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Electro Oxidation Process for Wastewater Treatment in Petroleum Refineries

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ABSTRACT

In this research, successive electro-oxidation (EO) process was utilized to eliminate some of the primary organic contaminants in effluent wastewater, specifically phenol and chemical oxygen demand (COD). The performance of the electro-oxidation process was studied by using two graphite electrodes as anodes and three stainless steel electrodes as cathodes, which is a new strategy in this field. Taguchi method has been used to design experiments to approach the best experimental conditions for phenol and COD removal as significant responses. The best operating conditions that resulted in the maximum reduction of phenol and COD were current density (CD=25 mA/cm^2), pH=4, support electrolyte (NaCl=2g/l), the distance between electrodes (Dist.=5mm), and time of 60 minutes. At these operating conditions, phenol and COD removal were 99.27% and 99.96%, respectively. This work provides important insights into a novel water and wastewater treatment method with a detailed analysis of the results.

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INTRODUCTION

The rapid expansion of chemical, petrochemical, and oil refining industries in the past few decades has led to an increasing worry regarding the extensive pollution of surface and groundwater with diverse organic substances (Awad et al., 2022; Beauregard et al., 2020; Mohammed Ali et al., 2022). Accordingly, phenolic compounds, pervasive in various industrial wastes, hinder the efficacy of conventional biological treatment processes, often rendering their elimination difficult or unachievable (Noorani et al., 2024; Rahman and Mustafa, 2022). Given their capacity to harm organisms even at low concentrations, phenols have attained the status of priority pollutants; moreover, several have been labeled hazardous due to the probable threat they pose to human health (EPA, 2002; Kalash et al., 2020; Kalash et al., 2019). Wastewater from the oil industry will frequently be produced and discharged into the world's leading water bodies, causing severe environmental issues. The amount and characteristics of pollutants in refinery wastewater depend on the type of oil being processed, the plant configuration, operation procedures, and the processing unit in the oil refinery (Diya'uddeen et al., 2011).

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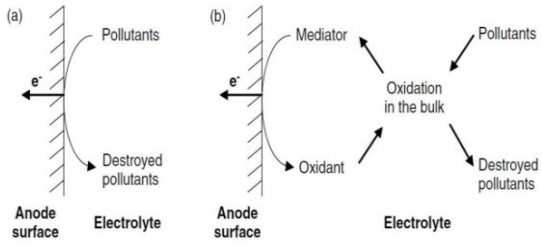

Fig. 1. Diagram of (a) direct pollutant oxidation and (b) indirect pollutant oxidation (Anglada et al., 2009)

In refineries, wastewater contaminants frequently encompass unrestrained hydrocarbons, suspended particulates, inorganic substances with a high salt concentration, greasy substances, essential nutrients, substantial metals, benzene, phenol, organic carbon, ammonia, sulfides, and incorporated chemical agents (Yavuz et al., 2010).

Wastewater treatment uses coagulation and flocculation as crucial methodologies (Alalwan et al., 2022; Mohammed et al., 2022). The pollutants become absorbed in these processes, separated by formed products. Key benefits of these methods include their simplicity, availability of a diverse range of commercially accessible chemicals, affordability, efficient reduction of chemical and biochemical oxygen demand, and whole organic carbon (Alalwan et al., 2023).

Electrochemical oxidation (EO) is one of the promising electrochemical advanced oxidation processes that utilize electrons as green agents for the in-situ generation of oxidants (dos Santos et al., 2021). Electricity is utilized to produce oxidants, which oxidize the contaminant of the organic compounds in wastewater. Wastewater of electrochemical oxidation might follow two pathways to oxidize pollutants: direct and indirect oxidation. The former happens where organic pollutants oxidize directly on the anode's surface, as seen in Fig. 1. Throughout the direct oxidation, two oxidant species may be electro-chemically produced on the anodes of the oxide (MOx). One via Eq. (1) as active oxygen in oxide lattice (MOx^{+1}) referred to as chemisorbed, whereas the other is via adsorbed radicals of the hydroxyl (•OH) or physiosorbed active O as shown in Eq. (2).

$$R + MOx^{+1} \rightarrow RO + MOx \qquad (1)$$

$$R + n\left(OH^{\cdot}\right) \Rightarrow CO_2 + nH^+ + ne^- + Mox \qquad (2)$$

R denotes the organic pollutant materials, and *n* represents the number of •*OH* absorbed on an electrode. Indirect pathway of the oxidation advantages from precarious intermediates that are produced electro-chemically like hypochlorite (ClO_2), chlorine (Cl_2), hydrogen peroxide (H_2O_2), and ozone (O_3). Those powerful oxidizing agents hold the responsibility for destroying the organic pollutants. The rate of generating oxidant, temperature, and pH determine the oxidation rate in the indirect oxidization (Bayar et al., 2013).

The anodic reactions are as follows:

$$2Cl^- \Rightarrow Cl_2 + 2e^- \qquad (3)$$

$$2H_2O \Rightarrow O_2 + 4H^+ + 4e^- \qquad (4)$$

The bulk reactions are as follows:

$$Cl_2 + H_2O \Rightarrow HOCl + H^+ + Cl^- \qquad (5)$$

$$Cl_2 + OH^- \Rightarrow HOCl + Cl^- \quad (6)$$

$$HOCl \Rightarrow H^+ + OCl^- \quad (7)$$

$$R + OCl^- \Rightarrow 2CO_2 + Cl^- + H_2O \quad (8)$$

The cathodic reactions are as follows:

$$2H_2O + 2e^- \Rightarrow 2OH^- + H_2 \quad (9)$$

$$OCl^- + H_2O + 2e^- \Rightarrow Cl^- + 2OH^- \quad (10)$$

When appropriate chloride ions are present in wastewater, Cl_2 *is* produced, as in Eq. (3), and forms OCl^-, as shown in Eqs. (4) to (7), which may be utilized for the destruction and elimination of oxidizable organic pollutants such as phenol, dyes, glucose, and aniline (Martínez-Huitle and Brillas, 2009).

EO has been used in various investigations due to its numerous advantages: (i) easy operation with simple equipment; (ii) environmentally friendly process with clean reagents (electrons) and without additional chemicals; (iii) versatile technology that can be used for the removal of a wide range of pollutant (Ahmad et al., 2021; Tong et al., 2021). The main objectives of the present work are Choosing the optimum cell from the several electrode arrangements and the most effective oxidation process regarding removal. Using the best arrangement achieved through the modification of both electrodes (anode and cathode) in the electro-oxidation process, an assessment was conducted to gauge the efficiency of this novel batch electrochemical reactor. Determine the effect of various process parameters such as the initial pH, phenol concentration, electrolyte concentration, current density, and time.

EXPERIMENTAL PROCEDURES

Wastewater

As a case study, the petroleum refinery wastewater used in this study was obtained from the Dora petroleum refinery plant in Baghdad, Iraq.The significant features related to this refinery wastewater are specified in Table 1.

Body Oxidation Cell Reactor

The cell's body has been created from Perspex with inner dimensions of 12 cm width*12 cm length*12 cm height and a discharge valve located 2.5 cm above the bottom of the cell, as shown in Fig 2. The Perspex cover used for electrode fixing has dimensions of 12 cm * 12 cm

Table 1. Characteristics of refinery wastewater supplied by the Department of Laboratories (Refinery of Al-Dora)

Test	Inlet to Unit	Outlet of Unit
pH	8.4	8.1
TDS (PPT)	0.955	1.290
Oil mg/l	7.6	3.7
BOD mg/l	78	15
COD mg/l	565	79
Phenol mg/l	0.1402	0.008

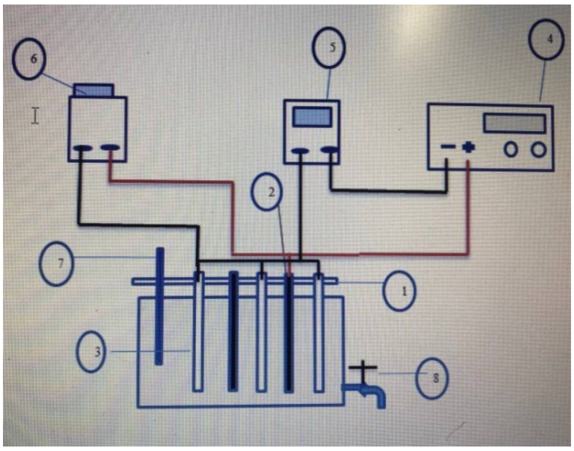

Fig. 2. Diagram of EO experiment. 1. Cell cover, 2.Anode (porous graphite), 3.Cathode (stainless steel), 4. Power supply, 5.Voltmeter, 6.Ammeter, 7.Thermometer, 8. Discharge valve.

area and 0.4 cm thickness and contains five slots with 13cm length * 6 cm width * 0.4 cm width at a distance of 0.5, 1, and 1.5 cm from each. Three holes were made in the Perspex cover to insert the thermometer, electrical conductivity, and pH probes inside the cell,. The electrolytic cell was comprised of two graphite plates as anodes and three stainless steel plates as cathodes. Each electrode's effective anodic surface area was 97.2 cm^2 with dimensions of 8.1 cm height * 6 cm width. These materials were chosen due to the following reasons: (a)Their high physical stability; prerequisite for the electrode material involves robust mechanical durability, effective resistance against erosion, and the ability to withstand cracking. (Moradi et al., 2020) **(b)** Their high chemical stability; where a fundamental requirement is that the electrode material needs to withstand corrosion, prevent the emergence of undesirable oxides or hydrides, and avoid the accumulation of hindering organic films in any circumstance. (Li et al., 2022) (c)Their suitable physical shape; the attainability of shaping the material as necessary, to facilitate dependable electrical connections, and enable easy attachment and substitution across different scales is important. (Tan et al., 2022)(d) Their low cost-to-life ratio; where preference should be given to utilizing electrode materials that are both reasonably priced and long-lasting (Sohal et al., 2023; Sun et al., 2022).

Chemicals

HCl (Sigma-Aldrich with a concentration of 37%) has been utilized to prepare a 0.1M solution for modifying initial pH related to treated wastewater effluents. In addition, NaOH pellets (Sigma-Aldrich with purity ≥ 97.0 %) have been utilized for preparing (0.1M) solution. Sodium chloride (NaCl, Fisher Chemicals with a purity of 99.5% by weight) was used to increase the conductivity between the electrodes. The volume of wastewater used in the experiment was 1.5 litters

Taguchi Experimental Design

Diverse operational parameters significantly impact the behavior of organic pollutant degradation in refinery wastewater treatment. Examining these impacts can be conducted using a range of experimental designs, such as mixture design, response surface methodology, factorial design, and Taguchi methods (Martinez-Villafane and Montero-Ocampo, 2010). The Taguchi approach was used to investigate the impact of numerous inputs on the process performance using the orthogonal array (OA) design of experiments theory (Jin et al., 2014; Nandhini et al., 2014). It plays a crucial role in evaluating the model's performance. Loss functions are how one

Table 2. Operating parameters and levels

Control Parameters	Levels
CD (mA/ cm ^{2})	(5, 15, 25)
pH	(4, 6.5, 8)
NaCl (g/l)	(0, 1, 2)
Distance (mm)	(5, 10, 15)
Time(min)	(20, 40, 60)

Table 3. Layout of L27 (3^5) experimental design

NO	Current density (mA/ cm^2)	pH	NaCl (g/l)	Distance(mm)	Time (min)
1	5	4	0	5	20
2	5	4	0	5	40
3	5	4	0	5	60
4	5	6.5	1	10	20
5	5	6.5	1	10	40
6	5	6.5	1	10	60
7	5	8	2	15	20
8	5	8	2	15	40
9	5	8	2	15	60
10	15	4	1	15	20
11	15	4	1	15	40
12	15	4	1	15	60
13	15	6.5	2	5	20
14	15	6.5	2	5	40
15	15	6.5	2	5	60
16	15	8	0	10	20
17	15	8	0	10	40
18	15	8	0	10	60
19	25	4	2	10	20
20	25	4	2	10	40
21	25	4	2	10	60
22	25	6.5	0	15	20
23	25	6.5	0	15	40
24	25	6.5	0	15	60
25	25	8	1	5	20
26	25	8	1	5	40
27	25	8	1	5	60

measures the difference between the predicted and true values, and they guide the model during the training process to find the optimal set of parameters – minimizing the loss (Ebert-Uphoff et al., 2021).

The present study aims to elucidate the impact of critical factors on phenol and COD removal efficiency from refinery wastewater by employing the electro-oxidation technique. Additionally, the optimal set of operating parameters that can maximize process efficiency is sought to be determined. The selection of operating parameters depended on the literature. This study takes into account certain governing factors, namely current density (CD), pH, time (min), distance between electrodes(mm), and NaCl(g/l). Table (2) depicts operating parameters and levels. Table 3 illustrates the OA design of L27 (3^5) used in this study, which is a technique that allows you to test multiple factors and interactions with a minimal number of test cases. It is based on the concept of orthogonal arrays, which are mathematical structures that ensure balanced and uniform coverage of all possible combinations. The number of levels is three, number of factors is five, and number of runs is 27. The numeric 5 signifies the presence of five parameters, each of which was investigated across three distinct levels. The time of experiments was variable (20, 40, and 60) min while the date of the samples were on the (4^{th}, 5^{th}, 6^{th}) March ,2023. The experiment was conducted per the arrangement in Table 3, wherein each row within the table represents a single experiment run.

RESULTS AND DISCUSSION

The Signal-to-Noise Analysis

By analyzing the signal-to-noise ratio (S/N), the Taguchi method can determine the best removal conditions and identify variation in response around the mean value. By utilizing the (S/N) ratio, TED identifies the specific levels of controlling factors that result in reduced response variance due to the impact of noise factors. The (S/N) ratio is a quantitative measure that represents the ratio between the mean (desired magnitude) and the standard deviation (undesired magnitude) of the output value (Chaulia and Das, 2008). By analyzing means and (S/N) ratios, Taguchi proposes a conceptual method by plotting effects and identifying significant factors visually. Three distinct categories are identified within the (S/N) ratio: smaller the better characteristics (STB), nominal the better characteristic (NTB), and larger the better characteristics (LTB). The selection of the (S/N) ratio type is determined by the goal of the experimental process and the recognition of the sought process quality characteristic. As an illustration, the characteristic signal-to-noise ratio (LTB) is chosen to optimize the efficiency of phenol and COD removal. A formula can be used to determine the (LTB) response utilizing Eq. (11) for this kind of characteristic [13].

$$\left(\frac{S}{N}\right)i = -10log\frac{1}{n}\sum_{i=1}^{n}\frac{1}{Y^2 ij} \qquad (11)$$

Where i is the number of trials, Yij denotes the response of the determined value for i the trial and j the run, and n is the replications' number for the experimental combination. The (S/N) ratios obtained through experimentation for phenol and COD removal can be found in Table (4).

Linear Model Analysis for Means of the Removal of Phenol and COD

The relative importance of each factor concerning the concentration of phenol and COD removal is indicated by the absolute value of the coefficients' order. The parameter with the highest sum of squares and biggest coefficient holds the most significant impact. The Taguchi approach utilizes linear model analysis to perform means with LTB (Eq. 11). Figs. 3 and 4 illustrate the means plot for NaCl, pH, CD, distance between electrodes, and phenol and COD removal time, respectively. The parameter level yielding the highest means led to the most efficient phenol and COD removal.

The maximum phenol and COD removal is achieved by combining specific parameters (NaCl = 2 g/l, pH = 4, CD = 25 mA/cm^2, and t = 60 min). Every operating parameter impact on phenol and COD removal is evident in the results displayed in Fig. (3) and (4), along with the representation in tables 5 and 6. Delta statistics are utilized to determine the ranks in these tables. Delta statistics represent the difference between each factor's highest and lowest averages. The delta value assigned to Rank 1 is the biggest, followed by the delta magnitude assigned to Rank 2,.. etc (Fıl et al., 2014). The experiment's time has the most significant impact on phenol and COD removal responses. Current density emerged as the second significant factor influencing the removal of phenol and COD. The Tables (5 and 6) emphasize that the phenol and COD removal have the lowest change by varying the distance between electrodes.

Table 4. Taguchi's experimental design of L27 (3^5) standard OA, phenol, COD removal, and (S/N) ratios.

Run No.	Phenol Removal %	Phenol (S/N)	COD Removal %	COD (S/N)
1	57.80	35.20	58.10	35.28
2	74.66	37.46	75.00	37.50
3	90.50	39.13	91.00	39.18
4	57.12	35.13	57.50	35.19
5	69.10	36.78	70.10	36.91
6	87.55	38.84	88.40	38.92
7	54.10	34.66	55.00	34.80
8	69.00	36.77	70.22	36.92
9	88.10	38.89	89.35	39.02
10	62.55	35.92	63.25	36.02
11	79.85	38.04	80.45	38.11
12	93.33	39.40	94.00	39.46
13	62.50	35.91	63.45	36.04
14	75.40	37.54	76.65	37.69
15	91.55	39.23	92.66	39.33
16	56.20	34.99	57.89	35.25
17	71.50	37.08	73.00	37.26
18	87.90	38.87	89.15	39.00
19	68.22	36.67	69.95	36.89
20	83.13	38.39	84.88	38.57
21	99.60	39.96	98.50	39.86
22	61.00	35.70	62.85	35.96
23	74.50	37.44	76.00	37.61
24	90.65	39.14	92.43	39.31
25	61.20	35.73	63.12	36.00
26	74.00	37.38	75.37	37.54
27	92.00	39.27	93.89	39.45

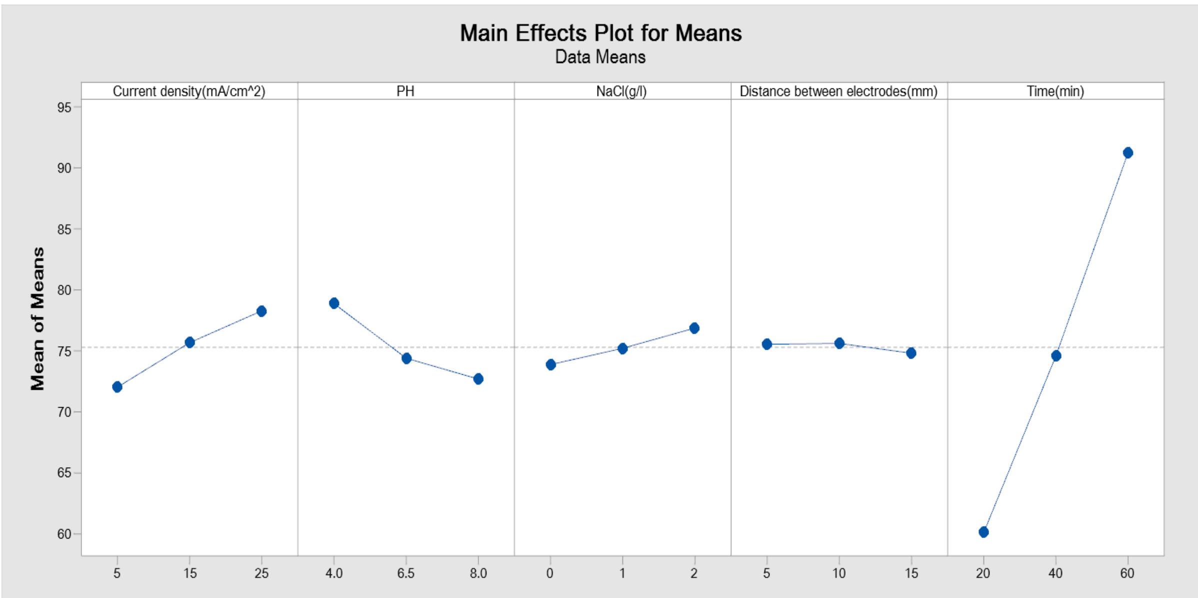

Fig. 3. Main effects plot for phenol removal %

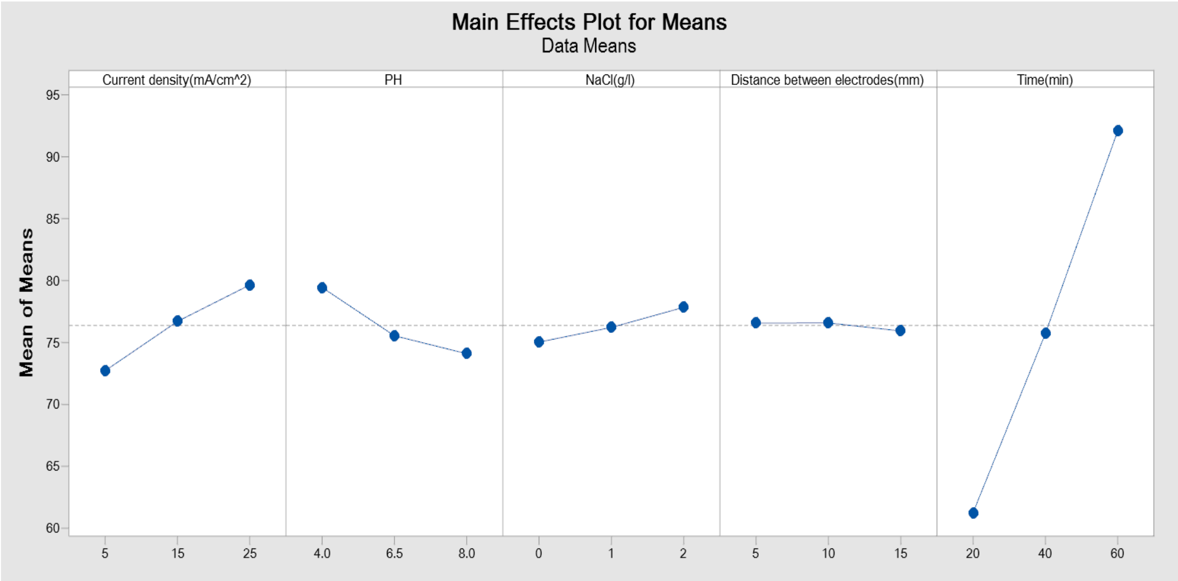

Fig. 4. Main effects plot for COD removal %

Table 5. Response of means for phenol removal

Level	Current density (mA/cm^2)	pH	NaCl (g/l)	Distance between electrodes (mm)	Time (min)
1	71.99	78.85	73.86	75.51	60.08
2	75.64	74.37	75.19	75.59	74.57
3	78.26	72.67	76.84	74.79	91.24
Delta	6.26	6.18	2.99	0.80	31.17
Rank	2	3	4	5	1

Table 6. Response of means for COD removal

Level	Current density(mA/cm^2)	pH	NaCl (g/l)	Distance between electrodes(mm)	Time(min)
1	72.74	79.46	75.05	76.58	61.23
2	76.72	75.56	76.23	76.60	75.74
3	79.67	74.11	77.85	75.95	92.15
Delta	6.92	5.35	2.80	0.65	30.92
Rank	2	3	4	5	1

Operating Parameters Influence the Removal of Phenol and COD

Influence of current density

Current density is one of the most influential variables in the electro-oxidation process. The data depicted in Fig. 5 (a and b) shows the degradation of phenol and COD over time with numerous current densities applied. The results showed that the maximum removal rate is 99.27% and 99.95% for phenol and COD, respectively, at a current density value of 25mA/cm^2 while keeping other process variables constant (pH=4, NaCl=2 g/l, and the distance between electrodes is 5 mm). Increasing the current density from 5 mA/cm^2 to 25 mA/cm^2 will raise the efficiency of the removal rate of the phenol and COD, where the generated hypochlorite OCl^- oxidizes organic compounds predominantly via employing the current applied. However, further increasing the current density will also increase the energy cost consumed in the process.

In addition, if the current density exceeds the optimum values, that will affect the process by generating O_2 instead of OCl^- oxidizes from H_2O.

Influence of NaCl concentration

The impact of NaCl amount on phenol and COD removing efficacy is shown in Fig. 6 (a and b). At an abatement time of 30 min, the removal efficiency increased from 72.91 to 75.89% and from 73.96 to 76.88% for phenol and COD, respectively, adding 2.00g/L of sodium chloride compared to no addition. A 60-minute abatement time at various concentrations of NaCl resulted in the highest removal of phenol and COD. Increasing the electrolyte concentration increases conductivity and decreases resistance, resulting in increasing current passing within the cell, which increases metal hydroxide production. These results confirm that chlorine ions from the solution can degrade pollutants even at low concentrations if they are initially present in the solution.

Influence of pH value

The pH impact on phenol and COD removal efficiency has been studied, and the results are illustrated in Fig. 7 (a and b). Phenol and COD removal effectiveness are significantly influenced by pH initial value. The pH values equal to or lower than four are optimal for the ultimate efficiency of phenol and COD removal. Similarly, higher COD removal was observed by Fil et al. when graphite anodes and NaCl were used in the anodic oxidation of wastewater from a pistachio processing industry (Fıl et al., 2014). In their investigation (Fıl et al., 2014), they found that neutral circumstances are more effective in removing COD than alkaline medium.

a b

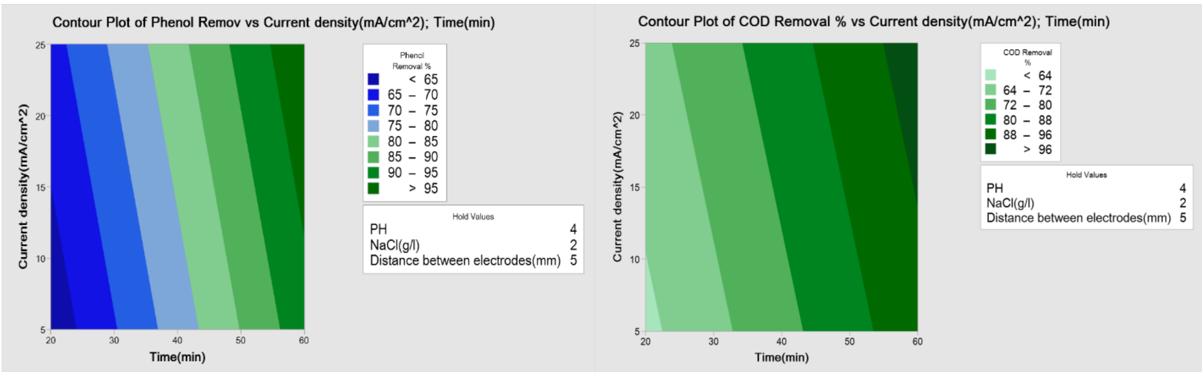

Fig. 5. Effect of applied different CD at different times on the removal rate of (a) phenol and (b) COD.

a b

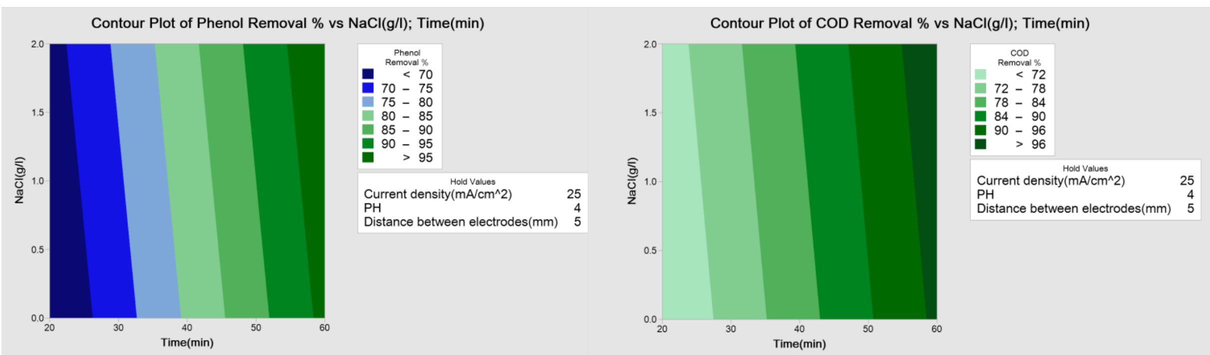

Fig. 6. The effect of NaCl concentration with time on the removal rate of (a) phenol and (b) COD.

This is due to the formation of hypochlorous, a more potent oxidant than hypochlorite. Anodic oxidation commonly provides better outcomes in acidic or neutral conditions than in primary conditions when chlorine ions are present in the electrolytic process (Ibrahim et al., 2013).

Influence of the distance between electrodes

Fig. 8 (a and b) illustrate the effect of changing the distances between electrodes from 5 mm to 15 mm on phenol and COD removal efficiency while keeping other parameters constant (current density = 25 mA/cm^2, NaCl = 2 g/l, and pH = 4) with variety times. If the distance between electrodes is 5 mm, the phenol and COD removal rates increase slightly from 98.54 to 99.27% and 99.33 to 99.96%, respectively. Thus, the 5 mm distance was the ideal distance between electrodes because the closer the distance between the cathode and anode resulted in a more significant reduction in resistance drop (Ohm) through the electrolyte, resulting in a lower equivalent cell voltage and energy consumption. (Yörük et al., 2023)

Optimization and Multiple Regression Model

The multiple regression equations (Eq. 12 and 13), acquired by MINITAB 19 software are shown as follows, representing the relationship between phenol and COD removal with the studied factors.

$$\text{Phenol Removal }\% = 48.35 + 0.3132 * X1 - 1.570 * X2 + 1.494 * X3 - 0.0726 * X4 + 0.7791 * X5 \quad (12)$$

$$\text{COD Removal }\% = 47.88 + 0.3462 * X1 - 1.360 * X2 + 1.402 * X3 - 0.0632 * X4 + 0.7730 * X5 \quad (13)$$

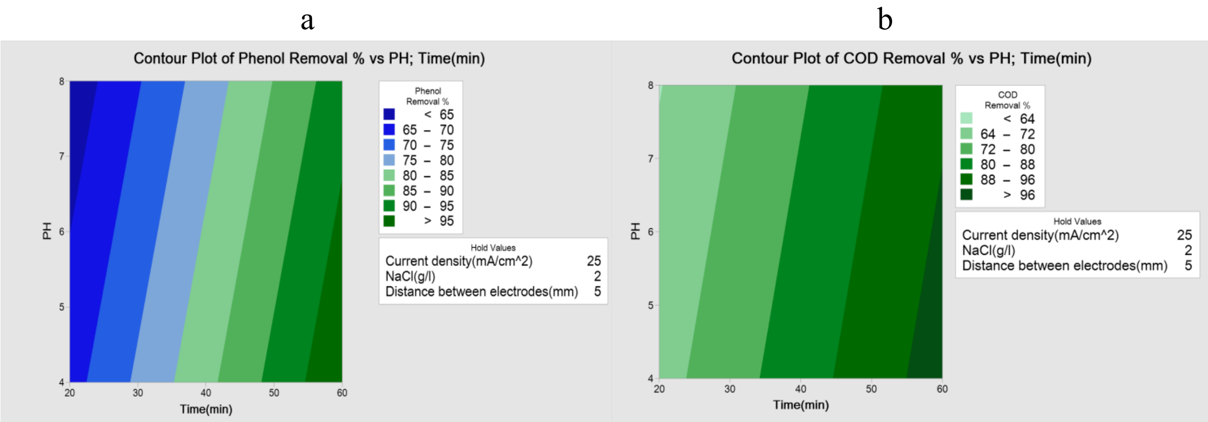

Fig. 7. The effect of pH value with time on removing (a) phenol and (b) COD.

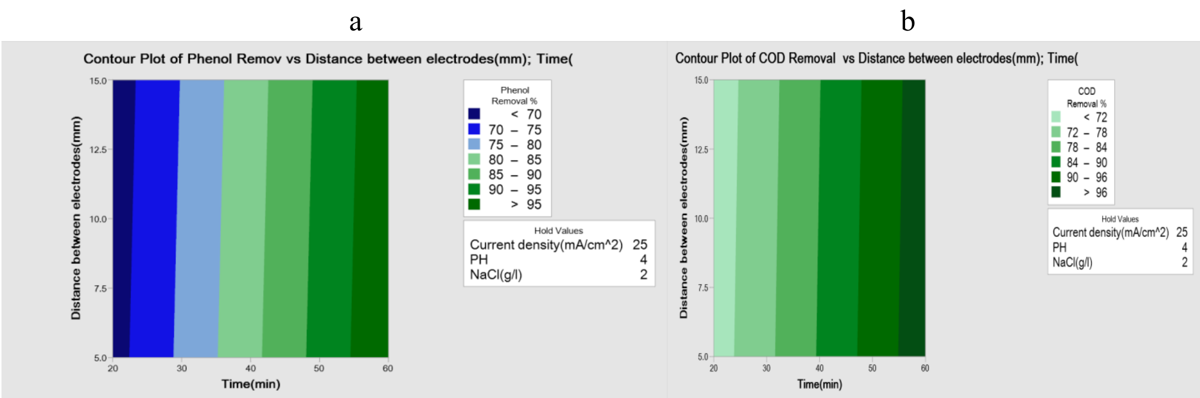

Fig. 8. The effect of the distance between electrodes with time on the removal rate of (a) phenol and (b) COD

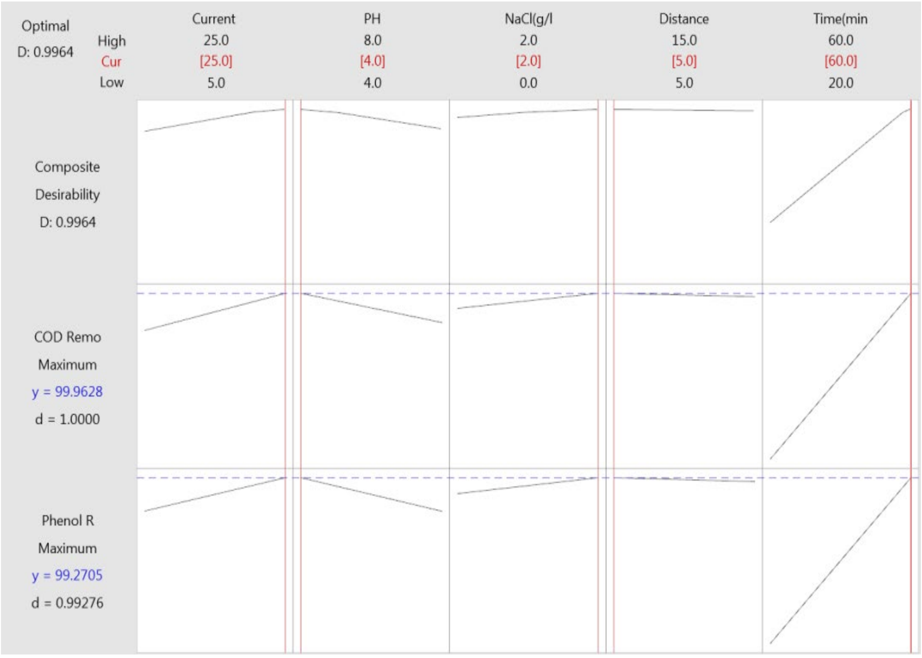

Fig. 9. Response optimization of phenol and COD removal rate

Where X1: is the current density, X2: is the pH value, X3: is the amount of NaCl, X4: is the distance between electrodes, X5: is the time of the run.

The two predicted equations yield squared correlation coefficient (R^2) values of 99.27% and 99.20% for phenol and COD removal, respectively. Optimal conditions for each experimental design are depicted in Fig. 9. Phenol and COD removal efficiency is impacted by current density, pH value, NaCl concentration, distance between the electrodes, and time. The primary parameter for phenol and COD removal is time, while CD, pH, and NaCl have secondary roles in effectiveness. The distance between electrodes has the lowest impact on the process's efficiency. Results from the means analysis are consistent with the obtained results. Considering all these outcomes, the optimal response are time = 60 min, distance = 5, NaCl = 2 g/l, pH = 4, and CD = 25 mA/cm^2.

CONCLUSION

In this research, wastewater generated from Al-Dora Petroleum Refinery has been treated by electro-oxidation method using a jacketed Perspex glass lab scale, where phenol and (COD) were eliminated to final concentrations lower than standard limits using the Taguchi method for the design of experiments. The outcomes of the electro-oxidation process showed that the experiment time, current density, pH initial value, and NaCl concentration significantly affect the phenol and COD removal. The removal efficiency of phenol and COD is 99.27% and 99.96%, respectively.

In addition, these results found that increasing the concentration of electrolyte increases conductivity and decreases resistance, resulting in increasing current passing within the cell, which increases metal hydroxide production. This confirms that chlorine ions from

the solution can degrade pollutants even at low concentrations if they are initially present in the solution. Furthermore, the results confirm that the formation of hypochlorous, a more potent oxidant than hypochlorite, anodic oxidation commonly provides better outcomes in acidic or neutral conditions than in primary conditions when chlorine ions are present in the electrolytic process. According to the optimization response based on the Taguchi method, the optimum operating values are as follows: current density is 25 mA/cm^2, pH value is four, NaCl concentration is two g/l, distance between electrodes is five mm, and time is 60 min. For future work, it is recommended to explore the long-term stability of the EO process and its applicability to different types of wastewater, to guide further studies in the field.

NOMENCLATURE TABLE

List of Abbreviations Symbol	*Definition*
EO	electro-oxidation
COD	chemical oxygen demand
CD	current density
L27	Taguchi orthogonal array on which process variables varied on 27 runs
LTB	Larger the better
NTB	Nominal the better
SBT	Smaller the better
OA	Orthogonal array
TED	Taguchi experiment design

DECLARATIONS

GRANT SUPPORT DETAILS

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CONFLICT OF INTEREST

The authors declare that there is not any conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/ or falsification, double publication and/or submission, and redundancy has been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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