removals greater than 85% are achieved in less time, they present a greater energy consumption than at lower voltages, this is because the energy is transformed into heat as the voltage increases, presenting a direct relationship between the voltage and the final temperature of the treated solution, it is then concluded that the best conditions found in this research were 0.5 g/L of NaCl as electrolyte, pH 7, 2 electrodes, 2 cm separation between them with a voltage of 10V for 60 min achieving a removal of 85.29% with 14.08 kJ of energy consumed.

Funding

Not applicable.

Autor Contributions

Á. Villabona-Ortíz: Methodology, Experimental Work, Validation, Resources, Data Curation, Writing—Original Draft Preparation, Writ-Ing—Review And Editing, Visualization, Supervision, Project Administration, Funding Acquisition.



Figure 11. FTIR analysis (a) aluminum sludge at 10 V; (b) aluminum sludge at 15 V; (c) aluminum sludge at 20 V

C. Tejada-Tovar: Methodology, Experimental Work, Formal Analysis, Investigation, Resources, Writing— Original Draft Preparation, Visualization, Project Administration, Funding Acquisition. D. Navarro-Romero: Experimental Work, Software, Investigation, Resources, b Writ-Ing—Review And Editing.

All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest

The authors declared that they have no conflicts of interest to this work.

Institutional Review Board Statement

Not applicable.

Informed Consent Statement

Not applicable.

Disclosure Statement

The authors reported no potential conflict of interest.

Declaration of Competing Interest

The authors declared that they have no conflicts of interest to this work.

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