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Optimization of the photo-Fenton process for Congo red removal from synthetic wastewater using the Box-Behnken

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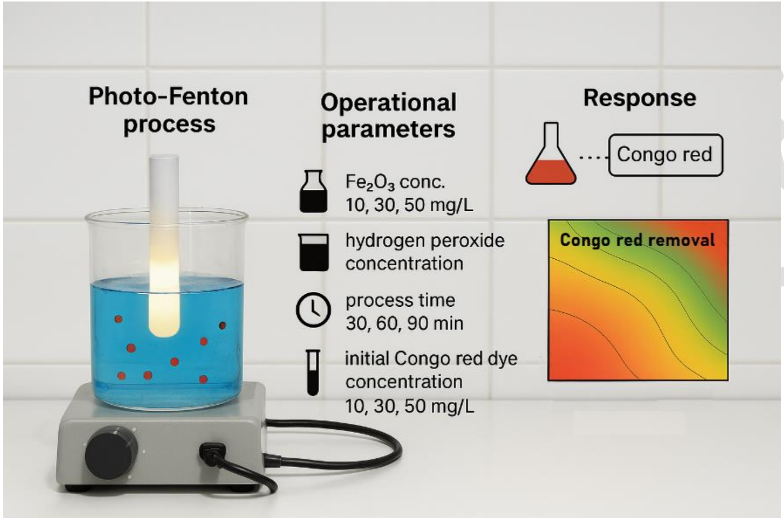
GRAPHICAL ABSTRACT



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ABSTRACT

This study investigates the efficiency of the photo-Fenton process in removing Congo red dye from synthetic wastewater. Considering the importance of eliminating colored pollutants from the environment, this research examines the effect of key operational parameters, including Fe_2O_3 concentration (10, 30, and 50 mg/L), hydrogen peroxide concentration (100, 300, and 500 mg/L), process time (30, 60, and 90 min), and initial Congo red dye concentration (10, 30, and 50 mg/L), on the percentage of dye removal in the photo-Fenton process. The experiments were conducted in a glass reactor using a UV-C lamp as the radiation source, at ambient temperature and with a stirring speed of 350 rpm. To optimize the process and examine the interaction of parameters, the Box-Behnken experimental design method was used, and 27 experiments were designed and performed randomly. Analysis of variance on the results showed that the proposed quadratic model has acceptable accuracy for predicting dye removal efficiency. Furthermore, examination of contour plots revealed that Fe_2O_3 concentration and initial Congo red dye concentration have the greatest impact on process efficiency. Finally, the optimal conditions for achieving the maximum percentage of dye removal from synthetic wastewater were determined. Under optimal conditions (process time of 30 min, hydrogen peroxide concentration of 100 mg/L, Fe_2O_3 concentration of 40.99 mg/L, and initial Congo red concentration of 30 mg/L), the maximum Congo red removal efficiency of 99.59% was achieved.

1. Introduction

Today, water is recognized as a vital national asset worldwide, and its scarcity has become an increasing and worrying challenge (Samimi and Moghadam, 2024). This scarcity not only threatens ecosystems but also makes access to safe drinking water difficult for approximately 1.2 billion people worldwide, seriously endangering human health and the environment (Raghav, Painuli and Kumar, 2018; Samimi and Amiri, 2024; Mohadesi *et al.*, 2024b). Among various industries, the textile industry, as a major water consumer, plays a significant role in producing large volumes of industrial wastewater (Mohadesi *et al.*, 2024a). These wastewaters often contain a wide range of colored pollutants that can have adverse effects on water quality and the health of living organisms (Masalvad and Sakare, 2021; Sharifi and Mohadesi, 2024).

Traditional wastewater treatment methods, such as physical, chemical, and biological processes, although effective in removing some pollutants, face limitations in completely removing color and fully mineralizing pollutants (Fuentes-Gandara *et al.*, 2024; Samimi, 2025). For example, some synthetic dyes are difficult to remove by these

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methods due to their complex structure and resistance to biodegradation. As a result, advanced oxidation processes (AOPs) have gained widespread attention as emerging and efficient methods for removing organic and inorganic pollutants, especially dyes, from water and wastewater (Salameh *et al.*, 2026; Sirés *et al.*, 2014).

AOPs function by creating conditions that lead to the production of strong free radicals, particularly hydroxyl radicals (•OH). These radicals, as very strong oxidants, are capable of breaking down and degrading a wide range of resistant and non-biodegradable pollutants (Al-Kdasi *et al.*, 2004). These processes include Ozonation (O_3), hydrogen peroxide (H_2O_2)-based processes, Fenton and photo-Fenton processes, as well as the use of various catalysts such as titanium dioxide (TiO_2) and iron oxide nanoparticles (Atout *et al.*, 2025; Phan and Nguyen, 2024; Leichtweis *et al.*, 2026; Gökdağ *et al.*, 2025; Ain *et al.*, 2024). The Fenton process, which involves the reaction of iron ions (Fe^{2+}) with H_2O_2 under acidic conditions, is known as a cost-effective and efficient method for removing pollutants, especially dyes (Ghribi and Bagane, 2023). However, this process faces challenges such as the formation of iron sludge and the need to adjust the pH in the acidic range, which can increase operating costs. To improve the efficiency and reduce the limitations of the Fenton process, the use of ultraviolet (UV) radiation as a stimulating agent in the photo-Fenton process has been considered (Masalvad and Sakare, 2021; Atout *et al.*, 2023). UV radiation can help accelerate the production of hydroxyl radicals and increase the rate of pollutant degradation.

Numerous studies have investigated the efficiency of AOPs in removing dyes and other pollutants from industrial and synthetic wastewaters. For example, da Silva Brito et al. showed that solar radiation can be used as an effective and sustainable method in biodiesel wastewater treatment (da Silva Brito *et al.*, 2019). They found that the use of sunlight in the photo-Fenton process can help reduce energy costs and improve process sustainability. Jiménez et al. also examined various AOPs for water treatment and found that ozonation combined with H_2O_2 showed the best results in removing organic compounds, including compounds resistant to biodegradation (Jiménez *et al.*, 2019). In addition, other studies have shown that the use of iron oxide nanoparticles, zeolites, and other nanostructured materials can increase the efficiency of AOPs in removing dyes and other pollutants (Bouras *et al.*, 2019; Phan and Nguyen, 2024; Leichtweis *et al.*, 2026; Gökdağ *et al.*, 2025; Ain *et al.*, 2024). These materials can act as heterogeneous catalysts and increase the contact surface between pollutants and free radicals.

In the field of Congo red (CR) dye removal from wastewater, which is an azo dye widely used in the textile and dyeing industries, numerous studies have investigated the efficiency of advanced treatment processes, especially Fenton and photo-Fenton-based processes (Khelassi-Sefaoui *et al.*, 2025). For example, one study examined the effectiveness of the photo-Fenton reaction for treating textile wastewater containing CR dye and investigated the effect of various parameters such as Fe^{2+} concentration, initial dye concentration, pH, and hydrogen peroxide level (Masalvad and Sakare, 2021). In this regard, researchers have sought to optimize process conditions, identify factors affecting dye removal efficiency, and understand reaction mechanisms (Masalvad and Sakare, 2021; Ghribi and Bagane, 2023).

Recently, the modification of photocatalysts and optimization of operational parameters have been shown to significantly enhance dye degradation efficiency. For instance, a B–ZnO/TiO_2 nano-photocatalyst achieved complete degradation of Direct Red 16 under acidic conditions and visible light irradiation, mainly due to the generation of hydroxyl radicals and reduced electron–hole recombination rate (Habeeb, Zinatizadeh and Zangeneh, 2023). These findings emphasize the importance of catalyst design and statistical optimization approaches in advanced oxidation processes for dye removal.

In another study, the catalytic activity of cobalt molybdate ($CoMoO_4$) was investigated using a UV-vis assisted peroxydisulfate (PDS) activated system for CR dye degradation. The results of this study showed that the dye degradation efficiency reaches approximately 97% under optimal conditions, and the excellent catalytic activity was attributed to the synergistic effect of photocatalysis and $CoMoO_4$-activated PDS degradation (Zhou *et al.*, 2022). These findings suggest that the use of novel catalysts and advanced activation systems can help improve the efficiency of photo-Fenton processes in CR dye removal.

Additionally, one study investigated the photocatalytic and photo-Fenton oxidation of CR dye using treated natural iron chromite. The results of this study showed that iron chromite, after the addition of H_2O_2, exhibits photocatalytic properties under UV irradiation and photo-Fenton oxidation (Shaban *et al.*, 2017). These findings suggest that the use of natural and inexpensive materials, such as iron chromite, can lead to the development of more sustainable and cost-effective methods for treating wastewaters containing CR dye.

Furthermore, researchers have explored the use of nanostructured materials, such as iron-modified geopolymers (Fe/GP), to improve CR dye decontamination. In one study, a geopolymer with iron obstruction was synthesized from natural iron-containing kaolinite and optical waste, and it was found that this material, as a catalyst, has significant activity towards photo-Fenton oxidation even at high concentrations of CR dye (Adly *et al.*, 2022). These findings suggest that the use of nanostructured materials can help increase the contact surface area, improve adsorption properties, and enhance the efficiency of photo-Fenton processes in CR dye removal.

Also, studies have investigated the use of electrochemical advanced oxidation processes for CR dye degradation. In one study, the degradation of CR dye was investigated using an electrochemical advanced oxidation process, focusing on hydroxyl radicals, and it was found that mineralization of the dye under optimal conditions resulted in a total organic carbon removal rate of 81.1% after 300 min (Mansour *et al.*, 2024). These findings suggest that electrochemical processes can be used as an efficient and environmentally friendly alternative to chemical processes in the treatment of wastewaters containing CR dye.

Given the variety of methods, materials, and operating conditions used in different studies, there is still a need for further research in the field of optimizing advanced treatment processes, especially the photo-Fenton process, for the effective and sustainable removal of Congo red dye from wastewater. In this regard, the present study aims to investigate the effect of key parameters, including process time, hydrogen peroxide concentration, Fe_2O_3 concentration, and initial Congo red dye concentration, on the removal efficiency of this pollutant from synthetic wastewater using the photo-Fenton process. In addition, by using the Box-Behnken experimental design method, the goal is to provide a mathematical model for predicting and optimizing this process.

2. Materials and methods

2.1. Materials

In this study, the following laboratory-grade chemicals were used: iron(II) sulfate heptahydrate ($FeSO_4.7H_2O$) as the source of iron ions, hydrogen peroxide 30% solution, and Congo red dye. All chemicals were purchased from Sigma-Aldrich. Double-distilled water was used to prepare all solutions to prevent any contamination and interference with the experimental results.

2.2. Experimental setup

The experiments were conducted in a 100 mL glass beaker. Two 15W UV-C lamps manufactured by Philips were used as the light source. These lamps were placed horizontally in the center above the beaker and inside a box to ensure uniform light irradiation to the reaction solution. The beaker containing the reaction solution was placed on a magnetic stirrer to ensure proper mixing and homogeneity of the solution throughout the experiment. All experiments were performed at room temperature, and temperature changes were not controlled. The concentration of Congo red dye before and after the treatment process was measured using a spectrophotometer (PG Instrument, T80+) at the maximum absorption wavelength (λ_{max}=497 nm).

2.3. Experimental procedure

In this study, the effect of four main parameters on the Congo red dye removal efficiency was investigated: H_2O_2 concentration, Fe_2O_3 concentration, initial CR dye concentration, and process time. In each experiment, the required amount of $FeSO_4.7H_2O$ and H_2O_2 were accurately weighed. Then, a 100 mL solution of CR dye with a specific concentration was prepared and poured into the beaker. The prepared solution was placed on a magnetic stirrer and the photo-Fenton process was initiated under UV irradiation and simultaneous stirring by a magnetic stirrer. At specific time intervals, samples were taken from the solution and, after passing through filter paper (to remove suspended particles and possible precipitates), the CR dye concentration was measured using the spectrophotometer. The percentage of CR dye removal was calculated using the following equation (Nouri Molalar, Banaei and Massoudi, 2025):

$$\text{CR removal, \%} = \frac{C_0 - C}{C_0} \times 100 \qquad (1)$$

where C_0 and C are the initial and final CR concentrations in mg/L, respectively.

2.4. Experimental design

Experimental design is a powerful method for improving the quality and increasing the efficiency of processes. In this method, by performing a series of planned experiments and making informed changes to the input variables, the effects of these changes on the performance characteristics of the process are investigated. Experimental design is essentially the systematic manipulation of a number of variables and the evaluation of the effects of these manipulations on the desired responses. There are three basic principles in experimental design: Replication, Blocking, and Randomization. Replication and blocking are used to increase the accuracy of the experiment and reduce the effects of disturbing noises, while randomization is used to reduce error and prevent bias in the results.

There are various methods for experimental design, including Factorial, Simplex, Central Composite Design, and Box-Behnken Design methods. The choice of the appropriate method depends on factors such as the type of relationship between the input variables and the response, the number of variables and their levels, and the constraints related to resources and time. If the relationship between the response and the variables can be approximated by a linear equation, a factorial or simplex design is used. However, if a quadratic equation is needed to describe the relationship more accurately, central composite or Box-Behnken design methods are more appropriate (Bano and Jusoh, 2025; Samimi and Nouri, 2025; Samimi and Moeini, 2020). Also, the amount of available raw materials is effective in choosing the experimental design method. If the amount of raw materials is limited, designs that require fewer experiments are prioritized (Montgomery, 2017).

In this study, to investigate the effect of four variables - process time (30-90 min), hydrogen peroxide concentration (100-500 mg/L), iron ion concentration (10-50 mg/L), and initial Congo red concentration (10-50 mg/L) - on CR dye removal, the Box-Behnken experimental design method was used. This method suggests a total of 27 experiments in a randomized order for an experiment with four variables at three levels (low, medium, and high levels). The variables examined in this study and the values of each of their levels are presented in Table 1.

Table 1. Operating parameters and their levels.

Factor	Symbol	Unit	Levels		
			Low	Middle	High
Treatment time	t	Min	30	60	90
H_2O_2 concentration	$[H_2O_2]$	mg/L	100	300	500
Fe_3O_4 concentration	$[Fe_2O_3]$	mg/L	10	30	50
Initial CR concentration	C_0	mg/L	10	30	50

3. Results and discussion

3.1. Experimental design results and statistical analysis

In this section, the results obtained from the experimental investigation of CR dye removal from synthetic wastewater containing this dye are reported and analyzed using Design-Expert 7.0.0 software (Sattar, Khalid and Yusoff, 2024).

After conducting the experiments with the photo-Fenton process, the results of CR removal analysis from synthetic wastewater for all samples are presented in Table 2. As mentioned earlier, the experimental variables included t, $[H_2O_2]$, $[Fe_2O_3]$, and C_0, and the percentage of CR removal was considered as the response.

Based on the results, the highest CR removal rate (99.90%) was achieved in experiment number 24 under the following conditions: t=30 min, $[H_2O_2]$=300 mg/L, $[Fe_2O_3]$=50 mg/L, and C_0=30 mg/L. The lowest CR removal rate was also obtained in experiment number 5 (79.27%) under the conditions t=60 min, $[H_2O_2]$=300 mg/L, $[Fe_2O_3]$=10 mg/L, and C_0=50 mg/L.

The main effect plots in Fig. 1 show the effect of coded operating variables on the percentage of CR removal. These plots are drawn for each variable under conditions where the other variables are kept constant at their average value. As can be seen in Fig. 1, the effect of process time and hydrogen peroxide concentration is almost negligible, which can be attributed to the scavenging behavior of excess hydrogen peroxide that consumes hydroxyl radicals and forms less reactive species, as well as the rapid degradation of Congo red during the early stage of the photo-Fenton reaction, after which the system reaches a quasi-steady state. In contrast, Fe_2O_3 concentration and initial CR concentration have a significant effect on the percentage of CR removal, because Fe_2O_3 directly participates in hydroxyl radical generation and the initial dye concentration determines the availability of pollutant molecules for oxidation. As the Fe_2O_3 concentration increases from 10 to 50 mg/L, the percentage of CR removal increases. Also, the initial CR concentration has an optimal range between 10 and 30 mg/L, which is consistent with previous studies on Congo red degradation using Fenton and photo-Fenton processes (Ghribi and Bagane, 2023; Masalvad and Sakare, 2021).

Table 2. Experimental design using the Box-Behnken method and the response.

No.	t, min	$[H_2O_2]$, mg/L	$[Fe_2O_3]$, mg/L	C_0, mg/L	$CR_{rem.}$, %
	Manipulated variables				Response
1	30	100	30	30	98.96
2	30	300	30	50	89.61
3	60	500	30	10	99.23
4	30	300	10	30	88.32
5	60	300	10	50	79.27
6	90	300	30	10	99.02
7	60	500	10	30	88.79
8	90	300	30	50	87.12
9	60	300	50	10	99.47
10	60	100	10	30	89.33
11	60	100	50	30	99.95
12	60	500	30	50	91.44
13	60	300	30	30	99.84
14	90	300	10	30	84.23
15	60	300	30	30	98.25
16	60	300	10	10	88.43
17	90	500	30	30	98.35
18	30	300	30	10	95.48
19	30	500	30	30	99.28
20	90	100	30	30	99.67
21	60	100	30	10	97.59
22	60	500	50	30	99.28
23	90	300	50	30	99.84
24	30	300	50	30	99.90
25	60	300	50	50	93.93
26	60	300	30	30	99.28
27	60	100	30	50	89.66

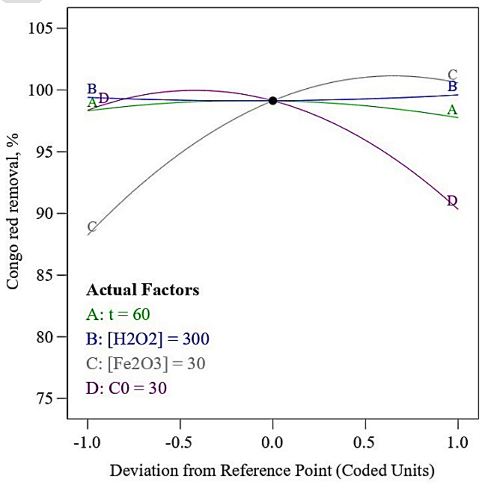

Fig. 1. Main effect plots for examining the effect of operating variables on Congo red removal.

In Table 3, the results of the analysis of variance (ANOVA) for the response ($CR_{rem.}$) are presented. The p-value for the quadratic model is less than 0.0001, indicating that the model is significant (Khanbolouk *et al.*, 2025). The p-value for lack of fit is 0.2845, indicating that the lack of fit of the model is non-significant and the model is statistically adequate (Samimi, 2024). The p-values for the terms $[Fe_2O_3]$, C_0, $t.C_0$, $[Fe_2O_3]^2$, and C_0^2 are less than 0.05, indicating that these variables have a significant effect on CR removal efficiency. The term t^2 also has a p-value of 0.0743, indicating its potential importance. Other terms in the quadratic model are non-significant and do not have a significant effect on CR removal efficiency. The quadratic model equation for Congo red removal in terms of operating variables is as follows:

$$\mathrm{CR_{rem.}} = 99.12 - 0.28\mathrm{X_t} + 0.10\mathrm{X_{[H_2O_2]}} + 6.17\mathrm{X_{[Fe_2O_3]}} - 4.02\mathrm{X_{C_0}} - 0.41\mathrm{X_t}.\mathrm{X_{[H_2O_2]}} + 1.01\mathrm{X_t}.\mathrm{X_{[Fe_2O_3]}} - 1.51\mathrm{X_t}\mathrm{X_{C_0}} - 0.03\mathrm{X_{[H_2O_2]}}.\mathrm{X_{[Fe_2O_3]}} + 0.04\mathrm{X_{[H_2O_2]}}.\mathrm{X_{C_0}} + 0.90\mathrm{X_{[Fe_2O_3]}}.\mathrm{X_{C_0}} - 1.10\mathrm{X_t^2} + 0.37\mathrm{X_{[H_2O_2]}^2} - 4.73\mathrm{X_{[Fe_2O_3]}^2} - 4.79\mathrm{X_{C_0}^2} \quad (2)$$

Table 3. Analysis of variance of the quadratic model for CR removal.

Source	Sum of Squares	DF	Mean Square	*F*-Value	*p*-Value Prob > *F*	Level of Significance
Model	901.00	14	64.36	38.44	<0.0001	highly significant
T	0.92	1	0.92	0.55	0.4731	not significant
$[H_2O_2]$	0.12	1	0.12	0.073	0.7918	not significant
$[Fe_2O_3]$	456.33	1	456.33	272.57	<0.0001	highly significant
C_0	193.52	1	193.52	115.59	<0.0001	highly significant
$t.[H_2O_2]$	0.67	1	0.67	0.40	0.5381	not significant
$t.[Fe_2O_3]$	4.06	1	4.06	2.43	0.1454	not significant
$t.C_0$	9.09	1	9.09	5.43	0.0381	significant
$[H_2O_2].[Fe_2O_3]$	4.22×10^{-3}	1	4.22×10^{-3}	2.52×10^{-3}	0.9608	not significant
$[H_2O_2].C_0$	4.90×10^{-3}	1	4.90×10^{-3}	2.93×10^{-3}	0.9577	not significant
$[Fe_2O_3].C_0$	3.28	1	3.28	1.96	0.1872	not significant
t^2	6.40	1	6.40	3.82	0.0743	posibly significant
$[H_2O_2]^2$	0.74	1	0.74	0.44	0.5178	not significant
$[Fe_2O_3]^2$	119.09	1	119.09	71.13	<0.0001	highly significant
C_0^2	122.20	1	122.20	72.99	<0.0001	highly significant
Residual	20.09	12	1.67			
Lack of Fit	18.79	10	1.88	2.89	0.2845	not significant
Pure Error	1.30	2	0.65			
Cor Total	921.09	26				

In this equation, the percentage of CR removal is expressed as a function of the coded operating variables. X_t, $X_{[H_2O_2]}$, $X_{[Fe_2O_3]}$, and X_{C_0} represent the coded process time, hydrogen peroxide concentration, Fe_2O_3 concentration, and initial CR concentration, respectively. The values of these variables at low, medium, and high levels are -1, 0, and +1, respectively.

To evaluate the modeling ability, the predicted values of CR removal are plotted against the experimental values in Fig. 2. As can be seen, the predicted values are very close to the experimental values, and there is a high correlation between the experimental and predicted values ($R^2 = 0.9782$, $R^2_{adj.} = 0.9527$, and $R^2_{pred.} = 0.8793$).

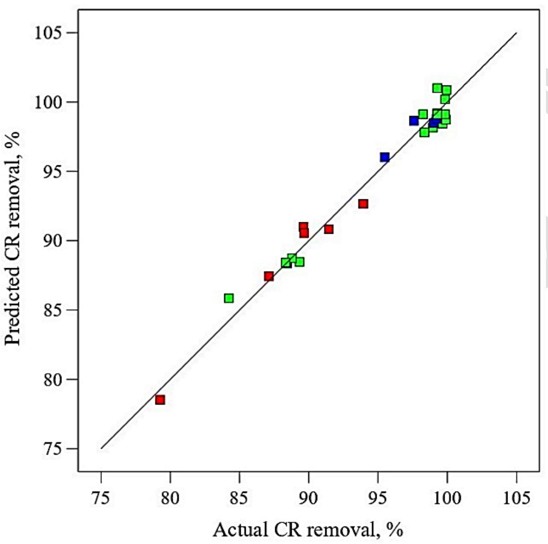

Fig. 2. Comparison of actual and predicted values of Congo red removal percentage.

3.2. Investigating the effect of operating variables on Congo red removal

The contour plots for the percentage of Congo red removal as a function of the four operating variables (process time, H_2O_2 concentration, Fe_2O_3 concentration, and initial CR concentration) are shown in Fig. 3.

Fig. 3-a shows the changes in the percentage of CR removal as a function of process time and H_2O_2 concentration at constant values of $[Fe_2O_3]$=30 mg/L and C_0=30 mg/L. By changing the H_2O_2 concentration, no significant change in the percentage of CR removal is observed. At a constant concentration of H_2O_2, the percentage of removal first increases and then decreases with increasing time (the optimal process time is between 30 and 60 minutes). This behavior can be attributed to the rapid generation of hydroxyl radicals during the initial stage of the photo-Fenton reaction and the subsequent formation of intermediate by-products that inhibit further oxidation of Congo red molecules at longer reaction times (Ghribi and Bagane, 2023; Masalvad and Sakare, 2021; Zhou *et al.*, 2022).

Fig. 3-b shows the removal of CR dye as a function of process time and Fe_2O_3 concentration, under conditions of C_0=30 mg/L and $[H_2O_2]$=300 mg/L. The effect of process time on CR dye removal is non-linear, and the percentage of removal increases slightly with increasing time. Increasing the Fe_2O_3 concentration from 10 to 30 mg/L increases the percentage of CR removal, but further increasing the Fe_2O_3 concentration from 30 to 50 mg/L does not create a significant change in the percentage of CR dye removal. This trend may be explained by the saturation of active catalytic sites and the recombination of excess hydroxyl radicals at higher iron dosages, which limits further improvement in removal efficiency (Ghribi and Bagane, 2023; Masalvad and Sakare, 2021; Adly *et al.*, 2022).

Fig. 3-c shows the contour plot of CR dye removal as a function of process time and initial CR concentration, under conditions $[H_2O_2]$=300 mg/L and $[Fe_2O_3]$=30 mg/L. At low initial concentrations of Congo red dye (less than 20mg/L), the lower the dye concentration, the more time is required to reach a certain percentage of removal. At high initial concentrations of Congo red dye (more than 30mg/L), the process time and the percentage of CR dye removal are not linearly related, and there is an optimal time for removing a higher concentration of CR dye. This behavior is due to the limitation of available hydroxyl radicals relative to the number of dye molecules at higher pollutant loadings, resulting in competition for reactive species and reduced degradation efficiency (Ghribi and Bagane, 2023; Mansour *et al.*, 2024; Shaban *et al.*, 2017).

Fig. 3-d shows the contour plot of CR dye removal as a function of H_2O_2 concentration and Fe_2O_3 concentration, under conditions C_0=30 mg/L and t=60 min. Increasing the Fe_2O_3 concentration causes significant changes in the amount of dye removal, but changing the H_2O_2 concentration does not have a significant effect on the amount of dye removal. Such a trend was also observed for the effect of H_2O_2 concentration and Fe_2O_3 concentration in Figs. 3-a and 3-b, respectively. This observation confirms that iron dosage is the dominant parameter controlling hydroxyl radical generation in the photo-Fenton process, whereas excess hydrogen peroxide mainly acts as a radical scavenger rather than an oxidizing agent (Ghribi and Bagane, 2023; Masalvad and Sakare, 2021; Zhou *et al.*, 2022).

Fig. 3-e shows the contour plot of CR dye removal as a function of H_2O_2 concentration and initial CR dye concentration, under conditions $[Fe_2O_3]$=30 mg/L and t=60 min. Changing the H_2O_2 concentration does not have a significant effect on the amount of dye removal. In other words, at a constant dye concentration, slight changes in the percentage of CR dye removal are observed with increasing H_2O_2 concentration. The highest CR dye removal is achieved at an initial CR concentration of about 20 mg/L. This result can be attributed to a balance between sufficient pollutant availability and effective hydroxyl radical production, which leads to optimal oxidation conditions (Ghribi and Bagane, 2023; Zhou *et al.*, 2022; Adly *et al.*, 2022).

Fig. 3-f shows the contour plot of CR dye removal as a function of Fe_2O_3 concentration and initial CR dye concentration, under conditions $[H_2O_2]$=300 mg/L and t=60 min. The Fe_2O_3 concentration has a significant effect on the amount of CR dye removal, and at a constant dye concentration, the percentage of CR dye removal increases dramatically with increasing Fe_2O_3 concentration. The highest CR dye removal is obtained at an initial concentration of about 20 mg/L (similar to Figs. 3-c and 3-e). This behavior is mainly related to enhanced

hydroxyl radical generation and reduced competition among dye molecules for reactive oxidative species at moderate pollutant concentrations. These trends are in good agreement with previous studies on Congo red degradation using Fenton and photo-Fenton based processes (Ghribi and Bagane, 2023; Masalvad and Sakare, 2021; Zhou *et al.*, 2022; Adly *et al.*, 2022).

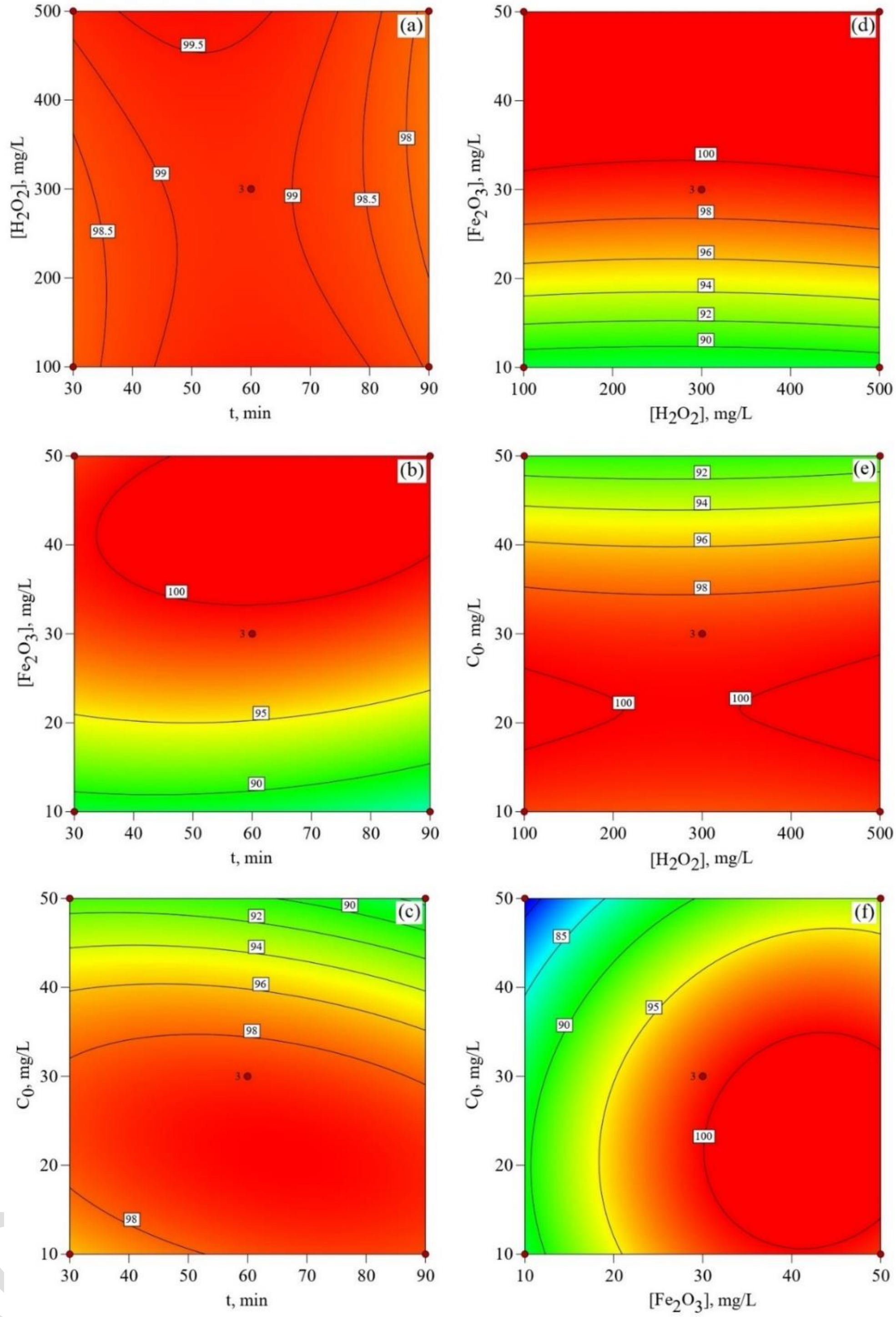

Fig. 3. Effect of operating variables on Congo red removal; (a) effect of process time and H_2O_2 concentration, (b) effect of process time and Fe_2O_3 concentration, (c) effect of process time and initial CR concentration, (d) effect of H_2O_2 and Fe_2O_3 concentrations, (e) effect of H_2O_2 concentration and initial CR concentration, and (f) effect of Fe_2O_3 concentration and initial CR concentration.

3.3. Determining the optimal conditions of the photo-Fenton process

According to the plots in Fig. 3, the percentage of CR dye removal can reach its maximum value under various conditions. To determine the optimal values of the variables in order to maximize the percentage of CR removal, Design Expert optimizer 7.0.0 software was used. Considering that the percentage of CR dye removal can have the highest possible value under different operating conditions, the conditions in which the process time is as short as possible and the concentration of H_2O_2 and Fe_2O_3 is as low as possible are introduced here as optimal conditions. These conditions were determined for different initial concentrations of CR dye and are presented in Table 4. As can be seen in this table, the time is considered at its lowest value (30 min). Also, considering the ineffectiveness of H_2O_2 concentration on the amount of Congo red removal, this variable is also considered at its lowest value (100 mg/L). By changing the initial CR concentration from 10 to 50 mg/L, the optimal value of Fe_2O_3 concentration changes, and the highest CR removal is obtained at an initial Congo red concentration between 20 and 30 mg/L.

The highest dye removal efficiency at moderate initial Congo red concentrations (20-30 mg/L) can be attributed to an appropriate balance between the number of dye molecules and the availability of hydroxyl radicals generated in the photo-Fenton process. At higher initial dye concentrations (40-50 mg/L), the removal efficiency decreases because the amount of generated hydroxyl radicals becomes insufficient relative to the increased pollutant loading, and light penetration into the solution is also reduced. In addition, the minor influence of H_2O_2 concentration under optimal conditions can be explained by the scavenging effect of excess hydrogen peroxide, which leads to radical recombination and limits further improvement in oxidation efficiency. These observations are consistent with previously reported results for Congo red degradation using Fenton and photo-Fenton based advanced oxidation processes (Ghribi and Bagane, 2023; Mansour *et al.*, 2024; Shaban *et al.*, 2017; Masalvad and Sakare, 2021).

Table 4. Optimal operating values for determining the maximum CR dye removal rate.

t, min	[H_2O_2], mg/L	[Fe_2O_3], mg/L	C_0, mg/L	$CR_{rem.}$, %
30	100	39.06	10	96.89
30	100	40.03	20	99.43
30	100	40.99	30	99.59
30	100	41.44	40	97.38
30	100	42.90	50	92.80

4. Conclusion

In this study, the efficiency of the photo-Fenton process was evaluated to reduce Congo red dye in synthetic wastewater using the Box-Behnken experimental design. The effect of hydrogen peroxide concentration, Fe_2O_3 concentration, initial CR concentration, and process time, as key parameters, on dye removal efficiency was investigated. The results showed that increasing the Fe_2O_3 concentration up to a certain value increases the removal efficiency, while the hydrogen peroxide concentration does not have much effect on this process. Also, the initial concentration of CR dye has a significant effect on the removal efficiency, and the highest efficiency is obtained at lower initial concentrations. In the photo-Fenton process (UV=30 W), the maximum CR dye removal rate (99.59%) was achieved under the following conditions: process time 30 min, hydrogen peroxide concentration 100 mg/L, Fe_2O_3 concentration 40.99 mg/L, and initial Congo red concentration 30 mg/L. These results indicate that the photo-Fenton process can be used as an efficient method for removing Congo red dye from synthetic wastewaters, provided that optimal operating conditions are selected.

Author contributions

Majid Mohadesi: Supervision, Methodology, Software, Writing-Reviewing and Editing
Navid Amjadian: Data curation, Writing- Original draft preparation

Conflict of Interest

The authors declare no conflicts of interest.

Data Availability Statement

All data generated or analyzed during this study are included in this published article.

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