

# Treatment of Aqueous Solution Containing Acid red 14 using an Electro Peroxone Process and a Box-Behnken Experimental Design

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## A-R-T-I-C-L-E-I-N-F-O

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## A-B-S-T-R-A-C-T

**Background & Aims of the Study:** Azo dyes utilized in industrial processes, such as the textile manufacturing, lead to the creation of huge amounts of colored wastewaters that contain non-organic and organic constituents. Therefore, it is necessary to search for remedies in this regard. This study investigated the degradation and mineralization of Acid red 14 (AR14), which is a mono Azo dye generally employed in textile manufacturing, using an Electro peroxone process. The Electro-peroxone is a grouping of ozone and electrochemically generated hydrogen peroxide that can result in the production of strong hydroxyl radicals.

**Materials and Methods:** This project was accompanied on synthetic wastewater that holds a high concentration of Acid red 14(400 mg/l) based on a Box-Behnken experimental design using an Electro-peroxone process for the remediation. Moreover, the influence of operational parameters was investigated in this study.

**Results:** The results obtained from an Electro-peroxone process in a cylinder-shaped reactor showed 100% AR14 removal after 30 min with an initial dosage of dye at 400 mg/l at an optimum condition (current intensity at 0.7 A, pH at 10, reaction time at 30 min, and electrolyte concentration at 0.1 M). Moreover, the removal percentage of the chemical oxygen demand was obtained at 69% after 30 min indicating the great performance of Electro-peroxone in the mineralization of AR14.

**Conclusion:** The hydrogen peroxide is produced electrochemically from O<sub>2</sub> in the O<sub>2</sub>-O<sub>3</sub> mixture, which was entered into the reactor. Subsequently, the hydroxyl radicals were shaped via the peroxone reaction. Based on the high removal percentages of COD in short reaction time, it can be found that the Electro-peroxone process produces no secondary pollutants. Therefore, it can be regarded as an environmentally-friendly water treatment method.

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## Background

The organic dyes originated from textile productions utilize a large volume of water over the dyeing process. Following that, wastewaters

containing dyes are discharged into rivers or public sewages (1, 2). Therefore, it is necessary to find an operative way to eliminate the color from the textile wastes. The conventional approaches are not appropriately efficient since dyes are scarcely transferrable due to their low

molecular weight and high solubility in water (3). Although conventional techniques are not destructive, they only transfer the pollution from one phase to another leading to secondary pollution, which is necessary to be handled using extra treatments (4-6). Several advanced oxidation processes have been recommended and extensively employed for the degradation of many toxic, as well as non-biodegradable, carcinogenic, and mutagenic pollutants (7, 8).

The Electro-peroxone is a new branch of advanced oxidation processes exceeding ozonation or peroxone due to its benefits. According to the findings of some studies, the Electro peroxone can remove pollutants from wastewaters at a more accelerated rate, compared to electrolysis, ozonation, and the combined O<sub>3</sub>-electrolysis method. Hydroxyl radicals produced from several mechanisms resulted in the improvement of this process (9, 10). Generally, ozone is employed along with other techniques, like H<sub>2</sub>O<sub>2</sub>, TiO<sub>2</sub> and UV in the treatment of colored wastewater to improve pollutant mineralization efficiency (11). The peroxone process is the utilization of H<sub>2</sub>O<sub>2</sub> together with O<sub>3</sub>, which has an important synergistic effect on the degradation of pollutants. This combined effect resulted mainly from the reaction of H<sub>2</sub>O<sub>2</sub> with O<sub>3</sub> to form hydroxyl radicals (12). Since the only by-products of H<sub>2</sub>O<sub>2</sub> and O<sub>3</sub> are O<sub>2</sub> and H<sub>2</sub>O, they are not able to make secondary toxins (13).

With this background in mind, this project aimed to study the possibility of decolorization and mineralization of AR14 using an Electro peroxone process. Moreover, this study attempted to appraise the effect of different

effective factors, like current intensity, pH, time of reaction, and electrolyte concentration.

## Materials & Methods

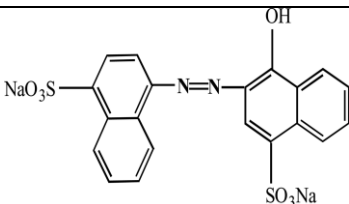
### Materials

Acid red 14 (AR14) was acquired from Afrand Tuska company (Iran) and employed without further purification. In addition, H<sub>2</sub>SO<sub>4</sub>, Na<sub>2</sub>SO<sub>4</sub>, and NaOH are all obtained from Merck. Table 1 tabulates the chemical properties of AR14. It should be noted that distilled water was employed during the work.

### Experimental Setup

The removal of synthetic wastewater containing 400 mg/l of AR 14 is performed by an Electro peroxone process in a glass column reactor (Figure 1). The ozone was generated from a capsule containing high-purity of Oxygen (99.9%) passing through an ozone generator (Arda Co., Iran). The concentration of O<sub>3</sub> in the outlet of the generator (O<sub>3</sub>-O<sub>2</sub> mix), can be attenuated using the varying power of the ozone producer. Subsequently, the ozone generator discharge was entered to the bottom of reactor at a fixed flow rate of 0.25 L/min through a fine diffuser. Moreover, a DC power supply was utilized to perform the treatment under galvanostatic settings. The anode and cathode were a platinum (Pt) sheet and a carbon-PTFE electrode, respectively. The carbon-PTFE electrode was organized by anhydrous alcohol, Vulcan XC-72 carbon powder (Cabot Corp., USA), and PTFE dispersion (15). It is worth mentioning that each electrode had an exposed

Table 1) Chemical structure and maximum absorption of Acid red 14.

Pollutant	Molecular Structure	$\lambda_{\max}$ , nm	M <sub>w</sub> (g mol <sup>-1</sup> )
Acid Red 14		515	501

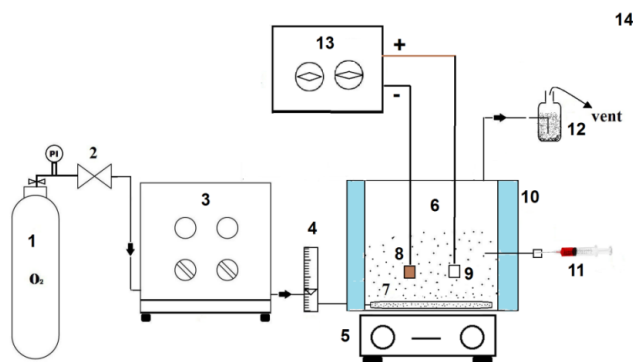


Figure 1) Graphic diagram of used setup in experimental scale.

1. Capsule of Oxygen, 2. Cut off valve, 3. Ozone Generator, 4. Rotameter, 5. Magnetic Stirrer- Semi bath reactor, 7. Diffuser, 8. Cathode, 9. Anode, 10. Water bath jacket, 11. Sampling point, 12. Ozone destructor (containing 2% KI solution), 13. DC power, 14. Wooden Box

area of 25 cm<sup>2</sup>.

The space between the cathode and anode was 2.5 cm, and the supporting electrolyte was Na<sub>2</sub>SO<sub>4</sub> solution. Regarding the treatment of the Electro peroxone, the ozone generator and DC power supply were turned on concurrently. In all tests, the temperature was fixed at 25°C using a thermo bath. Furthermore, the temperature of the solution in the reactor was adjusted by recirculation of water through a thermostat.

The setup included a cylindrical Pyrex reactor (1 L), which could be mixed magnetically. The top of the reactor was closed, and the initial pH was controlled through a dilute solution of caustic and sulfuric acid by a Sartorius pH meter PT-10P (Germany).

### Analytical Procedure

About 400 mg/l of AR14 was prepared by dissolving the suitable amount in 1 L distilled water. During the experiment, 3 ml aliquots were taken at every each 10 min until 30 min. in the next stage, the samples were taken and then considered at 515 nm using a UV-Vis spectrophotometer (Agilent, 5453, American). Regarding the absorbance versus concentration, the calibration plot was used based on the Beer Lambert's law. The reduction in the absorbance at 515 nm was due to the removal of the AR14 solution. The chemical oxygen demand (COD) was determined according to

standard method (16).

The appropriate efficiencies were assessed in terms of its initial value, and the removal percentages of AR14 and COD were obtained as follow (Equations 1-2):

$$\text{Removal of AR14}(\%) = \left( \frac{(AR14)_0 - (AR14)}{(AR14)_0} \right) \times 100 \quad (1)$$

$$\text{Removal of COD}(\%) = \left( \frac{(COD)_0 - (COD)}{(COD)_0} \right) \times 100 \quad (2)$$

Where (COD)<sub>0</sub> and (AR14)<sub>0</sub> are amount of COD and concentration of AR14 at the beginning of the reaction, respectively. In addition, COD and AR14 are the amounts of COD and the concentrations of AR14 at time t, respectively.

### Experimental Design and Statistical Analysis

The Box-Behenken experimental method was used to remove AR 14 from synthetic wastewater completely. The effects of current intensity, pH, reaction time, and electrolyte concentration were investigated on the removal of AR 14. The input variables and their values were offered in Table 2. Since the adjustment of the ozone flow rate was difficult in the reactor, it was fixed at 0.25 L/min in all runs, and the concentration of ozone was not measured at this flow rate. Moreover, the concentration of AR 14 was fixed at 400 mg/l in all experiments (near to actual concentration

rate in some industries).

The Box-Behnken experimental design requires a smaller sum of tests, related to all response surface methodologies (RSM) (17). The resulting Equation (2) is suggested for the response variable (Y) as a polynomial equation of free variables.

$$Y = b_0 + \sum b_i x_i + \sum \sum b_{ij} x_i x_j + \sum \sum b_{ii} x_i^2 + \varepsilon \quad (2)$$

Where  $b_0$  is a constant number,  $\varepsilon$  signifies the remainder of the equation,  $b_i$  denotes the slope of the variable,  $b_{ii}$  indicates the quadratic

coefficient ( $i = 1, 2, 3, 4$ ), and  $b_{ij}$  presents a linear interaction among the input variables of  $x_i$  and  $x_j$  ( $i = 1, 2$  and  $j = 1, 2, 3$ ). Regarding the evaluation of the significance of each variable in the polynomial equation (2), an analysis of variance (ANOVA) was employed in this study (18). A p-value fewer than 0.05 was considered statistically significant. Moreover, F-value was employed to define the statistical significance of the second-order models. The p-value will be much minor if the F-value is lower than the calculated F-value in Table 3, thereby identifying the significance of

**Table 2) Levels and range of the variables.**

Variables	Symbol	Range and levels		
		0.3	0.6	0.9
Current Intensity(A)	$X_{CI}$	0.3	0.6	0.9
pH	$X_{pH}$	4	7	10
Reaction time(min)	$X_t$	10	20	30
Electrolyte concentration(M)	$X_{EC}$	0.025	0.075	0.125

**Table 3) Experimental design for four independent variables and their responses**

Run no.	$X_{CI}$	$X_{pH}$	$X_t$	$X_{EC}$	Exp	Pred.
1	0.3	4	20	0.075	60.7	61.24
2	0.6	10	20	0.125	76.0	76.73
3	0.9	4	20	0.075	68.0	67.96
4	0.6	10	30	0.075	99.6	97.50
5	0.6	10	20	0.025	59.0	59.62
6	0.9	7	30	0.075	90.6	90.22
7	0.6	7	20	0.075	72.5	72.60
8	0.6	7	20	0.075	72.6	72.60
9	0.3	7	20	0.025	45.0	42.70
10	0.6	4	30	0.075	98.3	96.30
11	0.3	10	20	0.075	63.0	63.04
12	0.9	7	20	0.025	47.5	48.51
13	0.6	7	10	0.025	29.0	28.26
14	0.3	7	20	0.125	61.0	59.21
15	0.6	4	10	0.075	43.0	44.33
16	0.6	4	20	0.125	75.1	75.23
17	0.9	10	20	0.075	70.0	69.46
18	0.6	7	30	0.025	73.0	74.37
19	0.6	7	20	0.075	72.7	72.60
20	0.3	7	10	0.075	31.0	32.13
21	0.9	7	10	0.075	34.0	32.40
22	0.6	7	10	0.125	41.5	40.12
23	0.6	7	30	0.125	96.3	97.04
24	0.3	7	30	0.075	75.0	77.35
25	0.6	4	20	0.025	57.8	57.82
26	0.9	7	20	0.125	65.0	66.53
27	0.6	10	10	0.075	45.2	46.43



the statistical model.

## Results

This study included 27 tests. The removal percentages of AR 14 and the predicted values by the model are summarized in Table 3.

### Optimization and Modeling of AR 14 removal in an Electro Peroxone Process

This study investigated the effect of four independent variables on the response function using the Box-Behnken design (BBD) and response surface methods (RSM) to acquire the optimum conditions. The mathematical relationship between four significant variables and responses is obtained by a quadratic polynomial equation. The equation for the removal of AR 14 is presented as the following (Equation 3):

$$\begin{aligned} \text{Removal of AR 14(\%)} = & -40.8 + 123.9X_{Cl} - \\ & 4.11X_{pH} + 3.365X_t + 546.2X_{EC} - 112.69X_{Cl}^2 + \\ & 0.33X_{pH}^2 - 0.0442X_t^2 - 3287X_{EC}^2 - 0.08X_{pH}X_{Cl} + \\ & 1.05X_{Cl}X_t + 25X_{Cl}X_{EC} - 0.0075X_tX_{pH} - \\ & 0.5X_{EC}X_{pH} + 5.4X_tX_{EC} \end{aligned} \quad (3)$$

Table 4 presents the achieved results obtained from the BBD with residuals for all runs. The predictions about the response for the given levels of each factor can be made using the equation of real factors. At this point, the levels should be stated in the unique units for each factor.

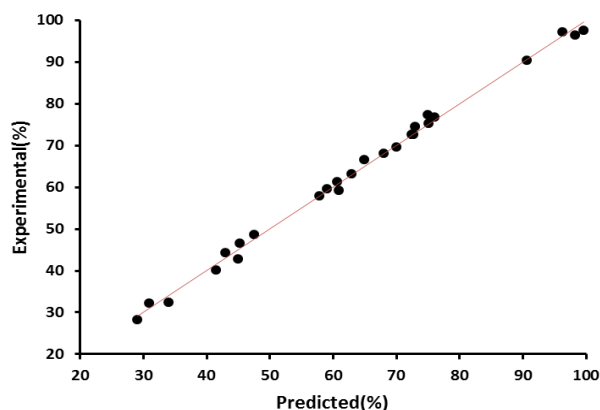
As can be seen in Table 4, with respect to the significance of the coefficients, the F-value of the model is 219.66, which signifies the significance of the model. P-values less than 0.05 are considered statistically significant. On the other hand, values larger than 0.10 were regarded as insignificant.

The linear terms (i.e.,  $C_{Cl}$ ,  $C_t$ ,  $C_{EC}$ ) and their

**Table 4) An analysis of variance for quadratic models in the removal of AR 14 using an Electro peroxone process**

Source	DF	Adj SS	Adj MS	F-Value	P-Value
<b>Model</b>	14	10130.4	723.60	219.66	0.00
<b>Linear</b>	4	8993.8	2248.46	682.54	0.00
$X_{Cl}$	1	129.4	129.36	39.27	0.00
$X_{pH}$	1	8.2	8.17	2.48	0.141
$X_t$	1	7961.9	7961.90	2416.92	0.00
$X_{EC}$	1	894.4	894.41	271.51	0.00
<b>Square</b>	4	1066.9	266.72	80.97	0.00
$X_{Cl}^2$	1	548.6	548.55	166.52	0.00
$X_{pH}^2$	1	47.1	47.07	14.29	0.003
$X_t^2$	1	104.6	104.63	31.76	0.00
$X_{EC}^2$	1	360.1	360.07	109.30	0.00
<b>2-Way Interaction</b>	6	69.7	11.61	3.52	0.03
$X_{pH}X_{Cl}$	1	0.0	0.02	0.01	0.935
$X_tX_{Cl}$	1	39.7	39.69	12.05	0.05
$X_{Cl}X_{EC}$	1	0.6	0.56	0.17	0.687
$X_{pH}X_t$	1	0.2	0.20	0.06	0.808
$X_{pH}X_{EC}$	1	0.0	0.02	0.01	0.935
$X_tX_{EC}$	1	29.2	29.16	8.85	0.012
Error	12	39.5	3.29		
Lack of fit	10	39.5	3.95	395.11	0.03
Pure error	2	0.0	0.01		
Total	26.0	10169.9			
Model Summary	S	R <sup>2</sup>	R <sup>2</sup> <sub>adj</sub>	R <sup>2</sup> <sub>pred</sub>	
	1.815	0.9961	0.9916	0.9776	



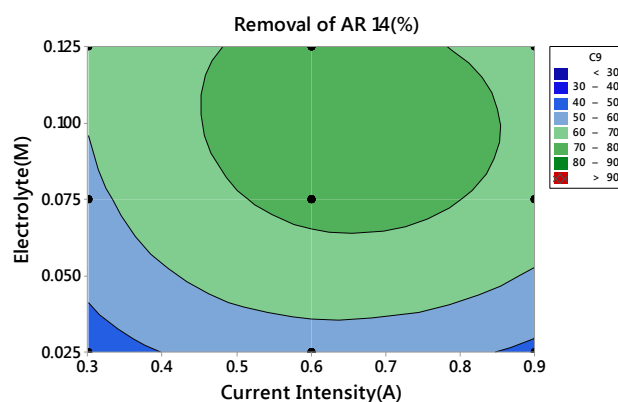


**Figure 2) Evaluation of the experimental data and predicted values in the removal of AR 14 in Electro peroxene process**

quadratic terms obtained p-values less than 0.01; therefore, they were regarded as statistically significant. However, the p-value of linear  $C_{pH}$  was estimated at 0.141 (larger than 0.05); accordingly, pH seems to be an insignificant factor. As it is clear from the experimental results, the response rate is high at acidic and alkaline pH; however, it is low in a neutral condition. Nonetheless, the p-value of the binary interaction terms among the variables was determined at 0.05 indicating an insignificant interaction among variables. Therefore, the insignificant terms were removed to improve the model (Table 4).

As presented in Table 4, the lack of fit is significant, associated to the pure error because its p-value (0.03) is less than 0.05. This signifies the suitability of the model to predict AR 14 removal efficiencies in the ranges of the process variables. The predicted value of  $R^2$  (0.9776) is completely in line with the adjusted value of  $R^2$  (0.9916) (i.e., the difference is less than 0.2).

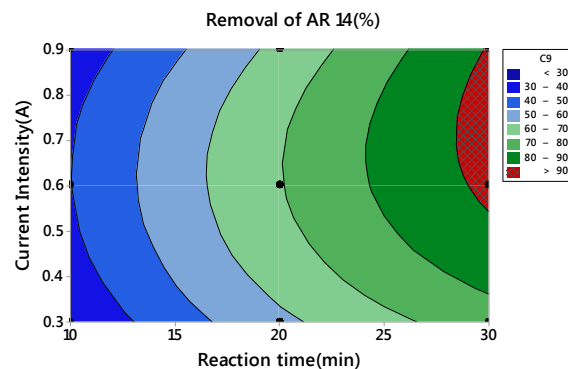
The precision of the model (Figure 2) compared the experimental values with the predicted responses obtained from the model regarding the degradation of AR 14. The results indicated the consistency between the predicted responses from the model and the experimental data.



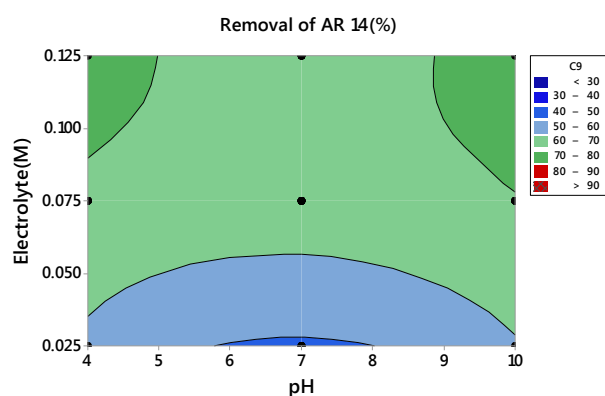
**Figure 3) Counter plot of the interaction between current intensity and electrolyte concentration on the removal of AR 14**

### *Influence of the Current Intensity*

Figures 3-4 illustrate the influence of applied current on the electro peroxene method. It was clear that an increase in the current intensity from 0.1 to 0.7 A could increase the removal of AR 14, whereas an additional increase in current to 0.7 A had no more influence. Conversely, the dye degradation efficiency was reduced by an increase in current to 0.9 A. As reported by previous studies (19), an increase in degradation efficiency originated from the increased current intensity, which was related to the growth in hydrogen peroxide reduction at the cathode surface. More hydrogen peroxide can cause more hydroxyl radical regeneration, which in turn progresses AR 14 removal. This



**Figure 4) Counter plot of the interaction between the time of reaction and current intensity on the removal of AR 14**

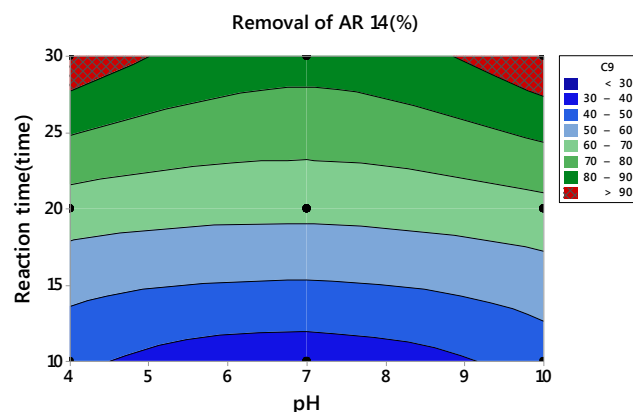


**Figure 5) Counter plot of the interaction between pH and electrolyte concentration on the removal of AR 14**

process occurs commonly at the applied current of 0.7 A. The growth in current up to 0.7 A has a slight or even negative impact on the dye removal efficiency. In this condition, the production of hydroxyl radicals is saturated since the dissolution rate of ozone in water is low, and extra hydrogen peroxide reacts with the hydroxyl radicals. It can be mentioned that the degradation of hydrogen peroxide at the surface of cathode was increased at high current. Moreover, the rising of different potential may result in the reduction of hydrogen peroxide instead of oxygen at the surface of cathode (20).

#### ***Effect of Electrolyte Concentration***

The electrolyte concentration can have effects on the electrochemical process. Based on the previous studies,  $\text{Na}_2\text{SO}_4$  is one of the top electrolytes for the Electro peroxone and electrochemical processes (21). Therefore, as presented in Figure 5, different concentrations of  $\text{Na}_2\text{SO}_4$  (i.e., 0.025, 0.075, and 0.125) are considered on the removal of AR 14. Electrolytes can increase the speed of the ions exchange and the electrostatic resistance between electrodes and ions, thereby intensifying the performance of electrodes and reactions, like the reduction of oxygen to hydrogen peroxide at the surface of the cathode. Accordingly, the generation of hydrogen



**Figure 6) Counter plot of the interaction between pH and reaction time on the removal of AR 14**

peroxide by an electrode was reduced in low electrolyte concentration (22) leading to the reduction of the Electro peroxone process efficiency. It should be noted that the high concentrations of  $\text{Na}_2\text{SO}_4$  can result in the production of sulfate radicals that have a high oxidation potential or even hydroxyl radical depletion (23). Therefore, the optimum concentration of electrolyte was obtained in this study.

#### ***Effect of Initial pH***

The pH was prepared using 0.1 M solutions of NaOH and HCl with no control during the reaction. The pH was reduced slightly due to the production of acidic intermediates. The influence of initial pH (4, 7, and 10) was investigated on the removal of AR14, and the results were presented in Figure 6.

#### ***Optimization of the Operational Conditions***

The removal percentages of AR 14 can be high in some circumstances. The values of different variables were designated to magnify the removal percentages of AR 14 using the Minitab software. Table 5 summarizes the optimal values and the removal percentages of AR 14.

The predicted optimum conditions by the model were current intensity at 0.7 A, pH at 10, reaction time at 30 min, and electrolyte

**Table 5) Optimal conditions of parameters and AR 14 removal (%)**

Parameters	Unit	Value
Current Intensity	Amper	0.7
pH		10
Reaction time	min	30
Electrolyte Concentration	mg/l	30
Predicted AR 14 removal	%	102.43
Experimental AR 14 removal	%	100

concentration at 0.1 M. Under the predicted optimum conditions, the experiment was repeated and the removal efficiency of AR 14 was 100%. Moreover, the predicted value commended by the software in the removal of AR 14 was estimated at 102.6 %. The COD was reduced from 600 mg/l to 190 mg/l, and the pollutant was degraded completely; however, the intermediate product had COD.

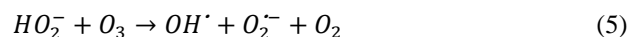
## Discussion

The Electro peroxone process can be easily utilized with a carbon-based cathode. On the other hand, the metal electrodes may decay  $H_2O_2$  catalytically, and therefore, cannot produce  $H_2O_2$  from  $O_2$  (24). The carbon-based electrodes (e.g., carbon-PTFE) can change  $O_2$  to  $H_2O_2$  because of their high over potential for  $H_2$  evolution and little catalytic activity for  $H_2O_2$  degradation (25, 15). The results of this project revealed that the unity of ozonation and electrolysis in the Electro peroxone process might lead to gain a major synergy for the removal of AR14 in synthetic wastewater. This concurrence can be generally related to some mechanisms increasing  $O_3$  transformation to hydroxide in the Electro peroxone system. Moreover, it results in an increase in the electro-generation of  $H_2O_2$  from  $O_2$  at the carbon-PTFE cathode and the following electro-reduction of  $O_3$  to hydroxide ion near the cathode.

Therefore, a key parameter in the high removal of pollutants in the Electro peroxone process is the operative generation of  $H_2O_2$

from  $O_2$  at the cathode. It should be noted that high current intensity can result in the production of high hydrogen peroxide molecules and subsequently hydroxyl radicals. Additionally, in high concentrations, they can scavenge each other and react with hydrogen peroxide to produce hydro peroxyl as a less oxidative agent than hydroxyl radicals.

Given the following reactions (Equations 4-5), the ozonation efficiency is usually more optimized in alkaline pH based on the reaction of ozone with hydroxide ions, production of  $HO_2^-$ , and its reaction with ozone to produce hydroxyl radical.



This mechanism also happens in the Electro peroxone process at alkaline conditions; however, in this process, it is explored a scavenging effect because of the existence of hydrogen peroxide, consumption of soluble ozone, and slowness of hydroxyl radical production. As can be seen from the counterplots, the high effectiveness of Electro-peroxone in an acidic condition might be originated from better performance of carbon-based electrodes in producing hydrogen peroxide (26).

## Conclusion

The Electro peroxone is a new branch of advanced oxidation processes that exceeds ozonation or peroxone due to its benefits. In this instrument, the joining ozone and electrochemically produced hydrogen peroxide lead to the generation of hydroxyl radicals, which are the robust oxidizing species. The results showed 100% removal after 30 min with an initial concentration of AR 14 at 400 mg/l at optimum conditions (i.e., current intensity at 0.7 A, pH at 10, reaction time at 30 min, and



electrolyte concentration at 0.1M). Furthermore, the removal of COD was determined at 69% for 400 mg/l initial concentration after 30 min indicating the great performance of Electro-peroxone regarding the treatment of wastewaters. In a similar vein, H<sub>2</sub>O<sub>2</sub> was manufactured electrochemically from O<sub>2</sub> in the O<sub>2</sub>-O<sub>3</sub> mixture, which was entered into the experimental setup. Subsequently, hydroxyl radicals were shaped via the peroxone reaction of in-situ produced H<sub>2</sub>O<sub>2</sub> with sparged O<sub>3</sub>. Based on the high removal percentages of COD in a short reaction time, it can be found that the Electro peroxone process produces no secondary pollutants. Therefore, Electro peroxone can be considered an environmentally-friendly treatment method.

## Footnotes

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### Conflict of Interest

The writers announce that they have no conflicts of interest concerning the publication of the study.

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