



REDUCING THE PHENOL IN AL-NAJAF PETROLEUM REFINERY WASTEWATER BY USING ELECTROOXIDATION TECHNIQUE

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ABSTRACT

Industrial effluents, hazardous biological waste, and wastewater from crude oil refining all contribute to water contamination. All of these pollutants are emitted into the environment and have emerged as a modern issue owing to their hazardous organic and inorganic constituents. This research aimed to decrease the concentration of organic phenol (C₆ H₅ OH), a contaminant found in the wastewater from the Najaf refineries in Iraq. Laboratory research used electrical approaches, including electro-oxidation (EO), which has shown significant efficacy in eliminating dissolved phenol. Graphite electrodes served as the anode in the electrolytic cell, while steel (SS) electrodes functioned as the cathode inside a tough plastic casing. The starting phenol content in the treated water was 50 ppm under the following parameters: electric current density (10, 15, 20) mA/cm², sodium chloride (NaCl) concentration (0, 1.5, 3) g/L, acidity (pH) (3, 7, 10), and duration (2–4 h). The results showed that the higher the current density and NaCl concentration, the better the removal rate in both strong and moderate acidity, with the best conditions for removal being a current density of 15 mA/cm², a pH of 3, a NaCl concentration of 3 g/L, and a duration of 3 hours. A removal rate of 96.3% was attained under the specified circumstances.

Keywords: phenol, wastewater, electrooxidation, treatment.

INTRODUCTION

Recent years have seen a notable rise in interest regarding the treatment of petroleum and petrochemical wastewater, attributable to the growth of industrial operations in these industries. The composition of refinery effluent depends on the quality of crude oil and the

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existing operational parameters. (Al-Tameemi, Sukkar, and Abbar 2024). found that the volume of oil effluent generated during crude oil processing ranges from 0.4 to 1.6 times the quantity of raw oil processed. It is well recognized that the discharge of oil sewage with a substantial concentration of organic matter into aquatic ecosystems requires 2 mg/L of dissolved oxygen for the proper survival of aquatic organisms. This problem leads to a reduction in dissolved oxygen levels as a consequence of bacterial activity (Dalanta and Kusworo 2022).

Phenols (phenol and phenolic compounds) are frequently found in aqueous effluents from diverse manufacturing processes, including oil refineries, petroleum refineries, gas and coke oven industries, pharmaceuticals, explosives production, plastic and varnish industries, pesticides, and phenolic resin facilities (Busca et al. 2008). Phenolic compounds, such as phenol formaldehyde resins or Bakelite, are noted for their significant stability and environmental pollution, possessing carcinogenic properties even at low concentrations. Consequently, they can induce acute toxicity, manifesting as sweating, cyanosis, loss of reflex activity, and death due to respiratory failure (Pimentel et al. 2008). Furthermore, phenol values above 10,000 mg/L have been seen in several industrial effluents (Busca et al. 2008). The Environmental Protection Agency (EPA) mandates a reduction of phenol concentration in wastewater to below 1 mg/L. The phenol-formaldehyde resin manufacturing industries produce wastewater with elevated levels of organic debris (Akyol et al. 2020).

PRW was addressed by many methodologies, including physical, chemical, and biological processes, such as flotation, filtration, sedimentation, coagulation/flocculation, adsorption, and ion exchange (CAMCIOĞLU et al. 2016). Nonetheless, using biological methods to decompose complex refractory organic pollutants in wastewater presents significant challenges (Camcioglu, Ozyurt, and Hapoglu 2017). The generation of additional pollution from unreacted chemicals and the difficulties in managing substantial quantities of hazardous sludge produced during traditional wastewater treatment indicate that physical-chemical methods are not consistently effective (Camcioğlu et al. 2017). Physical-chemical methods aren't always effective because they create more pollution when chemicals don't react and it's hard to handle large amounts of dangerous sludge that is made when wastewater is treated the old way (Tonini and Ruotolo 2017).

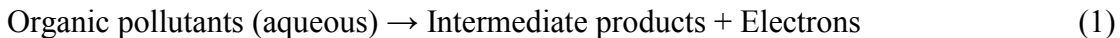
In contrast to the previously mentioned conventional procedures, electrochemical technology, including electrodeposition (Daghrir, Drogui, and Zaviska 2013). Electro disinfection (Ghernaout and Ghernaout 2010). Electro-Fenton (Nidheesh and Gandhimathi 2014). Electro sorption (Huang and Su 2010). Electro oxidation (EO) and Electrocoagulation (EC) (Hakizimana et al. 2017). The unique advantages of electrochemical treatment—namely, cost-effectiveness, energy efficiency, automation, and flexibility—have garnered more attention lately (Kariyajjanavar, Narayana, and Nayaka 2011).

EO process typically reduces pollutants in wastewater by mineralizing them via the action of free radicals generated inside the system. The generated hydroxyl radicals will target the contaminants in wastewater (Al-Tameemi, Sukkar, and Abbar 2024). EO operations may be categorized into direct and indirect processes (AlJaberi et al. 2023).

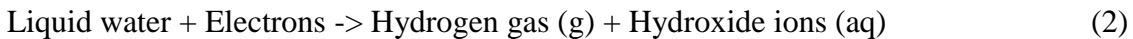
Presently, there is significant interest in EO as an effective method for eradicating various pollutants. EO technology is very reliable and often used owing to its efficacy and eco-friendly outcomes (Salvestrini et al. 2020). In actuality, pollutants are degraded in the direct electrooxidation process by direct electron transfer between the anode surface and the impurities (Radjenovic et al. 2020). In contrast, indirect electrolysis EO involves the uniform interaction of organic pollutants with strong oxidants produced during the electrolysis process, including Cl_2 , H_2O_2 , HClO , ClO^- , SO_4^{2-} , and O_3 (Najafinejad et al. 2023). The EO approach has many benefits, including the generation of disinfection chemicals, complete mineralization of persistent organic pollutants, straightforward equipment and operational requirements, and low electrode maintenance expenses (Özyurt and Camcıoğlu 2018). Still, the EO method has some problems, such as not being able to get rid of particles that are spread out and possibly losing its effectiveness if a layer of impermeable film forms on the cathode (Özyurt and Camcıoğlu 2018). The reactions below (5, 6, 7, 8, and 9) happen at the anode, the cathode, and in the bulk solution during the EO process. They happen through direct and indirect oxidation (Najafinejad et al. 2023):

1- Direct oxidation Reaction (Najafinejad et al. 2023)

Anodic reaction:



Cathodic reaction:

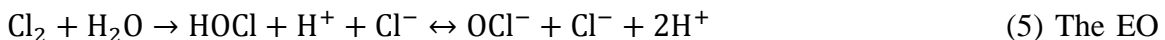


2- Indirect oxidation Reaction Involving the Addition of NaCl (Ibrahim 2022):

Anodic reaction



Cathodic reaction:



The EO method has many benefits, such as simple operating conditions and equipment, complete mineralization of organic contaminants, low electrode maintenance costs, and the production of compounds that kill microbes. Still, the EO method has some problems, such as not being able to get rid of particles in suspension and not working as well as it could because a resistive film layer forms on the cathode (Sirés et al. 2014).

Researchers have used EO in several research projects due to its advantages, including ease of use with basic instruments, utilization of clean reagents (electrons) without

additional chemicals, and its versatility in eliminating a broad spectrum of contaminants (Ahmad et al. 2021).

Specifically, this study looks at how well the electro-oxidation process works for treating the wastewater from the Najaf refinery in Iraq, with the goal of lowering the concentration of phenol. It also looks at how time, sodium chloride concentration, density, and pH affect how well the electro-oxidation process works. With this method, phenol was effectively removed while important factors for treating wastewater from crude oil refining were being identified.

METHOD OF SOLUTION FOR EO

80 liters of effluent from the crude oil refining process were extracted from the Najaf refinery and preserved in a laboratory refrigerator to maintain its characteristics shown in Table 1.

Table 1. Qualities of Wastewater.

Properties	Values
<i>COD (ppm)</i>	1340
<i>TDS (ppm)</i>	3400
<i>Turbidity(NTU)</i>	40.4
<i>POD (ppm)</i>	122.6
<i>Phenol (ppm)</i>	50
<i>Oil (ppm)</i>	30.6
<i>Cl⁻ (ppm)</i>	2340
<i>PO₄(ppm)</i>	0.15

Chemicals Used

1. HCl, conc. (36%), Thomas Indian Bakers in brands, has been used to create a (1M) solution for the purpose of modifying the initial pH of wastewater and also prepare 5% (v/v) for the purpose of cleaning and reactivating the electrodes.
2. NaOH, purity $\geq 97.0\%$, pellets, Thomas Indian Bakers in brands, , has been used to create a (5M) solution for the purpose of modifying the initial pH of wastewater.
3. NaCl, that has purity (99.9%), Barcelona, Spain, has been employed for the preparation of concentrated solutions with appropriate electrical conductivities.
4. Distilled water for clean.

Tools and Equipment

As shown in Fig. 1, 2 we conducted the experiments in a single-electrode electrochemical cell for electrochemical (EC) and electrooxidation (EO) in batch mode at ambient temperature. The cell was made from a plastic container with dimensions of 20 cm in length, 6 cm in width, and 15 cm in height. The cell was then placed on a magnetic stirrer functioning at 200 rpm (DAIHAN LABTECH CO, 0-450 rpm). The cathode electrodes in the electrochemical process included three sheets of 316-AISI stainless steel, whereas the anode electrodes consisted of two aluminum plates. In the electrochemical oxidation process, the anode electrodes were two graphite plates, and the cathode electrodes were three identical stainless-steel sheets. The electrodes measured uniformly at $18 \times 5 \times 0.3$ cm. The submerged dimensions of each side of the anode were 8×5 cm, yielding an effective surface area of 160 cm^2 . Each anode is positioned between two cathodes, with a separation of 2 cm, and the effective area of the aluminum and graphite electrodes has been calculated. The anode electrode, potentially composed of aluminum in the process or graphite in the electrolysis procedure, is connected to the positive terminal of a DC power supply (MCH-305 D-II, 0–30V, 0–5A dual output), while the stainless-steel cathodes are attached to the negative terminal. An RMS multimeter (UNI-T, UT803) is then connected in series with the anode. We conducted a number of exploratory tests to evaluate the range of objects under examination and to gain a comprehensive understanding of the proposed method. The investigations revealed that the EC procedure lasted around 1 hour, but the EO process required 2.5 hours. During the first hour of each run in the EC and EO tests, aluminum electrodes function as anodes in the electrochemical process. Subsequently, we substitute the aluminum electrodes with graphite electrodes and use the electrooxidation procedure for the remainder of the period. Samples were collected and filtered using Whatman filter paper and thereafter evaluated to establish therapeutic effectiveness concerning phenol.

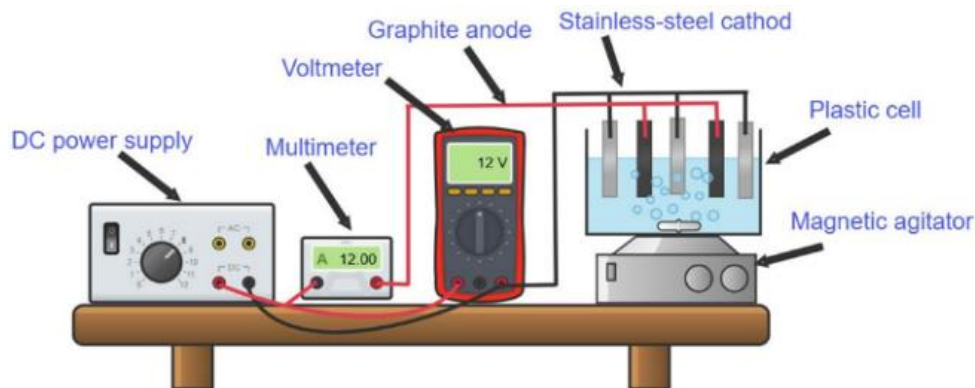


Figure 1. Diagram of the sequential EO process.

Measurement and Analysis Method

A phenol solution with a known concentration of 100 ppm was prepared. The material was examined using an ultraviolet (UV) light spectrometer to determine the wavelength of phenol. The results were then compared with those of a previous reference solution. Phenol was detected in a wastewater sample from the Najaf refinery. Several measurements of known concentrations were then performed to establish a calibration curve and phenol measurement equation. Three tests on the samples showed an S value of less than 0.05.

$$Y = 0.011x - 0.079 \tag{6}$$

$$R^2 = 98.83\%$$

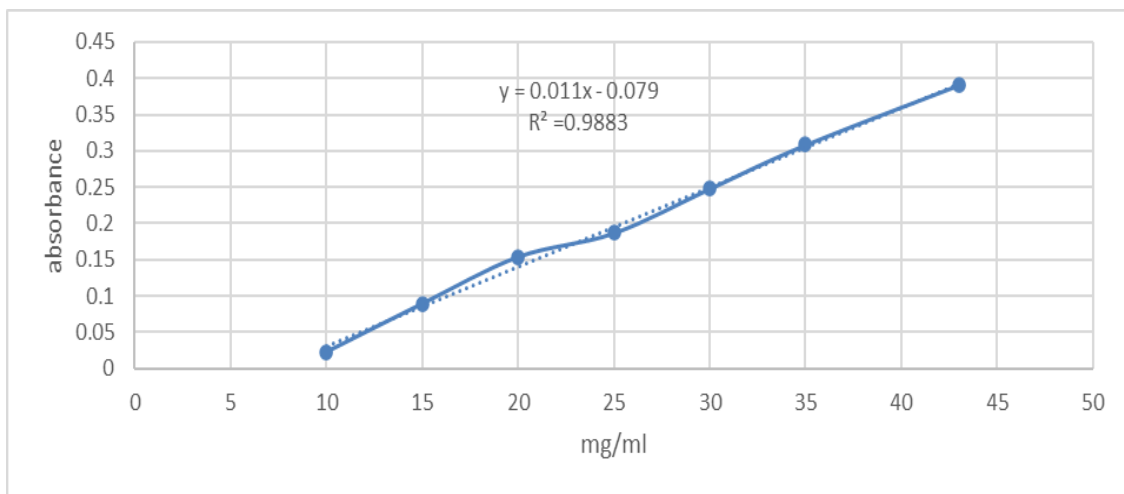


Figure 2. Calibration curve for absorbance of phenol for (EO).

RESULTS AND DISCUSSION

Four adjustable factors—sodium chloride (NaCl) concentration (g/L), current density (CD) (mA/cm²), solution pH, and time—are investigated in this paper. The four tiers of these qualities are shown in Table 5. Wastewater treatment was improved by NaCl. These criteria were chosen for their relevance in affecting wastewater treatment.

Table 5. Levels that are specific to the experimental parameter levels.

Parameters	Symbols	Level 1	Level 2	Level 3
CD mA/cm ²	A	10	15	20
PH	B	3	7	10
Time / hour	C	2	3	4

NaClcontent g/l	D	0	1.5	3
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According to table 6 Six optimization tests are conducted using response surface methodology (RSM) with Box-Behnken design (BBD) to find the important operational factors that influence phenol removal from wastewater(Kassob and Abbar 2022).

Table 6. Optimization of experimental parameters.

Run No.	pH	CDmA/cm ²	NaCl g/L	Time /hour	Phenol ppm	Econs KWh/m ³
1	3	10	1.5	3	76.48	26.4
2	3	20	1.5	3	83.6	78.72
3	10	10	1.5	3	52.22	31.68
4	10	20	1.5	3	44.8	97.92
5	7	15	0	2	39.8	45.2
6	7	15	0	4	50.76	90.24
7	7	15	3	2	82.52	33.6
8	7	15	3	4	78.9	68.16
9	7	10	0	3	52.53	37.44
10	7	20	0	3	36.45	96.96
11	7	10	3	3	76.44	29.28
12	7	20	3	3	83.62	78.72
13	3	15	1.5	2	71.55	33.6
14	10	15	1.5	2	56.34	33.6

15	3	15	1.5	4	90.49	70.08
16	10	15	1.5	4	55.34	79.68
17	7	10	1.5	2	77.23	20.48
18	7	20	1.5	2	60.34	58.24
19	7	10	1.5	4	70.32	41.6
20	7	20	1.5	4	75.92	112.64
21	3	15	0	3	47.44	59.04
22	10	15	0	3	39.24	47.52
23	3	15	3	3	96.3	46.08
24	10	10	3	3	56.73	41.76

Multivariate Regression Analysis

Conclusive Equation Represented in Coded Variables

$$\text{Phenol} = 57.45 - 1.34 * 13.43 * B + 3.23 * C + 18.08 * D - 3.82 * AB + 5.62 * AC + 5.82 * AD - 5.15 * BC - 7.63 * BD - 3.65 * CD + 6.15 * A^2 + 1.84 * B^2 + 8.60 * C^2 + 0.0000 * D^2 \quad (7)$$

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

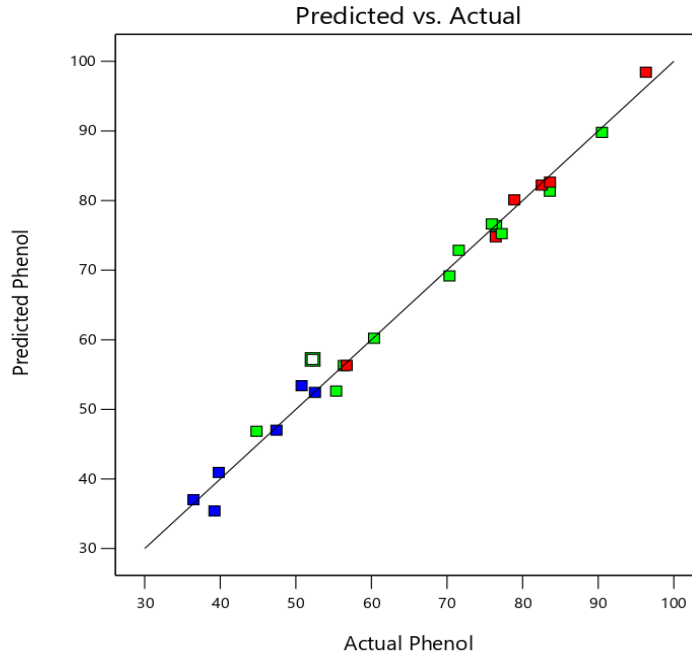


Figure 3. Phenol behavior against Phenol actual.

Optimization Analysis of (ANOVA)

To evaluate how acceptable the results are and how well the tests worked in controlled conditions, we use ANOVA analysis to look at how each controllable factor affects the process response, specifically the success of phenol removal(Sutrisno et al. 2024).

The data indicates that a substantial proportion of writers hail from the United States, India, and China. Furthermore, several writers are associated with esteemed higher education institutions, like the University of São Paulo, São Paulo State University "Júlio de Mesquita Filho," and Tehran University of Medical Sciences. This report offers a comprehensive analysis of the quantity and distribution of contributions to the progression of ANOVA research(GilPavas, Dobrosz-Gómez, and Gómez-García 2020). As seen in Table 7.

The model's F-value of 64.73 indicates statistical significance. The likelihood of an F-value of this size resulting from random noise is around 0.01%.

P-values less than 0.0500 indicate that model terms are statistically significant. In this case, B, C, D, AB, AC, AD, BC, BD, CD, A², and C² are relevant model terms. Values beyond 0.1000 indicate that the model terms are insignificant. Model reduction may improve your model if several insignificant terms are present, except those essential for hierarchical support.

Table 7. ANOVA analysis data.

Source	Sum of squares	df	Mean of Square	F-value	P-value
Model	6951.58	13	534.74	64.73	<0.0001
A-CD	21.11	1	21.11	2.56	0.1410
B-PH	2165.18	1	2165.18	262.11	<0.0001
C-Time	128.79	1	128.79	15.59	0.0027
D-Salt	3821.79	1	3821.79	462.66	<0.0001
AB	59.05	1	59.05	7.15	0.0233
AC	126.45	1	126.45	15.31	0.0029
AD	135.26	1	135.26	16.37	0.0023
BC	107.59	1	107.59	13.03	0.0048
BD	235.96	1	235.96	28.56	0.0003
CD	53.14	1	53.14	6.43	0.0295
A ²	151.41	1	151.41	18.33	0.0016
B ²	12.96	1	12.96	1.57	0.2389
C ²	295.67	1	295.67	35.79	0.0001
D ²	0.0000	0			
Residual	82.60	8.26			
Cor Total	7034.19	23			

Effect of Operational Parameters on Phenol Removal

Impact of PH

While The results demonstrated that pH 3 yielded the greatest and most cost-effective

phenol removal rate, as seen in Figure 4. Experiments conducted under these conditions demonstrated the formation of sodium hydroxide radicals and active chlorine compounds, such as Cl_2 , HOCl , and ClO^\bullet , which are potent oxidizers that efficiently decompose phenol, while simultaneously augmenting the concentration of (H^+) , thereby facilitating oxidation reactions on the electrode surface. However, several researches have stated that the optimal acidity level for the highest clearance rate is pH 7, as noted in the preceding publication (GilPavas, Dobrosz-Gómez, and Gómez-García 2020).

Impact of CD

While experiments conducted with this method at different current levels (10, 15, and 20 mA/cm^2), the best phenol removal occurred at a current level of 15 mA/cm^2 . Other factors, such as time and sodium chloride concentration, depend on the current. This result is consistent with previous studies (Salvestrini et al. 2020).

In electrolysis, increasing the current boosts the production of hypochlorous acid (HOCl) or sodium hypochlorite (NaClO) when chloride ions (Cl^-) are in the solution, which speeds up the reactions at the electrodes (anode and cathode) (Li et al. 2023). as seen in figures 4, 5 and 6.

Impact of NaCl

The incorporation of sodium chloride substantially influences the phenol elimination process. Experiments demonstrate that the incorporation of sodium chloride favorably influences the electrooxidation process by improving conductivity and reducing the potential. The anode electrodes produce many chlorine species, including hypochlorous acid, throughout this process. These forms promote the breakdown of organic molecules by a process termed indirect oxidation.

Figure 5 This study illustrates the impact of NaCl supplementation on the process of phenol removal. We performed laboratory experiments with and without the incorporation of salt, particularly at concentrations of 0, 1.5, and 3 g/l . Research demonstrates that salt enhances the removal process at all current densities, as supported by previous studies (Rubí-Juárez et al. 2015).

Impact of Time

The results showed that how long the electrooxidation process takes greatly affects how quickly phenol is removed, depending on other factors like current density and sodium chloride concentration. The optimal removal rate was achieved in three hours with a current density of 15 mA/cm^2 , a sodium chloride concentration of 3 g , and a pH of 3. The values fluctuate during the duration of the procedure. The study results indicate that time significantly influences CD density and the efficacy of phenol removal. This is evident in Figure 6.

The likelihood of phenol decomposing into less dangerous chemicals or carbon

dioxide and water increases with time. Excessive processing time may result in heightened energy consumption without substantial gains in efficiency and the generation of unwanted oxidation byproducts, as shown in a previous study (Fajardo et al. 2017).

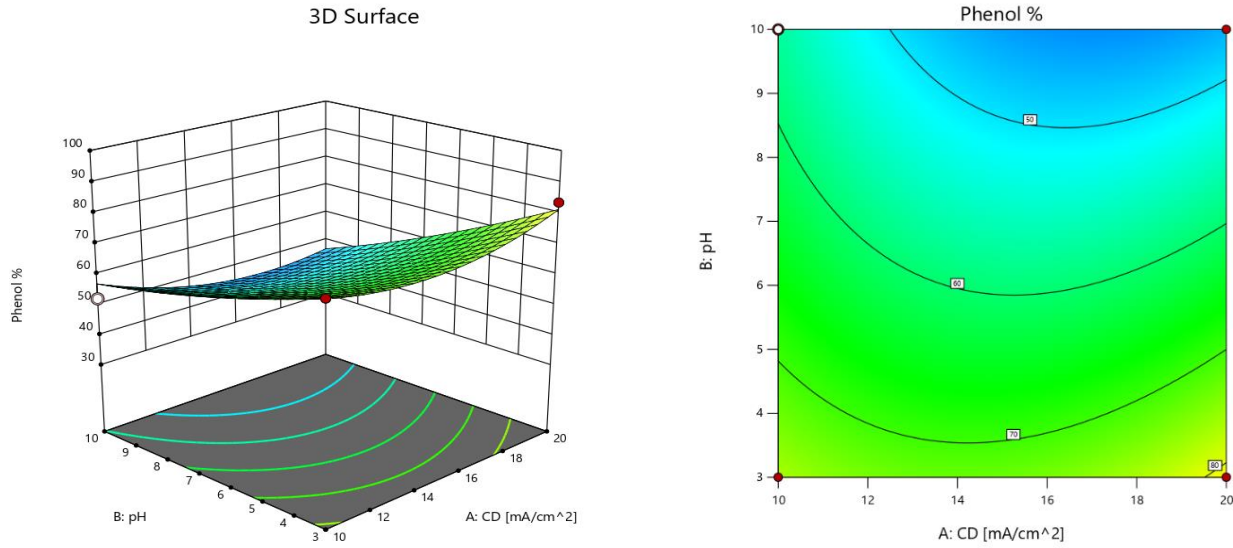


Figure 4. Contour and 3D for PH and CD vs. Phenol %.

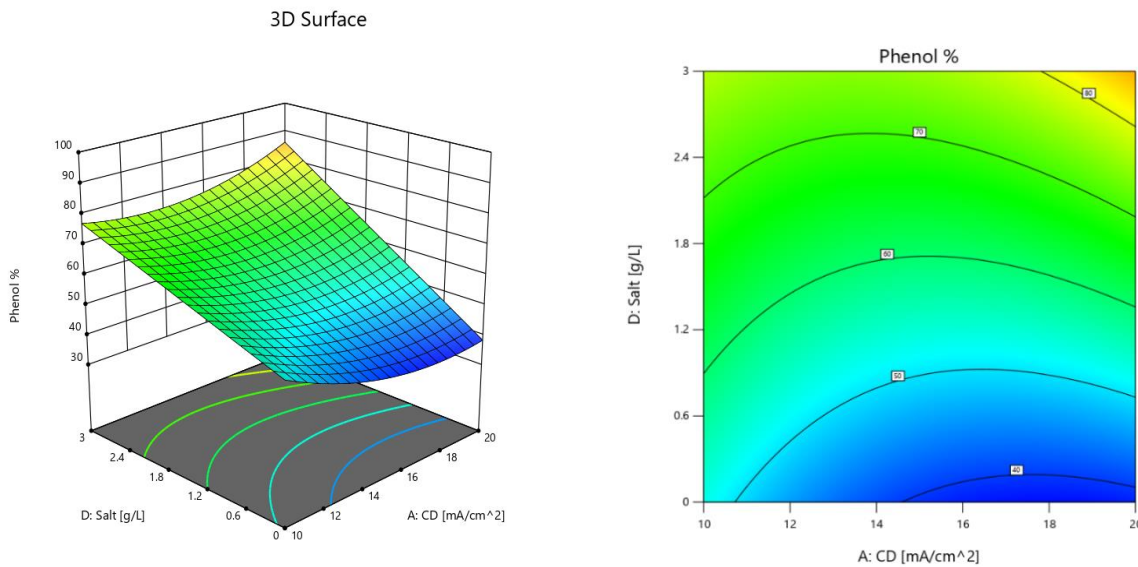


Figure 5. Contour and 3D for salt and CD vs. Phenol.

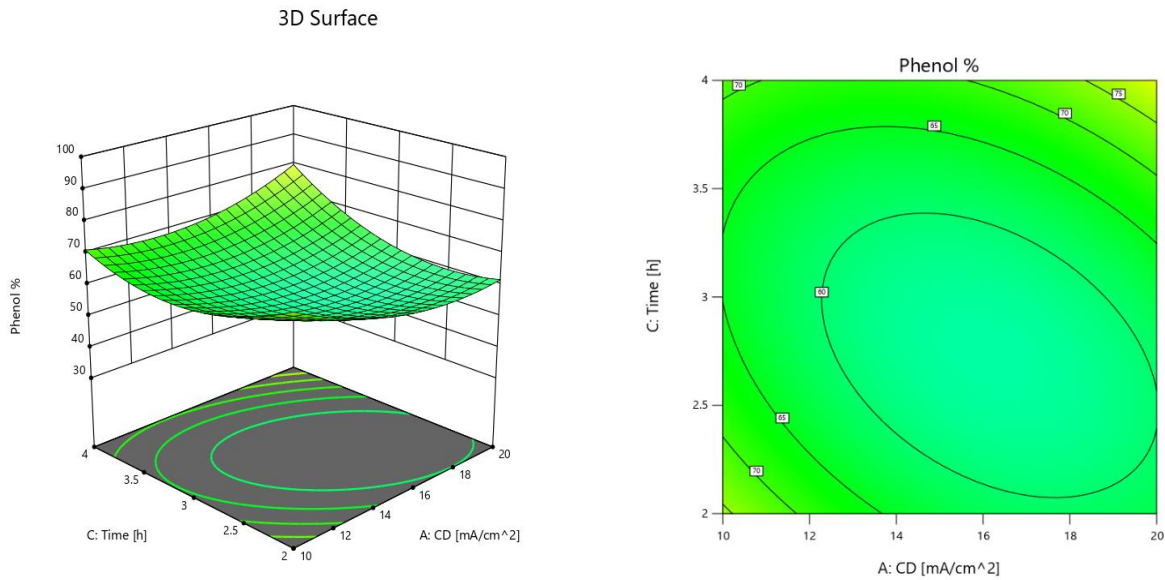


Figure 6. Contour and 3D for Time and CD vs. Phenol %.

ENERGY CONSUMPTION

The wastewater treatment procedure needs electrical energy. Consequently, Econs must be articulated and analyzed to circumvent costly endeavors. The Econs value was calculated using equation 8.

All calculations were conducted at a capacity of one liter. The EO process duration was established at 2-4 hour.

$$E_{cons} = \frac{E \times I \times t}{1000V} \quad (8)$$

CONCLUSIONS

Industrial water, including that from oil refining, includes several hazardous organic and inorganic compounds detrimental to the environment and living organisms. Phenol, a significant hazardous and carcinogenic chemical compound, is present in the oil refining water sourced from the Najaf Refinery. The quantity of this material was diminished by efficient electrolytic (EO) techniques and was successfully eliminated under certain operational parameters including CD, pH, NaCl, and duration. The Design-Expert program was used to compute the duration for each of the 24 experimental trials. The results demonstrated that medium CD levels, along with high or medium acidity and medium

reaction duration, enhanced phenol removal more efficiently as the NaCl concentration rose. The rise in conductivity during the process is attributed to NaCl (Time = 3, pH = 3, CD = 15, NaCl = 3). The analysis of variance (ANOVA) findings indicated that the P and F values in this research demonstrate that all process factors influence the phenol removal process, either directly or in relation to one of the process-specific variables. We aspire that our research has accomplished a measurable decrease in environmental contamination.

RECOMMENDATIONS

- 1-To make coagulation work better and use fewer electrodes, use aluminum or coated iron electrodes.
- 2- Creating ways to reuse or remediate the sludge that comes from the coagulation process to lessen its effect on the environment.
- 3- To make elimination more effective, use electrocoagulation with membrane filtration or activated carbon.
- 4-Try using intermittent current operation instead of direct current to save energy and make the electrodes last longer.
- 5- Try different processing techniques, such the Electro-Fenton procedure, and see how they compare to the outcomes you researched.

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