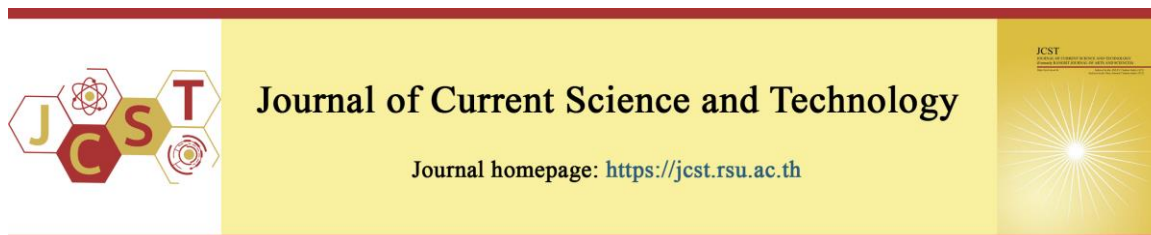


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Development of Electro-Fenton Process for Methylene Blue Degradation using NiFeCo Foam Alloy Cathode and Porous Graphite Anode

Hind H. Thwaini^{1,*}, ORCID: 0009-0001-0075-9784
Rasha H. Salman¹, ORCID: 0000-0001-9353-9096
Zahraa M. Hameed¹, ORCID: 0009-0001-5334-9244
Khalid M. Abed^{1,2}, ORCID: 0000-0003-4791-219X
Sunarti Abd Rahman², ORCID: 0000-0003-0986-5548
Wan Jeffrey Basirun^{3,4}, ORCID: 0000-0001-8050-6113

¹Department of Chemical Engineering, College of Engineering, University of Baghdad, Baghdad, Iraq

²Faculty of Chemical and Process Engineering Technology, University Malaysia Pahang Al-Sultan Abdullah, Lebuhraya Persiaran Tun Khalil Yaakob, Kuantan, Pahang 26300, Malaysia

³Nanotechnology and Catalysis Research (NanoCat), Institute of Advanced Studies (IAS), University Malaya, Kuala Lumpur 50603, Malaysia

⁴Department of Chemistry, Faculty of Science, University Malaya, Kuala Lumpur 50603, Malaysia

*Corresponding author; E-mail: hind.abd2107m@coeng.uobaghdad.edu.iq

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Abstract

The low efficiency of electro-Fenton (EF) technology in generating hydrogen peroxide (H_2O_2) is one of the most frequently encountered obstacles. Thus, the search for high-performance electrodes and reactors is necessary to increase system efficiency. In this study, porous graphite served as the control group to increase oxygen mass transfer in the reactor, while foam alloy was used as the cathode for producing in situ H_2O_2 . X-Ray diffraction analyzer (XRD) and scanning electron microscope (SEM) were utilized to investigate the microstructure of the electrodes. Response surface methodology (RSM) was used to examine the various operational parameters affecting the reduction of methylene blue (MB) dye. A $FeSO_4 \cdot 7H_2O$ concentration of 0.5 mM, a current density of 8 mA/cm², and a reaction time of 30 min were the optimal conditions for the electro-Fenton technique. Under these conditions, the MB removal efficiency (RE%) was 97.73%, and the energy consumption was 11.76 kWh/kg MB. The two most important factors controlling dye reduction in the electro-Fenton process are the $FeSO_4 \cdot 7H_2O$ concentration and the current density, which contributed 68.33% and 15.44%, respectively, in this model. The coefficient of multiple correlation (R^2) was 99.59%, which demonstrated the statistical significance of the regression analysis. Therefore, the foam alloy electrode represents a new and viable strategy for contaminant degradation in the electro-Fenton process.

Keywords: foam alloy, analysis of variance, methylene blue, electro-Fenton, hydroxyl radical, RSM

1. Introduction

Industrialization is a major factor in a nation's progress. The textile manufacturing industry represents both innovation and prosperity in an era of rapid globalization and consumer-driven economies (Islam et al., 2025). However, serious environmental

and public health concerns are associated with the growing pollution of natural water sources by synthetic dyes used in the cosmetic, pharmaceutical, and textile industries. (Ho, 2022; Mohammed et al., 2023a; Saleem & Al-Jubouri, 2024; Salman et al., 2024). Methylene blue (MB), a heterocyclic aromatic

cationic dye with extensive industrial applications, stands out among thousands of dyes used worldwide (Hasnaoui et al., 2024). MB has high solubility in water and stability in aqueous solutions; it imposes considerable negative environmental effects, especially on the aquatic organisms, such as color pollution, decreased light penetration, and increased toxicity (Wetchakun & Wetchakun, 2022). It is difficult to fully degrade MB by common wastewater treatment methods such as coagulation-flocculation (Ratchawet et al., 2025; Mohammed et al., 2023b), adsorption (Üner et al., 2016), biological degradation (Uddin et al., 2024), and membrane filtration (Periyasamy, 2024). This necessitates the development of an efficient treatment process that could completely eliminate these persistent organic contaminants. Advanced Oxidation Processes (AOPs) have become a well-known efficient method for degrading various types of organic contaminants (Hameed & Salman, 2024).

The EF method is one of the most promising technologies due to its high efficiency, cost-effectiveness, and environmental sustainability. The foundation of the EF process is the Fenton reaction, which occurs between hydrogen peroxide (H_2O_2) and ferrous ions (Fe^{2+}) and produces hydroxyl radicals $\text{HO}^\bullet$ in situ, one of the most potent oxidizing agents (Yin et al., 2025). The non-selective attack by these radicals oxidizes organic molecules, which are eventually mineralized into CO_2 and H_2O (Abbas & Abbas, 2022; Minnalkodi Senguttuvan et al., 2024; Thwaini & Salman, 2023b). A variety of operational factors, such as the applied current, pH, electrolyte concentration, reaction duration, space between electrodes, composition and structure of the electrode materials, significantly affect the efficacy of the electro-Fenton process (Nair et al., 2021; Nuhma et al., 2025). Carbon-based cathodes and soluble iron anodes have been used previously, but new developments have prompted research into new electrode materials to improve H_2O_2 generation, enhance stability, and reduce operating costs (Abbas & Abbas, 2021). In electrochemical applications, foam electrodes have attracted the interest of numerous researchers due to their high porosity, large surface area, and excellent mass transport capabilities (Fahim & Abbar, 2020). Foam alloy cathodes are a key component of the electro-Fenton method, especially those based on metals such as nickel, iron, and cobalt. These metals are known for their catalytic activity, high electrochemical conductivity, and structural stability (Kim et al., 2012).

The NiFeCo foam alloy particularly provides synergistic effects due to the electrochemical properties of each constituent metal. Cobalt controls catalytic activity for the oxygen reduction reaction (ORR) in the redox cycle for Fe^{2+} regeneration (Hsieh et al., 2022; Karnjanakom et al., 2024), while iron acts as a source of Fe^{2+} to improve the Fenton's reaction (Benjumea-Hoyos et al., 2024), and nickel supports mechanical toughness and high electrochemical conductivity (Thwaini & Salman, 2023a; Zhu et al., 2020). The foam structure enhances peroxide synthesis efficiency and the formation of $\text{HO}^\bullet$ by improving reactant mass transfer and assisting bubble detachment (Ali et al., 2021).

Furthermore, it is just as important to select the appropriate material. Porous graphite anodes are highly important due to their chemical inertness, environmental compatibility and high electronic conductivity (Zhang et al., 2021). The large surface area of porous graphite facilitates an efficient electron transfer and supports the anodic oxidation of Fe^{2+} to Fe^{3+} , which is a necessary process for sustaining the Fenton cycle (Deng et al., 2023). In addition, anodic reactions confine the action of the hydroxyl radicals and improve overall mineralization, thereby enhancing the oxidative breakdown of intermediates during the EF process (Khan et al., 2023).

Electro-Fenton optimization requires several essential components such as the design of experiment (DOE), and its application through response surface methodology (RSM). RSM makes it possible to identify ideal conditions and create models with a minimum number of experimental runs. It also improves the understanding of interactions among process parameters, either positively or negatively. (Faraj et al., 2024; Jankovic et al., 2021).

This study proposes an efficient, economical, and ecologically beneficial solution for eliminating dye contaminants from industrial wastewater by combining material innovation with process optimization. The use of NiFeCo foam alloy as the cathode in the electro-Fenton system for MB dye degradation, offering a unique innovation in the electrochemical treatment for organic pollutant removal. Although multiple cathode materials have been used in previous studies, this ternary foam alloy has not been considered, as it is expected to enhance free radical production and improve contaminant degradation efficiency. Its combination with a porous graphite anode and the use of a response surface optimization methodology represents a new pathway toward

developing a more effective and integrated process for treating polluted water.

2. Objectives

This study aimed to improve the electro-Fenton cathode by using a NiFeCo foam alloy for in situ hydrogen peroxide generation for methylene blue degradation and to assess the influence of operational parameters, including iron concentration, current density, and reaction time on methylene blue removal efficiency. It also aimed to optimize the electro-Fenton operating conditions using response surface methodology (RSM) based on the Box–Behnken design (BBD) as well as to evaluate the dye removal efficiency and energy consumption to demonstrate the effectiveness of the proposed electrode for effective wastewater treatment.

3. Materials and Procedures

3.1 Chemicals

All chemicals used in the study were of high purity and did not require further purification. Distilled water and solutions of appropriate concentrations were used to prepare the experimental setup. Methylene blue powder was sourced from BDH Company. Ferrous heptahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) was from CDH Company, Sodium sulfate (Na_2SO_4 , 99% purity) was from SDFCL and served as the electrolyte. Sulfuric acid (0.1 M, 98% pure, Himedia Chemical Company) and sodium hydroxide (0.1 M, 98% purity, Sigma Chemical Company) were used to adjust the solution pH to 3. The foam alloy, with a porosity of 110 PPI, was supplied by Xiamen Top New Energy Technology (China), and the porous graphite anode was obtained from the local market

3.2 Methods

The electro-Fenton process was conducted in a 1.0 L beaker containing 0.8 L of aqueous solution with 100 mg/L MB dye for the batch experiments. An 8 mm-thick Perspex cover with four holes was placed above the glass reactor; two holes were used to secure the two electrodes, the third hole was used to insert the thermometer, and the fourth was used to insert the airflow. The electrodes were separated by 30 mm and consisted of a porous graphite electrode (150 mm $\times$ 50 mm $\times$ 10 mm) as the anode and NiFeCo foam alloy (150 mm $\times$ 50 mm $\times$ 10 mm) as the cathode. Then, 0.1 M H_2SO_4 and ultra-pure water were used to remove

the native oxide layer from the foam alloy. An electromagnetic stirrer (Heidolph) was used to continuously mix the aqueous solution at 250 rpm (Abbas & Abbar, 2020).

Figure 1 shows a schematic representation of the electro-Fenton process. The solution was aerated with an airflow rate of 20 L/h for 15 minutes before and throughout the experiments using an air pump (ACO-001 electromagnetic, China). A DC power supply (UNI-T, UTP3315PE type (0-30 V, 0-5 A)) was used to establish a steady current density. Na_2SO_4 (0.01 M) was added to enhance conductivity, and a catalytic quantity of Fe^{2+} was added to initiate the reaction. Solution pH measured with a pH meter (HANNA, Romania) after adjustment to pH 3. MB content was measured using an ultraviolet spectrometer (UV-9200, UK) at a wavelength (λ) of 665 nm. Dye removal efficiency (RE%) was calculated using Equation (1) (Abbas & Abbas, 2019):

$$\text{RE\%} = [(C_i - C_f) / C_i] \times 100 \quad (1)$$

where C_i and C_f are initial and final dye concentrations, respectively. Energy consumption (EC) in kWh/kg was calculated using Equation (2):

$$\text{EC} = \frac{I \times E \times t \times 1000}{\Delta\text{MB} \times V} \quad (2)$$

where EC is the energy consumption (kWh/kg MB), I is the applied current (A), E is the cell voltage (V), t is the electrolysis time (h), $\Delta\text{MB} = (C_i - C_f)$ is the concentration of MB removed (mg/L), V is the volume of the treated solution (L), and 1000 is the unit-conversion factor.

3.3 Characterization Method of Electrodes

The cathode was NiFeCo foam, and the anode was porous graphite. Structural properties were determined using an X-ray diffraction analyzer (Shimadzu 6000, Japan). The X-ray analysis was performed at 40 kV and 30 mA with a scanning rate of 5 degrees per minute using a $\text{Cu K}\alpha$ radiation tube source. A scanning electron microscope (SEM, FEI-Company Netherlands, Inspect S50) operated at 25 kV and 100 microamperes was used to investigate the electrode surface. Energy-dispersive X-ray spectroscopy (EDX, Bruker Germany, XFlash-6110, 25kV, 100 μA) was used to determine the chemical composition of the electrodes

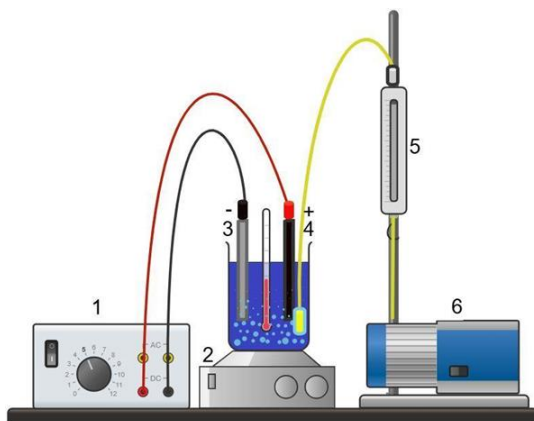


Figure 1 Electrochemical schematic system: (1) power supply, (2) magnetic stirrer, (3) NiFeCo foam cathode, (4) porous graphite anode, (5) flow meter, (6) air pump

3.4 Design of Experiment

The relationship between process response and influencing factors was determined using statistical and mathematical data collection techniques based on the design of experiments (DOE), specifically response surface methodology (RSM) with Minitab-18. A Box–Behnken design (BBD) was employed to identify optimal parameters for dye elimination efficiency. The three independent variables examined were current density (X_1), electrolysis time (X_2), and iron dosage (X_3) at three different levels. Each variable was examined at three coded levels (-1 , 0 , $+1$), with the experimental ranges summarized in Table 1. A second-order model and least-squares method were used to establish the correlation between RE% and the independent variables, as shown in Equation (3) (Demirel et al., 2022):

$$Y = \alpha_0 + \sum \alpha_i x_i + \sum \alpha_{ii} x_i^2 + \sum \alpha_{ij} x_i x_j \quad (3)$$

the regression coefficient (R^2) was calculated, and an analysis of variance was performed to demonstrate the model's applicability. The resulting BBD experimental matrix, comprising 15 runs including three center-point replicates, is presented in Table 2.

Table 1 Experimental variables of design

Independent variable	levels		
	-1	0	1
X_1 , Current Density, mA/cm ²	4	6	8
X_2 , Time, min	10	20	30
X_3 , FeSO ₄ ·7H ₂ O, mM	0.1	0.3	0.5

Table 2 MB elimination using the BBD design, Bk. = 1

Run	Bk.	Coded value			Real value		
		x_1	x_2	x_3	X_1 Current Density, mA/cm ²	X_2 Time, min	X_3 FeSO ₄ ·7H ₂ O, mM
1	1	0	1	-1	6	30	0.1
2	1	1	0	1	8	20	0.5
3	1	0	1	1	6	30	0.5
4	1	0	0	0	6	20	0.3
5	1	-1	0	1	4	20	0.5
6	1	-1	0	-1	4	20	0.1
7	1	1	1	0	8	30	0.3
8	1	-1	-1	0	4	10	0.3
9	1	1	-1	0	8	10	0.3
10	1	0	-1	1	6	10	0.5
11	1	0	0	0	6	20	0.3
12	1	-1	1	0	4	30	0.3
13	1	0	0	0	6	20	0.3
14	1	1	0	-1	8	20	0.1
15	1	0	-1	-1	6	10	0.1

4. Results

4.1 Characterization of the Produced Porous NiFeCo Foam Alloy

4.1.1 XRD Phase Analysis

The composition of the NiFeCo foam alloy cathode in its crystalline phase was determined using XRD analysis, and the result is shown in Figure 2. Phase identification, which was performed using the ICDD database, confirmed successful formation of a multi-phase metallic alloy which consist of nickel, cobalt, and iron without presence of significant oxide or hydroxide phases, which indicates the metallic character of the electrode.

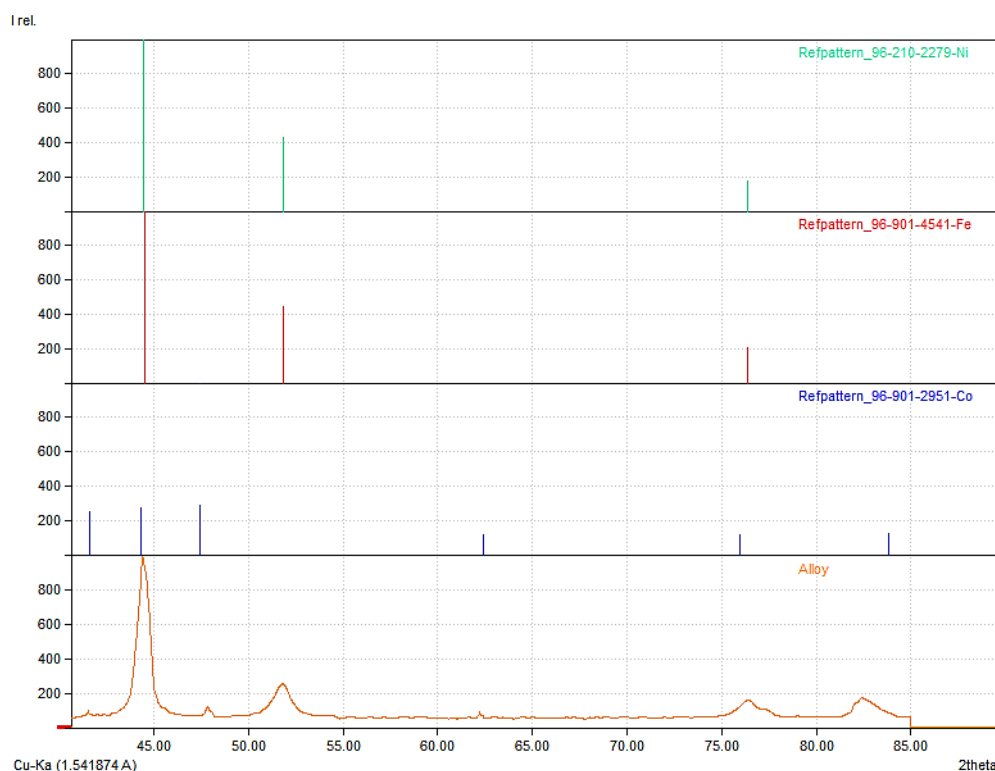


Figure 2 XRD pattern of the NiFeCo foam alloy compared with the standard diffraction patterns of pure Ni, Fe, and Co

Figure 2 shows the XRD pattern of the foam alloy. The peaks are centered at approximately 2θ values of 42° (100), 44.4° (111), 48.1° (101), 51.6° (200), and 76.4° (202). The main peaks at 44.4° , 51.6° , 76.4° correspond to nickel according to the Crystallography Open Database standard card 96-210-2279 (Hu et al., 2019), while the cobalt peaks are present at 42° , 44° , 48° according to Standard Card 96-901-2951 (Khezri et al., 2013; Manjunatha et al., 2019; Yang et al., 2025). The main peak of iron appears at 44.6° from the according to Standard Card 96-901-4541 the figure identified by using ICDD data base program.

4.1.2 Surface Morphology (SEM Analysis)

The NiFeCo foam alloy cathode surface morphology was detected using SEM analyzer, and its micrograph is shown in Figure 3. The SEM image reveals a well-defined, three-dimensional microporous skeleton structure characterized by a smooth band surface, and a cross-linked skeleton network. This

structure provides a large specific surface area, which is fundamental for increasing the active electrochemical sites essential for the oxygen reduction reaction (ORR).

The 3D interconnected porosity enables efficient transport of dissolved oxygen and electrolyte ions through the electrode body to the electrolyte and electrode interface. This is particularly very important in the electro-Fenton process, which needs a continuous and abundant supply of dissolved O_2 at the cathode surface is required to support in situ H_2O_2 electro generation through the two-electron ORR pathway as shown in Equation (4). Furthermore, the skeleton geometry of the foam promotes efficient bubble separation from the electrode surface, preventing gas accumulation that would block active sites and reduce current efficiency (Li et al., 2023). These morphological characteristics combined show that the NiFeCo foam is structurally well-suited to serve as a high-performance cathode in the electro-Fenton process.

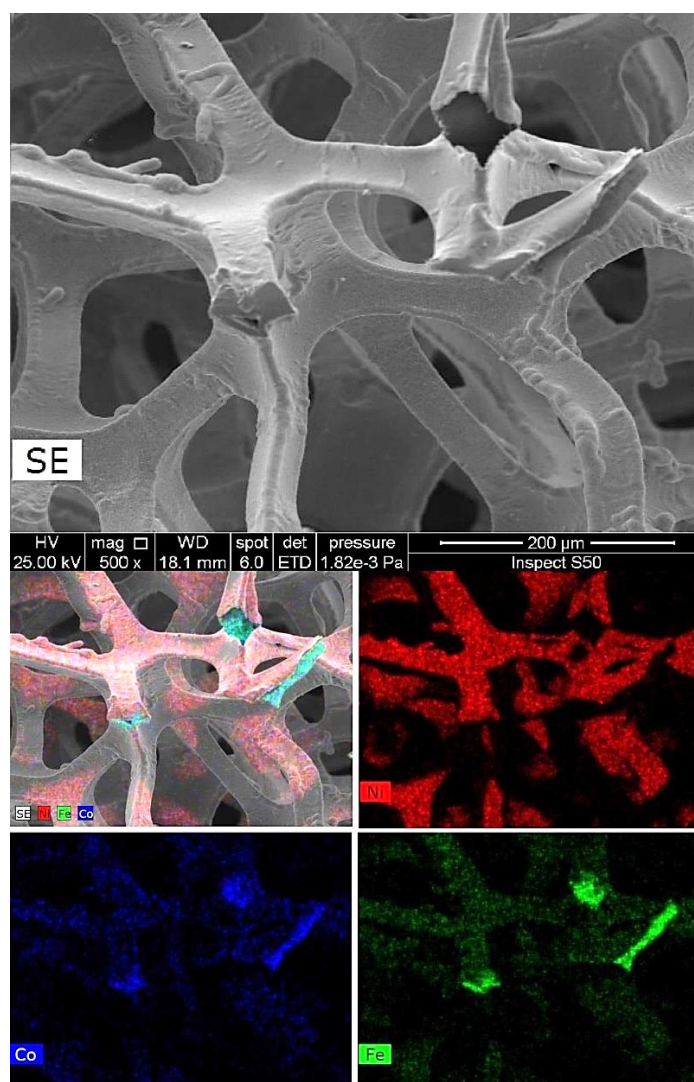


Figure 3 SEM image and EDX elemental mapping of the NiFeCo foam showing its porous structure and the distribution of Ni, Co, and Fe

4.1.3 Elemental Composition (EDX Analysis)

The elemental composition of the NiFeCo foam cathode surface was quantified by EDX spectroscopy, and the identical spectrum is shown in Figure 4. The results showed that successful incorporation of all three metallic elements within the alloy structure. Nickel (Ni) is the dominant component at 60.19 wt.%, serving as the primary structural matrix of the foam. Iron element (Fe) and cobalt element (Co) are present at 20.07 wt.% and 11.65 wt.%, respectively. Carbon (C) and nitrogen (N) were also detected at 3.61 wt.% and 4.48 wt.%, respectively.

This distribution of elements within the alloy is significant in the electro-Fenton mechanism. Each

constituent metal plays a distinct functional role: (Ni) provides high electrical conductivity and, mechanical stability to the foam structure, which improving charge transfer efficiency; Co enhances catalytic activity toward the (ORR) and may facilitate Fe^{2+} regeneration from Fe^{3+} at the cathode surface, aiding the Fenton catalytic cycle; and (Fe), serves as an additional source of Fe species which may participate in homogeneous EF pathway driven by dissolved Fe^{2+} catalyst (Ghjair & Abbar, 2022). The synergistic combination of these three elements within a single porous electrode provides both structural and catalytic advantages compared with single-metal foam cathodes and supporting the reason for adopting the NiFeCo foam alloy configuration in this study.

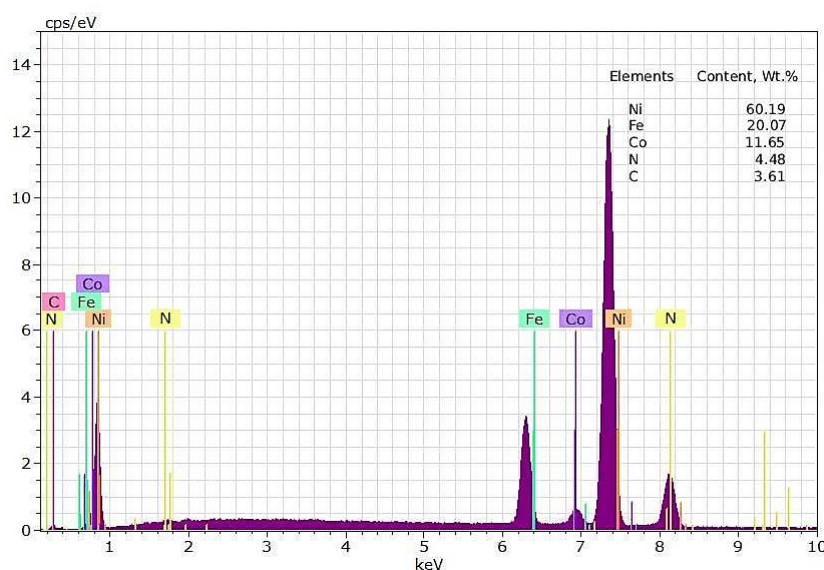


Figure 4 EDX spectrum of the NiFeCo foam showing the elemental composition of Ni, Fe, and Co

Table 3 Results of experiments utilizing the BDD for dye removal efficiency and energy consumption

Run	Bk.	Current Density, mA/cm ²	Time, min	FeSO ₄ .7H ₂ O, mM	Actual MB RE%	Predicted MB RE%	E, volt	EC, (kWh/kg MB dye)
1	1	6	30	0.1	81.125	81.0567	5.2	9.615
2	1	8	20	0.5	97.123	96.8527	5.7	7.83
3	1	6	30	0.5	94.853	95.3642	5.2	8.22
4	1	6	20	0.3	90.001	90.4647	5.4	6.11
5	1	4	20	0.5	93.027	92.7177	4.1	2.99
6	1	4	20	0.1	78.341	78.6112	4.2	3.57
7	1	8	30	0.3	95.151	94.9100	5.7	11.98
8	1	4	10	0.3	85.204	85.4450	4.5	1.69
9	1	8	10	0.3	91.021	91.2230	6.2	4.35
10	1	6	10	0.5	89.765	89.8332	5.5	2.94
11	1	6	20	0.3	91.170	90.4647	5.2	5.71
12	1	4	30	0.3	89.136	88.9340	3.8	4.26
13	1	6	20	0.3	90.101	90.4647	5.4	6.11
14	1	8	20	0.1	84.881	86.2302	5.8	9.29
15	1	6	10	0.1	79.923	79.4117	5.6	3.36

4.2 Statistical Analysis

Fifteen runs were conducted according to the BBD to examine the ideal conditions for MB dye elimination. Table 3 summarizes these experimental findings, for energy consumption (EC) and removal efficiency (RE%).

The MB dye removal efficiency ranged from 78.341 to 97.123%, according to the results, while the energy consumption ranged from 1.69 to 11.98 kWh/kg MB dye. The comparison of runs 5 and 6 as a first step shows that iron concentration significantly affects the efficiency of dye removal, with RE%

increasing from 78.341 to 93.027 %, representing a difference of 14.686%, as the FeSO₄.7H₂O concentration increased from 0.1 to 0.5 mM at a constant current density of 4 mA/cm² and reaction time of 20 min. The comparison between runs 7 and 12 was performed to show the effect of current density on the MB dye removal efficiency, with the RE% increasing from 89.136 to 95.151%, resulting in a 6.015% difference, at a constant FeSO₄.7H₂O concentration of 0.3 mM and a reaction time of 30 min. The comparison of runs 1 and 15 shows that the reaction time had a lower effect on the MB dye

removal by a difference of 1.202%, with a constant $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ concentration of 0.1 mM and a current density of 6 mA/cm².

By analyzing dye removal efficiency using the Minitab-18 application, an empirical model of removal efficiency was developed based on the actual process parameter values.

$$\begin{aligned} \text{MB RE\%} = & 65.09 - 0.7 X_1 + 0.523 X_2 + 76.06 X_3 + 0.2312 \\ & X_1^2 - 0.01262 X_2^2 - 69.66 X_3^2 + 0.0025 X_1 X_2 - 2.178 \\ & X_1 X_3 + 0.486 X_2 X_3 \end{aligned} \quad (4)$$

where $X_1 X_2$, $X_1 X_3$, and $X_2 X_3$ represent the interaction effects of the model parameters. The predicted dye removal efficiency values for dye removal were studied using Equation (4).

A positive value before any parameter in Equation (4) indicates that the RE% increases with the parameter, and vice versa.

4.3 Analysis of Variance (ANOVA)

The adequacy of the BBD was assessed using analysis of variance (ANOVA). It is an analytical

technique that determines the significance of model parameters using the Fisher's F-test and the P-test. Table 4 presents the ANOVA results, and the terminologies used in statistics are exemplified by the terms "sequential sum of squares" (Seq. SS), "adjusted sum of squares" (Adj. SS), and "adjusted mean square" (Adj. MS). DF represents the model and its degrees of freedom of the model and its parameters, whereas Contr. indicates the contribution of each variable.

The relevance of the developed model is indicated by the high model F-value (134.7) and low probability value ($p = 0.0001$), with a contribution value of 99.59% demonstrating the significance of the regression model. Only 0.41% of the total variation was not explained by the model, according to the multiple correlation coefficient R^2 of 99.59%. This demonstrates the statistical significance of the regression. In this model, the predicted multiple correlation coefficient (pred. $R^2 = 95.8\%$) and the adjusted multiple correlation coefficient (adj. $R^2 = 98.85\%$) were well matched as the difference between them was less than 0.2.

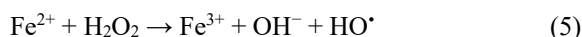
Table 4 ANOVA analysis for the electro-Fenton method of dye elimination

Source	DF	Seq SS	Contr.	Adj SS	Adj MS	F-Value	P-Value
Model	9	445.654	99.59%	445.654	49.517	134.70	0.000
Linear	3	400.587	89.52%	400.587	133.529	363.23	0.000
X_1	1	69.078	15.44%	69.078	69.078	187.91	0.000
X_2	1	25.747	5.75%	25.747	25.747	70.04	0.000
X_3	1	305.762	68.33%	305.762	305.762	831.75	0.000
Square	3	38.247	8.55%	38.247	12.749	34.68	0.001
$X_1 X_1$	1	5.503	1.23%	3.159	3.159	8.59	0.033
$X_2 X_2$	1	4.073	0.91%	5.877	5.877	15.99	0.010
$X_3 X_3$	1	28.671	6.41%	28.671	28.671	77.99	0.000
2-Way Interaction	3	6.820	1.52%	6.820	2.273	6.18	0.039
$X_1 X_2$	1	0.010	0.00%	0.010	0.010	0.03	0.877
$X_1 X_3$	1	3.035	0.68%	3.035	3.035	8.25	0.035
$X_2 X_3$	1	3.775	0.84%	3.775	3.775	10.27	0.024
Error	5	1.838	0.41%	1.838	0.368		
Lack-of-Fit	3	1.067	0.24%	1.067	0.356	0.92	0.558
Pure Error	2	0.771	0.17%	0.771	0.385		
Total	14	447.492	100.00%				
Model Summary				S	R^2	R^2 (adj.)	R^2 (pred)
				0.606311	99.59%	98.85%	95.80%

5. Discussion

5.1 Process Parameters' Impact

The effect of a particular parameter on the response can be shown graphically using RSM. The impact of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ concentration on RE % at various current densities (4 – 8 mA/cm^2) over a fixed reaction time of 20 min is shown in Figure 5. Figure 5a shows the contour plot, while Figure 5b shows the surface response plot. The surface plot indicates that the MB dye removal efficiency increases at the same current density (4 mA/cm^2) when the Fe^{2+} concentration increases from 0.1 to 0.5 mM. This behavior can be explained by the fact that a higher concentration of Fe^{2+} enhances the oxidation of H_2O_2 in breaking down large molecules (Gopalakrishnan et al., 2023). Equation (5) shows that iron is very important in the Fenton process because it enhances the production of $\text{HO}^\bullet$ radicals in the solution through reaction with H_2O_2 .



It is possible to control the amount of iron ions released into the solution during the process. At the cathode, Fe^{3+} can be simultaneously reduced to Fe^{2+} , and the foam alloy cathode provides iron in addition to the iron released from the dissolved iron salt. Together, these processes overcome the disadvantages

of the conventional Fenton method and increases the effectiveness of pollution treatment (Wang et al., 2022). From 4 to 8 mA/cm^2 , the RE% increases linearly with the current density at any iron concentration. Since the current is considered the driving force for the reduction of oxygen at the cathode surface which produces H_2O_2 , the increase in current density enhances the release of $\text{HO}^\bullet$ from the reaction between H_2O_2 and ferrous ions (Ni et al., 2024).

The contour plot reveals that a narrow region with a current density of 7.3–8 mA/cm^2 and a Fe^{2+} concentration of 0.35–0.5 mM achieved a dye removal efficiency of >95%.

Figures 6a and 6b show how reaction time affects the MB RE% at various current densities (4 – 8 mA/cm^2) at a $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ concentration of 0.3 mM. For all current densities, Figure 6b demonstrates that the dye removal efficiency increases exponentially until approximately 30 min, after which it becomes constant. This outcome implies that reaction time has a positive influence on the EF process development (Majed & Abbar, 2025). Figure 6a shows that the maximum dye removal efficiency of >94% occurs in a small area at a current density between 7.8 and 8 mA/cm^2 and a reaction time of 20–30 min.

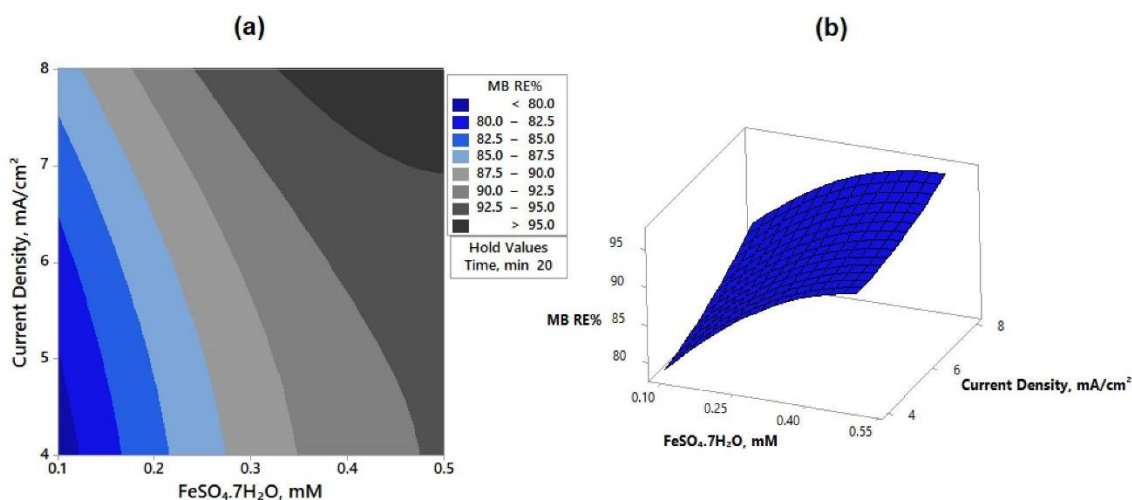


Figure 5 Effect of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ concentration and current density on MB removal efficiency at a constant reaction time of 20 min: (a) contour plot and (b) three-dimensional response surface plot

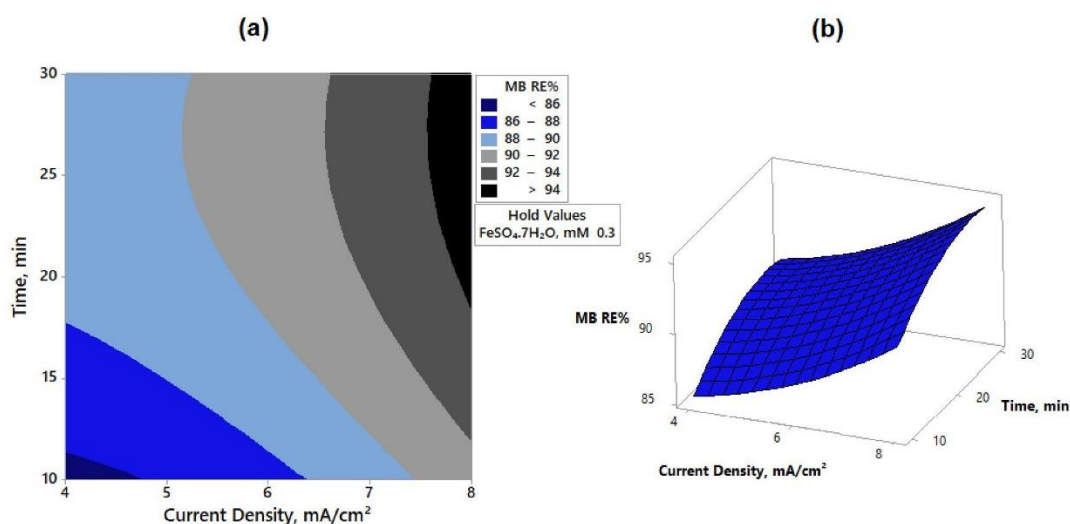


Figure 6 Effect of current density and reaction time on MB removal efficiency at 0.3 mM $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$: (a) contour plot and (b) three-dimensional surface plot

The effect of time on RE% within a range of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ concentrations of 0.1-0.5 mM, and a constant current density of 6 mA/cm^2 is shown in Figures 7a and 7b. Figure 7b shows that the dye removal efficiency increases exponentially with time at any $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ concentration, while Figure 7a shows that the removal efficiency of up to 95% occurs between 0.45-0.5 mM and time at a reaction time of 25 – 30 min. These findings indicate that the RE% increases rapidly at the initial stage and then slows at the final stage when the concentration of FeSO_4 rises to ≥ 0.4 mM. Consequently, the use of RSM makes it possible to determine the feasible optimal values for the parameters under investigation and to obtain

important data on the relationships among these variables.

5.2 Optimization, Confirmation Test, and Comparison with Previous Studies for MB Removal by EF Process

The primary purpose of the optimization process was to specify the ideal parameter values to optimize MB dye removal efficiency. The main objective for dye removal efficiency is to "optimize to maximize" the performance, to obtain the best removal efficiency using the electro-Fenton approach. Table 5 presents the optimal values followed by Table 6, which shows the results of the confirmation test.

Table 5 The optimization of process parameters to get the best MB dye removal efficiency RE%

Response	Goal	Lower	Target	Upper	Weight	Importance
MB RE%	Maximum	78.341	97.123		1	1
Parameters		Several solutions Prediction				
Current Density, mA/cm^2	Time, min	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, mM	Fit	SE Fit	95% CI	95% PI
8	30	0.5	98.4062	0.716	(96.565, 100.248)	(95.994, 100.819)

Table 6 Confirmation of MB dye removal efficiency and energy consumption

Run	Current Density, mA/cm^2	Time, min	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, mM	E, volt	EC, (kWh/kg MB dye)	Actual MB RE%	Average
1	8	30	0.5	5.7	11.69	97.45	97.73
2	8	30	0.5	5.8	11.83	98.01	

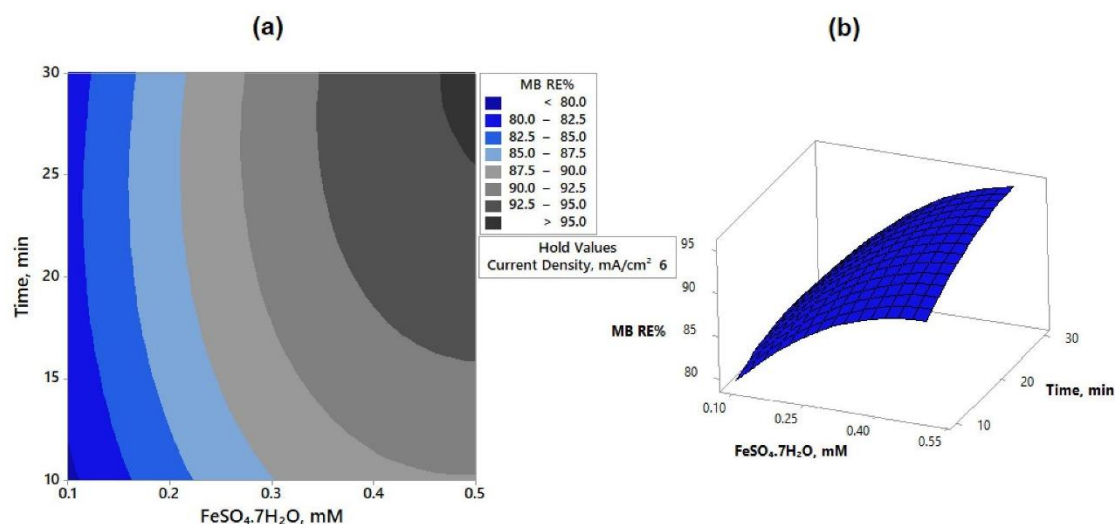


Figure 7 Effect of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ concentration and reaction time on MB removal efficiency at a constant current density of 6 mA/cm^2 : (a) contour plot and (b) three-dimensional surface plot

Table 7 Comparison of EF process for MB removal

Cathode type	Catalyst type	The best operating conditions	RE%	References
Graphite felt	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	[catalyst] = 0.2 mM, voltage = 7 V, time = 20 min, $[\text{MB}]_0 = 20 \text{ mg/L}$, pH = 3, $[\text{Na}_2\text{SO}_4] = 10 \text{ mM}$	97.80%	(Li et al., 2019)
Iron rod	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	[catalyst] = 0.4 mM, voltage = 9 V, time = 60 min, $[\text{MB}]_0 = 10 \text{ mg/L}$, pH = 3, $[\text{Na}_2\text{SO}_4] = 0.1 \text{ M}$	36.56%	(Teng et al., 2020)
Carbon felt	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	[catalyst] = 0.1 mM, CD = 2.5 mA/cm^2 , time = 60 min, $[\text{MB}]_0 = 50 \text{ mg/L}$, pH = 3, $[\text{Na}_2\text{SO}_4] = 0.05 \text{ M}$	73.77%	(Le et al., 2021)
Carbon felt	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	[catalyst] = 0.25 mM, E = - 0.6 V (vs. SCE), time = 60 min, $[\text{MB}]_0 = 100 \text{ mg/L}$, pH = 3, $[\text{Na}_2\text{SO}_4] = 0.05 \text{ M}$	96.70%	(Chai et al., 2022)
NiFeCo foam	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	[catalyst] = 0.5 mM, CD = 8 mA/cm^2 , time = 30 min, $[\text{MB}]_0 = 100 \text{ mg/L}$, pH = 3, $[\text{Na}_2\text{SO}_4] = 0.01 \text{ M}$	97.73%	This study

Under optimized conditions, a control experiment was conducted in the absence of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (Fenton catalyst) to evaluate the role of electro-oxidation, including anodic oxidation (AO) in MB removal. The results show that MB RE% by AO was 37.53% at the end of 30 min while EF achieved 97.73% over the same period. This result is in agreement with the findings reported by (Fayazi & Ghanei-Motlagh, 2020) which used graphite as the cathode and Pt as the anode for removing MB by AO and under the same conditions, used sepiolite/pyrite (Sep/ FeS_2) nanocomposite as Fenton catalyst for MB removal under EF. The removal efficiencies were 21.4% and 96.6% of MB removal by AO and EF, respectively. This performance may be explained by the mechanism of AO in which

pollutant degradation occurs on the anode surface, governed by mass transfer toward the anode and oxidant generation, depending on the anode type and applied current density (CD) (Hameed & Salman, 2025).

To highlight the capability of the NiFeCo foam cathode in enhancing MB degradation by EF, Table 7 summarizes the results reported by earlier studies for degradation of MB by EF compared with present the study. The comparison clearly demonstrates the superiority of the foam alloy electrode over its counterparts in previous studies. This superiority is evident in its ability to handle higher contaminant concentrations (100 mg/L), require a shorter treatment time (30 min) and achieve a high dye removal rate

(97.73%). This enhanced performance may be ascribed to the synergistic catalytic activity and porous structure of the foam alloy, which facilitate oxidant generation and mass transfer during the EF process.

6. Conclusion

The electro-Fenton (EF) process, which utilizes porous graphite as the anode and NiFeCo foam as the cathode, was successfully applied in this study to eliminate methylene blue (MB) dye from aqueous solution. The impacts of current density, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ concentration, and reaction time on the system's energy consumption and decolorization efficiency were efficiently estimated and optimized using response surface methodology (RSM). The EF process achieved a high removal efficiency of 97.73% for MB dye with an energy consumption of 11.76 kWh/kg MB under ideal conditions, which consisted of a current density of 8 mA/cm², $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ concentration of 0.5 mM, and a reaction time of 30 min. The suggested quadratic model was statistically significant, which was confirmed by an ANOVA analysis where the *p*-value less than 0.0001. The high *R*² value of 99.59% supports the accuracy of the RSM model in predicting the effects of process parameters on removal efficiency and energy consumption. The $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ concentration and current density had the greatest effects on removal efficiency among the examined parameters due to the strong oxidation reaction generated throughout the process, the NiFeCo foam alloy and porous graphite demonstrated greater degradation efficiency and lower energy demand compared with results from previous investigations. Thus, the electro-Fenton process with porous electrodes is a secure, economical, and highly effective method to remove MB dye from wastewater, providing a viable option for long term wastewater treatment applications.

7. Abbreviations

Abbreviation	Full Term
EF	Electro-Fenton
RSM	Response Surface Methodology
MB	Methylene Blue
DOE	Design of Experiment
BBD	Box–Behnken Design
XRD	X-Ray Diffraction
EDX	Energy Dispersive X-Ray Spectroscopy
AO	Anodic-oxidation

8. CRediT Statement

Hind H. Thwaini: writing - original draft preparation, investigation, data analysis, Methodology, Software, Data curation, Format analysis.

Rasha H. Salman: conceptualization and reviewing and editing.

Zahraa M. Hameed: reviewing and editing.

Khalid Abed: reviewing and editing.

Sunarti Abd Rahman: reviewing and editing.

Wan Jeffrey Basirun: reviewing and editing.

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