Al-Qadisiyah JournalFor Engineering Sciences 18 (2025) 163 – 169

Contents lists available at: http://qu.edu.iq

Al-Qadisiyah Journal for Engineering Sciences

Journal homepage: https://qjes.qu.edu.iq

Research Paper

Petroleum refinery wastewater treatment through the successive process of electrocoagulation and electrooxidation

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ARTICLE INFO

Article history:
Received 23 June 2024
Received in revised form 25 July 2024
Accepted 02 April 2025

keyword:
COD
Electrocoagulation
Electrooxidation
Wastewater
Treatment

ABSTRACT

Water pollution and scarcity are problems of the current time due to the industrial and biological wastes that are thrown into the aquatic environment, especially the water produced from petroleum refineries, because it contains organic and inorganic pollutants. In this study, work was done to reduce the chemical oxygen demand (COD), which represents some major pollutants such as organic materials in real wastewater collected from the Najaf refinery in Iraq, using successive electrocoagulation (EC) and electrooxidation (EO) processes. Graphite and aluminum (Al) electrodes were used as the anode and a stainless-steel electrode (SS) as the cathode. The Box-Behnken design (BBD) of experiments was used. Starting from COD (1250 ppm), the effect of current density 10,15 and 20 (mA/cm^2), time 2,3.5 and 5 h, NaCl concentration 0,1.5 and 3 (g/l), and pH 4,7 and 10 on the removal efficiency was studied. The results indicate that the removal efficiency is directly proportional to the increase in current density, time, and NaCl conc, whereas it is inversely proportional to the increase in pH, as the optimal conditions for removal were at current density $(15mA/cm^2)$, time $(5\ h)$, NaCl concentration 1.5 (g/l), and pH (4) in this case. Conditions: About 97.5% COD removal was achieved. Through the results of the ANOVA analysis, it was found that current density and time have a high effect on removal, while NaCl concentration and pH have a lower effect on removal.

1. Introduction

Water contamination became the serious environmental problem that has adverse effect on public health [1] Oil refining generates enormous amounts of wastewater with high levels of organic contaminants, severe toxicity, and low biodegradability [2] Petroleum oil is a major source of energy in the globe. Oil and grease (O&G) and varied amounts of emulsified oil, heavy metals, and organic pollutants are among the many characteristics that make Petroleum Refinery Wastewater (PRW) a significant source of pollution. It is also known for its high salinity and the chemical oxygen demand (COD) [3]. PRW was treated using a variety of methods, including physical, chemical, and biological processes, i.e., flotation, filtration, sedimentation [4] coagulation/flocculation, adsorption, and ion exchange [4] aerated lagoons, and membrane bioreactors [5] nevertheless, using biological techniques to break down the complex refractory organic contaminants in wastewater is challenging [6]. The production of extra pollution from unreacted chemicals and the challenges associated with treating significant amounts of hazardous sludge created during conventional wastewater treatment, therefore physical-chemical approaches are not always effective [7]. To preserve clean water quality in the face of stringent environmental restrictions regarding wastewater discharge, it is imperative to develop large scale, effective technologies and methodologies for the treatment and management of PRW [8]. Compared to the traditional techniques previously discussed, electrochemical technology, such as electrodeposition [9], electro disinfection [10], electro Fenton [11], electro sorption [12], electro oxidation (EO) [13], and electrocoagulation (EC) [14] significantly contribute to environmental protection through the introduction of effluent treatment, waste reduction, and the reduction of hazardous substances [15] The special benefits of electrochemical treatment such as cost effectiveness, energy efficiency, automation, and flexibility have received more attention in recently [16] EC for drinking water was initially conducted in 1946 by Fred E. Stuart [17]. Many studies were conducted in the second half of the 20th century due to the growing interest [18]. The foundation of EC is the idea that coagulant species, such as hydroxide precipitates, are created in situ by the electrolytic oxidation of the sacrificial anodic material. This material is then dissolved as ions by applying electric current through metal electrodes like iron and aluminum [19]. In terms of sludge formation, the EC approach beats the coagulation/flocculation method, which uses metal salts and polyelectrolytes as coagulants and flocculants [20]. EC seeks to eliminate the particles from the wastewater by neutralizing or weakening the repulsive forces that maintain the suspended particles in the water [5]. Growing into bigger particles that may precipitate when the repulsive forces are offset, suspended particles could isolate from water more readily [5]. Moreover, EC offers simultaneous cathodic reaction-based pollutant removal, either deposition on the cathode or flotation based on hydrogen gas production at the cathode [14]. EC has been touted as an affordable, effective, and simple-to-use technique in recent years. EC method has advantages such as, it requires minimal equipment and operating conditions, additional chemicals are needed the treated water is clear, colorless, and odorless. In additional there is minimal sludge formation, which is easily stabilized and dehydrated; the effluent contains fewer total dissolved solids than with chemical coagulation; and the gas bubbles created in the cathode make it simple to separate the pollutants by floating them to the surface [21]. EC method's shortcomings are sacrificial electrodes need to be frequently changed. The process may become less effective if an impermeable film layer forms on the cathode, and the removal of persistent dissolved organic pollutants will not be accomplished efficiently [21].

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https://doi.org/10.30772/qjes.2024.151030.1274
2411-7773

Nomenclature

BBD	Box Behnken Design	*EO*	Electrooxidation
BOD	Biological Oxygen Demand	$NaCl_{conc}$	*NaCl* Concentration (g/L)
CCD	Central Composite Designs	*O&G*	Oils and Greases
CD	Current Density (mA/cm^2)	*PRW*	Petroleum Refinery Wastewater
COD	Chemical Oxygen Demand (ppm)	*RSM*	Response Surface Methodology
COD%	Removal efficiency	*TDS*	Total Dissolved Solids (ppm)
EC	Electrocoagulation		

When an Al anode is utilized, the following reactions 1, 2, and 3 take place [22, 23].

$$Al \rightarrow Al^{3+} + 3e^- \quad (1)$$

$$2H_2O \rightarrow 4H^+ + O_2 + 4e^- \quad (2)$$

$$xAl^{+3} + yOH^- \rightarrow Al_x + (OH_y^{z+}) \quad (3)$$

However, the following reaction happens in case of Al is cathode [22, 23]:

$$2H_2O + 2e^- \rightarrow H_{2(g)} + 2OH^- \quad (4)$$

Currently, there is a lot of interest in EO as a highly successful technique for eliminating a variety of contaminants. EO technology is very dependable and often employed due to its efficiency and environmentally friendly results [24]. Two methods that define EO processes: direct electro-oxidation, and indirect electro-oxidation. In reality, pollutants degrade in the direct EO process due to the direct electron transfer between the anode surface and contaminants [25]. Conversely, indirect electrolysis EO entails the homogenous interaction of organic contaminants with powerful oxidants generated during the electrolysis process, including Cl_2, H_2O_2, $HClO$, ClO^-, SO_4^{2-}, *and* O_3 [26]. EO technique has some advantages as: it generates disinfection compounds, entirely mineralizes persistent organic pollutants, needs simple equipment and operating conditions, and has minimal electrode maintenance costs [21]. Nevertheless, the EO approach has many disadvantages including its inefficiency in removing suspended materials and the possibility that the process's efficiency may be lowered if an impermeable film layer forms on the cathode [21]. The following reactions (5, 6, 7, 8, and 9) occur during the EO process at the anode, cathode, and in the bulk solution through direct and indirect oxidation reactions:

1. Direct oxidation Reaction [26]
   Anodic reaction:

   $$Organicpollutants(aq) \rightarrow Intermediateproducts + Electrons \quad (5)$$

   Cathodic reaction:

   $$Water(liquid) + ElectronsßHydrogengas(g) + Hydroxideions(aq) \quad (6)$$

2. Indirect oxidation Reaction with Addition of NaCl [27]:
   Anodic reaction:

   $$2Cl^- \rightarrow Cl_2 + 2e^- \quad (7)$$

   Cathodic reaction:

   $$2H_2O + 2e^- \rightarrow 2OH^- + H_2 \quad (8)$$

   At bulk:

   $$Cl_2 + H_2O \rightarrow HOCl + H^+ + Cl^- \leftrightarrow OCl^- + Cl^- + 2H^+ \quad (9)$$

Combining the two procedures will create a workable hybrid process, where EC is incomplete and fast process whole EO is complete and slow process [28]. This hybrid process can also remove the contaminants efficiently and instantaneously [28]. The current research aims to evaluate the practical application of using EC and EO techniques to treat actual wastewater obtained from the Najaf refinery by reducing the COD value and studying the effect of factors (time, NaCl concentration, CD, and pH) on the effectiveness of successive EC and EO procedures. It is clear that the current work has super estimation than previous works in the selection of the influence factors in the field of petroleum refinery wastewater treatment which led to advanced removal.

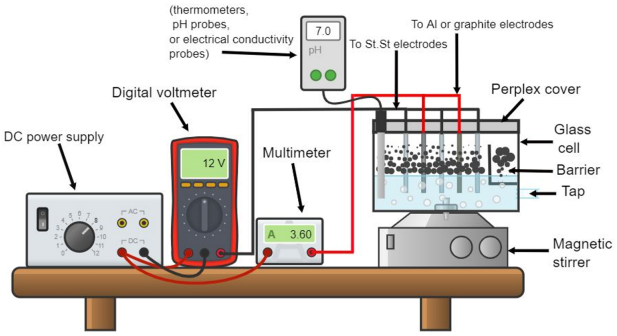


Figure 1. Diagram of the sequential EC &EO process.

2. Experimental Work

2.1 Materials and Tools

In the current study, 70 liters of wastewater samples have been collected from the feeding tank to the treatment unit of the Najaf refinery in Iraq. The sample are stored in resistant plastic containers at $4C°$to prevent any changes in their properties. Table 1 lists the variety of wastewater properties. In each experiment, one liter of collected wastewater has been filtrated into two stages using a cotton filter and a CTO filter to remove impurities, clay, and some fat and grease before treatment. Thomas Indian Bakers in brands of 1 M HCl (HCl,36%) and 5 M NaOH (NaOH, 97%) are used to adjust the pH measured using a microprocessor pH meter (HANNA, pH 211) and added NaCl (sodium chloride, 99.9%, Co. Barcelona, Spain) to increase the conductivity of the solution, as salt is one of the variables studied. Moreover, in each experiment, the electrodes are cleaned and activated by immersing the Al electrodes and SS in 5% (v/v) HCl for 10 minutes and then washing them carefully with distilled water. However, the graphite electrode has been washed by distilled water only.

Table 1. Wastewater properties

Properties	Values
pH	0008.30
TDS(ppm)	3500.00
Po_4(ppm)	0000.17
Cl^-(ppm)	2460.00
Turbidity (NTU)	0042.40
Oil (ppm)	0035.60
BOD (ppm)	0122.70
COD (ppm)	1250.00
Phenol (ppm)	0016.10

2.2 The apparatus

The experiments were conducted at room temperature ($25 \pm 5C°$) in a batch monopolar EC and EO electrochemical cell shown in the Fig. 1. The cell designed from a glass container with dimensions 20 cm length, 6 cm width and 15 cm height containing one side of a closed barrier with dimensions 6 cm length, 6 cm width, and 8 cm height used to collect the suspended sludge formed during the experiment, below this barrier, there is an outlet (tap) for collecting treated samples. After then, the cell was placed on a magnetic stirrer 250 rpm (DAIHAN LABTECH CO, 0-450 rpm) to achieve optimal mass transfer. The cathode electrodes in the EC process are three SS plates of type 316-AISI, whereas the anode electrodes are two Al plates. The cathode electrodes in the EO process were the same three SS plates, while the anode electrodes were two graphite plates. The electrodes possess identical dimensions, measuring 20×5×0.2 cm. The submerged dimensions of each side of the anode are 9×5 cm, where the effective surface area is 180 cm^2. The electrodes fixed in the cell

using a perplex cover with dimensions of 17 cm length, 5 cm width and 0.7 cm height which makes five incisions of the cover to insert the electrodes, and drilled a hole to insert (thermometers, pH probes, or electrical conductivity probes). Each anode is placed between two cathodes with a distance of 1.5 cm between them, the effective area of the aluminum and graphite electrodes was calculated. The anode electrode which could be Al in the process or graphite in the EO process connected to the positive port of a DC power supply (MCH-305 D-II, 0-30V, 0-5A dual output), while the cathodes SS connected to the negative port. Additionally, an RMS multimeter (UNI-T, UT803) was connected in series with the anode. Several initial experiments were undertaken to evaluate the range of elements under study and to get a general understanding of the hypothesized process. According to the data collected from these trials, the period of the EC process was about one hour, while the remaining time was allocated to the EO process. During the first hour of each run in the EC and EO tests, Al electrodes are used as anodes in EC process. Afterward, Al electrodes are substituted with graphite electrodes, and the EO process is used for the remaining duration. During each experiment, readings of the applied current and corresponding voltages using a digital voltmeter (Winapex, ET8101) for each electrode. The samples were collected, filtered using Whatman filter paper, and then evaluated to determine the treatment's performance in terms of COD.

2.3 Measurement and analysis method

The performance of the EC and EO cascade processes is evaluated based on the COD removal efficiency according to the following stages: stage 1, collecting 2 *ml* of both original and treated effluents. Stage 2, add the collected samples to a prepared vial (kit) containing an oxidizing agent and stir gently. Stage 3 placed it in a thermal reactor and expose it to a temperature of 150 for 120 minutes. Stage 4, kit taken out after the digestion process and left for 24 hours for the solution to stabilize. Finally, stage 5, analyzed using a spectrophotometer to determine the number of COD in ppm. The removal effectiveness can be expressed mathematically in equation 10 [29]. All experiments were conducted with high precision, and the experiment we doubt was repeated again.

$$COD\% = \frac{COD_{in} - COD_{out}}{COD_{in}} \times 100 \tag{10}$$

3. Results and discussion

The present research focused on four variables with controllable characteristics. NaCl concentration (g/l), CD (mA/cm^2), pH of the solution, and time (h). Table 2 represents three levels of these factors. To improve wastewater treatment, NaCl was added. These factors were chosen because they are important factors affecting wastewater treatment. Optimization experiments are conducted utilizing response surface methodology (RSM) using Box-Behnken design (BBD) to determine the statistically important operational parameters that impact the COD removal from wastewater [30], as shown in Table 3. BBD was used instead of central composite designs (CCD) because the experiments had few design points, where BBD will require fifteen experiments for three factors as compared to CCD (twenty experiments for three factor), BBD can be less expensive than CCD with the same factors' numbers. Box-Behnken designs do not have axial points while CCD usually have axial points outside the cube. thus, all points of design fall within safe operating zone. Hence, BBD is easy to predict the upper and lower limits at three level point.

Table 2. Levels of the experimental parameters with the customized levels.

Parameters	Symbols	Level 1	Level 2	Level 3
CD mA/cm^2	A	10.0	15.0	20.0
PH	B	04.0	07.0	10.0
Time h	C	02.0	03.5	05.0
NaCl conc g/l	D	00.0	01.5	03.0

3.1 Multiple regression model

Equation 11 represents the multiple regression equation. It shows the correlation between COD% and the parameters under study obtained using Design Expert 13 software. The model fits the data well, as shown by the correlation coefficient (R2) of 99.68%, as shown in Fig. 2 by how close the actual results are to those Predicted.

$$\begin{aligned} COD\% = {} & +80.37 + 5.77A + 0.2875B + 7.24C + 2.44D + 0.7725AB \\ & - 1.03AC + 0.8025AD - 4.65BC - 1.05BD + 0.9200CD + \\ & + 2.34A^2 + 4.03B^2 + 1.46C^2 + 1.15D^2 \end{aligned} \tag{11}$$

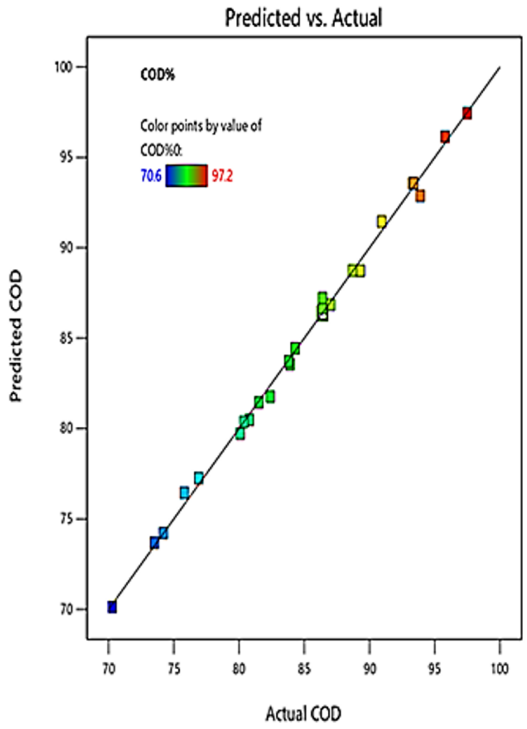


Figure 2. COD behavior against COD actual.

3.2 Analysis of variance (ANOVA)

Analysis of Variance (ANOVA) is a statistical tool used for optimization procedure. It may be used to determine the effect of the controllable factors. ANOVA gives a comprehensive grasp of the consistency of the perceived outcomes [31, 32]. The calculation of error variance may be achieved by ANOVA which determines whether the observed variation in the response is caused by changes in level adjustments or experimental standard errors. Furthermore, the influence of each component on the response may be determined using an F-value. F-values locate the impact of the operation on the response of the process, The results showed that F-values are greater than unity, this indicates that significant impact as shown in Table 4 [32, 33]. P-values measure whether the tests are controlled or not as shown Table 4. P-values are less than 0.05 which means that the tests are under control settings except.

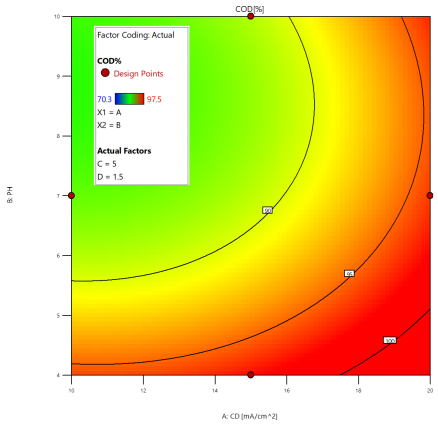
3.3 Effect of operational parameters on COD removal

3.3.1 Effect of PH

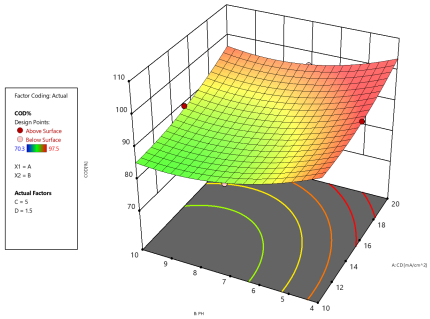
The results showed that the most expensive removal of COD% at the best time of 5 hours is at pH 4, as shown in Fig. 3 and 6. The reason is attributed to the formation of OH radicals and active chlorine species such as Cl_2, HOCl, and ClO^-. This result agreed with previous studies [34], which explained that the adsorption rate of OH radicals on the anode surface decreases when the pH increases [35]. Moreover, Al ion $Al(H2O)_6^{+3}$ which is formed at this pH value, contributes to the coagulation and treatment process [36]. In conclusion, acidic medium is preferred to obtain the highest COD% removal during the successive EC and EO processes.

3.3.2 Effect of Current Density (CD)

Current density is a fundamental factor in any electrochemical treatment process. The current experiments are adopted different current densities (10, 15, and 20mA/cm^2). It was found that the best removal of COD is at CD 20mA/cm^2, and this is shown in the Fig. 3, Fig. 4, and Fig. 5. This COD behavior is agreed with previous studies [27, 36]. In the EO process, increasing the current leads to an increase in the formation of HCLO, while in the EC process, increasing the current leads to an increase in the masses as a result of the release of a large amount of Al ions by anodic dissolution (according to Faraday's law) [37], which leads to an increase in the formation of Al hydroxides which is necessary for the formation of coagulants. It is clear that high currents mean increasing of electrodes decomposition which means an increase in costs, we resort to increasing the process time to reduce the current and obtain the same result as shown in Fig. 4.

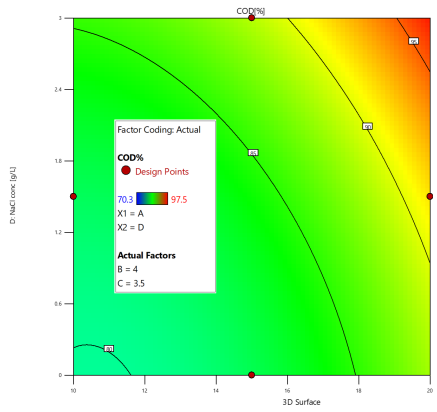


(a) pH

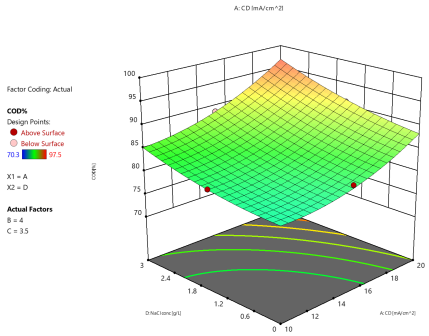


(b) CD

Figure 3. The contour and 3D for COD% vs. pH. and CD.

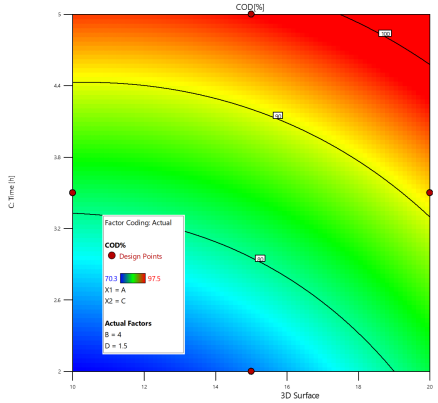


(a) pH

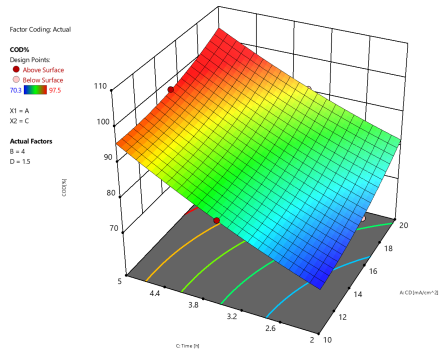


(b) CD

Figure 5. The contour and 3D for COD% vs. NaCl conc. and CD.

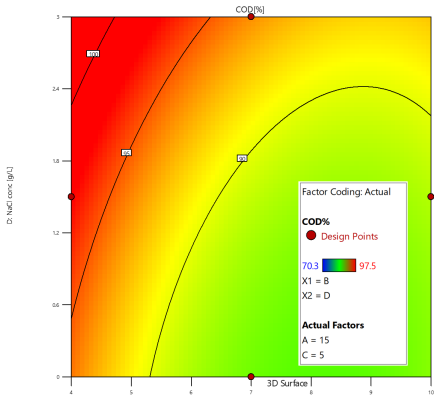


(a) pH

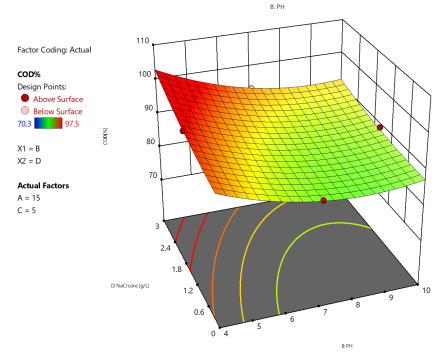


(b) CD

Figure 4. The contour and 3D for COD% vs. time. and CD.



(a) pH



(b) CD

Figure 6. The contour and 3D for COD% vs. NaCl conc. and pH.

Table 3. Box-Behnken model runs with operational parameters and effectiveness of removal.

Run No.	CD (mA/cm^2)	PH	Time, (h)	NaCl (g/l)	COD (ppm)	COD (%)	Run No.	CD (mA/cm^2)	PH	Time, (h)	NaCl (g/l)	COD (ppm)	COD (%)
01	10	04	03.5	01.5	231.000	81.52	14	15	10	02.0	1.5	201.250	83.90
02	20	04	03.5	01.5	113.375	90.93	15	15	04	05.0	1.5	031.250	97.50
03	10	10	03.5	01.5	240.000	80.80	16	15	10	05.0	1.5	133.750	89.30
04	20	10	03.5	01.5	083.750	93.30	17	10	07	02.0	1.5	371.250	70.30
05	15	07	02.0	00.0	322.500	74.20	18	20	07	02.0	1.5	202.500	83.80
06	15	07	05.0	00.0	162.250	87.02	19	10	07	05.0	1.5	170.000	86.40
07	15	07	02.0	03.0	288.750	76.90	20	20	07	05.0	1.5	052.500	95.80
08	15	07	05.0	03.0	082.500	93.40	21	15	04	03.5	0.0	220.000	82.40
09	10	07	03.5	00.0	302.500	75.80	22	15	10	03.5	0.0	196.250	84.30
10	20	07	03.5	00.0	170.000	86.40	23	15	04	03.5	3.0	141.250	88.70
11	10	07	03.5	03.0	248.875	80.09	24	15	10	03.5	3.0	170.000	86.40
12	20	07	03.5	03.0	076.250	93.90	25	15	07	03.5	1.5	245.375	80.37
13	15	04	02.0	01.5	331.250	73.50							

Table 4. Analysis of Variance (ANOVA) for COD%.

Source	Sum of Squares	df	Mean Square	F-value	p-value		Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	1262.63	14	090.19	222.81	< 0.0001	Significant	BC	86.49	01	86.490	213.68	< 0.0001
A-CD	0399.28	01	399.28	986.45	< 0.0001		BD	04.41	01	04.410	010.90	0.0080
B-PH	00.9919	01	0.9919	002.45	0.1486		CD	03.39	01	03.390	008.36	0.0160
C-Time	0628.14	01	628.14	1551.85	< 0.0001		A^2	15.440	01	15.440	038.14	0.0001
D-NaCl	0071.39	01	071.39	176.38	< 0.0001		B^2	45.790	01	45.790	113.13	< 0.0001
AB	0002.39	01	002.39	005.90	0.0355		C^2	06.03	01	06.030	014.89	0.0032
AC	0004.20	01	004.20	010.38	0.0091		D^2	03.72	01	03.720	009.18	0.0127
AD	0002.58	01	002.58	006.36	0.0302		Residual	04.05	10	0.4048		

3.3.3 Effect of Time

According to Faraday's law, time is an important factor in the treatment process in terms of its effect on the formation of active chlorine and Al ions. The present experiments spent 1 h for the EC process and the remaining time for the EO process (1-4 h). Figure 4, demonstrates that increasing in time leads to an increase in the COD removal rate for all current densities, which aligns with previous studies [38, 39]. As mentioned previously, increasing the time gives permission to reduce CD and thus control operational costs. The EC process is fast and effective in removing the suspended particles, while there are many limitations for treating organic materials, on the other hand the EO process is highly effective in reducing COD, however it requires longer time [27].

3.3.4 Effect of NaCl Concentrations

During the treatment process, NaCl is added to increase conductivity and to reduce the voltage and resistance of the solution. As shown in Eqs. 12 and 13, the different kinds of chlorine which are formed at the anode electrode, such as hypochlorous acid and hypochlorite ions, which breaks down the organic compounds through a process called indirect oxidation [36]. In this study, some experiments are conducted without adding NaCl, because of the working with the direct oxidation process. Compared with the experiments with added NaCl in quantities 0, 1.5 and 3 g/l. It is observed that when salt is added, its quantity is increased, an increase in the COD removal rate is observed at all current densities, as shown in Fig. 5 and Fig. 6. This is due to the increase in hypochlorous acid as well as the increase in ions absorbed on the electrode, thus reducing the use of high currents. This is consistent with previous studies [40].

$$2Cl^- + 2e^- \rightarrow Cl_2 \tag{12}$$

$$Cl_2 + H_2O \rightarrow HCl + HClO \tag{13}$$

4. Conclusion

The contaminants produced by petroleum refinery affect human health by polluting the water and the environment. There are many studies focused on possible technologies to control this toxies. In the present work, two techniques have been adopted to reduce the chemical oxygen demand COD on industrial wastewater taken from the Najaf refinery using successive EC and EO technology and the COD removal rate was reached below the standard limit. Several variables, such as CD, pH, NaCl concentration, and time, were measured and improved in 25 experiments using the RSM method using BBD in the Design-Expert 13 program. It was concluded that increasing CD and time led to an increase in COD removal efficiency, considering energy consumption and increased cost, as increasing the amount of NaCl leads to an increase in the conductivity of wastewater and thus reduces energy consumption and cost. COD removal is inversely proportional to the increase in pH value. It was found that the optimal conditions for treating industrial wastewater using the sequential EC and EO process were pH (4), time 5 h, CD 15mA/cm^2 and salt dose 1.5 g/l. In these conditions, about 97.5% of remove COD. The results of the analysis (ANOVA) and the F and P values in the current study showed that all variables influence COD removal, either directly or in comparison with another variable. Those results are hoped to be a starting step in the way to control pollutants of the environment.

Authors' contribution

All authors contributed equally to the preparation of this article.

Declaration of competing interest

The authors declare no conflicts of interest.

Funding source

This study didn't receive any specific funds.

Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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How to cite this article:
Laith Louay Abdullah, and Shaker Salih Bahar. (2025). 'Petroleum refinery wastewater treatment through the successive process of electrocoagulation and electrooxidation', Al-Qadisiyah Journal for Engineering Sciences, 18(2), pp. 163-169. https://doi.org/10.30772/qjes.2024.151030.1274