The Eurasia Proceedings of Science, Technology, Engineering & Mathematics (EPSTEM)
ISSN: 2602-3199

The Eurasia Proceedings of Science, Technology, Engineering and Mathematics (EPSTEM), 2025

Volume 37, Pages 1124-1134

ICEAT 2025: International Conference on Engineering and Advanced Technology

Electro-Fenton Oxidation for Degradation of Direct Red Organic Pollutants Using Stainless Steel and Iron Electrodes

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Abstract: The most common pollutant in industrial wastewater is organic compounds. Traditional wastewater treatment systems do not remove effect of toxic and effect of dangerous pollutants from the wastewater. The newest and most powerful method for treating wastewater from organic pollutants is the electro-Venton method, and it is considered an environmentally friendly method. Response surface methodology (RSM) and Minitab-17 method were used to study the impact of operational parameters such as current (0.5-2 mA), electrolysis duration (10–30 min), and Hydrogen peroxide concentration (0.0003 –0.0015 M). The pH, agitation speed, and the sodium chloride solution were all adjusted to 250 rpm, 3 g, and 0.25 g, respectively. Pollutants and their intermediate reaction products were found to be promptly eliminated and mineralized. When the operating variables were tuned, more than 98% of organic material removal efficiency and 39.67 kWh/m3 energy consumption were attained.

Keywords: Electro-Fenton; Dyes; Wastewater; Hydroxyl radicals

Introduction

Textile wastewater pollution is one of the major environmental problems that the earth and life are facing nowadays (Casado, 2019). Textile production is one of the greatest industrial wastewater emitters, releasing around 125–150 L of water per kilogram of textile output (Rajkumar & Muthukumar, 2017). Even at extremely low quantities, the presence of dyes in textile effluents is noticeable and unwanted. Colored wastewater impacts the aesthetic quality of the water and inhibits light penetration as well as photosynthesis in aquatic organisms (El Messaoudi et al., 2016). These an effluents are a major source for the an aquatic environmental pollutions. In general, effluents are composed of multiple inorganic and organic components derived from the nature of the

- Selection and peer-review under responsibility of the Organizing Committee of the Conference

feedstock, which is mostly composed of hydrocarbons in addition to a variety of other components (Diya'uddeen et al., 2011; Hasan et al., 2012).

Because of the growing production and distribution of dye all around the world, the threats of dye effluent to freshwater and marine habitats have escalated. Wastewater is a worldwide problem owing to its economic and environmental consequences (Abdelwahab et al., 2017). Textile wastewaters if not efficiently treated would consititue a serious environmental issue for water pollution (Li et al., 2017). The high concentrations of dyes and hazardous chemicals found in industrial wastewater make its treatment extremely difficult. Therefore, effective treatment technologies must be developed to reduce or remove these pollutants before they are discharged into the environment, as discharging them in large quantities causes significant environmental damage (Fakhru'l-Razi et al., 2009).

Conservative treatment techniques have been implemented to be less aggressive in removing impurities from a wastewater due to their toxic, intractable nature also non-biodegradable (Alvarez-Corena, Bergendahl, & Hart, 2016). These positive reuses reduce the outflow of potable water, which is more very precious commodity in many places around the world (Fathy et al., 2018; Galvão et al., 2006; Okiel et al., 2011). To remove pollutants, including dyes, from wastewater, various traditional techniques have been used. Biological-treatment (Mousa & Al-Hasan, 2017), coagulation /flocculation (Li et al., 2005), adsorption (Hathal et al., 2023; Mohsen et al., 2024), and separation by membrane (Abed et al., 2025; Naeem & Hassan, 2018). All these techniques are costly and frequently produce harmful byproducts. In any case, these procedures are non-destructive since they just converted non-biodegradable waste from one-form to another, Due to the emergence of a new type of pollution, it has become necessary to devise additional methods to address it (Naeem, Hassan, & Al-khateeb, 2018). Among the several physicochemical strategies researched in the treatment of wastewater over the years, One of the most prominent methods used to eliminate the organic pollutants found in wastewater is the Advanced Oxidation Processes (Hassan et al., 2018). AOPs are widely described as near temperature of ambient treatment methods that use highly radicals reactive, particularly the hydroxyl radical (OH•) , as the principal oxidant (Nidheesh & Gandhimathi, 2012). The most stubborn pollutants in wastewater can only be removed using the AOPS method. These techniques may entirely breakdown the pollutants into inoffensive inorganic elements such as CO2 and H2O under tolerable circumstances (Caliman et al., 2011). Recently, huge attention has been focused on chemical oxidation advancements such as electro-fenton (Baiju et al., 2018).

The reaction between hydrogen peroxide (H_2O_2) present in the system and the electrically produced ferrous ions will lead to the generation of hydroxyl radicals (OH•). This reaction occurs at a pH between 2 and 3. It is worth noting that iron remains in the treated water (Davarnejad et al., 2014). Electrochemical oxidation processes are considered successful and important solutions for treating many organic pollutants, noting that this method does not require the addition of chemicals because it is essentially formed during oxidation and reduction reactions. This method was the leading method that emerged during the last decade (Hammouda et al., 2019). Mixing the catalyst (SC-Fe^{2+}) with an agent that oxidize (diverse doses of H_2O_2) leads to the synthesis of the oxidized dye. This is achieved using the efficient and environmentally friendly electro-Fenton process. It is worth noting that the electro-Fenton process is particularly effective because ferrous ions are not rapidly oxidized by citrate ions (Buftia et al.,2018; Dang et al., 2019).

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO^- + HO^\bullet \quad (1)$$

$$O_2 + 2H^+ + 2e^- \rightarrow H_2O_2 \quad (2)$$

$$O_2 + H_2O + 2e^- \rightarrow HO_2^- + HO^- \quad (3)$$

The current research focused on removal of the organic pollutants from wastewater using electro-Fenton-oxidation with how to determine the optimal hydrogen peroxide concentration value, as well as examining the effects on current and electrolytic duration in electro-Fenton process. Also, the organic material removal efficiency and energy consumption were studied according to the operating variables.

Materials and Methods

Materials

The chemical structure of Direct red is shown in Fig.1. In this investigation, H_2SO_4 (purity 98% SDFCL), H_2O_2 from (45% wt /wt Germany), NaCl (purity 99% India), and sodium hydroxide (Thomas baker) were employed.

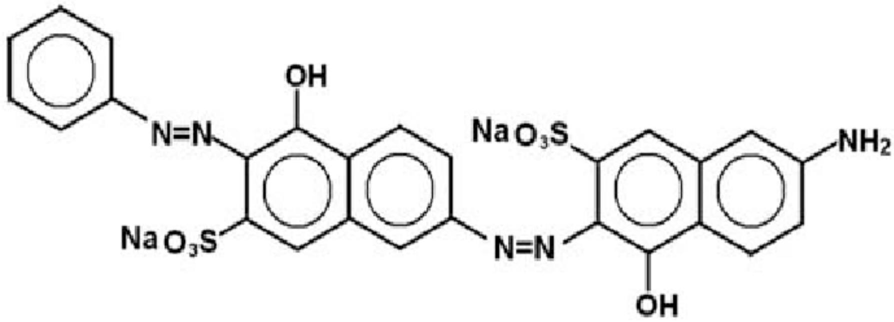

Figure 1. Discretion of the structure of direct-red.

Electrodes

The anode and cathode electrodes used in the study were stainless -steel and iron, respectively. The iron electrode was 10 cm * 8 cm * 0.3 cm in size. The stainless steel electrode measured 0.1 cm*8 cm*10 cm. In the Electro-Fenton technique, the effective electrode area is 30 square centimeters. All internal distances between the electrodes are 5 cm.

Electro Oxidation Procedure

All the experiments were carried out in an electro-Fenton glass reactor (batch reactor) consisting of an 800 ml. The starting pH of 300 mL of Direct-red was corrected to (3) by using (NaOH) sodium hydroxide with (H_2SO_4) sulfuric acid (1N) solutions. 0.25 gram of sodium chloride was added to the electrodes, and then they were supplied with DC electricity source (RXN-305D). The DC voltage remained at 29.6. As indicated in Figure 2, use the power source for ten minutes to swept ferrous ions inside the reactor and then add amounts from the H2O2 with 250 rpm for all trials as stirring speed to set.

Figure 2. The electro-oxidation reactor scheme.

To separate the sludge, all the studied samples were separated by centrifugation at a speed of 2000 rpm for ten minutes. These samples were taken at regular intervals. The floating material obtained was organically analyzed. The percentage of residue organic at the time t (Ct) to original dye was used to calculate dye reduction (C0).

Further research was conducted by altering the H2O2 dose, current, and time. All electrodes were properly washed with water before to use in the E-Fenton procedure to remove any additional debris. The electrodes then were immersed in 1M Hydrochloric acid for one hour before being immersed in 1M NaOH for another hour time. It is worth noting that the electrodes used in this study are stored in distilled water when not in use. To remove of any potential contamination, the electrodes were rinsed in 1M Hydrochloric acid and 1M NaOH after each usage.

The following equation was used because energy consumption (kWh/m³) is an important factor in any handling procedures (AlJaberi, 2018):

$$E = \frac{U.I.t}{1000\, V} \tag{4}$$

The symbol U (volte) refers to used voltage, t: contact time (h), I (Amps) refers to the applied current, V (m^3) the amount of the direct red wastewater.

AOPS Treatment and Kinetic Study

The fluctuation in organic contents in direct red throughout the advanced oxidizing process was observed utilizing a UV spectrophotometer (Japan, Shimadzu, UV-1800) at 540 nm, all results were reconstructed into the suitable concentrations (A). The efficiency (DR) was computed as follows (Equation (5):

$$\eta = \frac{C_{\bullet} - C_t}{C_{\bullet}} \times 100 \tag{5}$$

The percentage of dye removed was expressed as (η), the value of the concentration after treatment (parts per million) is expressed as Ct, and Co the measured concentration before treatment (mg/L).

Experimental Design

The central composite design (CCD) approach was used in this work to optimize experimental settings for decolonization and direct-red wastewater reduction using electro-Fenton process. For both the quadratic model extraction data analysis experimental design, and graph display, the program Design Expert Minitab-17 was utilized. (X_1) electrolysis time, (X_2) H_2O_2 concentration, and current are independent factors (X_3). As stated in Table 1, these factors were coded with two levels (high and low) into the composite design central. Table 2 displays both natural and coded operating variables for experimental systems using the Design Minitab program.

Table 1. The parameters of the operation.

Parameters	The ranges
X_1 – The electrolysis time (min)	10 -30
X_2 - H_2O_2 concentration (ppm)	10 -50
X3 – The current (Amps)	0.5 -2

Table 2. The operational variables

Natural Variable (X_i)	Coded Variables				
	- 2	- 1	0	1	2
X_1 - Electrolysis time (min.)	10	15	40	25	30
X_2 - H_2O_2 concentration (ppm)	10	20	30	40	50
X_3 - Current (mA)	0.5	0.88	1.25	1.63	2

The adequacy and correctness of the correlation -coefficients were tested in the current study using Chi-square (χ^2) as defined by the following equation (Eq. 6), in all of this indication the value should be the lowermost. The applicability of the mode is best represented by the high correlation (R_2) with lower (χ^2).

$$X^2 = \frac{(Y^{exp} - Y^{coded})^2}{Y^{coded}} \tag{6}$$

Where the coding experiments were represented via Y^{coded}, and the responses were represented via Y^{exp}.

Results and Discussion

The Regression Models

The below a least-squares technique with second-order model was used in this investigation to determine correlations between the independent variables and the responses (Davarnejad et al., 2014):

$$Y = B_0 + \sum_{i=1}^{q} B_i X_i + \sum_{i=1}^{q} B_{ii} X_i^2 + \sum_i \sum_j B_{ij} X_i X_j + \varepsilon \tag{7}$$

Where X_1, X_2, and X_q are the operational variables; Y denotes the studied responses; Bi denotes the linear regression-coefficient, Bo refers to the regression constant, Bij indicates the cross-product regression-coefficient, and Bii indicates the squared regression coefficient; and ε indicates to the random error. the operational variables' values, as well as the % reduction of the researched responses, namely dye removal, final pH, and energy consumption displays with Table 3.

Table 3. Studied variables results

Run	Current (Amp)	H_2O_2 concentration (ppm)	Electrolysis time (min)	E (kWh/m^3)	Final PH	Dye removal (%)
1	0.88	20	15	12.81	3.51	93.24
2	0.88	20	25	21.36	4	94.62
3	0.88	40	15	12.81	4.2	94.85
4	0.88	40	25	21.36	3.26	96.46
5	1.63	20	15	23.81	3.27	96.92
6	1.63	20	25	39.67	3.57	98.07
7	1.63	40	15	23.80	3.42	94.85
8	1.63	30	25	39.67	3.25	96.46
9	1.25	30	10	12.20	3.76	95.31
10	1.25	10	30	36.62	4.1	96
11	1.25	50	20	24.42	3.27	95.77
12	1.25	30	20	24.41	3.51	93.93
13	0.5	30	20	9.77	3.2	91.4
14	2	30	20	39.06	3.41	98.07
15	1.25	30	20	24.41	3.48	96.46
16	1.25	30	20	24.41	3.51	96.23
17	1.25	30	20	24.35	3.44	96.46
18	1.25	30	20	24.41	3.35	96.23
19	1.25	30	20	24.37	3.25	96.46
20	1.25	30	20	24.35	3.28	96.46

Effect of Current

All experiments were conducted with currents ranging from 0.5 to 2 mA to investigate the influence of current on an electro-Fenton process performance, the direct red elimination improved with increasing current, reaching a high of 98.07% for 1.63 mA with 20 ppm hydrogen peroxide at 25 minutes with pH 3. At currents of 0.5 mA, the lowest direct removal rates of the red dye were obtained, as shown in Figure 3, which were 91% with 30 ppm of H_2O_2 and 20 minutes. A similar trend was seen for ibuprofen (isobutylphenyl propionic acid) by the heterogeneous electro-Fenton degradation (Liu et al., 2018). The current applied to the electrode will increase proportionally. As a result of the higher rate of H_2O_2 creation, the rate of radical generation also rises, resulting in better removal efficiency at 1.63 V as to compare for 0.5 V and 1 V. The removal efficiency decreased to 88% when the applied voltage was increased to 2 volts due to the occurrence of undesirable reactions: i) hydrogen's two evolutions of at the cathode (ii) hydrogen peroxide oxidation at the anode.

$$2H_2O_2 + 2e^- \rightarrow H_2 + HO^- \tag{8}$$

$$H_2O_2 \rightarrow O_2 + 2H^+ + 2e^- \tag{9}$$

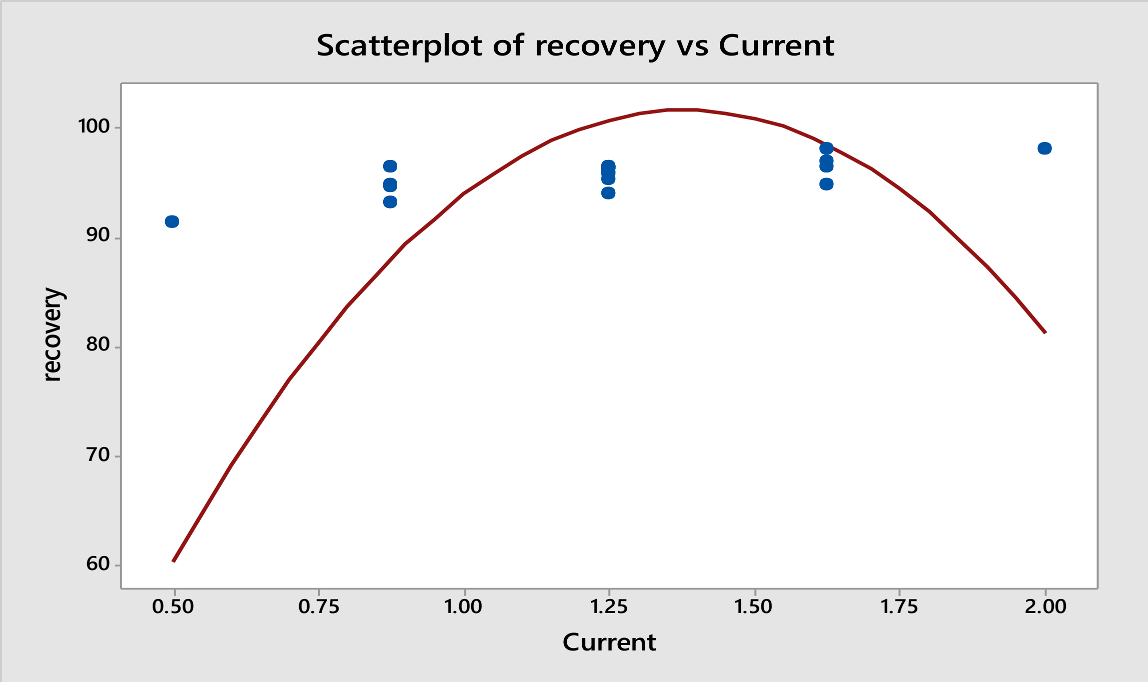

Figure 3. Current's impact on removal efficiencies of 20 mg/ L direct red wastewater (electrolysis time = 30 minutes)

Effect of Electrolysis Time

To investigate effect of electrolysis duration impact, studies were done using operational treatment settings that were consistent with 20 ppm direct red and 3 pH. Maximum removal was achieved using H_2O_2 concentration of 30 ppm and 1.63 Am at least of 20-minute the efficiency of the organics removal from wastewater is increasing with an increase of electrolysis duration, as shown in Figure 4 below. This has been coincident with Gaber et al. study (Gaber et al., 2013). The duration of electrolysis is directly proportional to the efficiency of organic pollutant removal, based on the increased the adsorption process activity occurring during the electrochemical reactor as the electrolysis period prolonged (Apaydiın, 2014). In adding, lengthy oxidation durations led to a detoxication one step by one till the synthesis of inorganic tin and Carbone dioxide (Yong et al., 2017), thus achieving was attained to full mineralization.

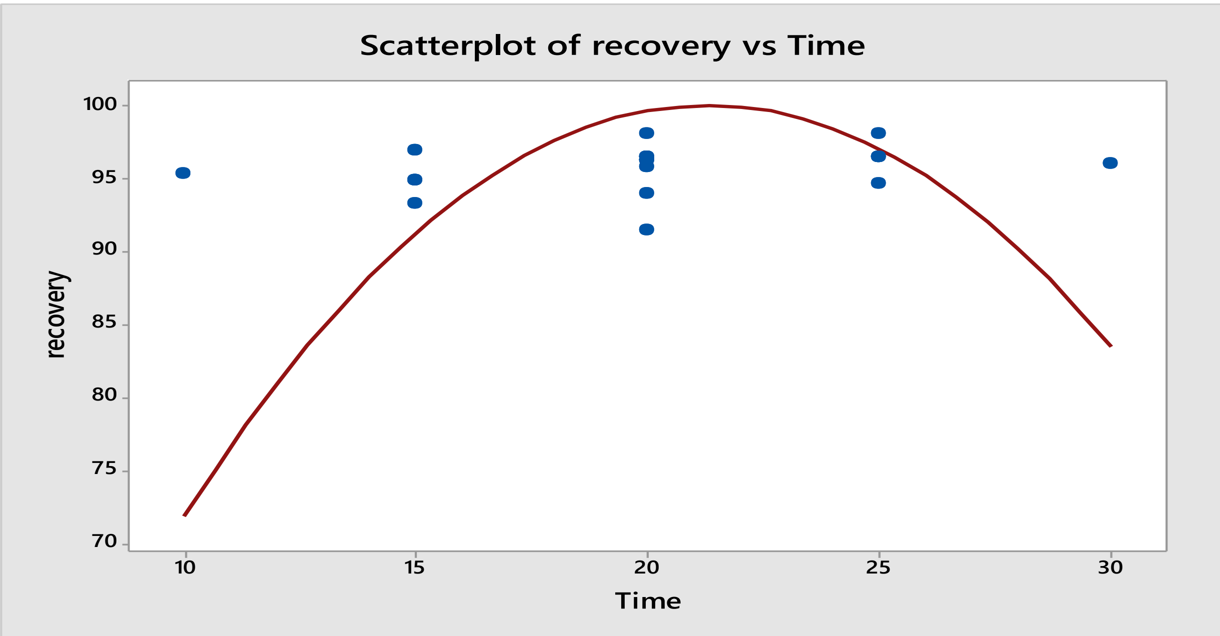

Figure 4. The effect of electrolysis time on the removal efficiencies of 20 mg/ L direct red wastewater

Hydrogen Peroxide Concentration

The hydrogen peroxide has a substantial impact on the reaction efficiency of an electro-Fenton since it directly determines theoretical peak quantity of generated. An increase in H_2O_2 dose often enhances overall efficiency due to an increase in free radicals. This association has been proven in several investigations. Furthermore, unused H_2O_2 with reductive capacity might utilize organic oxidant during electro-oxidation, resulting in overestimation of organic values, with the magnitude of inaccuracy equivalent to H_2O_2 concentration. As a result, excess H_2O_2 is likely to lead organics reduction; also, excess hydrogen peroxide is hazardous to many organisms and significantly reduces overall effectiveness in circumstances when the electro-Fenton technique is used. To enhance efficiency during an electro-Fenton system while avoiding associated disadvantages, an appropriate H_2O_2 concentration must be calculated (Nidheesh & Gandhimathi, 2012).

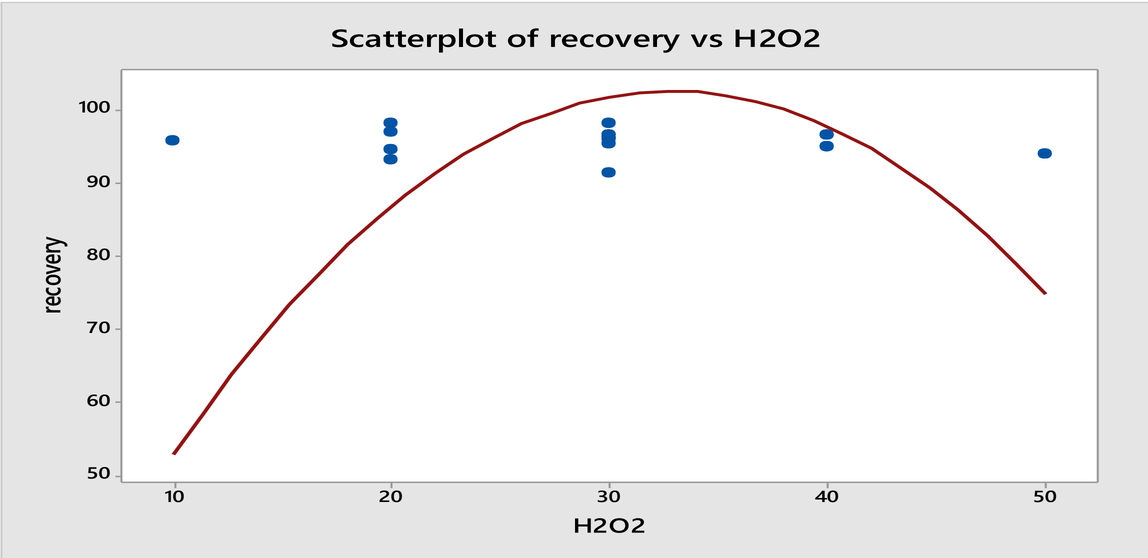

Figure 5. The effect of hydrogen peroxide on the removal efficiencies of 20 mg/L direct red wastewater

Optimization of Operational Variables

Using a statistics software tool, the optimal parameters of electrolysis duration, current, and H_2O_2 concentration were determined (Minitab-17). the D-optimization measurement findings depict in figure 6. The optimal organic compound removal efficiency was much more than 98%.

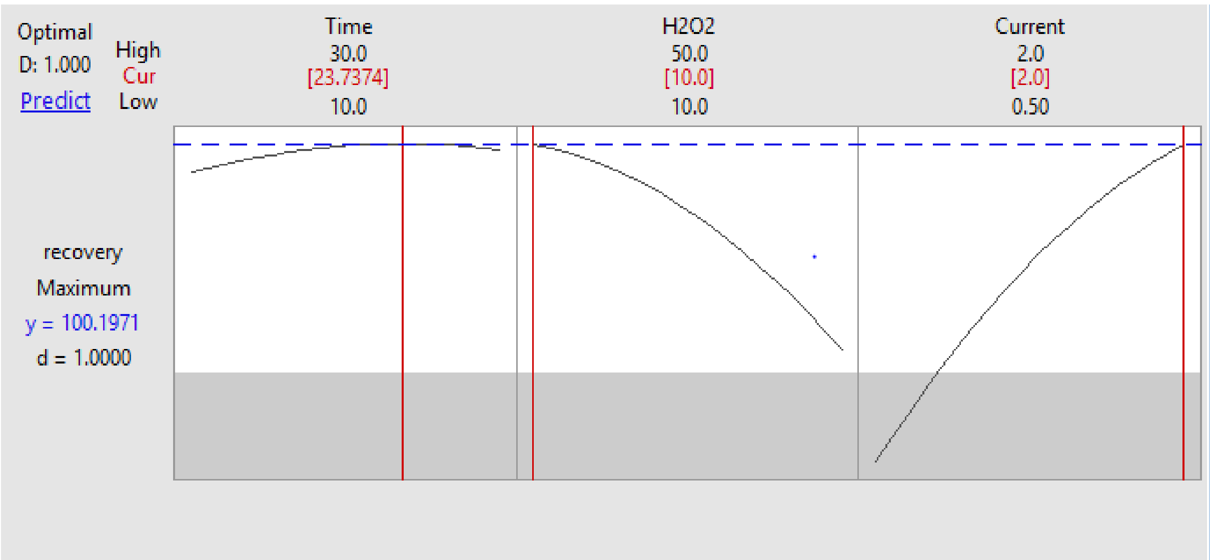

Figure 6. The optimal operational variable values and verified values for the responses for treatment of 20 mg/ L direct red wastewater.

The Responses Studied Mathematical Correlation

Equation (7) was applied to check the mathematical relationships for the removal of the content of the organic responses to the factors of the operation. Table 4 shows the quadratic relationships for removal of dye in terms of real and coding factors.

Table 4. Real and coded mathematical relationships for the responses

Responses	terms	correlations	R^2	R^2 (adjusted)	R^2 (predicted)
Dye Removal: Y_{OCR}	Coded	Y_{OCR}^{coded} % = 73.17+ 0.291X_1 + 0.447 X_2+17.54X_3 - 0.00586 X_1^2 - 0.00348 X_2^2-2.67 X_3^2+0.00173 X_1 X_2 -0.015X_1X_3-0.2377X_2X_3	0.89	0.9801	0.990
	Real	$Y_{OCR}^{exp.}$ % = 73.17+ 0.291X_1 + 0.447 X_2+17.54X_3 - 0.00586 X_1^2 - 0.00348 X_2^2-2.67 X_3^2+0.00173 X_1 X_2 -0.015X_1X_3-0.2377X_2X_3			

The findings in Table 4 demonstrate that the regression coefficients have high values for both answers, which is desirable, and that the projected the regression-coefficients values are in very good agreement with adjusted values. To assess the applicability and precision of the correlation-coefficients, the employment of Chi-square (χ^2) was carried out in accordance with the equation 6 as shown in Table 5. The lower (χ^2) with the higher correlation-coefficients (R^2) are the best indication of the model's applicability, as demonstrated.

Table 5. The studied responses by Chi-square (χ^2) test.

Run	(X^2) Chi-square	Y_{OCR}^{code} (%)	Y_{OCR}^{exp} (%)
1	1.44175E-06	93.23	93.24
2	0.003974468	94.01	94.62
3	0.002637267	94.35	94.85
4	0.010111537	95.48	96.46
5	0.005335421	97.64	96.92
6	0.000584354	98.31	98.07
7	0.001287955	95.20	94.85
8	0.000629333	96.21	96.46
9	0.001438064	94.94	95.31
10	0.005588752	96.73	96
11	0.000662946	95.52	95.77
12	0.004012267	94.54	93.93
13	0.009727705	92.349	91.4
14	0.003370063	97.5	98.07
15	1.37067E-05	96.42	96.46
16	0.000389162	96.42	96.23
17	1.37067E-05	96.42	96.46
18	1.37067E-05	96.42	96.23
19	0.000389162	96.42	96.46
20	0.000389162	96.42	96.46

Estimation of Energy Consumption

To make the use of electro-chemical technology as an effluent treatment practicable, various factors must be considered (performance of anode material, operating cost and consumption energy). The findings of consumption energy as a function of current and electrolysis duration through treatment by electro-Fenton for organic in direct-red wastewater are shown in Figure 7. During electrochemical treatment, it is discovered that values were related to the time and current applied for all forms of water (Da Silva et al., 2013).

Based on the previously established optimal values of electrolysis duration and current, the values of electrodes with expenditure energy are 39.67 kWh/m^3. The factor of the energy is a crucial operating factor in the electro-oxidation decontamination process, as illustrated in Figure 7. It is worth noting that energy consumption during electrolysis is lower compared to energy consumption with the current. This is an important indicator that current is significant factor in measuring energy consumption, a fact consistent with findings in numerous studies cited in the literature (Ganiyu et al., 2018). Figure 7 illustrates this important point.

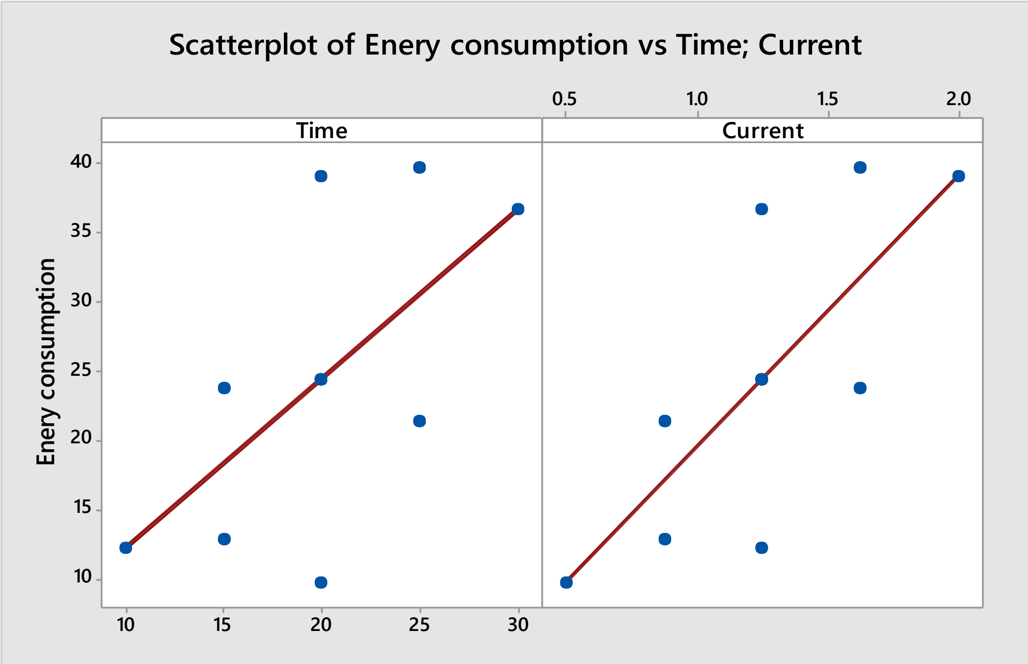

Figure 7. The operational variables effect on the consumption energy for the handling of 20 ppm direct-red wastewater.

Conclusion

Electro-oxidation provides diversity, energy competence, automation capability, and environmental friendliness. It is a complex technology used to remediate direct-red wastewater. The electro-Fenton approach was introduced in this study to treat wastewater from industry direct-red wastewater. Finally, the working conditions were upgraded and made available in order to get the best outcomes with the greatest amount of dye removal. The revealed mathematical connections exhibit substantial regression coefficients for all evaluated responses, confirming the model as polynomial for second order of appropriate adjustment. The best removal dye (98%) was achieved with a pH of 3, a current of 1.63 mA, a reaction time of 25 minutes, and an H_2O_2 concentration of 30 ppm. Nanoporosity plays a crucial role in accelerating the decomposition of hydrogen peroxide, resulting in an increase in the formation ions of the hydroxyl. Due to the electro-Fenton technique efficiency, the current approach might be to recommend it as a straightforward solution for direct- red wastewater treatment.

Scientific Ethics Declaration

* The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

Conflict of Interest

* The authors declare that they have no conflicts of interest

Funding

* No funds have been allocated for this purpose.

Acknowledgements or Notes

* This article was presented as an oral presentation at the International Conference on Engineering and Advanced Technology (ICEAT) held in Selangor, Malaysia on July 23-24, 2025.

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To cite this article:

Nuhma M.J., Mohsen O.A., Abed K.M., Zageer K.F., Wasmi B.A., & Hassan A.A. (2025). Electro-fenton oxidation for degradation of direct red organic pollutants using stainless steel and iron electrodes. *The Eurasia Proceedings of Science, Technology, Engineering and Mathematics (EPSTEM), 37,* 1124-1134.