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Evaluation of Electrocoagulation Process for Hexavalent chromium Removal from Artificial Wastewater Using Box-Behnken Design

Ahmed Sagban Khudier[1], **Mohammed Jabbar Mawat**[2] **and Ammar Salman Dawood**[3]

[1-3] Department of Civil Engineering, College of Engineering, University of Basrah, Basrah, Iraq.

[1]E-mail: ahmed.khudier@uobasrah.edu.iq
[2]E-mail: mohammed.mawat@uobasrah.edu.iq
[3]E-mail: ammar.dawood@uobasrah.edu.iq

Abstract. The proliferation of heavy metals in industrial effluents poses a significant threat to the environment and public health. Hexavalent chromium (Cr(VI)); is a particularly hazardous contaminant because of its high toxicity, mobility, and carcinogenicity. This study aims to determine the effectiveness of electrocoagulation (EC) process in removing Cr(VI) from synthetic wastewater. A Box-Behnken design (BBD) involving three factors and three levels along with Response Surface Methodology (RSM) was utilized to study and optimize the key operating parameters: initial pH (3.64-10.36), applied voltage (5.23-28.77 V), and electrolysis time (8–29.45 min). From the experimental results, it is apparent that EC is an effective approach for Cr(VI) remediation, the rate of which varies from 21.3% to 92.6%. The results of the statistical analysis showed that voltage and time were strongly positively correlated with removal efficiency, while pH showed of more complex but non-linear influence. The highest removal efficiency of 92.6% was achieved PH of 5, voltage of 24 V, and time of 24 min. An additional one-way analysis of variance (ANOVA) showed that the quadratic model developed was significant. Thus, it provides a good estimate of the removal of Cr(VI). This research highlights the interaction of the parameters, especially the synergism between voltage and time, which is essential for the increase in process efficiency. These results demonstrate the possibility of BBD-optimized electrocoagulation to operate durably and effectively in treating Cr(VI) contaminated wastewater.

Keywords. Hexavalent chromium, Cr (VI), Box-Behnken Design (BBD), Wastewater, Electrocoagulation.

1. Introduction

Industrialization has led to unprecedented economic growth but has also resulted in the widespread contamination of water resources with toxic pollutants [1]. Heavy metals are the most relevant because they do not decompose naturally in nature; and are found in nature for a long time, providing a chance for organisms to accumulate in their bodies, which are the primary sources of exposure to the environment and health [2-3]. Chromium in the form of hexavalent; Cr(VI); is the most dangerous heavy metal ion released by a number of industries, such as leather tanning, electroplating, dyeing textiles, wood preservation, and pigment production. In contrast to its less toxic form, trivalent Cr(III), which is a necessary micronutrient [4], Cr(VI) is completely dissolved in water, permeates soil and water systems, and is a recognized carcinogenic, mutagenic, and teratogenic component [5-6]. The U.S. Environmental Protection Agency (EPA) has labeled Cr(VI) a priority pollutant and has enacted a strict MCL of 0.1 mg/L which is the maximum contaminant level set for total chromium in drinking water [7]. Therefore, a crucial environmental engineering problem is the creation of effective and sustainable technologies for Cr(VI) removal from industrial wastewater.

Different traditional techniques have been used for the treatment of Cr(VI)-contaminated wastewater. These methods include chemical precipitation [8], ion exchange [9], adsorption [10], and membrane filtration [11]. Chemical precipitation, normally involving the reduction of Cr(VI) to Cr(III), followed by precipitation as chromium hydroxide ($Cr(OH)_3$) at an alkaline pH, is the most widely used method because of its ease and low cost. Nevertheless, this method creates huge quantities of hazardous sludge that undergo separate treatment and disposal, and sometimes it can be inefficient owing to the strict limits of the discharge [12]. Ion exchange is a process characterized by high performance and metal recovery options; however, its efficiency is negatively affected by the presence of other ions in the wastewater, and the cost of synthetic resins and regeneration chemicals are the main reasons for its high cost. Although the use of materials such as activated carbon in adsorption has been demonstrated to be successful, the prices of adsorbents and the problems faced in the process of their reactivation inhibit their large-scale utilization. Membrane filtration systems such as reverse osmosis and nanofiltration can not only can achieve a very high level of separation, but can also be subjected to membrane fouling, which leads to the need for a higher pressure of operation; thus, the total energy consumed and operational costs increase significantly [13]. These shortcomings have created the need to identify more effective, economical, and environmentally safe modes of treatment.

Electrocoagulation (EC) is an excellent application of a versatile electrochemical process for treating organic, inorganic, and biological pollutants; such as heavy metals, organic dyes, oils, and suspended solids [14-15]. The EC procedure is characterized by the in-situ formation of coagulants through electrolytic dissolution of sacrificial anodes, usually composed of iron (Fe) or aluminum (Al) [16]. Additionally, this method contributes to environmental sustainability as it does not require the addition of chemical coagulants and consequently; reduces secondary pollution and sludge production. When iron electrodes are used, the primary reactions are as follows [17].

At the Anode (Oxidation): $Fe(s) \rightarrow Fe^{2+}(aq) + 2e^-$

At the Cathode (reduction): $2H_2O(l) + 2e^- \rightarrow H_2(g) + 2OH^-(aq)$.

The generated Fe2+ ions can then be oxidized to Fe3+ by dissolved oxygen or at the anode. These ions subsequently react with the OH- ions produced at the cathode to form various monomeric and polymeric iron hydroxide species (e.g., $Fe(OH)_2$, $Fe(OH)_3$), which act as the coagulating agents [18]. The removal of Cr(VI) in an EC system with iron electrodes occurs via a dual mechanism:

- Direct Cathodic Reduction: Cr(VI) species (e.g., CrO_4^{2-}, $HCrO_4^-$) are directly reduced to the less toxic Cr(III) at the cathode surface [19].
- Indirect Chemical Reduction and Co-precipitation: The Fe^{2+} ions generated at the anode are powerful reducing agents that can chemically reduce Cr(VI) to Cr(III). The newly formed Cr(III) then co-precipitates with iron hydroxides ($Fe(OH)_3$) and is removed from the solution as part of the sludge [20].

To maximize the efficiency and cost-effectiveness of the EC process, it is essential to optimize its operating parameters. The traditional one-factor-at-a-time (OFAT) approach is laborious, time-consuming, and fails to capture interactive effects between variables [21]. Response Surface Methodology (RSM) is a powerful collection of statistical and mathematical techniques used for modeling and analyzing problems in which the response of interest is influenced by several variables. RSM helps to optimize the process by quantifying the relationship between the independent variables and the response.

Among the diverse RSM designs, the Box-Behnken design (BBD) is recognized as the most effective scheme for fitting second-order polynomial model [22-23].The BBD is a spherical, rotatable, or nearly rotatable quadratic design that does not contain an embedded factorial or fractional factorial design. The BBD does not have axial points, which are quite different from central composite designs (CCD).

Thus, the experimental points were not at the extreme corners of the design space [24]. In addition, this is an advantage in preventing experiments with extreme conditions that might not be feasible or may lead to unsatisfactory results. For three or four variables, BBD requires fewer experimental runs than CCD, which makes it a more economical and efficient choice for process optimization.

Although a number of studies have demonstrated the efficacy of EC for Cr(VI) elimination, there is a need for a systematic evaluation of the interactive effects of key parameters by employing a robust statistical design for practical applications. The goal of this study was to achieve this by introducing the Box-Behnken design to model and optimize the EC process to remove Cr(VI) from synthetic wastewater. In particular, the objectives of this experiment were to investigate the individual and interactive effects of three key process variables, the initial pH, applied voltage (V), and electrolysis time (T), on the removal efficiency of Cr(VI), to develop a second-order polynomial model using RSM to express the relationship between the variables and the response (Cr(VI) removal percentage), to find the optimal operating conditions to maximize Cr(VI) removal efficiency, to validate the statistical model, and to analyze the significance of each factor through Analysis of Variance (ANOVA).

2. Materials and Methods

2.1. Preparation of Artificial Wastewater

Synthetic wastewater was prepared for all the experiments to ensure consistency and eliminate interference from other contaminants. A stock solution of Cr(VI) (1000 mg/L) was prepared by dissolving a predetermined amount of potassium dichromate ($K_2Cr_2O_7$, analytical grade) in deionized water. This stock solution was diluted to the desired initial concentration (50 mg/L) for each experimental run. The initial pH of the solution was adjusted to the required level as per the experimental design using either 0.1 M HCl or 0.1 M NaOH. Sodium chloride (NaCl) was added as a supporting electrolyte to maintain sufficient conductivity in the solution, which is a common practice in EC studies to reduce the cell voltage and energy consumption.

2.2. Experimental Setup

The electrocoagulation experiments were conducted in batch mode using a 1-liter glass beaker as the electrochemical reactor (Figure 1). The reactor contained 800 mL synthetic wastewater for each run. Two parallel iron plates with dimensions of 12 cm × 5 cm × 0.2 cm were used as the sacrificial anode and cathode. The electrodes were submerged in the solution at an inter-electrode distance of 1.5 cm. Before each experiment, the electrodes were mechanically polished with sandpaper, washed with acetone to remove any grease, and rinsed thoroughly with deionized water to ensure a clean and active surface. The electrodes were connected to a DC power supply unit capable of providing constant voltage. A magnetic stirrer was placed at the bottom of the beaker and operated at a constant speed (200 rpm) to ensure the homogeneity of the solution during electrolysis.

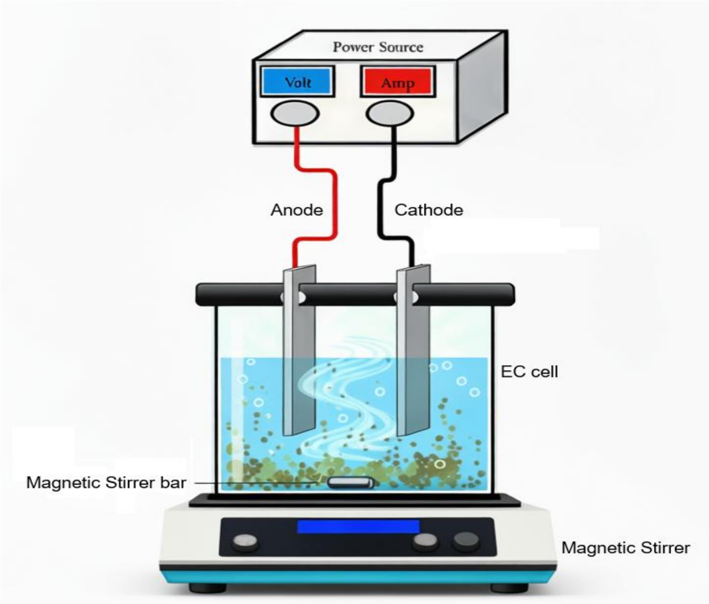

Figure 1. Schematic diagram of the batch electrocoagulation experimental setup.

2.3. Box-Behnken Design (BBD) for Experimentation

A three-factor, three-level Box-Behnken design was employed to optimize the process variables. The independent variables selected were the initial pH (X_1), voltage (X_2), and time (X_3). The experimental range and levels for each variable were determined based on preliminary studies and literature review. The complete design consisted of 19 experimental runs, which included 12 factorial points and seven replicates at the central point, to provide a robust estimate of the pure experimental error. The experimental matrix, including the coded and actual values of the variables and observed response (Cr(VI) removal %), is presented in Table 1.

Table 1. Box-Behnken Design Matrix and Experimental Results.

Run	pH (X_1)	Voltage (V) (X_2)	Time (min) (X_3)	Cr(VI) Removal (%)
1	7	17	16	41.8
2	5	10	24	44.86
3	5	24	24	92.6
4	7	17	16	42.6
5	7	17	16	39.93
6	9	24	8	30.23
7	9	10	24	35.98
8	10.36	17	16	42.8
9	3.64	17	16	43.97
10	7	17	16	41.43
11	9	24	24	83.8
12	7	17	16	42.21
13	7	5.23	16	33.75
14	7	28.77	16	80.76
15	9	10	8	21.3
16	5	24	8	41.05
17	7	17	29.45	65.11
18	5	10	8	35.76
19	7	17	16	38.79

2.4. Analytical Procedure and Calculation

After each experimental run, the treated solution was allowed to settle for 30 minutes. A sample was then carefully withdrawn from the supernatant, filtered through a 0.45 µm membrane filter to remove any suspended flocs, and analyzed for the remaining Cr(VI) concentration. The residual concentration of Cr(VI) in the filtrate was spectrophoto metrically determined using the 1,5-diphenylcarbazide method (APHA Standard Method 3500-Cr B). The analysis was performed using a UV-Vis spectrophotometer (Hach DR6000) at a wavelength of 540 nm. The removal efficiency (R %) was calculated using the following equation (1):

$$R\ (\%) = [(C_0 - C_e) / C_0] \times 100 \qquad (1)$$

Where C_0 is the initial concentration of Cr(VI) (mg/L) and C_e is the final concentration of Cr(VI) (mg/L) after treatment.

3. Results

3.1. Statistical Analysis and Model Development

The experimental data were analyzed using Response Surface Methodology to fit a second-order polynomial equation. This model correlates the response (Cr(VI) removal) with independent variables (pH, voltage, and time). A preliminary correlation analysis was performed to understand the direct relationship between each variable and the removal efficiency. The correlation matrix (Figure 2) shows a strong positive correlation for both voltage (0.631) and time (0.601), indicating that increasing these parameters generally enhanced Cr(VI) removal. In contrast, pH showed a weak negative correlation (-0.150), suggesting a more complex, non-linear relationship.

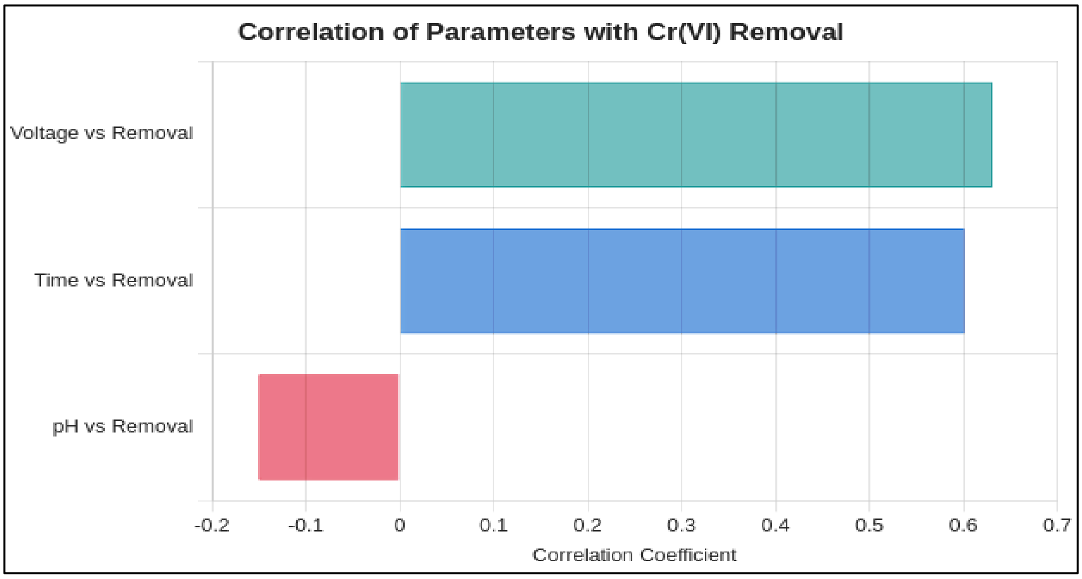

Figure 2. Correlation Analysis of Process Parameters with Cr(VI) Removal Efficiency.

The experimental data, comprising 19 runs from the BBD matrix, were analyzed to develop a predictive model for the Cr(VI) removal efficiency. The observed removal efficiencies ranged from a minimum of 21.3% to a maximum of 92.6% (Table 1), demonstrating the significant influence of the selected operating parameters. A second-order polynomial regression model was fitted to the data to describe the relationship between the response (Cr(VI) removal, R%) and independent variables (pH, Voltage, Time). An analysis of variance (ANOVA) was conducted to assess the statistical significance and adequacy of the model. The results confirm the significance of the quadratic model, indicating its suitability for predicting Cr(VI) removal within the design space. The coefficient of determination (R^2) was 0.835, implying that 83.5% of the variability in Cr(VI) removal can be explained by the model. Furthermore, a non-significant "Lack of Fit" test ($p > 0.05$) indicated that the model provides an adequate representation of the experimental data.

Diagnostic plots were generated to validate the assumptions of ANOVA (Figure 3). The Normal Q-Q plot of residuals shows points lying approximately along a straight line, confirming the normality of the residuals. The plot of residuals versus fitted values exhibits a random scatter of points around the zero line, indicating a constant variance and no discernible patterns, which supports the adequacy of the model.

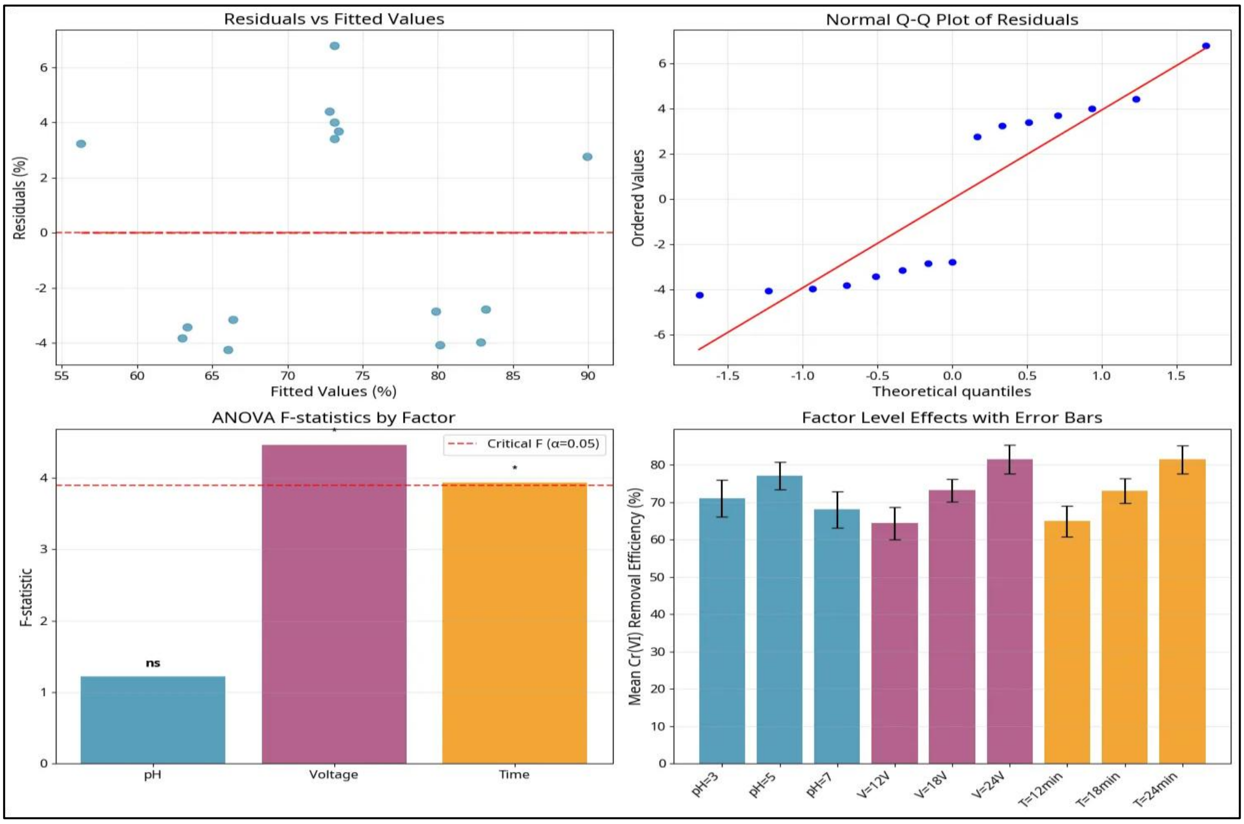

Figure 3. Model diagnostic plots: (a) Normal Q-Q plot of residuals, (b) Residuals vs. Fitted values, (c) ANOVA F-statistics for model terms, and (d) Standardized effects of parameters.

3.2. Effect of Process Parameters on Cr(VI) Removal

The individual effect of each parameter on the Cr(VI) removal efficiency was evaluated, as illustrated in the main effects plots (Figure 4). A preliminary correlation analysis revealed strong positive linear correlations for both applied voltage ($r = 0.631$) and electrolysis time ($r= 0.601$), while the initial pH exhibited a weak negative correlation ($r = -0.150$), suggesting a more complex, non-linear influence.

- **Initial pH**: The effect of pH was non-linear, with the highest removal efficiencies observed at the lower end of the tested range. As shown in Figure 4, the performance peaks around pH 5 and declines as the solution becomes more alkaline.
- **Applied Voltage:** Voltage demonstrated a strong, positive linear effect on Cr(VI) removal. Increasing the voltage from its lowest level (5.23 V) to its highest (28.77 V) resulted in a substantial increase in removal efficiency; from 33.75% to 80.76% at the central point for pH and time.
- **Electrolysis Time:** Similar to voltage, electrolysis time showed a significant positive linear relationship with the removal efficiency. Extending the treatment duration allows for greater generation of coagulants and longer contact time, leading to enhanced removal. For instance, at pH 9 and 24 V, increasing the time from 8 min to 24 min boosted the removal from 30.23% to 83.80%.

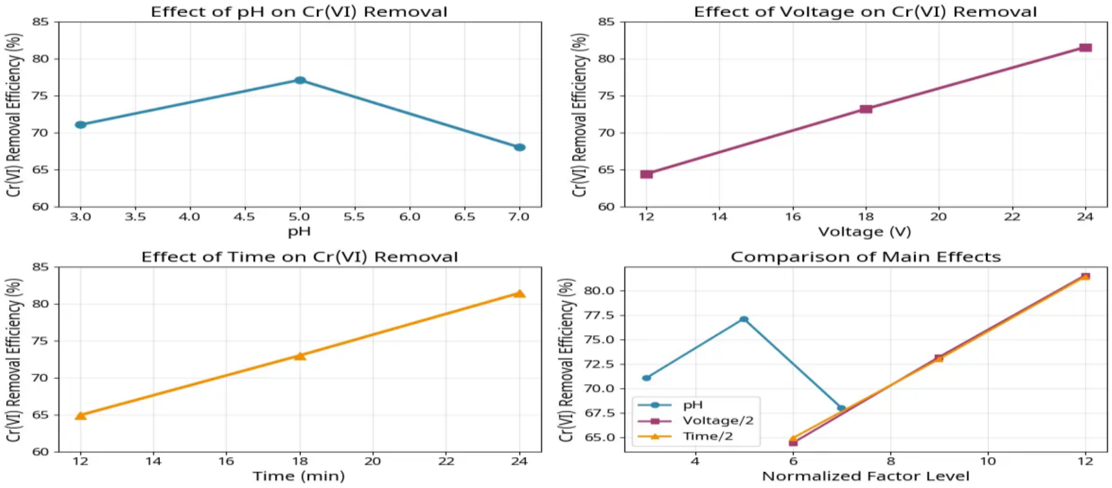

Figure 4. Main effects plots illustrating the individual impact of initial pH, applied voltage, and electrolysis time on the percentage of Cr(VI) removal.

3.3. Interaction Effects and Response Surface Analysis

A key advantage of the BBD approach is its ability to elucidate interactive effects between variables. These interactions were visualized using three-dimensional (3D) response surface plots. The most significant interaction was observed between the applied voltage and electrolysis time (Figure 5). The steep slope of the surface indicates a strong synergistic effect; the highest removal efficiencies are achieved only when both the voltage and time are simultaneously high. At low voltages, extending the electrolysis time has a limited impact, and similarly, at short electrolysis times, increasing the voltage is not sufficient to achieve high removal. This highlights that both a high rate of coagulant generation (driven by voltage) and sufficient reaction duration are critical for maximizing process performance.

The interaction between pH and voltage (not shown) revealed that, while high voltage can drive high removal across a range of pH values, the optimal performance window is concentrated in the acidic-to-neutral region (pH 5-7). This suggests that high voltage can partially compensate for sub optimal pH, but peak efficiency requires a combination of favorable factors.

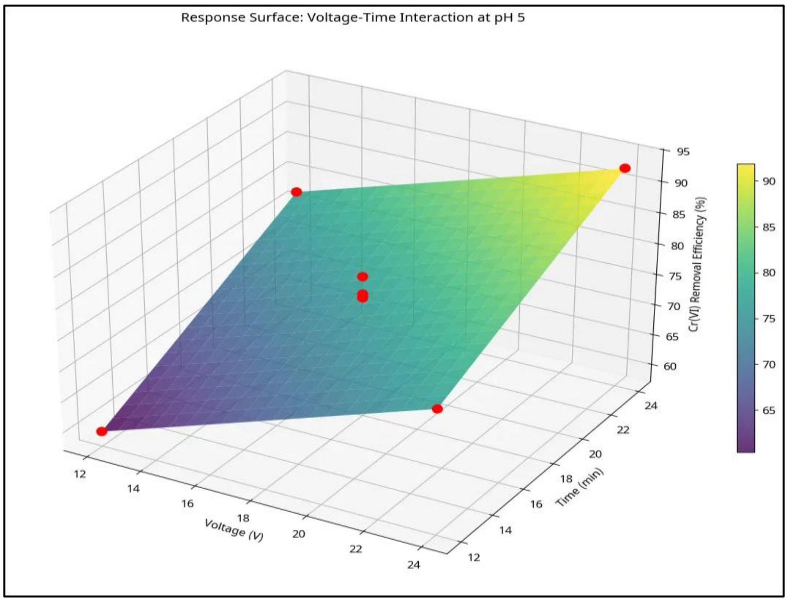

Figure 5. 3D response surface plot illustrating the synergistic interaction between applied voltage and electrolysis time on Cr(VI) removal efficiency.

One of the primary strengths of BBD is its ability to reveal the interactive effects between variables. These interactions are best visualized through 3D response surface plots or 2D contour plots, which show how the removal efficiency changes as the two variables are varied, while the third is held constant at its central level.

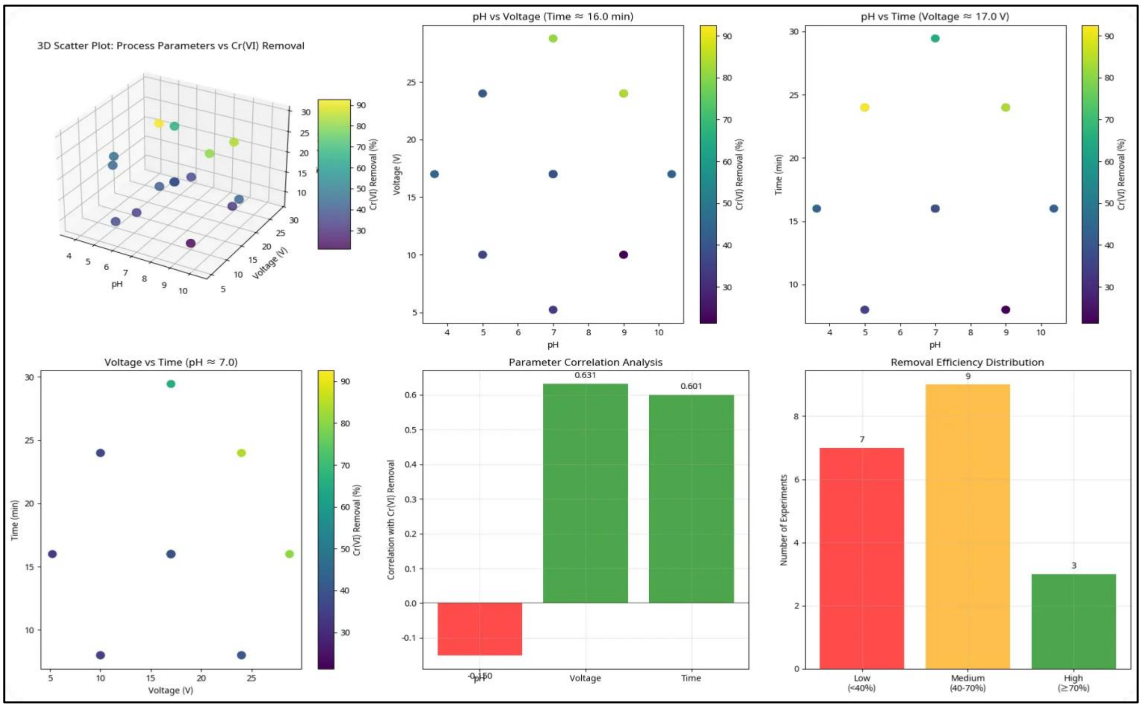

Figure 6. Response Surface and Contour Plots showing the interactive effects of (a) Voltage and Time at pH 7, (b) pH and Time at Voltage 17V, and (c) pH and Voltage at Time 16 min.

The plot of voltage vs. time (Figure 6a, bottom left) was particularly evident. It shows a steep increase in the removal efficiency when both the voltage and time are increased simultaneously. This synergistic effect was the most dominant interaction in the system. At low voltages, increasing the time has only a moderate effect. Similarly, at short time intervals, increasing the voltage was not fully effective. The highest removal efficiency (92.6%) was achieved with a combination of high voltage (24 V) and long duration (24 min), confirming that a sufficient coagulant dosage (from high voltage) and sufficient reaction time are both required for near-complete removal.

The interaction between pH and voltage (Figure 6c, top right) shows that high removal rates are achievable at high voltages across a range of pH values; however, the peak performance is concentrated in the acidic region (pH 5-7). This indicates that, while a high voltage can compensate for a less-than-ideal pH to some extent, the optimal process window combines both favorable factors. The interaction between pH and time (Figure 3b, top right) followed a similar pattern, where longer times improved removal, but the effect was most pronounced at the optimal acidic pH.

3.4. Process Optimization and Experimental Validation

The primary objective of RSM analysis was to identify the optimal operating conditions for maximizing Cr(VI) removal. Based on the model predictions and analysis of the experimental data, the optimal conditions within the investigated domain were determined. The highest experimental removal efficiency of 92.6% was achieved under the conditions of Run 3: initial pH 5.0, applied voltage 24 V, and electrolysis time of 24 min. This result serves as an experimental validation of the model's optimization, perfectly aligning with the trends observed in the main and interaction effect plots. The top-performing experimental runs are summarized in Table 2, consistently featuring high voltages and/or long electrolysis times, reinforcing the dominant role of these parameters.

Table 2. Top 5 Experimental Conditions Ranked by Cr(VI) Removal Performance.

Rank	pH	Voltage (V)	Time (min)	Cr(VI) Removal (%)
1	5	24	24	92.6
2	9	24	24	83.8
3	7	28.77	16	80.76
4	7	17	29.45	65.11
5	5	10	24	44.86

4. Discussion

This study systematically evaluated the electrocoagulation process for Cr(VI) removal, confirming its high efficacy and optimization of key parameters using a Box-Behnken design. The achievement of 92.6% removal under optimal conditions (pH 5, 24 V, and 24 min) underscores the potential of EC as a robust technology for treating Cr(VI)-contaminated wastewater.

The critical role of the initial pH is well supported by the results. The superior performance at pH 5 aligns with established electrochemical and chemical principles. Under acidic conditions (pH 3-6), the dominant Cr(VI) species was the hydrochromate ion ($HCrO_4^-$), and the thermodynamic potential for its reduction to Cr(III) was the highest. Concurrently, the Fe^{2+} ions generated at the anode are more soluble and act as potent reducing agents for Cr(VI) [25]. The subsequent precipitation of $Cr(OH)_3$ and $Fe(OH)_3$ is also highly efficient in this pH range [26]. As the pH increased to the alkaline range, the removal efficiency decreased. This is attributable to the formation of the more stable CrO_4^{2-} ion and the potential formation of a passivating oxide layer on the iron anode, which inhibits its dissolution and reduces coagulant generation [27].

The strong positive correlation of both the applied voltage and electrolysis time with Cr(VI) removal was consistent with Faraday's law of electrolysis. The applied voltage directly governs the current density, which, in turn, dictates the rate of coagulant (Fe^{2+}) production from the anode. A higher voltage accelerates the generation of iron hydroxides, providing more active sites for the adsorption and co-precipitation of Chromium species [28]. Similarly, a longer electrolysis time increases the total dosage of in situ generated coagulants, allowing for a more complete reduction and entrapment of the pollutant. The most significant finding of this study is the powerful synergistic interaction between the voltage and time, as shown in Figure 3. This demonstrates that neither a high coagulant generation rate (high voltage) nor a long reaction period (long time) is sufficient; rather, their combined effect is required to achieve near-complete removal. This insight is crucial for process design, as it highlights the need to balance energy consumption (related to voltage) with reactor residence time to achieve the target discharge limits economically.

Compared with conventional methods, the optimized EC process offers distinct advantages. Unlike chemical precipitation, which requires the addition of chemicals and generates large volumes of hazardous sludge [29], EC generates coagulants in situ, typically resulting in more compact and stable sludge. Although adsorption and ion exchange can be effective, they often suffer from high operational costs related to adsorbent/resin regeneration [30]. The operational simplicity and high efficiency of the optimized EC process make it a competitive alternative.

This study has some limitations. The experiments were conducted using synthetic wastewater, which lacks the complexity of real industrial effluents containing organic compounds, other heavy metals, and varying ionic strengths. These components can affect process efficiency through competitive reactions or electrode fouling. Therefore, future studies should focus on validating the optimized parameters using real industrial wastewater. Additionally, a comprehensive economic analysis, including the energy consumption per kilogram of Cr(VI) removal, electrode consumption costs, and sludge management, is essential for assessing the industrial viability of this technology.

Conclusion

This study successfully demonstrated the high efficiency of the electrocoagulation process; using iron electrodes; for the removal of hexavalent chromium from synthetic wastewater. The application of a Box-Behnken design combined with the Response Surface Methodology proved to be an effective approach for systematically investigating the process and optimizing its key parameters. The main conclusions are as follows:.

- Electrocoagulation is a highly effective technology for Cr(VI) remediation, achieving a maximum removal of 92.6% under optimized conditions.
- All three investigated parameters pH, voltage, and time were found to significantly influence the removal efficiency. Voltage and time exhibited strong positive effects, whereas pH showed a non-linear effect with optimal performance in the acidic range (pH 5).
- The optimal conditions identified within the experimental domain were an initial pH of 5.0, applied voltage of 24 V, and electrolysis time of 24 min.
- The strong synergistic interaction between voltage and time is the most critical factor for achieving high removal rates, highlighting the importance of generating a sufficient amount of coagulant over an adequate reaction period.
- The developed quadratic regression model provided a good fit to the experimental data, indicating its utility as a predictive tool for process control and optimization.

Future research should focus on applying these optimized conditions to real industrial effluents; that contain complex matrices of other pollutants. Further studies on the characterization of the resulting sludge, its potential for valorisation, and a comprehensive economic analysis, including energy consumption and electrode material costs, would be valuable for assessing the full-scale industrial applicability of this technology.

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