

ORIGINAL RESEARCH ARTICLE

Electrode performance via electrochemical treatment of textile wastewater: Analysis and characterization

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ABSTRACT

This study investigated a novel real-time thermodynamic diagnostic approach via current-interruption during active EC for track of the open circuit potential (OCP) during short current interruptions over the course of the experiment throughout the performance of a batch electrocoagulation reactor (BER) that using aluminum (Al)-iron (Fe) electrodes for the treatment of simulated wastewater containing a mixture of cation and anion dyes. The study considered various factors, including pH (2–10), current (0.6–1.4 A), concentration of dyes mixture (100–500 ppm), and electrolysis time (4–90 min). Surface response design process based on the central composite design (RSM-CCD) has been used to design the experiments and optimize the present operational parameters. The COD reduction, electrode consumption, open circuit potential (OCP), and energy consumption were predicted under the mentioned conditions with the aid of the analysis of variance (ANOVA). The optimization process was conducted to categorize the optimal conditions required to achieve the highest reduction of COD and minimum real consumption of energy and the anode electrode. The highest predicted reduction of COD of 94.57% vs. real removal of 88.9% under minimum consumption of energy and the anode electrode were obtained at the optimal conditions of 0.6 A, pH=7.98, 4 min, and the highest initial COD of 4236.7 ppm. XRD, FESEM, and FTIR tests were used for anode characterization. The main conclusion revealed that the finding of treating of wastewater that contains different types of dyes that could affect the performance of the electrocoagulation process. Also, the use of current-interruption during active EC to insight the thermodynamic oxidation ability of the anode reflects a complex interaction between the studied variables and response of the OCP of the anode during operation of EC.

Keywords: Congo red dye; methylene blue dye; energy and anode consumption optimization; RSM/CCD; electrode characterization

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1. Introduction

Water is a vital resource that sustains ecosystems and supports numerous human activities, including industrial operations (Enock & Yaweri, n.d.). Water pollution from industrial wastewater discharge has become a significant environmental challenge, especially in rapidly developing of economies (Kamal et al., 2025). As processes such as manufacturing, mining, agriculture, and textiles continue to expand, the volume and diversity of pollutants entering freshwater bodies will be increased (Davis, 2024). The textile industry generates a substantial volume of effluents, including different chemicals (Sudarshan et al., 2023) (Ramadan et al., 2023).

Dyes are strongly regarded as the primary source of pollution among the diverse complex effluents observed in textile wastewater

(Nippatla & Philip, 2019). Therefore, it is imperative to efficiently eliminate dyes from wastewater to guarantee the safe release of treated liquid effluent into water bodies (Tan et al., 2015). Treating wastewater containing dyes is highly challenging issue due to the organic nature of the dyes, which exhibit resistance to aerobic digestion, heat, and oxidizing agents (Sadoon & M-Ridha, 2020). Consequently, in order to eliminate the dyes from textile effluents, it is essential to identify an effective method of wastewater treatment (Mohammed et al., 2020). Traditional technologies used for wastewater treatment including biological, physical, and chemical methods (Meshram et al., 2025). These methods are bounded in application because of the high cost, low efficiency, lack of applicability to a variety of dyes, and aggravated by the harmful pollution generated by excessive chemical usage ((Haran & Ali, 2022); (Ayad A. H. Faisal et al., 2022)).

Electrochemical wastewater treatment systems have garnered significant interest in recent years. These technologies include electrodeposition, electro-Fenton, electrooxidation (EOX), and electrocoagulation (EC) and their successive combinations for the decontamination of industrial discharged wastewaters (AlJaberi et al., 2023) (Aqeel et al., 2020). One of the most applied techniques is EC that has the advantages of its higher rate of pollutant reduction with minimal treatment duration, a reduced requirement for coagulant ions, simple maintenance and operation, decreased sludge generation with simplified disposal, easily settling and readily dewaterable flocs, and the absence of a need for chemical additives (Bhagawati et al., 2022).

The electrocoagulation process involve the in-situ generalization of a coagulant component through the application of external power to the electrodes, resulting in the release of metal ions (M^+) from the sacrificial electrode. Water electrolysis produces hydroxyl ions (OH^-) and hydrogen ions (H^+), which interact in the bulk solution to form and generate metal hydroxide and destabilizing dye particles, then adsorb them, and finally aggregate them together. Nevertheless, the gravitational force caused larger particles to precipitate, smaller particles remained suspended or floated because of gas bubbles of hydrogen (H_2) and oxygen (O_2) generated from the electrode surfaces, respectively. The following illustrates the mechanism of the electrochemical coagulation EC process (Jasim et al., 2024).



The most used electrodes in the EC process are aluminum and iron. Typically, Fe and aluminum are good coagulants with their inexpensive cost and high efficiency for treated wastewater. Flock products from Al electrode has more features in comparison to Fe flocks since flocks produced using Al are lighter ((El-Ashtoukhy et al., 2020); (Houssini et al., 2021); (Alardhi et al., 2024)).

The open circuit potential (OCP) provides a direct indication of the thermodynamics ability of oxidation at the working electrode. This indicates the spontaneous tendencies of both the anodic and cathodic reactions which can take place in the vicinity of the working electrode. In traditional corrosion polarization test, the OCP refers to the voltage records before the polarization test that is useful for both determining that scanning range of polarization, and gives understanding of thermodynamic ability. In Electrocoagulation (EC), some studies asses the dynamic variation in cell potential and current during the process time while the EC reactor was continuously powered. This yields a closed-circuit cell voltage, not open one (i.e., OCP). In EC process and to assess the working electrodes' thermodynamic capabilities to form ions needed for coagulation, OCP measuring during EC process through the circuit interrupted for few seconds can be performed. This permits the assessment of the thermodynamic oxidation ability of the electrode when it continuously interacts with the varying dye molecules. The greater the negative OCP value; the greater will be its ability to produce ions. Conversely, the more positive the OCP value, the smaller will be the working electrodes' ability to generate ions (Choudhary et al., 2016; Jang et al., 2025).

The utilization of diverse experimental design techniques, particularly the response surface method (RSM), is favored over other alternative factorial approaches. It minimizes the number of required experiments by concurrently assessing their effects, resulting in cost savings and time (Moghaddam & Seyyedi, 2022).

In this study, in contrast to other studies that had explored conventional EC process, a significant gap in understanding real-time, observation of anode surface states during EC process had been intensively explored via OCP tracking. To address this aspect, a novel real-time thermodynamic diagnostic approach via current-interruption during active electrocoagulation had been used to track the OCP measurement during the performance of EC process for the removal of anion and cation dyes mixture under specific parameters. This has been done using response surface methodology (RSM)-based central composite design method (CCD) to evaluate the effectiveness of the EC process, and develop predictable relations between the COD removal efficiency, combined with energy and anode consumption. This strategy of linkage between OCP and EC process provide a bridge between EC for dyes removal and actual insight on the thermodynamic variation in anode potential to oxidize during the operation and surface characterization techniques beside electrode weight losses.

2. Materials and methods

2.1. Chemicals

Two types of dyes, namely Congo red and methylene blue, were used in the present study to investigate the performance of a batch electrocoagulation reactor. Congo red dye is an azo-anion dye source (HIMEDIA) and is water-soluble, and its solubility is greater in organic solvents, yielding a red colloidal solution. Methylene blue is a cation-thiazine dye source (Loba Chemie) and works by converting the ferric iron in hemoglobin to ferrous iron.

3. Experimental setup

A one-liter EC reactor containing a work solution of 750 ml dye mixture was prepared using deionized water (**Figure 1**). 0.1 M of either NaOH or HCl was used to adjust the solution pH, and 0.75 g of NaCl salt per liter was used as a supporting electrolyte to maintain the desired conductivity in the experiments because the Cl^- anions can improve the anodic dissolution of the aluminum metal through the destruction of the passive layer on the metal surface (Moneer et al., 2021).

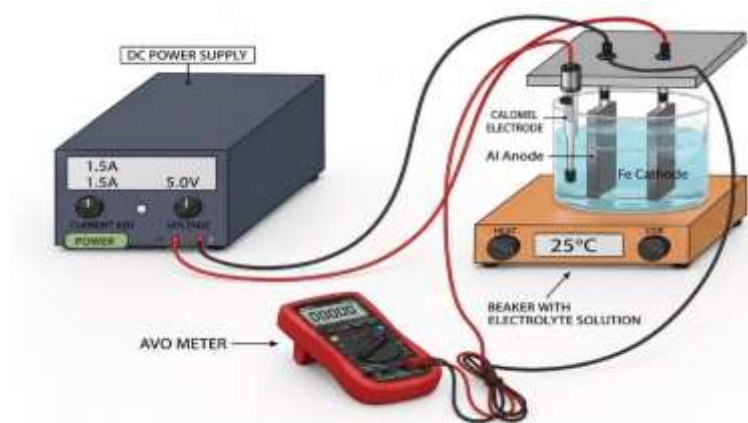


Figure 1. EC experimental setup.

Two flat sheets (23×6 cm) were used as electrodes with an effective immersed area of 45 cm^2 . The aluminum (Al) sheet was used as the anode, while the iron (Fe) sheet was performed as the cathode with a 2 cm interelectrode gap. This reactor was placed on a magnetic stirrer-hotplate from ISOLAB to provide the

required fixed mixing of 150 rpm. A direct-current (DC) power supply was used to maintain the required current intensity.

For tracking the open circuit potential (OCP), a calomel electrode was used, which is a standard reference electrode used to accurately measure the potential of the other electrodes without current flowing in it, as well as an AVO meter to measure the potential difference between the working electrode and the calomel electrode (Pruthviraj & Somashekariah, (2014).; Sahrani et al., 2014). For the measuring of the OCP during EC experiments, the DC circuit was interrupted for few seconds during operation intervals to prevent the Ohmic drop throughout the dye's solution during potential recording.

The studied responses of COD reduction, electrode consumption, and energy consumption were conducted in this investigation. The COD measurement of the raw and treated samples was conducted using dichromate closed flux according to the standard method (APHA, AWWA, and WFE, 1998). The required amount of sample was filled into the COD test cells and then heated in a Thermo reactor for 2 hours. After digestion, the COD test cells were removed and cooled to room temperature (Asghar et al., 2017).

The COD reduction efficiency from simulated dye wastewater containing cation and anion dyes was calculated using Eq. 4:

$$\text{COD reduction \%} = \frac{\text{COD}_{\text{initial conc.}} - \text{COD}_{\text{final conc.}}}{\text{COD}_{\text{initial conc.}}} \times 100 \quad (4)$$

The change in energy consumption (EC) per cubic meter of wastewater and theoretical anode consumption (TAC) are calculated according to Eqs. 5 and 6, as follows:

$$EC = \frac{U \times I \times t}{1000 \text{ V}} \quad (5)$$

$$TAC (g) = \frac{I \times t \times M}{96485.34 \text{ Z}} \quad (6)$$

Where I and U represent the constant current (A) and electric steady state potential of the electrolytic cell (V), respectively, that is supplied to the electrochemical system. The retention time (h) is t, and the volume of treated wastewater (m³) is V for EC in kWh m⁻³. M is the molecular weight of aluminum.

3.1. Experimental design

In this study, RSM was applied using statistical software (Minitab) to interactively design experimental trials and predict outcomes depending on operational variables. The primary impact of current (A) (Z_1), initial pH (Z_2), electrolysis time (min) (Z_3), and dye concentration (ppm) (Z_4) were systematically varied within the specified ranges shown in **Table 1**.

Table 1. Experimental design ranges of the operating variables.

Operating Variables	Values	Operating Variables	Values
Z_1 ; Current (A)	0.6-1.4	Z_3 ; Electrolysis time(min)	4-90
Z_2 ; Initial pH	2-10	Z_4 ; Dye Conc. (ppm)	100-500

The main purpose of this study, however, was not only to optimize the operating conditions with the aid of response surface methodology (RSM), but also to develop statistically validated models that relate the operating variables quantitatively to the process responses. There are several factorial and centre point runs included in the experimental design that act as reference runs for the evaluation of the effects of the variables investigated. Furthermore, the model predictions were experimentally validated under optimum operating conditions, and the results obtained were in good agreement with experimental ones, which demonstrates the reliability of the RSM models developed. This work is not intended to compare with other treatment technologies. The use of statistically designed experimental runs and validation experiments ensured an

adequate baseline for the evaluation of the influence of the operating parameters used and to indicate the robustness of the optimization approach used.

3.2. Statistical measures and analysis

ANOVA has been utilized to analyze the disparities across group means within a sample. It was conducted in this investigation to evaluate the correlation between the dependent variable (COD reduction percentage, energy consumption, electrode consumption, and OCP) and the independent variables (current, pH, electrolysis time, and dye concentration).

4. Results and discussion

The effects of operational parameters on the studied responses, COD reduction, energy consumption (kWh/m³), real anode consumption (g), and OCP were analyzed in this investigation. The experimental design was conducted using RSM-CCD with the aid of Minitab-18. **Table 2** shows the experimental conditions of the operating variables and the obtained results of the COD reduction response, energy consumption and real anode consumption. The relationships between independent variables (current (A), initial pH, electrolysis time (min), and initial COD (ppm)) and the responses have been expressed in Eqs. (7), (8), and (9).

Table 2. Results of COD reduction % response, energy and anode consumption.

Run No.	Z1	Z2	Z3	Z4	Actual COD %	Predicted COD %	Energy consumption (kW h/m ³)		Real Anode-Consumption (g)	
							Actual	Predicted	Actual	Predicted
1	0.8	4	26	200	62.525	64.3257	4.9867	2.1908	0.14	0.141250
2	1.2	4	26	200	64.529	66.0518	8.3640	8.9484	0.21	0.211667
3	0.8	8	26	200	76.553	74.5262	5.1227	5.3825	0.15	0.148333
4	1.2	8	26	200	82.164	79.5199	11.4240	12.7722	0.23	0.221250
5	0.8	4	69	200	88.176	85.7794	14.1262	15.3836	0.38	0.386667
6	1.2	4	69	200	90.782	89.5569	26.3040	26.3891	0.58	0.579583
7	0.8	8	69	200	83.166	84.1963	14.8569	16.3719	0.39	0.391250
8	1.2	8	69	200	91.984	91.2413	28.4960	28.0096	0.58	0.586667
9	0.8	4	26	200	80.975	80.4753	4.7147	5.8990	0.15	0.153333
10	1.2	4	26	400	82.218	80.9811	12.0360	10.7848	0.25	0.236250
11	0.8	8	26	400	81.453	82.5572	5.7120	5.8907	0.18	0.167917
12	1.2	8	26	400	84.990	86.3306	11.9680	11.4085	0.25	0.253333
13	0.8	4	69	400	90.631	93.0849	16.3182	15.2338	0.39	0.386250
14	1.2	4	69	400	94.742	95.6421	23.9293	24.3674	0.58	0.591667
15	0.8	8	69	400	85.946	83.3832	12.9084	13.0220	0.39	0.398333
16	1.2	8	69	400	91.013	89.2080	19.7280	22.7877	0.62	0.606250
17	0.6	6	47	400	83.798	83.7237	6.0160	6.1826	0.21	0.207083
18	1.4	6	47	300	90.040	91.3616	23.8342	22.7059	0.48	0.485417
19	1.0	2	47	300	73.440	72.1563	9.0867	10.3586	0.33	0.325417
20	1.0	10	47	300	73.971	76.5014	14.2044	11.9707	0.34	0.347083
21	1.0	6	4	300	75.033	74.7298	1.6533	2.6598	0.04	0.052083
22	1.0	6	90	300	98.141	99.6915	29.2000	27.2318	0.66	0.650417
23	1.0	6	47	300	73.413	75.1715	16.8156	16.4126	0.32	0.315417
24	1.0	6	47	100	88.749	88.3278	15.4578	14.8990	0.34	0.347083
25	1.0	6	47	500	83.134	81.7318	14.2044	14.2044	0.36	0.346667
26	1.0	6	47	300	79.814	81.7318	15.1444	14.2044	0.35	0.346667
27	1.0	6	47	300	82.337	81.7318	13.2644	14.2044	0.33	0.346667

$$\begin{aligned} \text{COD reduction (\%)} = & 35.8 - 76.7 Z_1 + 10.07 Z_2 + 0.59 Z_3 + 0.018 Z_4 + 36.3 Z_1 * Z_1 - 0.46 Z_2 * Z_2 \\ & + 0.0030 Z_3 * Z_3 - 0.0001 Z_4 * Z_4 + 2.04 Z_1 * Z_2 + 0.12 Z_1 * Z_3 - 0.0017 Z_1 * Z_4 - 0.069 Z_2 * Z_3 \\ & - 0.0011 Z_2 * Z_4 - 0.0001 Z_3 * Z_4 \end{aligned} \quad (7)$$

$$\begin{aligned} \text{Energy consumption (kW h/m}^3\text{)} = & -31.3 + 10.7 Z_1 + 3.89 Z_2 + 0.213 Z_3 + 0.043 Z_4 + 1.5 Z_1 * Z_1 \\ & - 0.19 Z_2 * Z_2 + 0.0004 Z_3 * Z_3 + 0.00004 Z_4 * Z_4 + 0.40 Z_1 * Z_2 + 0.25 Z_1 * Z_3 - 0.023 Z_1 * Z_4 \\ & - 0.013 Z_2 * Z_3 - 0.004 Z_2 * Z_4 - 0.00045 Z_3 * Z_4 \end{aligned} \quad (8)$$

$$\begin{aligned} \text{Real Anode-Consumption (g)} = & -0.040 - 0.038 Z_1 + 0.007 Z_2 + 0.00013 Z_3 + 0.00017 Z_4 - 0.003 Z_1 * Z_1 \\ & - 0.001 Z_2 * Z_2 + 0.000002 Z_3 * Z_3 - 0.0001 Z_4 * Z_4 + 0.002 Z_1 * Z_2 + 0.007 Z_1 * Z_3 + 0.0002 Z_1 * Z_4 \\ & - 0.00002 Z_2 * Z_3 + 0.00001 Z_2 * Z_4 - 0.0001 Z_3 * Z_4 \end{aligned} \quad (9)$$

Table 2 lists all 27 experimental runs of CCD and the actual as well as the model-estimated results of the three response variables, i.e. COD-reduction (%), specific energy consumption (kWh/m³), and real anode consumption (g):

The design contains 16 factorial points (Runs 1-16), 8 star or axial points (Runs 17- 24), and 3 replicates of the center-point ($Z_1 = 1.0\text{A}$; $Z_2 = 6$; $Z_3 = 47\text{min}$; $Z_4 = 300\text{ppm}$; Runs 23, 26, and 27). This provides an opportunity to estimate independently both the pure experimental errors, and subsequently, the lack-of-fit. Data inspection shows clearly physically logical trends. COD-reductions varied between 62.53 % (Run 1: current (0.8 A); acidic pH (4); electrolysis time (26 min)) and 98.14 % (Run 22: Axial Point at longest possible electrolysis time (90 min)).

Electrolysis time (Z_3) had the largest positive effect on COD removal: At constant current density, pH and dye concentrations increasing the time from 26 to 69 minutes increased significantly COD removal (e.g. 62.53% in Run 1 versus 88.18% in Run 5). This can be explained through the larger charge loadings ($Q = It$) due to longer times, as well as larger quantities of Al^{3+} coagulant species and $\text{Al}(\text{OH})_3$ flocs generated in situ via Faradays Law, causing both adsorptive and sweeping-coagulation of the methylene blue-Congo red mixture. An increase in the applied current from 0.8 to 1.2 A also resulted in a better COD removal (e.g. Runs 3 vs. 4: 76.55% to 82.16%), since higher anodic dissolution rates were observed and a more dense micro-bubble formation was observed leading to a better electro-flotation of the destabilized-dye/floccules-aggregates. The trade-offs between removal efficiencies and costs related to operation can also be seen in **Table 2**. The specific energy consumptions varied over two orders of magnitude (1.65 kwh/m³ (run 21; $t = 4$ min.) and 29.20 kwh/m³ (run 22; $t = 90$ min.)), while the real anode consumptions varied from 0.04 g to 0.66 g between these two runs. Therefore, it is shown that the best conditions regarding the COD removal (large currents and large electrolysis times) correspond exactly with the highest energy demands and anodic losses, showing therefore clearly that a multi-response optimization has to be carried out instead of a one-response optimization. The very good correlation between the measured and calculated anodic losses based on Faraday's law during all experiments show that there is nearly no parasitic reaction at the aluminum-anode, thus giving rise to almost perfect current efficiencies.

The reproduction of this process is demonstrated by the three center-point replicates (runs 23, 26, 27) whose COD reductions (73.41%, 79.81%, and 82.34%) and energy consumptions (16.82, 15.14, and 13.26 kwh/m³) determine the pure error in the ANOVA.

Finally, the small differences between the actual and predicted values for all three responses (with usually less than 2-3% deviations for COD, less than 2 kwh/m³ for energy, and less than 0.01 g for mass loss) indicate qualitatively the suitability of the fitted quadratic models, which will be proven quantitatively in **Table 3**.

Figures 2, 3, and 4 indicate favorable concordance between the experimental and predicted values of COD reduction efficiency, energy consumption, and electrode consumption. **Table 3**, also revealed the high validity of the mathematical relations predicted based on ANOVA test and regression factors.

Diagnostic parity plots serve as a visual representation of whether a mathematical model is able to accurately and reliably estimate measured values. If the modeled value were equal to the observed value at all times, then all experimental points would fall directly on the 45° line of equality. Any deviation from the 45° line represents either an error in the modeling process or some form of bias in the estimates produced by the model.

The COD reduction plot shown in **Figure 2** illustrates that all 27 data points representing COD reduction fall closely and randomly about the diagonal for nearly all concentrations (60–100%), illustrating that there is little or no curvature present. Also illustrated in **Figure 2** is that most of the data points show very little skewness toward one side or the other of the diagonal indicating that both the independence of the residuals and their normality have been met. These two assumptions regarding residuals are critical to the proper use of least squares regression analysis. Additionally, these two residual characteristics help explain why the coefficient of determination (R^2) calculated from the statistical analysis was so close to unity (96.39%) and also explains why the lack of fit term was found to be insignificant, as discussed in **Table 3**.

Similarly, **Figure 3** illustrates a similar trend concerning the energy consumption model when considering a wide range of operating conditions (between 1.6 – 29 kW h/m³). The relatively straight-line relationship exhibited throughout nearly two orders of magnitude illustrate that the quadratic equation used to describe energy consumption has effectively captured the strong non-linear relationship between current and electrolysis time through the I-t-type ohmic loss component. No systematic under or over-prediction is evident throughout any part of the design space.

Lastly, **Figure 4** illustrates the near perfect overlap of the experimental anode consumption values with the identity line between 0.04 and 0.66 g. This near-perfect alignment visually illustrates that the coefficients of determination for each experiment ($R^2 = 99.73\%$) are extremely high and reflect that aluminum dissolution occurs via Faradaic processes and thus that the estimated model may be used with extreme confidence to predict electrode wear (and thus replaceable costs due to electrode degradation) at any combination of experimental parameters considered in this study.

Collectively, **Figures 2, 3, and 4** demonstrate that the RSM/CCD-based models constructed during this research project are statistically validated, unbiased and capable of providing reliable predictions. Such graphical validation is a necessary step prior to continuing with further work including but limited to constructing response surface equations to understand how each parameter influences each variable of interest, determining which variables interact significantly with each other based upon their individual contributions to each variable, and performing multi-objective optimizations using the previously generated response surfaces to optimize the electrocoagulation treatment of mixtures of methylene blue and Congo red dyes.

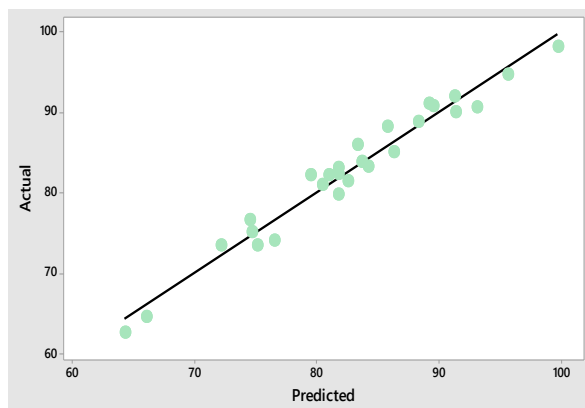


Figure 2. Observed and predicted values for COD reduction %.

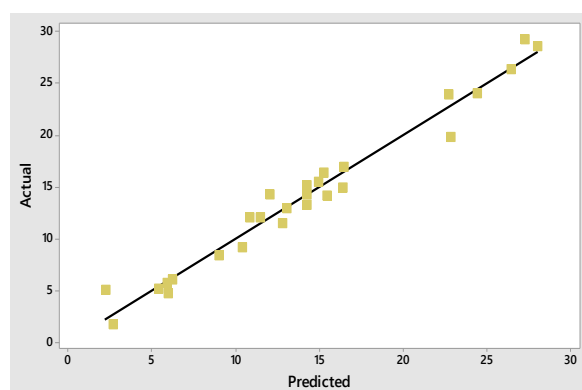


Figure 3. Observed and predicted values for energy consumption (kW h/m³).

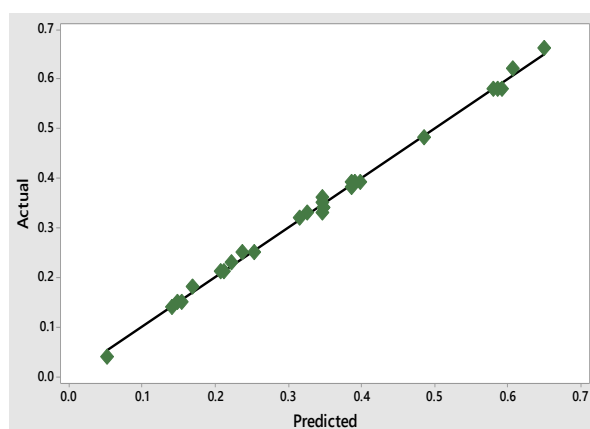


Figure 4. Observed and predicted values for real anode-consumption (g).

Table 3. ANOVA results for the studied responses.

Responses	Source	DF	Adj SS	Adj MS	F- Value	p- Value	Statuses
COD %	Model	14	1831.70	130.836	22.90	<0.0001	Highly significant
	Lack-of-Fit	10	62.55	6.255	2.08	0.368	Not significant
			R ² = 96.39%	R ² (adj)= 92.18%	R ² (pred)= 80.12%		
Energy consumption (kWh/m ³)	Model	14	1398.26	99.876	27.71	<0.0001	Highly significant
	Lack-of-Fit	10	41.49	4.149	4.70	0.188	Not significant
			R ² = 97.00%	R ² (adj)= 93.50%	R ² (pred)= 83.15%		
Real Anode-Consumption (g)	Model	14	0.67138	0.04796	315.33	<0.0001	Highly significant
	Lack-of-Fit	10	0.00136	0.00014	0.58	0.772	Not significant
			R ² = 99.73%	R ² (adj)= 99.41%	R ² (pred)= 98.68%		

Table 3 shows the results of the Analysis of Variance (ANOVA) for each of the three reduced quadratic models developed. In every case, the model F-value was high (COD% = 22.90, Energy Consumption = 27.71, Anode Consumption = 315.33) and the p-value was less than 0.0001. Since $p < .05$ defines statistical significance at a confidence interval of 95%, since these p-values were much lower ($.0001 < .0005$), the likelihood of obtaining such large F-Ratio values due to random chance is much less than .01%. Therefore, all three regression models are considered to be statistically significant and the selected independent variable(s) and their interactions/quadratic terms are truly explaining the measured variability of each response.

Just as importantly is the lack-of-fit test that determines how well the residual variation of the model fits the pure error determined from the replicated center points. The Lack-of-Fit F-values (2.08, 4.7, 0.58; Degrees

of Freedom: $n=10$) yielded p-values (0.368, 0.188 & 0.772 respectively) that are all greater than 0.05. Therefore, none of the lack-of-fit tests were statistically significant. When a lack-of-fit Test is statistically insignificant, it demonstrates that the second-order polynomial has successfully represented the true response surface across the experimental domain and the errors generated by the model are equivalent to the random errors associated with measuring each response.

Additionally, the goodness-of-Fit can also be expressed using coefficients of determination (R^2). The COD reduction model had an $R^2 = 96.39\%$, indicating that the model accounted for more than 96% of all variability in the data; whereas its adjusted R-Square ($R^2_{adj} = 92.18\%$) remained relatively similar to R^2 , demonstrating that adding additional terms did not result in over parameterization. Further, the predicted R-Squared (R^2_{pred}) of 80.12% was reasonably consistent with the Adjusted R-Squared Value (Difference < 0.20); collectively supporting reasonable prediction capabilities on new observations. The energy consumption model performed similarly ($R^2 = 97.00\%$; $R^2_{adj} = 93.50\%$; $R^2_{pred} = 83.15\%$) as did the anode consumption model ($R^2 = 99.73\%$; $R^2_{adj} = 99.41\%$; $R^2_{pred} = 98.68\%$), which performed exceptionally well given that anodic mass loss is governed nearly completely deterministically via Farady's Law through the charge passed, resulting in very small amounts of unexplained variability remaining. Overall, the ANOVA Results provide validation to utilize each of the three regression equations for Interpolation, Response Surface Analysis and Optimization within the experimental domains defined by **Table 1**.

5. Effects of the operating variables

5.1. Effect of solution pH

The pH of the wastewater is a critical parameter in EC processes. In the EC process, the pH is a problem because it determines the type of coagulant species that form in the solution (Can-Güven, 2021) (Singh & Ransingh, 2020). For acidic solutions of pH lower than 4, $Al(III)$ ions are formed, which have a moderate effect on the coagulant process. However, for pH levels ranging from 4 to 8, species with a high positive charge will be generated, such as $Al(OH)_2^+$, and $Al(OH)_3$. These positively charged species cause adsorption electrochemistry natural reaction. On the other hand, the effect of coagulants vanishes by forming $Al(OH)_4^-$ at a pH higher than 8. It has been noted that the dissolving rate of Al at pH larger than 3 is higher than at pH less than 3, although Al electrolysis is carried out with a constant specific volumetric loading charge. When the pH surrounding cathodic electrodes increases to a point where aluminum electrodes can dissolve chemically and aluminate anion are nearly generated (Liu et al., 2010).

5.2. Effect of electrolysis time

The efficiency of pollutant removal is primarily contingent on the electrocoagulation time. A higher rate of pollutant removal is achieved with longer times because of the generation of more metal flocs and coagulants at a constant current density. Nonetheless, the electrocoagulation effectiveness reaches a plateau at certain time when the pollutant removal rates stabilize due to the saturation of active sites forming coagulant pollutant flocs. The optimization of electrocoagulation time is essential since the pollutant removal rate is influenced by electrolysis time to prevent loss of resources and energy (Tahreen et al., 2020).

5.3. Effect of current

Current intensity is a crucial characteristic in EC, as it determines the size and growth of the flocs, coagulant dosage rate, and bubble production rate, which can influence the efficiency of the EC. The anode dissolution rate increases with an increase in the current intensity. This results in an augmentation of metal hydroxide flocs, hence enhancing pollution removal efficiency. An elevation in current density beyond the optimal level does not enhance pollutant removal effectiveness, as enough quantity of metal hydroxide flocks is present for pollutant sedimentation (Khandegar & Saroha, 2013). **Figure 5** depicts the influence of each

variable and the interactions among all variables on the COD reduction response. As observed, pH levels of 4, 6, and 8 are parallel and shows that while increasing the pH improves COD reduction, the improvement rate remains consistent with the current applied within the current 0.5 to 1.5 A, but there is a slight upward trend at higher currents, due to the increase in hydroxyl radical prediction via electrolysis. Moreover, the interaction effect of current (A) and electrolysis time confirms that electrolysis time is a dominant factor.

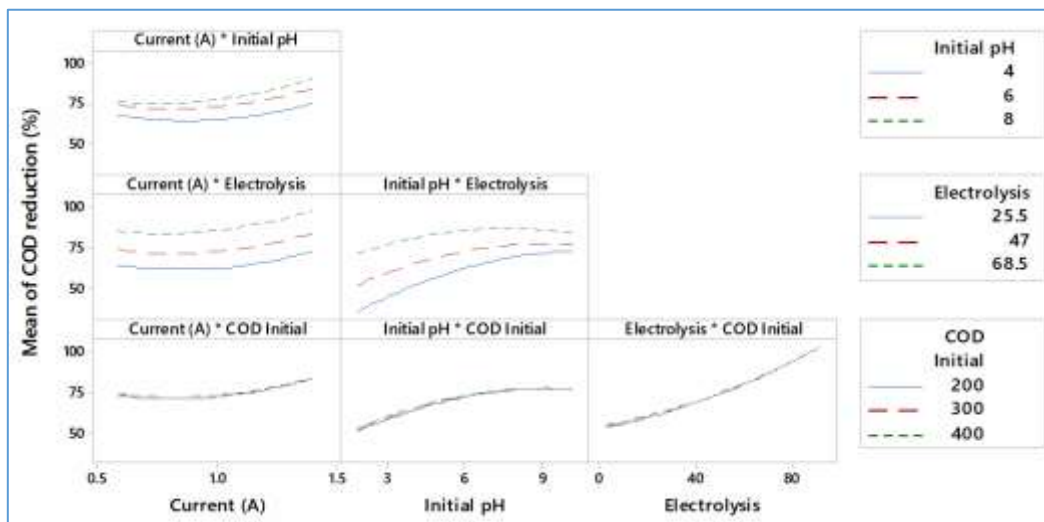


Figure 5. Effect of each operating variable and interaction among all variables on COD reduction.

The relatively constant vertical spacing suggests that increasing the current provides a steady boost to the reduction efficiency across all time intervals, likely by maintaining a higher current density for the electrical oxidation of organic matter. However, there is notable divergence for the interaction between pH and electrolysis time. At low electrolysis times, the gap between pH 4 and pH 8 is significant; as time increases, the lines tend to converge slightly. This indicates a synergistic interaction between high pH levels and alkaline conditions, which tend to accelerate the kinetics of COD reduction early on. At longer electrolysis times, even the lower pH settings begin to catch up, suggesting that time can partially compensate for less-than-optimal pH levels.

The effects of each variable and the interactions among all variables on the energy consumption response are shown in **Figure 6**. For the interaction between current intensity and electrolysis time, the slopes of the lines for different electrolysis times (25.5, 47, and 68.5) differ noticeably as current increases at low current (0.5 A). The energy gap between a short and long process is relatively small at high current (1.5 A). The gap widens significantly, which suggests a synergistic effect: the energy penalty for increasing electrolysis time is much harsher when operating at high current. The interaction between initial pH and electrolysis time exhibits a slight energy hump. Energy consumption appears slightly higher at neutral pH ~6 compared to extreme acidic or basic conditions. This is often because of changes in solution conductivity; very low or high pH levels typically have higher ion concentrations of H^+ or OH^- , which reduces the electrical resistance of the solution and can slightly lower the voltage required. However, for current and initial pH interactions, there is no significant crossing or divergence in these two factors that shows that they act independently on the response.

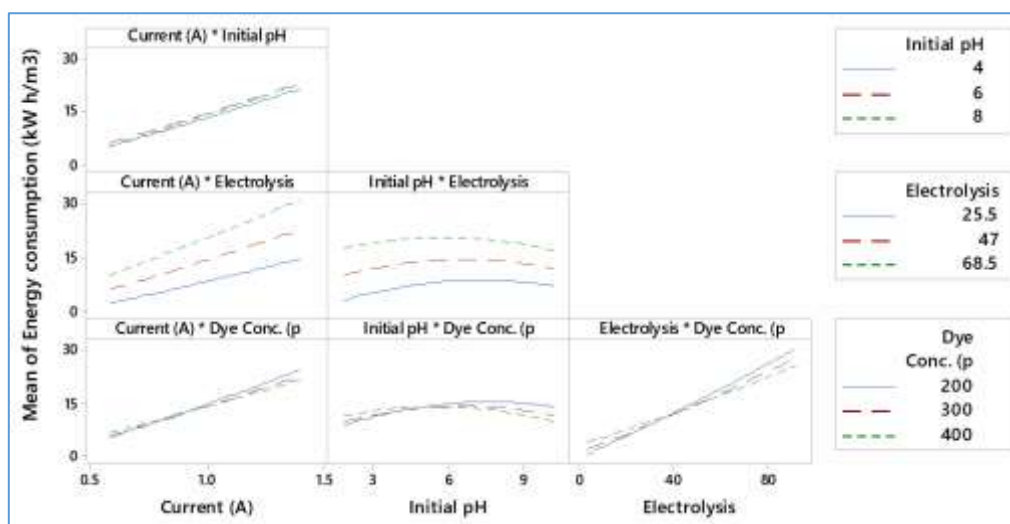


Figure 6. Effect of each operating variable and interaction among all variables on energy consumption.

The real anode consumption response's variable effects and interactions appear in **Figure 7**. As depicted, the current-electrolysis time interaction is the strongest effect; the diverging lines become much steeper as the electrolysis time increases. High current combined with a high electrolysis time system led to the maximum anode degradation, which is expected as both variables contribute to the total coulometric load on the system.

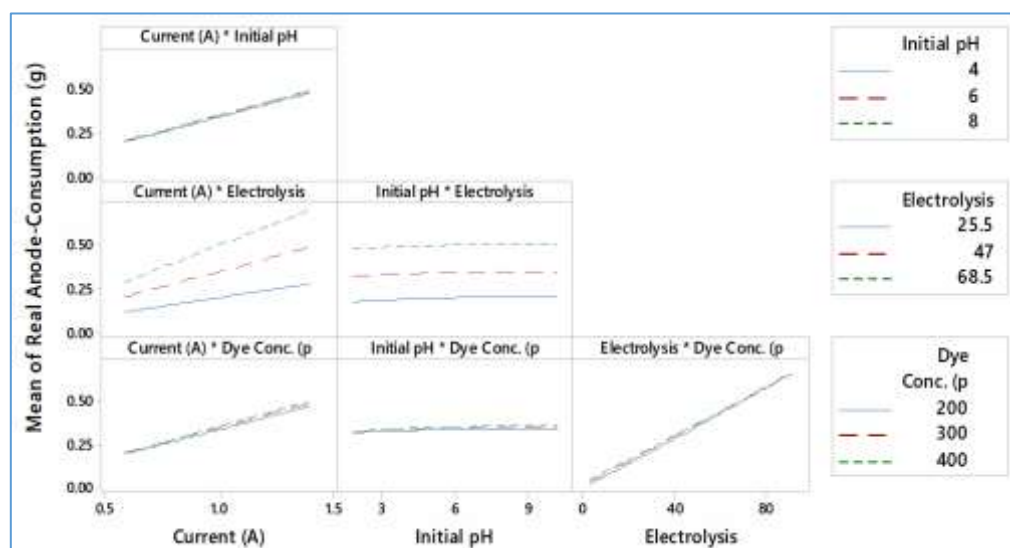


Figure 7. Effect of each operating variable and interaction among all variables on real anode consumption.

The behavior of the initial pH-electrolysis time horizontal nature suggests that initial pH is a weak factor in this specific electrochemical process; whether the pH is 4, 6, or 8, the anode consumption doesn't fluctuate significantly. This is common in electrocoagulation systems, where the electrical energy dominates that chemical environment. While the current-initial pH interaction had no significant crossing or divergence, they act independently on the response.

The influence of all operating variables on the open circuit potential (ΔOCP) response and interactions is presented in **Figure 8**. In this work, the tracking of the OCP change is a reliable, efficient, real-time oxidation diagnostic tool to observe the thermodynamic changes of the electrode surface ability to oxidize during treatment. To achieve this goal, the DC was shortly paused for few seconds, to prevent the IR ohmic drop across the solution during EC treatments, which provide the ability to record the potential relative to the SCE. The track of this relaxing potential allows us to have an insight on the electrode thermodynamic ability to

oxidize and surface transferring during the changes that occur in the solution as a result of dyes continuous removal, which affect the electrode surface, the solution acidity, consequently this will impact of electrode potential. It is well known that the change of OCP in the positive direction indicate that the activity of the anode will be suppressed due to the formation that will isolate the anode from solution, while the increase the OCP in the active direction will make the anode more active for the dissolution of ions (Dhongde et al., 2024). For current-initial pH interaction behavior, at lower currents (0.5 to 1.0 A), higher pH values (pH 8) result in a higher open circuit potential compared to lower pH values which suggest the formation of $\text{Al}(\text{OH})_3$ on the surface that can't be broken by low current. However, as the current increases toward 1.5 A, the lines converge, suggesting that the influence of pH diminishes at higher current densities due the degradation of the formed hydroxide that causes the transition to the trans-passive state and liberation of anions. While the behavior of both current-electrolysis time and current-dye concentration suggests that the impact of current on the open circuit potential is fairly consistent regardless of the electrolysis time of the initial dye concentration. The interaction of initial pH-electrolysis time indicates weak interaction; the rate at which ΔOCP increases with pH is not significantly altered by the duration of electrolysis. For the initial pH-dye concentration interaction, the response to the pH is consistent across different concentrations, showing a sharp increase in ΔOCP of the solution as it moves from acidic to alkaline conditions. While the interaction between electrolysis time and dye concentration shows intersecting lines at higher electrolysis times. At shorter durations, the 300-ppm dye concentration yields a higher ΔOCP , but as electrolysis time exceeds 60 min, the 200-ppm concentration, the solid blue line, becomes more dominant. This suggests a competitive or limiting kinetic interaction between the treatment time and the organic load. As overall view, the complicated interaction of the studied variables on the OCP reflect the complex effect of pH, dyes concentration, current, and electrolysis time, that all affect the transition from active to passive and trans-passive state of metal.

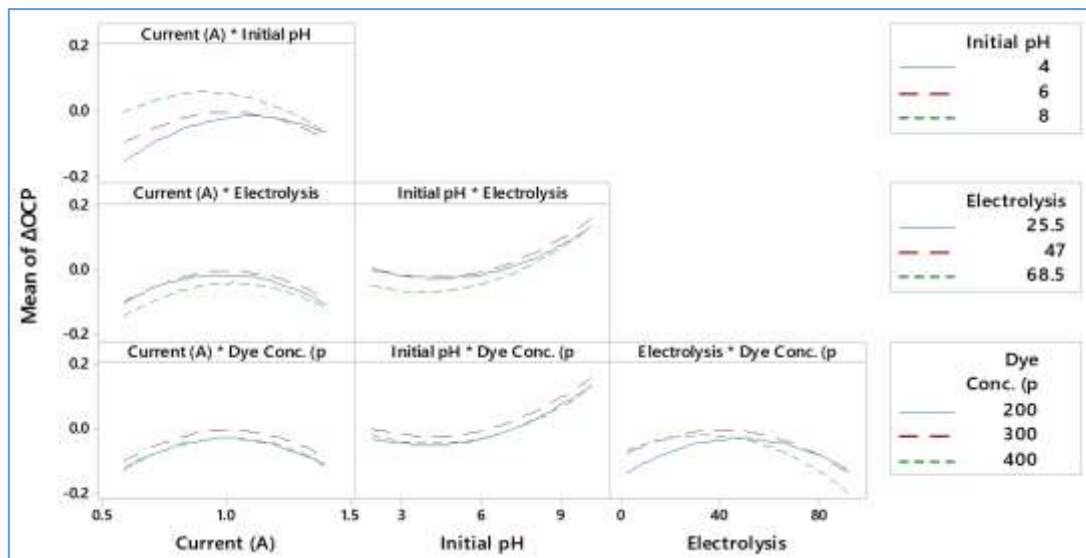


Figure 8. Effect of each operating variable and interaction among all variables on ΔOCP response.

As observed in **Figure 5**, the COD reduction efficiency improves with increasing current density and electrolysis time. This behavior occurs because higher current density promotes the generation of a coagulant species and oxidizing agents responsible for pollutant removal; this agrees with (Arbabi et al., 2022). While **Figure 6** shows that energy consumption increases significantly with an increase in current on the treatment time, it was expected because electrical energy input increases as more charge passes through the system; this behavior agrees with (Yakameran et al., 2025). The outcomes conducted from **Figure 7** indicate that increasing current density and electrolysis time parameters leads to higher electrode dissolution. This behavior is consistent with Faraday's law (electron dissolution is proportional to the charge passed through the system).

The amount of energy as well as the aluminum consumed increases steadily with the treatment duration (Naji et al., 2020). **Figure 8** shows that the change in the OCP is complex in experimental domain, due to the change of metal ability to oxidize, in which the negative change enhances anodic activity while the positive change signifies reduction of oxidation. Results show that the COD reduction efficiency improved as the electrolysis time and current intensity increased. But the electrical energy consumption increases, and the real anode consumption increases as well.

5.4. Statistical optimization of operating variables

For any electrochemical process to achieve the highest reduction of pollutants and lowest consumption of energy and electrodes, the optimization of system operating parameters is essential to be achieved by identifying numerous criteria to enhance the process, ultimately reaching the objective through optimizing of the desired function (DF) by detecting the importance or weight (Hameed & Salman, 2024). Minitab statistical software performed the optimization of operating variables. Five cases of optimization were separately performed for the studied responses. First, a complete COD reduction achieved at: Current = 1.4 A, Initial pH = 2.2, Electrolysis time = 90 min. The second case explains that the optimal conditions that achieved the lowest consumption of energy and electrode, as follows: Current = 0.6 A, Initial pH = 2, Electrolysis time = 4 min. While the third case depicts that the optimum conditions that achieved the highest COD reduction with the lowest electrode consumption, as follows: Current = 0.6 A, Initial pH = 6.8, Electrolysis time = 4 min. However, the fourth case shows that the optimal conditions that achieved maximum COD removal with the lowest energy consumption were: Current = 0.6 A, Initial pH = 8.1, Electrolysis time = 4 min. Finally, the fifth case shows the optimal conditions conducted that achieved maximum COD removal with the lowest energy and electrode consumption, and they were as follows: Current = 0.6 A, Initial pH = 7.98, electrolysis time = 4 min.

All five cases were applied to a mixture of Congo red and methylene blue dyes. The experimental results of the statistical optimization process experiment are as follows in **Table 4**.

Table 4. The statistical optimization process experiments results.

NO.	Energy consumption (kWh/m ³)	Theo. Anode Consumption (g)	Real Anode Consumption (g)	COD Reduction %
1	41.44	0.705	0.8	99.00
2	0.256	0.013	0.04	52.38
3	0.619	0.013	0.03	88.20
4	0.571	0.013	0.03	87.00
5	0.629	0.013	0.04	88.88

6. Characterization of anode in the five-cases

6.1. XRD analysis

Figure 9 represents the X-ray diffraction (XRD) analysis, which is widely used to identify crystalline phases formed during the EC process, using Al electrode dissolution of the anode to generate aluminum hydroxide that acts as coagulants and adsorbs dye molecules. Released Al ions hydrolyze in water to form Al hydroxide flocs capable of adsorbing organic pollutants such as dyes (Can et al., 2003).

All five diffraction patterns contain sharp peaks at the same locations ($2\theta \approx 44.7^\circ$, 65.1°) plus weaker peaks located at $2\theta \approx 38.5^\circ$, 78.2° , and 99.1° in three of the five patterns. These locations correspond to the (200), (220), (111), (311), and (400) planes of the body-centered cubic metal aluminum, space group Fm-3m, $a = 4.0496\text{\AA}$ (Cooper, 1962). Therefore, it appears that there were no additional crystalline phases formed on the anode substrates (i.e. the aluminum anode retained its metallic composition through the duration of the treatment).

In contrast to a randomly oriented aluminum powder where the (111) peak is the strongest reflection, the (220) and (200) peaks dominate in each pattern. A strong (111) peak may have appeared but did not appear. This behavior is indicative of the crystallographic texture associated with rolled aluminum sheets. Aluminum sheet that has undergone cold rolling develops a number of preferred orientations in addition to those developed by recrystallization. For example, a flat aluminum plate will primarily diffract from those planes of a unit cell that lie parallel to its surface due to the constraint of the plate's geometry (Hirsch & Locke, 1988); (Cullity & Stock, 2001). Thus, when different patterns exhibited differing ratios of (200) / (220) peak heights (for example, (200) dominant in Case 3 versus almost entirely (220) in Case 4), these differences reflected different textures on the surfaces of the plates exposed during electrocoagulation Prakash Pal & Sen, n.d.; (Zhen et al., 2014 and not the presence of any new phases (Mollah et al., 2004).

No crystalline peaks corresponding to either aluminum oxides or hydroxides, elemental sulfur or dye were observed. In electrocoagulation, the anode is consumed electrochemically as Al^{3+} and $\text{Al}(\text{OH})_3$ forms in the bulk solution rather than being deposited onto the surface of the anode (Sugai et al., 2023); however, the thin film formed on the anode surface is only an amorphous layer of native oxide approximately a few nm thick, which is below the sensitivity of laboratory XRD (Mollah et al., 2004); (Hakizimana et al., 2017). Similarly, $\alpha\text{-S}_8$ would produce its most intense peaks at 2θ values of approximately $23.0 - 31.3^\circ$ (Warren & Burwell, 1935); these regions are unoccupied in all five patterns. The sulfur present in the dye molecules is found only in sulfonated groups ($-\text{SO}_3^-$) that are chemically bonded into the organic matrix and desorb along with the adsorbed dye anions in a noncrystalline molecular state rather than forming crystalline compounds containing sulfur (Villanueva et al., 2022); (Chafi et al., 2011); (Moussa et al., 2017).

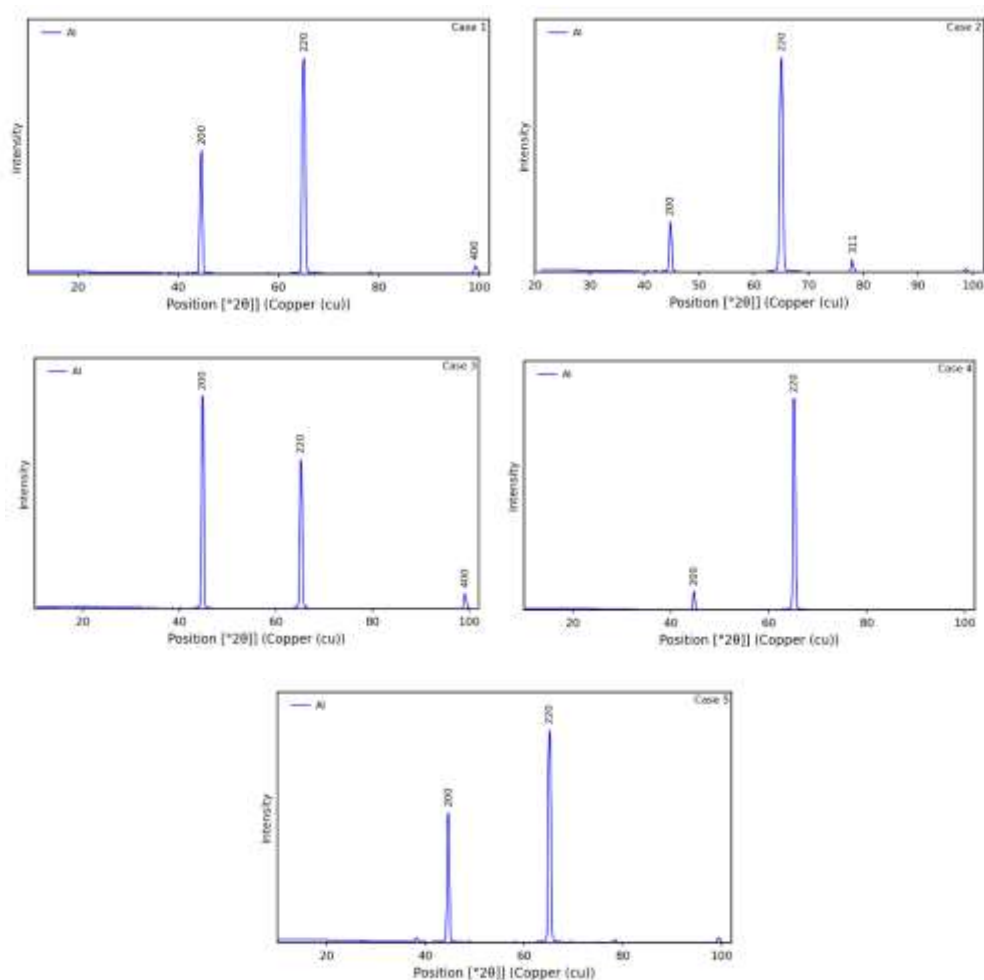
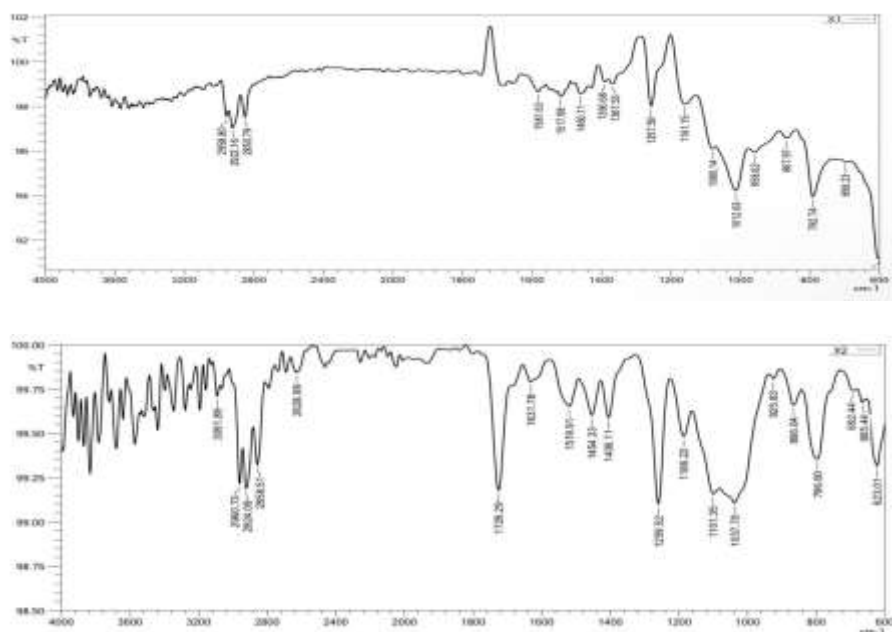


Figure 9. Results of XRD test for five cases.

6.2. FTIR analysis

Figure 10 depicts FTIR spectra analysis of five cases from X1 to X5. The spectra were recorded in the region of 4000-600 cm^{-1} . To identify the functional groups associated with aluminum hydroxide flocs and the adsorbed dye molecules. Absorption bands observed in the 2800-3000 cm^{-1} region is attributed to the aliphatic C-H stretching vibrations of the $-\text{CH}_2$ and $-\text{CH}_3$ groups present in the dye molecule structure. These peaks confirm the presence of organic dye molecules attached to the aluminum hydroxide flocs formed during the EC process. The strong bands observed in the 1700-1600 cm^{-1} region correspond to the $\text{C}=\text{O}$ stretching and aromatic $\text{C}=\text{C}$ vibrations (Gudić et al., 2025). These peaks are mainly associated with the aromatic structures present in MB and CR dyes. Additionally, the peak around 1630-1660 cm^{-1} may also correspond to the bending vibration of adsorbed water molecules on the surface of aluminum hydroxide.

The presence of these peaks indicates the interaction between molecules on the aluminum-based coagulant species generated during the EC. In the 1580 -1450 cm^{-1} region, several peaks appearing can be attributed to the aromatic skeletal vibrations and C-N stretching modes associated with the aromatic amine group's presence in the structure of the dye. These bonds further confirm the adsorption of dye molecules on the aluminum hydroxide flocs. 1360 to 1260 cm^{-1} absorption bands are mainly assigned to $\text{S}=\text{O}$ stretching vibrations of sulfonate groups $-\text{SO}_3^-$ (Gudić et al., 2025). This functional group is characteristic of CR dye, and its presence in the FTIR spectra indicates that the aluminum hydroxide precipitate formed during EC captured the dye molecules. Peaks observed in the 1180-1000 cm^{-1} region corresponded to the C-O (Tibebe et al., 2022) and C-N stretching vibrations of organic functional groups, which may also include contributions from Al-O stretching vibrations related to the aluminum hydroxide or aluminum oxide species generated during the EC process. lower wave NO. region 9900 to 600 cm^{-1} , several bands are attributed to aromatic C-H out-of-plane bending vibrations and Al-O or Al-O-H vibrations (Tibebe et al., 2022), indicating the formation of an aluminum hydroxide structure that acts as the primary coagulant responsible for the removal of dye. FTIR spectra confirmed the presence of hydroxyl groups and aluminum hydroxide species formed during EC, which play an important role in the adsorption mechanism of dye (Negash et al., 2023).



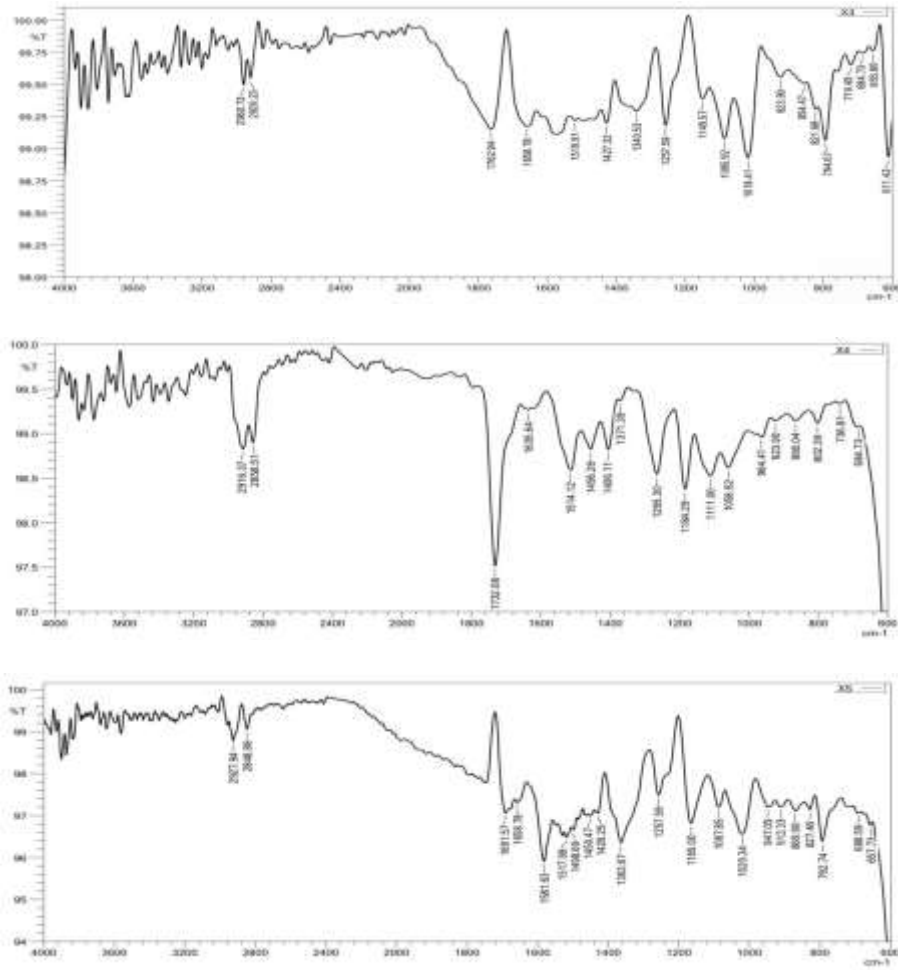


Figure 10. Results of FTIR test for five-cases.

6.3. FESEM analysis

The performance of any electrode can be concluded from its morphological structure. In **Figure 11**, Case 1 exhibits the most significant surface degradation. At a high current of 1.4 A and a prolonged electrolysis time of 90 minutes, the aluminum surface shows deep pitting corrosion and a highly porous, sponge-like structure. The acidic environment promotes the rapid dissolution of the Al^{3+} ions. The high current density accelerates electron transfer, leading to a honeycomb morphology. This indicates a high rate of coagulant generation, though at $\text{pH} = 2.24$, the monomeric $[\text{Al}(\text{H}_2\text{O})_6]^{3+}$ species predominate, which are less effective for coagulation than the polymeric hydroxides formed at neutral pH.

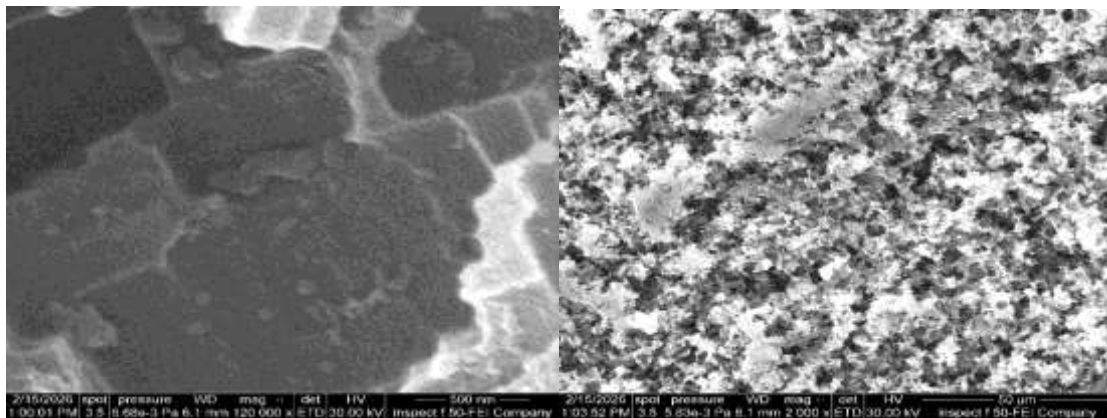


Figure 11. FESEM test result for case 1.

Compared to Case 1, **Figure 12** Case 2 shows a much smoother surface with minor jagged edges; the short duration (4 min) and lower current (0.6 A) result in limited material loss and more shallow pitting electrode corrosion. The surface appears relatively clean, suggesting that at pH = 2, the aluminum ions dissolve directly into the solution rather than forming a thick hydroxide precipitate layer on the electrode surface.

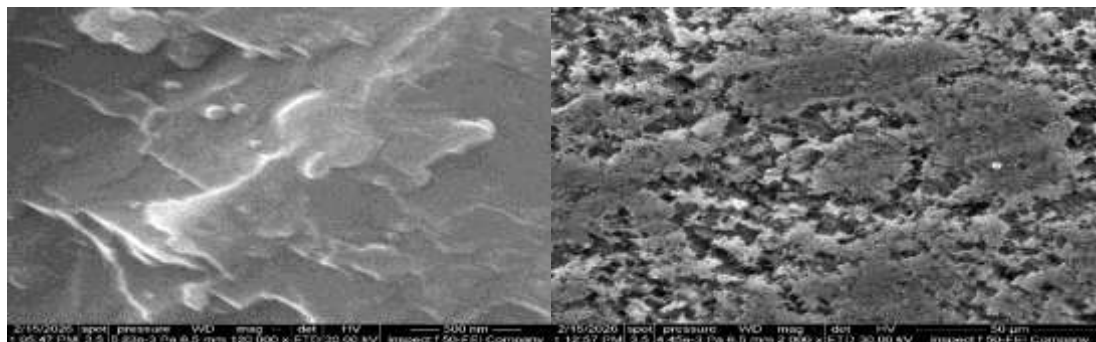


Figure 12. FESEM test result for case 2.

In **Figure 13**, the morphology shifts toward a more textured, "flaky" appearance. As the pH increases toward 6.8, the solubility of aluminum decreases. This leads to the initial formation of $\text{Al}(\text{OH})_3$ precipitates on the anode surface. These precipitates appear as small, irregular clusters. This pH range is often optimal for the removal of Congo Red and Methylene Blue because of the presence of positively charged amorphous hydroxides that facilitate charge neutralization.

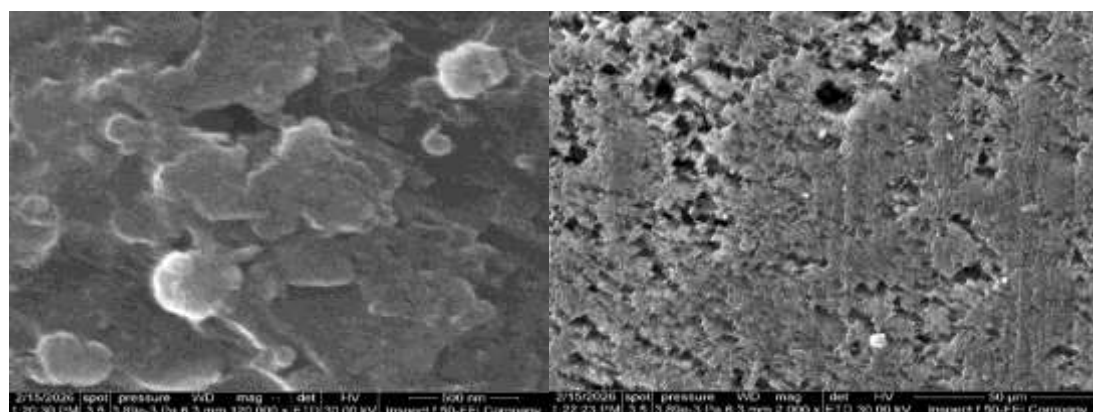


Figure 13. FESEM test result for case 3.

Figures 14 and 15 (Case 4 and Case 5) show the development of a dense, cauliflower-like coating over the aluminum grains. At pH = 8.14 and 7.9, the formation of solid $\text{Al}(\text{OH})_3$ is most favorable. The FESEM images reveal a thick layer of "flocs" adhering to the anode. While this provides a high surface area for the adsorption of dye molecules (especially the anionic Congo Red), an overly dense layer can lead to anode passivation. This layer increases the ohmic resistance of the cell, potentially decreasing the efficiency of the electrocoagulation process over time.

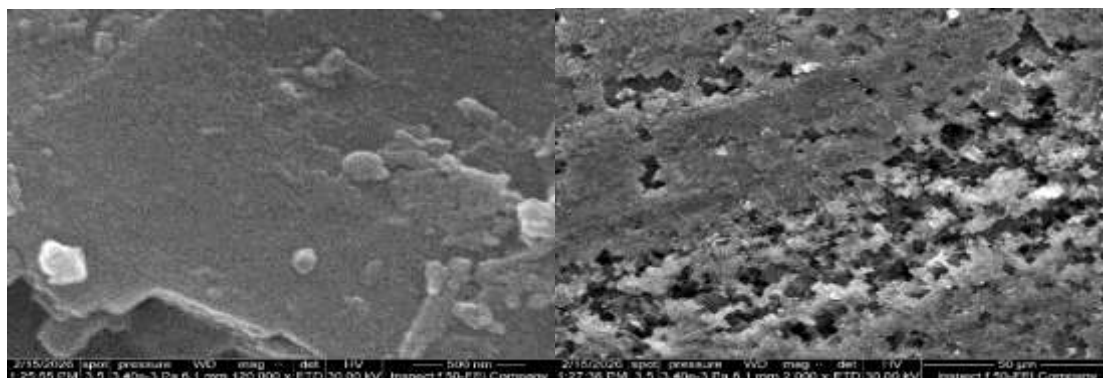


Figure 14. FESEM test result for case 4.

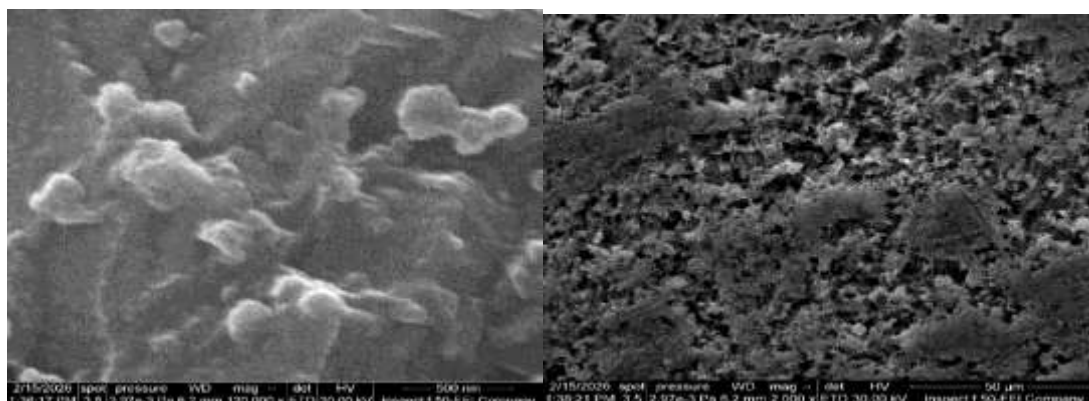


Figure 15. FESEM test result for case 5.

The similarity between Cases 4 and 5 confirms the reproducibility of the surface coating under alkaline conditions.

7. Conclusions

This study reveals the feasibility of the usage of transient current-interruption method to assess the electrode OCP throughout the EC using a batch reactor (BER) with aluminum (Al)-iron (Fe) electrodes. This study experimentally examined the treatment of simulated wastewater containing a mixture of cation and anion dyes while taking into account a number of variables, including pH (2–10), current (0.6–1.4 A), dye concentration (100–500 ppm), and electrolysis time (4–90 min). The tests have been designed, and the current operational parameters have been optimized using the surface response design technique based on the central composite design (RSM-CCD). The analysis of variance (ANOVA) was used to forecast the COD reduction, electrode consumption, the energy consumption, and the OCP under the specified parameters. The optimization procedure was used to classify the ideal circumstances needed to attain the greatest COD reduction and the lowest actual energy and anode electrode usage. The ideal circumstances of 0.6 A, pH=7.98, 4 min, and the highest beginning COD of 4236.7 ppm resulted in the greatest predicted reduction of COD (94.57%) vs. real value of 88.9% accomplished with the lowest energy and anode electrode usage. The study demonstrated that various dyes founded in wastewater may have an impact on the electrocoagulation process's efficiency and its optimization results. Furthermore, from scientific point of view, the experimental approach for real-time of the OCP of the working anode electrode give an insight on the thermodynamic condition of the surface changing through passivation and oxidation as it changes with variation of the organic condition of the solution during EC process. It is clear that there is a complex interactive manner with the studied variables, reflecting its complex thermodynamic ability.

Author contribution

Fatima H. Mohammed: Data curation, Investigation, Writing- Original draft preparation. Shaymaa A. Ahmed: Visualization, Supervision. Forat Yasir AlJaberi: Conceptualization, Methodology, Software, Supervision.

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Data availability

No datasets were generated or analysed during the current study.

Conflict of interest

The authors have no competing interests to declare that are relevant to the content of this article.

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