



Performance of Electro-Fenton Process for Phenol Degradation Using Nickel Foam as a Cathode

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Abstract

Toxic substances have been released into water supplies in recent decades because of fast industrialization and population growth. Fenton electrochemical process has been addressed to treat wastewater which is very popular because of its high efficiency and straightforward design. One of the advanced oxidation processes (AOPs) is electro-Fenton (EF) process, and electrode material significantly affects its performance. Nickel foam was chosen as the source of electro-generated hydrogen peroxide (H_2O_2) due to its good characteristics. In the present study, the main goals were to explore the effects of operation parameters ($FeSO_4$ concentration, current density, and electrolysis time) on the catalytic performance that was optimized by response surface methodology (RSM). According to the results, nickel foam made an excellent choice as cathode material. The pH value was adjusted at 3 and the airflow at 10 L/h for all experiments. It was found that the optimal conditions were current density of 4.23 mA/cm², Fe^{2+} dosage of 0.1 mM, and time of 5 h to obtain the removal rates of phenol and chemical oxygen demand (COD) of 81.335% and 79.1%, respectively. The results indicated that time had the highest effect on the phenol and COD removal efficiencies, while the impact of current density was the lowest. The high R^2 value of the model equation (98.03%) confirmed its suitability.

Keywords: Electro-Fenton; nickel foam; phenol; hydroxyl radical; response surface methodology.

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1- Introduction

Phenol is one of the major highly toxic contaminants that can be detected in many chemical and biochemical businesses and many industrial activities like oil refineries, petrochemical, coal conversion activities, medicines, pesticides, dyes, plastics, explosives, herbicides, and the manufacture of phenolic resin [1, 2]. Phenolic compounds are among the substances that warrant the most concern due to their poor biodegradability, the tendency to remain in the environment for extended periods, ecological implications, and hazardous consequences [3]. Several aromatic and aliphatic intermediates, which are more toxic than phenol, are produced during the electrochemical oxidation of phenol [4]. The health of humans and all other living beings is greatly influenced by water quality [5]. Even at low doses, the presence of phenol in drinking water and irrigation poses serious health risks to humans as a probable carcinogen [6, 7]. Since phenol and phenolic compounds are classified as priority pollutants by the United State Environmental Protection Agency (USEPA). Effluents containing such toxic contaminants must be removed or degraded before being released into the environment. According to strict USEPA standards, it is necessary to reduce the amount of

phenol in contaminated effluents to less than one mg/l [8, 9].

Numerous studies have been conducted to reduce and even eliminate some undesirable compounds from wastewater by many approaches like coagulation, adsorption, oxidation, biological processing, and electrochemistry [10]. Advanced oxidation processes (AOPs) such as Fenton and electro-Fenton are successfully used for the removal of phenol [11]. The electro-Fenton procedure is the most well-known EAOP method compared to regular techniques for treating and eliminating organic compounds [12], and it is an efficient oxidation process that has attained a great deal of interest [13].

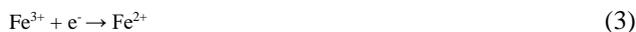
EF process has been used extensively in the treatment of wastewater because of the relatively simple handling of the reagents required for this procedure and the effectiveness that can be obtained at a cheap cost [14]. EF is one of the most important AOPs because it reduces the cost and risk of transporting hydrogen peroxide (H_2O_2) when compared to other methods. The power used for the EF process is pure and pollution-free; it also does not produce secondary pollutants [15]. AOPs have gained popularity in physic-chemical systems because they enable the production of hydroxyl radicals (OH^*) which



are non-selective oxidants that can react with several organic molecules very quickly [16].

The oxidation mechanism of Fenton process has been investigated for about 90 years. More than 20 chemical reactions are involved in the Fenton process [17]. Researchers have been studying the classic Fenton reaction, which is produced by combining H_2O_2 and a soluble Fe(II) salt, till the end of the nineteenth century [18]. When the environment is acidic, most organic materials can be non-selectively oxidized by the homogeneous Fenton reaction to produce hydroxyl radicals (OH^\cdot), which are harmless byproducts [19].

The cathodic production of H_2O_2 is widely recognized as the main mechanism in the EF process [20]. It depends on the electrochemical production of H_2O_2 (Eq. 1) over an extended period at a suitable cathode by the reduction of dissolved oxygen or air with adding an iron catalyst (Eq. 2) to the treated solution. Fenton's reaction produces OH^\cdot at the bulk solution, which is one of the most effective oxidizing agents, and it is the basis of the main mechanism of a Fenton system [21]. A homogeneous Fenton reaction (Eq. 2) in a Fenton system produces OH^\cdot which is the starting reaction for the subsequent chain Fenton reactions [22]. Hydrogen peroxide (H_2O_2) and a metal catalyst, such as (Fe^{2+}) transfer electrons in the EF process to produce the hydroxyl radical (OH^\cdot) [23].



Graphite is considered an ideal anode due to its low cost, and high energy density [24]. Also, due to their high surface area per unit volume, carbonaceous electrodes are frequently used as anodes in wastewater treatment [25]. Furthermore, nickel foam was a viable alternative to the production of H_2O_2 because its accumulation at nickel foam cathode was five times higher than that of graphite electrode, besides its superior advantages due to its porous structure and high conductivity. Faster electrons transfer in this electrode and generate O_2 on its surface, more H_2O_2 in the system is then produced which means more OH^\cdot would be formed [26]. The present work investigated the electrochemical Fenton technique for phenol removal from an aqueous solution using an electrochemical cell with nickel foam as a cathode and graphite as an anode and the result is optimized using a Box-Behnken design (BBD) based on the response surface methodology.

2- Experimental Work

2.1. Chemicals

All chemicals utilized in this study were of high purity and there was no need for any further purification. Phenol crystals (with 99.5% purity, Alpha Chemical Reagent Company, India), Sodium Sulfates (Na_2SO_4) (with purity

$\geq 99.0\%$, SDFCL). Ferrous sulfate heptahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) (CDH, Company), and sulfuric acid (H_2SO_4) (with 98% purity, Sigma-Aldrich) were the utilized chemicals. Deionized water was used to prepare the aqueous solutions. Nickel foam was purchased from (Xiamen Top New Energy Technology, China).

2.2. Experimental Methods

Batch reactors are more suitable for laboratory studies and pilot studies with a low working volume of the reactor [27]. Each batch experiment was performed in a glass reactor of 1.0 L filled with aqueous solution with 150 mg/l of phenol. A 10 mm thick Perspex cover was used with holes, two holes for firming the two electrodes and three holes for inserting the thermometer, airflow, and sample extraction. A nickel foam plate (140 mm \times 60 mm \times 10 mm) was used as the cathode and a graphite plate (140 mm \times 60 mm \times 5 mm) as the anode, and the distance between the electrodes was 30 mm. The nickel foam was cleaned with 0.1 M H_2SO_4 and de-ionized water to remove any oxides on the surface. The aqueous solution was continuously mixed at 250 rpm by a magnetic stirrer (Heidolph).

Air pump (ACO-001 electromagnetic, China) was used to aerate the solution with air at a rate of 10 L/h for 20 minutes before starting any run and continued until the end of the experiment. A glass tube attached to the diffuser circulates air through the solution. Digital (DC) power supply (0–30 V, 0–5 A) type (UNI-T, UTP3315PE) was utilized to provide constant current density. The required amount of Na_2SO_4 (0.01 M) was added to support the electrolyte and improve its conductivity. The catalyst amount was added to provide Fe^{2+} needed for the reaction. The pH of the solution was adjusted at 3 (with 0.1 M H_2SO_4 or 0.1 M NaOH) and was measured by a pH meter (HANNA, Romania), and the temperature of the electrolytic solution was kept constant at $27 \pm 1^\circ\text{C}$. Samples were analyzed to determine phenol concentration and the COD by the UV-9200 spectrometers, and RD125, Lovibond, respectively.

The simulated wastewater was prepared according to the following procedures: at 27°C , 150 mg/l of phenol was dissolved in 1.0 L of de-ionized water, and an amount of ferrous sulfate heptahydrate was added, the solution mixed for 15 minutes to release iron ions, then 0.01 M Na_2SO_4 was added. The solution's pH was adjusted at 3, and before each analysis, samples were withdrawn and filtered through a 0.45μ filter paper. A schematic drawing of the Electro-Fenton system is shown in Fig. 1. Based on Eq. 4, the effectiveness of phenol elimination was determined [28]:

$$\text{Re}\% = \frac{C_0 - C_f}{C_0} \times 100 \quad (4)$$

Where C_0 denotes the initial phenol (mg/l), C_f denotes the final phenol (mg/l), and Re% denotes the removal effectiveness. Energy consumption (EC) measures how much energy is used during the process of digesting one

kilogram of phenol. EC in (kWh/kg phenol) can be obtained by applying Eq. 5.

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

Where I is the operating current intensity, V is the volume of the solution, t is the electrolysis time, and Δphenol is the experimental phenol decay in solution during the experiments [28].

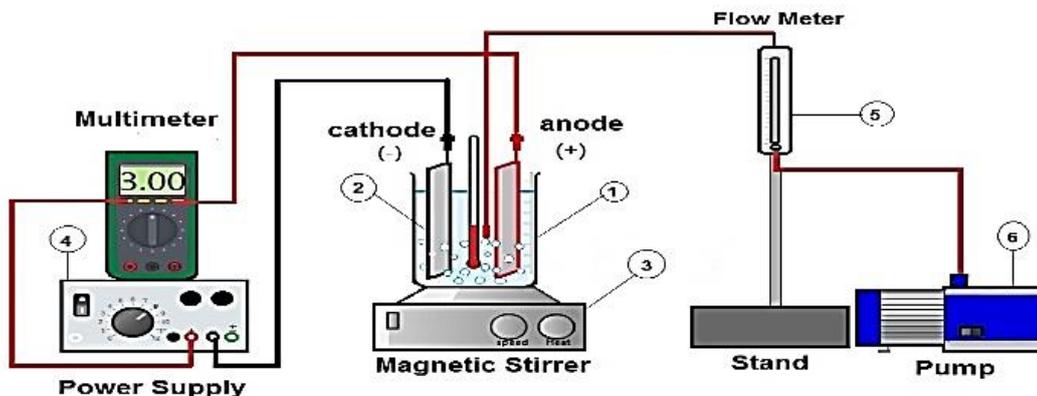


Fig. 1. Schematic Diagram of the Electrochemical System: (1) Cell, (2) Nickel Foam Cathode, (3) Magnetic Stirrer, (4) Power Supply, (5) Flow Meter, (6) Air Pump

2.3. Characterization of Electrodes

Graphite and nickel foam were utilized as anode and cathode, respectively. X-ray diffractometer (XRD) was used to determine the structural characteristics of these electrodes. The device characteristics were as follows: Model: XRD 6000, Shimadzu, Japan. The X-ray tube was operated at a scan speed of 5 degrees per minute using a 40 kV voltage and 30 mA current with $\text{CuK}\alpha$ radiation as the X-ray source. The electrodes' surfaces were examined using a scanning electron microscope (SEM), FEI-Company, Netherlands, Inspect S50 at 25Kv and 100 microamperes. The Energy Dispersive X-Ray Spectroscopy (EDX) from Bruker Company/Germany, XFlash-6110, 25Kv, 100 μA .

2.4. Design of experiment

It is possible to conclude the relationship between a process response and its factors through mathematical and statistical data collection using RSM [31]. In this study, three levels and three factor parameters that controlled the removal of phenol were verified and tested using the Box-Behnken experimental design (BBD) [32]. $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (X1), current density (X2), and time (X3) were chosen as process variables, while the response was the phenol removal efficiency. Each symbol has three levels of numbers to represent process variable scales. These numbers are -1 indicating the low level, 0 indicating the center point, and 1 indicating the high level. In BBD, the experimental points are distributed evenly around the central point on a hypersphere. The Minitab-18 software was utilized to analyze the results of phenol removal efficiency (Re%). Table 1 shows the experimental parameters with their levels. Table 2 shows the

experiments which would be accomplished according to the BBD.

Table 1. Process Factors and their Levels for Phenol Removal

Parameters and symbol	Range in Box-Behnken designs		
	Low (-1)	Center (0)	High (1)
X1- $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, mM	0.1	0.15	0.2
X2- Current density, mA/cm^2	2.5	3.5	4.5
X3- Time, h	3	4	5

Table 2. Box-Behnken Designs for Phenol Removal

Run	Bk.	Coded value			Real value		
		X1	X2	X3	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, mM	Current density, mA/cm^2	Time h
1	1	-1	1	0	0.10	4.5	4
2	1	1	-1	0	0.20	2.5	4
3	1	1	1	0	0.20	4.5	4
4	1	0	0	0	0.15	3.5	4
5	1	-1	0	-1	0.10	3.5	3
6	1	0	1	1	0.15	4.5	5
7	1	0	0	0	0.15	3.5	4
8	1	1	0	-1	0.20	3.5	3
9	1	0	-1	-1	0.15	2.5	3
10	1	0	-1	1	0.15	2.5	5
11	1	0	1	-1	0.15	4.5	3
12	1	0	0	0	0.15	3.5	4
13	1	-1	0	1	0.10	3.5	5
14	1	1	0	1	0.20	3.5	5
15	1	-1	-1	0	0.10	2.5	4

The empirical quadratic polynomial model depicted in Eq. 6 can represent the mathematical relationship between independent factors and response [33].

$$Y = \alpha_0 + \sum \alpha_i x_i + \sum \alpha_{ii} x_i^2 + \sum \alpha_{ij} x_i x_j \quad (6)$$

Where Y represents the response (Re%), i and j are the index numbers for independent variables, α_0 is the intercept term, $x_1, x_2 \dots x_k$ are the process variables (independent variables) in coded form. α_i is the first order (linear) main effect, α_{ii} second-order main effect, and α_{ij} is the interaction effect. Analysis of variance was performed [34].

3- Results and Discussion

3.1. Characterization of graphite and Ni foam

The XRD patterns of the graphite and nickel foam are presented in Fig. 2. The XRD pattern of graphite shows a sharp and tight peak at $2\theta = 26.5^\circ$ which corresponds to the diffraction line C (002) indicating the presence of carbon only [29]. While the Ni foam peaks are centered at about $2\theta = 44.5^\circ, 51.9^\circ,$ and 76.4° [30].

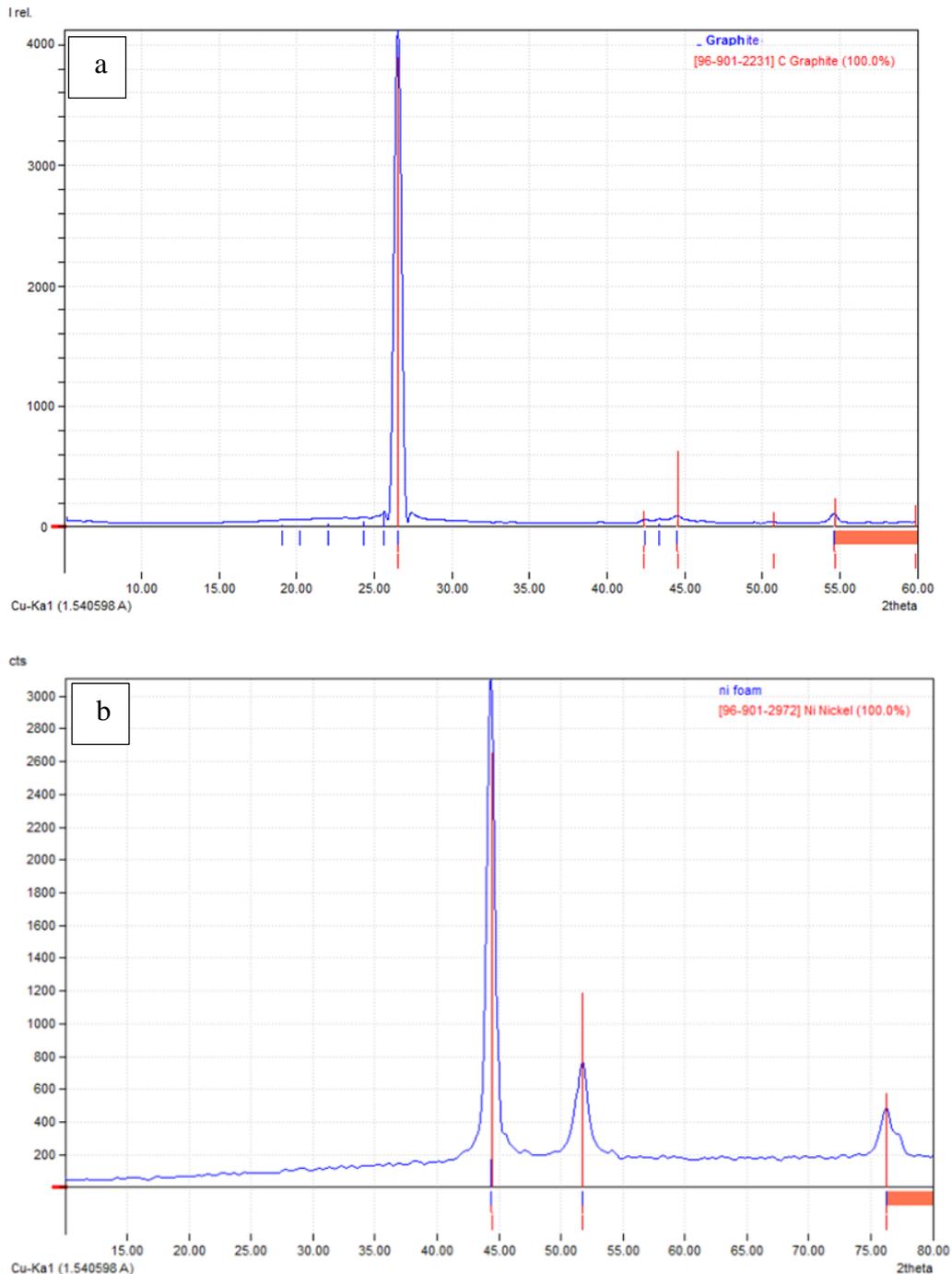


Fig. 2. XRD Pattern for (a) Graphite and (b) Nickel Foam

Fig. 3 a and b show the SEM of graphite and nickel foam, respectively. The SEM images of graphite illustrate that its surface was semi-porous and flaky, while the nickel foam has a typical porous structure with a high surface area. Fig. 3 b shows that nickel foam maintained its three-dimensional porous structure. In the presence of Ni foam, phenol was effectively removed, and the chemical reaction caused a significant variation in the surface shape.

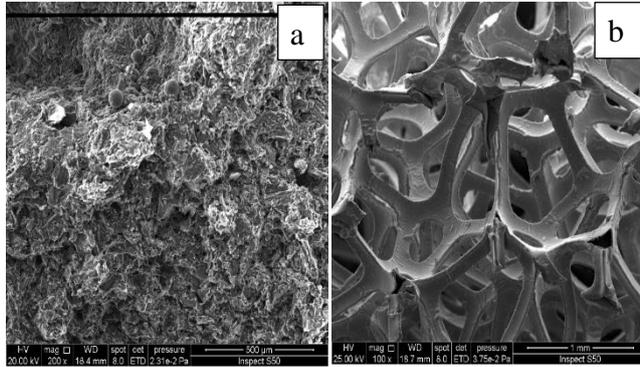


Fig. 3. SEM of (a) Graphite (with Zoom of 500µm) (b) Nickel Foam (with Zoom of 1 mm)

The results displayed in Fig. 4 show the presence of pure carbon due to the graphite and Ni, C, O, and N due to the Ni foam.

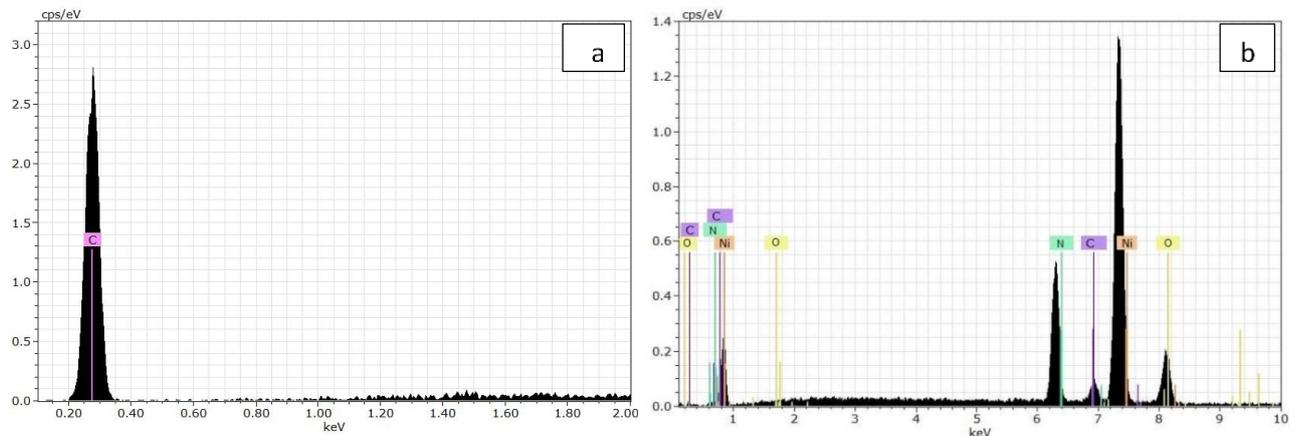


Fig. 4. EDX of (a) Graphite, (b) Nickel Foam

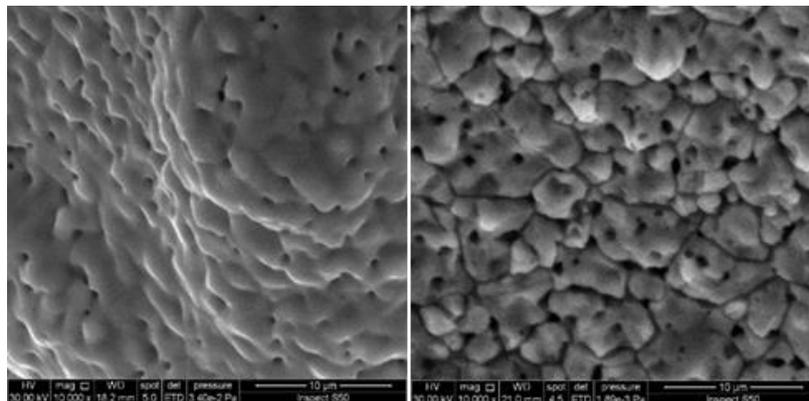


Fig. 5. SEM of Nickel Foam (a) before Reaction, (b) after Reaction

Fig. 5 a, and b represented the surface of nickel foam before and after the reaction. The original Ni foam featured a smooth surface in a microscale image (Fig. 5 a). The imperfections on the Ni foam's surface also show that nickel leached throughout the reaction, which was consistent with the removal efficiency being increased [35]. On the other hand, the increase in the electrode surface area is very important in the production of the Fenton reagent. By improving the conductivity and specific surface of the cathode, the production of hydroxyl radicals can be increased which ultimately improved the removal of pollutants [36].

3.2. Statistic evaluation

Fifteen batch runs were carried out at various process factor combinations to be optimized and to examine the combined effects of the independent parameters on the phenol removal efficiency. The experimental values of Re% and energy consumption results are shown in Table 3. The Minitab-18 program was used to assess the results of Re% and the following quadratic model of Re% in terms of coded units of process parameters was obtained:

$$\text{Phenol Re \%} = -25.0 + 379 X1 + 35.8 X2 - 13.2X3 - 959 (X1)^2 - 6.31 (X2)^2 + 1.27 (X3)^2 - 47.8 X1X2 - 0.3 X1X3 + 4.50 X2X3. \quad (7)$$

The predicted values of Re% based on Eq. 7 are summarized in Table 3.

Table 3. Experimental Result of Box-Behnken Design for Phenol Removal

Run	Bk.	FeSO ₄ .7H ₂ O, mM	Current density, mA/cm ²	Time, h	Actual Phenol Re%	Predicted Phenol Re%	E, volt	EC, (kWh/kg phenol)
1	1	0.10	4.5	4	65.300	63.5464	5.71	67.35
2	1	0.20	2.5	4	49.119	50.8726	3.93	29.44
3	1	0.20	4.5	4	52.770	51.0759	5.83	82.49
4	1	0.15	3.5	4	62.767	63.5263	4.14	38.78
5	1	0.10	3.5	3	52.433	53.5761	4.01	33.72
6	1	0.15	4.5	5	75.300	78.1373	5.85	72.50
7	1	0.15	3.5	4	64.240	63.5263	4.12	37.71
8	1	0.20	3.5	3	44.830	45.9136	3.94	38.75
9	1	0.15	2.5	3	50.680	47.8427	3.99	27.17
10	1	0.15	2.5	5	64.760	64.1495	3.85	31.70
11	1	0.15	4.5	3	43.210	43.8205	5.77	74.77
12	1	0.15	3.5	4	63.572	63.5263	4.31	39.86
13	1	0.10	3.5	5	80.000	78.9164	4.16	38.22
14	1	0.20	3.5	5	72.340	71.1969	4.23	42.97
15	1	0.10	2.5	4	52.090	53.7841	3.76	30.79

Based on the experimental results, the effectiveness of phenol removal ranged between 43.21– 80.0%, while the specific energy consumption was 27.18–82.49 kWh/kg phenol. The comparison between runs 5 and 13 demonstrates that the impact of time on phenol removal was superior. Phenol removal efficiency increased from 52.433% to 80.00% as time increased from 3 to 5 h at a constant current of 3.5 mA/cm² and 0.1 M of FeSO₄.7H₂O. While the comparison between runs 13 and 14 indicated that phenol removal efficiency increased from 72.340% to 80.00% as FeSO₄.7H₂O concentration increased from 0.1 to 0.2 mM at a constant current of 3.5 mA/cm² and 5 h of electrolysis time indicating FeSO₄.7H₂O concentration had the second influence on phenol removal.

Comparison between runs 1 and 15 showed that when the current density increased from 2.5 to 4.5 mA/cm², the phenol removal efficiency increased from 52.09% to 65.30%. Analysis of variance (ANOVA) results gave precise impact of these variables, and their interactions can be easily predicted.

ANOVA is an analytical method that estimates the significance of the model and its parameters by Fisher's (F)-test and P-test. In general, large F-values (higher than 4) and low p-values (lower than 0.05) imply the importance of the coefficient terms [37] [38] [39]. The results of ANOVA are shown in Table 4. Based on the results of ANOVA, time had the main impact on phenol removal with contr.% of 72.72 to the overall effect. The significance of FeSO₄.7H₂O concentration had a contr.% of 6.71. Current density had the lower effect on phenol removal with cont.% of 2.87. Based on the results of the P-value for the model (0.001 < 0.05), high value of F (27.58 > 4), and high value of the multiple correlation coefficient (R²) for the model (98.03%), it can be emphasized that the regression is statistically significant.

3.3. Main effect plot

The term "main effect" refers to the effect of a parameter on the process performance [40]. The ANOVA

analysis and the model regression clearly showed that time had the main impact on phenol removal efficiency, and this can be demonstrated from the main effect plot as shown in Fig. 6. The experimental results showed that the reduction of phenol increased when time increased. FeSO₄.7H₂O concentration decreased with an optimum value of current density of 3.5 mA/cm² to attain the highest value of phenol removal efficiency.

3.4. Effect of process parameters on the Phenol removal

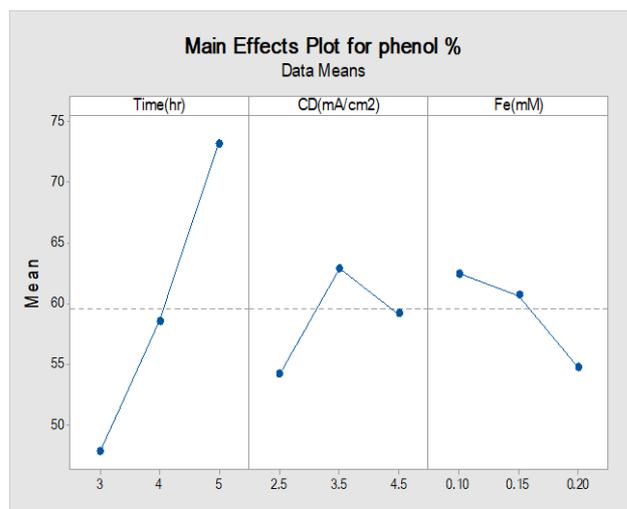
To investigate the interactions between operating parameters on the phenol removal efficiency more thoroughly, a contour three-dimensional response surface and two – dimensional plots were used as shown in Fig. 7 to Fig. 9. Fig. 7 shows the effect of time on the phenol removal efficiency over a range of current densities of 2.5– 4.5 mA/cm² at a constant FeSO₄.7H₂O concentration of 0.15 mM and time of 5h. Fig. 7 shows the contour, 3D response surface, and the 2D response surface plots. According to the contour plot shown in Fig. 7 a, the removal efficiency greater than 75% was obtained only within a narrow range of current density (3.4–4.5 mA/cm²) within an electrolysis time of 4.7–5 h. From the surface plot, at time = 3 h, a noticeable decrease in phenol removal efficiency occurred. While at a higher value of time = 5 h, the efficiency of phenol removal increased linearly with a current density of 2.5–3.5 mA/cm² and this result agreed with a previous study [41]. As shown in Fig. 7 b, the efficiency of phenol removal increased gradually as the current density raised from 2.5–4.5 mA/cm² and this result also can be detected clearly from the 2D plots. Current density had a positive effect on the phenol removal by the E-Fenton process, which could be explained by the fact that the current drives the reduction of oxygen on the cathode surface which resulted in producing H₂O₂. So, increasing the current density would result in more hydroxyl radicals because of the increased reaction of H₂O₂ with ferrous ions [42].

Table 4. ANOVA for Phenol Removal

Source	DF	Seq. SS	Contr. %	Adj. SS	Adj.MS	F-value	P-value
Model	9	1727.22	98.03%	1727.22	191.91	27.58	0.001
Linear	3	1449.33	82.25%	1449.33	483.11	69.44	0.000
X1	1	118.30	6.71%	118.30	118.30	17.00	0.009
X2	1	49.66	2.82%	49.66	49.66	7.14	0.044
X3	1	1281.37	72.72%	1281.37	1281.37	184.18	0.000
Square	3	173.96	9.87%	173.96	57.99	8.33	0.022
X1X2	1	15.49	0.88%	21.21	21.21	3.05	0.141
X2X2	1	152.50	8.66%	147.01	147.01	21.13	0.006
X3X3	1	5.97	0.34%	5.97	5.97	0.86	0.397
2-Way Interaction	3	103.93	5.90%	103.93	34.64	4.98	0.058
X1X2	1	22.84	1.30%	22.84	22.84	3.28	0.130
X1X3	1	0.00	0.00%	0.00	0.00	0.00	0.992
X2X3	1	81.09	4.60%	81.09	81.09	11.66	0.019
Error	5	34.79	1.97%	34.79	6.96	-	-
Lack-of-Fit	3	33.70	1.91%	33.70	11.23	20.65	0.047
Pure Error	2	1.09	0.06%	1.09	0.54	-	-
Total	14	1762.00	100.00%	-	-	-	-
Model Summary			S	R ²	R ² (adj.)	Press	R ² - (pred.)
			2.63765	98.03%	94.47%	541.614	69.26%

Fig. 8 depicts the impact of time on the phenol removal efficiency over a range of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ concentration at a constant current density of 3.5 mA/cm^2 . The efficiency increased linearly with time and exponentially with $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ concentration, and the contour plot shows that a small area of high removal efficiency can be obtained at $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ concentration of 0.1-0.17 mM and the time of 4.7 and 5h. The 3D response surface and 2D plots show that the increase in phenol removal efficiency at $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ concentration of 0.1 mM gave the optimum value of iron, and this result agrees with a previous study [43, 44]. According to Faraday's law, electrolysis time is an essential factor, and it influences the generation rate of active radicals that are responsible for reducing the pollutants [45].

Fig. 9 shows the relation between current density and iron dosage on the phenol removal efficiency at a constant electrolysis time of 4 h. The phenol removal efficiency increased as $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ concentration decreased from 0.2 to 0.1 mM, and the counterplot shows the highest removal efficiency at 0.1 and 0.16 mM of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ concentration and at 3.5 to 4.4 mA/cm^2 of current density. The higher values of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ dosage resulted in early high decomposition rate of H_2O_2 in the electrolytic solution which caused self-reaction of OH^\cdot , and quickly consume Fe^{+2} as shown in Eq. 8 [46]. Therefore, the removal efficiency of phenol decreased with increasing $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ concentration.

**Fig. 6.** Main Effect Plot of Phenol Removal Efficiency

3.5. The optimization and confirmation test

The electrochemical system must be improved to reduce energy losses. Many elements should be considered when optimizing the process to maximize the desirability function and achieve the desired goal [47]. The study's goal is to maximize phenol removal. As illustrated in Table 5, the response surface methodology can detect the optimal values of the influencing parameters. Two confirmation experiments were performed under optimal conditions, and the average phenol removal effectiveness was 81.335% as indicated in Table 6, with a COD value of 79.1%.

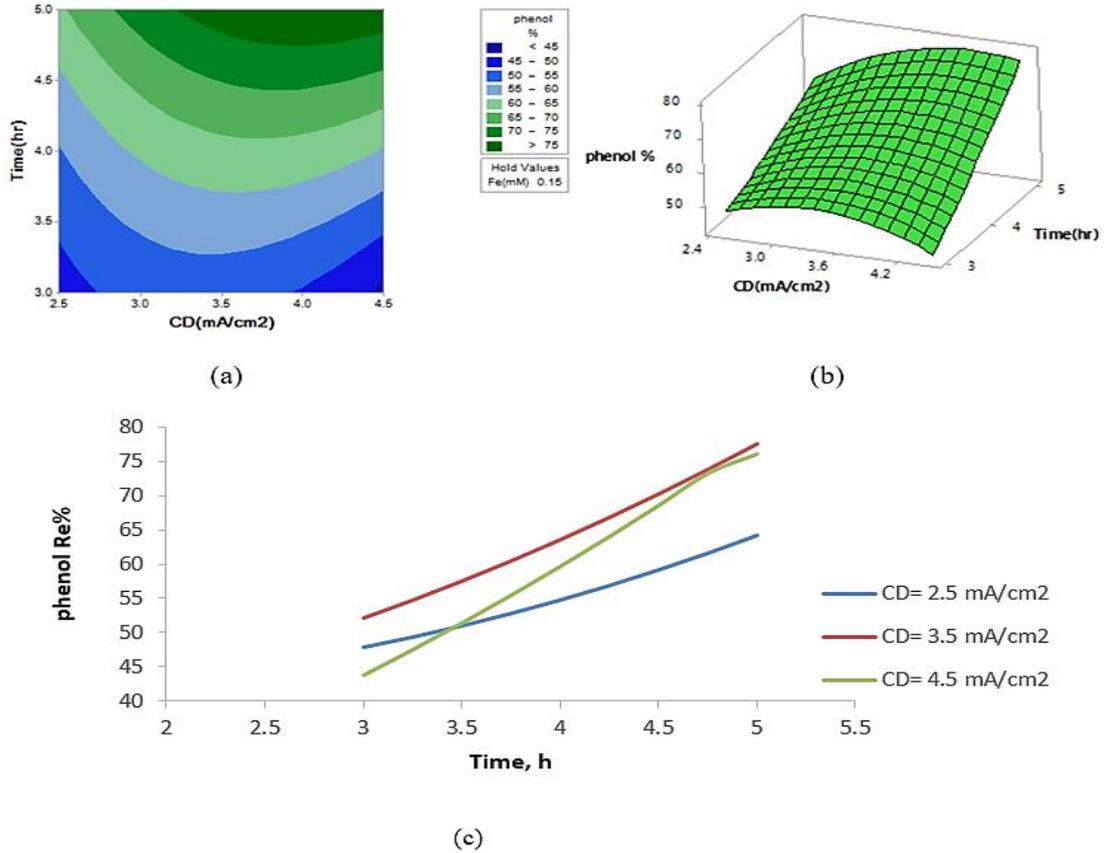


Fig. 7. (a) Contour Plot (b) 3D Plot (c) 2D Plot for Phenol Re % at $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ Concentration = 0.15mM, and Different Current Densities and Electrolysis Time

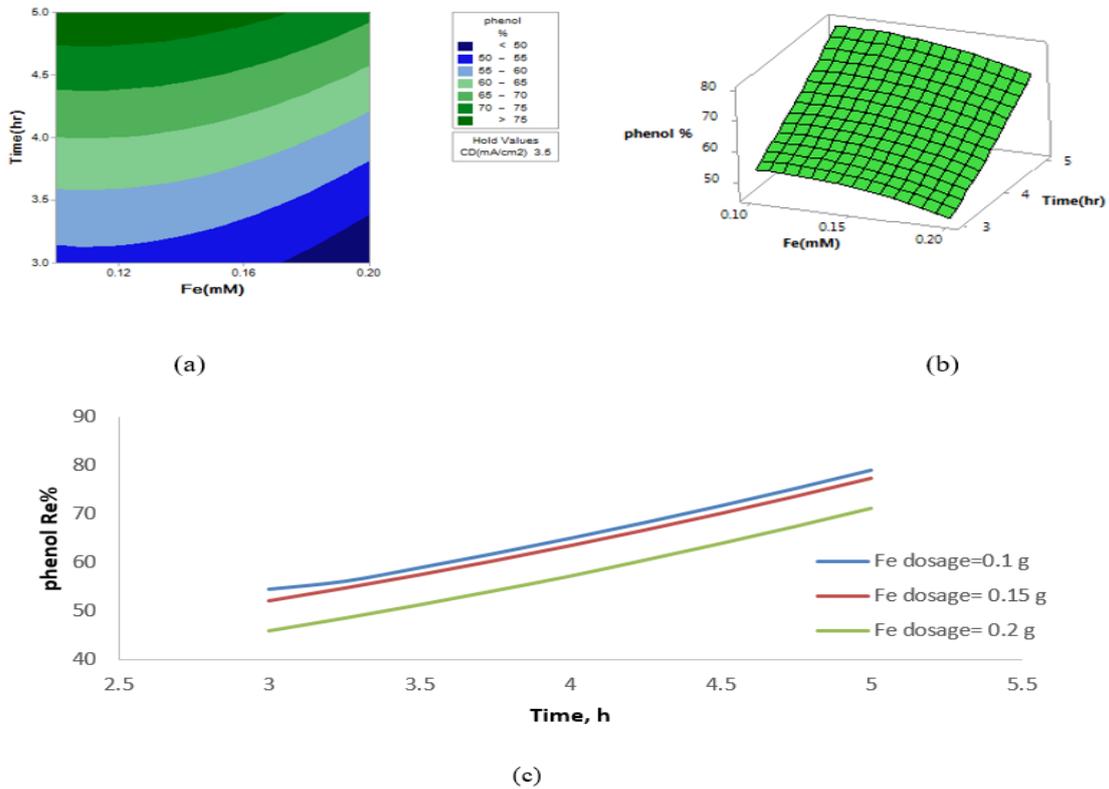


Fig. 8. (a) Contour Plot (b) 3D Plot (c) 2D Plot for Phenol Re%, Current Density = 3.5mA/cm², and Different Electrolysis Time and $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ Concentration

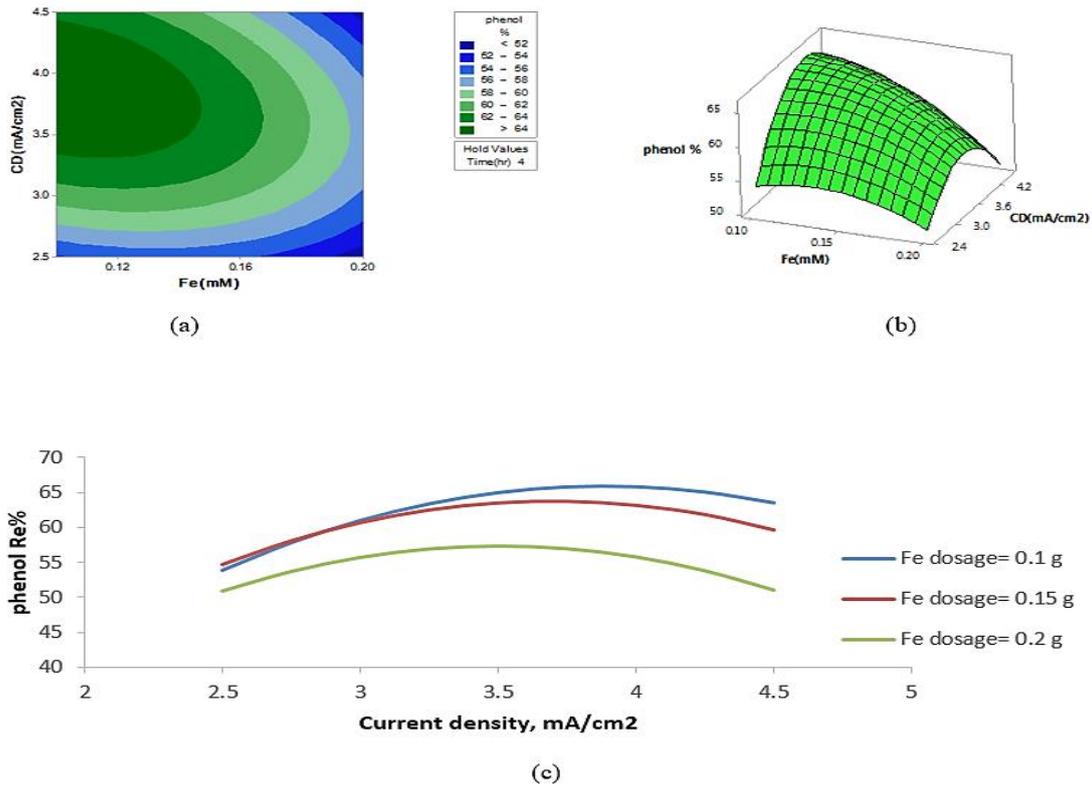


Fig. 9. (a) Contour Plot (b) 3D Plot (c) 2D Plot for Phenol Re%, Electrolysis Time = 4 h, and Different Current Density and FeSO₄.7H₂O Concentration

Table 5. Optimal Performance of System Variables for the Maximum Removal of Phenol

Response	Goal	Lower	Target	Upper	Weight	Importance	
Phenol%	Maximum	43.21%	80		1	1	
Solution of parameters			Multiple response Prediction				
FeSO ₄ .7H ₂ O, mM	Current density, mA/cm ²	Time, h	Phenol Re% Fit	SE Fit	95% CI	95% PI	Composite Desirability
0.1	4.23737	5	82.40	2.68	(75.51; 89.30)	(72.73; 92.07)	1

Table 6. Confirmation Experiments of Phenol Removal

Run	FeSO ₄ .7H ₂ O, mM	Current density, mA/cm ²	Time, h	E, volt	EC, kWh/kg phenol	Phenol Re% actual	Average
1	0.1	4.23000	5	4.23	46.03	81.62%	81.335%
2	0.1	4.23000	5	4.18	45.49	81.05%	

4- Conclusion

For modeling and optimizing phenol removal, a three level Box-Behnken design was used, with three variables; current density, electrolysis time, and FeSO₄.7H₂O concentration. Nickel foam was used as a cathode to provide a high surface area and increase phenol removal efficiency. The model's validity was confirmed by the P and F-values which showed that the overall acceptability model was significant. This model provided a good R² value, equal to 98.03%. The best phenol removal efficiency was acquired at 0.1 mM of FeSO₄.7H₂O concentration, 5 h of electrolysis time, and 4.237 mA/cm² of current density. These optimum conditions gave phenol

removal efficiency of about 81.335% and COD of 79.1%. Based on the results of ANOVA, the time has the main effect on the process efficiency and nickel foam was an efficient electrode.

Nomenclature

Nomenclature	Meaning	Unit
Y	Response (Re%)	----
ai	First-order (linear) main effect	----
aii	Second-order main effect	----
aij	Interaction effect	----
a0	Intercept term	----

CD	Current density	mA/cm ²
COD	Chemical Oxygen Demand	ppm
C _f	Final phenol concentration	ppm
C ₀	Initial phenol concentration	ppm
H ₂ O ₂	Hydrogen peroxide	mM
E	Voltage of cell	Volt
Fe ²⁺	FeSO ₄ .7H ₂ O concentration	mM
I	Operating current intensity	A
V	Volume of the solution	L
EC	Energy consumption	kWh/kg phenol
t	Time	h
RE%	Removal efficiency	%
X1	FeSO ₄ .7H ₂ O concentration	mM
X2	Current density	mA/cm ²
X3	Time	h

Symbol Definition

Adj. MS	Adjusted Mean of the Square
Adj. R2	Adjusted Correlation Coefficient
Adj. SS	Adjusted Sum of the Square
R ²	Multiple correlation coefficient
ANOVA	Analysis Of Variance
BBD	Box-Behnken Design
DF	Degree Of Freedom
RSM	Response Surface Methodology
XRD	X-ray diffraction
EDX	Energy-dispersive X-ray spectrometry
SEM	Scanning electronic microscopy
UV	Ultraviolet-visible
OH [•]	Hydroxyl radical

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اداء عملية Electro-Fenton لتحلل الفينول باستخدام رغوة النيكل ككاثود

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الخلاصة

يتم إطلاق مواد سامة في إمدادات المياه في العقود الأخيرة نتيجة التصنيع السريع والنمو السكاني. عملية فنتون الكهروكيماوية قدمت لمعالجة مياه الصرف الصحي والتي تحظى باهتمام كبير بسبب كفاءتها العالية وتصميمها المباشر. إحدى عمليات الأكسدة (AOPs) هي Electro-Fenton، وتؤثر مادة القطب بشكل كبير على أدائها. تم اختيار النيكل الرغوي كمصدر H_2O_2 المتولد كهربائياً للكشف عن تأثير معاملات التشغيل (تركيز $FeSO_4$ ، كثافة التيار، ووقت التحليل الكهربائي) على الأداء الذي تم تحسينه من خلال منهجية سطح الاستجابة. وفقاً للنتائج، يعتبر النيكل الرغوي اختياراً جيداً للغاية كمادة الكاثود. تم ضبط قيمة الرقم الهيدروجيني عند ٣، وتدفق الهواء عند ١٠ لتر/ساعة وفضل ازالة للفينول كانت عند كثافة التيار ٤,٢٣ ملي أمبير/سم^٢، وكانت كمية Fe^{2+} ٠,١ ملي مولار، وزمن ٥ ساعة للوصول إلى معدلات إزالة الفينول والطلب الكيماوي للأوكسجين بنسبة ٨١,٣٣% و ٧٩,١% على التوالي. أشارت النتائج إلى أن الوقت كان له تأثير أعلى على كفاءة إزالة الفينول، بينما كان تأثير كثافة التيار أقل. أشارت قيمة R^2 العالية لمعادلة وصف النظام (٩٨,٠٣%) كتأكيد لمدى ملاءمتها.

الكلمات الدالة: Electro-Fenton، رغوة النيكل، الفينول، جذور الهيدروكسيل، منهجية استجابة السطح.