

ENGINEERING RESEARCH JOURNAL (ERJ)

Volume (53) Issue (4) October. 2024, pp:179 -185 https://erjsh.journals.ekb.eg

Box-Behnken Design Wastewater Treatment Optimization for Fenton Technology Reuse in Irrigation

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Abstract: Wastewater treatment is one of the most critical environmental challenges because of the interactions that occur between sewage, soil, water, and human health. If wastewater is disposed of in banks and groundwater reservoirs without proper treatment, there is a serious risk to public health. The interplay of sewage, soil, water, and human health in the environment necessitates the development of efficient wastewater management techniques in order to prevent harmful consequences. Using Fenton technology for wastewater treatment is a great step toward achieving high organic substances, and as a result, the use of sustainable alternatives to treat that wastewater is an innovation in the field of wastewater treatment that will lead to increasing the standards of the water resulting from the treatment process. This study optimized the operating parameters necessary to obtain a greater COD elimination by the establishment of a laboratory experiment based on Fenton technology. The optimal pH value and quantities of peroxide of hydrogen (H2O2) and ferric salt (Fe2+) were calculated using a laboratory model. Fenton technique operates best at the following conditions: PH=4.2, Fe+2= 21.9 mM per liter, H2O2= 335 mM per liter. This allows for COD removal effectiveness of up to 71%, according to a numerical design carried out using Box-Behnken Design. To remove organic debris as efficiently as possible so that it may be used for irrigation, a second experimental model was created by putting a tank of aeration and filter unit next to the Fenton tank. As of right now, the final concentrations of COD, BOD, TSS, TN, and TP were 32.4, 14.9, 27, 12.1, and 1.12 ppm, each. The overall effluent concentrations were less than those allowed by the Egyptian regulation for irrigation reuse. Furthermore, the water may be recycled to irrigate green spaces in newly constructed cities, reducing the need for fresh water, which is becoming increasingly scarce and one of the greatest problems confronting mankind in the twenty-first century.

Keywords : Box–Behnken, wastewater, hydrogen peroxide, Optimization, Fenton Technology.

1. INTRODUCTION

There is a growing demand to develop more efficient methods for removing organic contaminants from wastewaters [1]. Traditional wastewater treatment approaches are ineffective against several organic pollutants [2]. Due to the deficiencies of standard wastewater treatment procedures in dealing with resistant contaminants, efforts to develop substitute solutions have accelerated [3] [4]. Several physicochemical techniques, such as adsorption, may decrease resistant pollutants from wastewater, although they are mostly separation techniques that may require subsequent remediation [5] [6]. The use of new technologies for the removal of pollutants from municipal wastewater has become globally necessary due to the complexity and facilities defined by conventional treatments. Advanced oxidative processes, specifically the Fenton process, have become widely applied given their low cost and ease of use. The Fenton process produces 'OH through a homogeneous reaction (takes place via the reaction from a mixture of hydrogen peroxide and ferrous salts). These oxidants are highly reactive and nonselective, reacting with a wide range of organic compounds and, consequently, degrading them into water, carbon dioxide, and inorganic ions. The principal advantages of the Fenton catalytic reactor process are less sludge formation, suitability for treatment of high volumes of wastewater, nonselective oxidation of organics, and its ability to be an effective polishing step for CETPs, municipal sewage treatment, and other uses [7].

Hydroxyl radicals (OH⁻) are created in a variety of ways during advanced oxidation processes (AOPs). OH could react with the pollutants in a reciprocal action, oxidizing them to simpler intermediates and, most likely, CO₂ and H₂O.AOP has gotten a lot of attention in the last ten years [8]. They are thought to be the best methods for degrading stubborn pollutants. Typically, AOPs are classified according to how the OH is produced. Fenton oxidation [9], photocatalysis [10], UV-based approches [11], ozonation [12], and electrochemical oxidation [13] are examples of such treatments. Fenton oxidation is a useful AOP that involves the catalytic decomposition of hydrogen peroxide (H_2O_2) by ferrous iron (Fe²⁺) to generate OH [14]. After that, the generated OH oxidizes the organic pollutant. The process begins with the quick breakdown of H_2O_2 by Fe^{2+} , which produces a considerable amount of OH and converts Fe²⁺ to Fe³⁺.

 $\begin{array}{ccc} H_2O_{2+}Fe^{2+} & \longrightarrow & Fe^{3+}+OH+OH^{-}\\ OH^{-}_{+} \mbox{ organic } & \longrightarrow & Product\\ {}^{\bullet}OH_{+}Fe^{2+} & \longrightarrow & Fe^{3+}+OH^{-} \end{array}$

The OH can interact with organic materials and/or the Fenton catalyst in mutual reactions in one of three ways: 1) hydroxyl addition, 2) hydrogen abstraction, or 3) electron transfer. Hydroxyl addition occurs in organic compounds with aromatic systems or numerous carbon-carbon bonds. Hydrogen abstraction occurs when unsaturated organic compounds contact inorganic ions, whereas electron transfer occurs when OH interacts with inorganic ions [14].

$$\begin{array}{c} OH_{+}R \longrightarrow R (OH) \\ OH_{+}RH \longrightarrow R+H_{2}O \end{array}$$

Because iron has a low poisonousness and Fenton approach (Fe^{2+}/H_2O_2) uses H_2O_2 , a non-toxic oxidant that breaks down into water and oxygen, it is a promising wastewater treatment technology. Fenton techniques are attractive due to their inexpensive cost, straightforward equipment, and ease of use and maintenance [15].

In order to maximize the performance of the Fenton technology and reuse the treated wastewater for irrigation with low cost and high efficiency, the effects of pH, ferrous salt (Fe²⁺), and H₂O₂ oxidation factors were examined in this experimental investigation. In order to optimize and research the impacts of three operational factors on COD reduction efficiency (pH, Fe⁺², and H₂O₂ concentrations) the current work also used Box–Behnken Design response surface design.

2. MATERIALS AND METHODS

2.1 Wastewater characteristics

Wastewater was collected from a wastewater treatment plant's collecting chamber in Beni-Suef, Egypt. Five samples were collected in five 20-liter containers, acidified to fix BOD and COD values, and then transferred to the lab, where the model was placed to conduct the tests.

2.2 Description of the Experimental procedure

Experiments were carried out in a batch reactor employing Fenton's higher removal efficiency. A wide variety of factors were evaluated in these trials, including pH, ferrous salt concentration, and H_2O_2 concentration, as reported in **Table 1**. Before the oxidation procedure, the pH was adjusted to the appropriate levels with H_2SO_4 and NaOH. The hydroxyl radical was then created by combining H_2O_2 and Fe⁺² solutions.

TABLE 1.	shows	the Control	variables	for	the
	Fentor	n process			

Control Variables	Levels of Concentrations		
Control Variables	Levels of ConcentrationMinimumMaximum2.56.5r)82250350	Maximum	
PH	2.5	6.5	
Ferrous salt (mM per 1 Liter)	8	22	
H ₂ O ₂ (mM per 1 Liter)	50	350	

2.3 Design of Box-Behnken Model

Box-Behnken was built using Design Expert Software to examine the PF response pattern in order to eliminate COD. A three-level Box–Behnken factorial design, including 15 runs with three replicates (central point), was employed by the researchers to develop the model.

 $\mathbf{Y} = \beta_0 + \sum_{i=1}^{K} \beta_i x_i + \sum_{i=1}^{K} \beta_{ii} x^2_i + \sum_{i=1}^{k-1} \sum_{i=1}^{K} \beta_{ij} x_i x_j$

Where Y is the predicted response (%), β_0 is the constant model coefficient, x_i , x_j are the independent variables, and β_i , β_{ii} , β_{ij} are the model coefficient terms.

2.4 Description of the Laboratory Pilot

Two experimental trials were performed in this study; the first batch reactor was established to estimate the optimum dosages of ferrous salt concentration, and H_2O_2 concentration in addition to pH. The second Experimental Trial was performed to apply the optimum conditions that are concluded from the first batch reactor to reuse this treated wastewater for irrigation purposes. The first model consists of two tanks; the primary sedimentation tank and the Fenton tank which are fully described in Table 2. The second model consists of four tanks a primary sedimentation tank, Fenton tank, aeration tank, and a filter media.

pilot					
Treatment unit	Parameters	Values			
Primary	$\begin{array}{c} \text{Length} \times \text{width} \times \\ \text{depth} \end{array}$	$50 \times 30 \times 30$ cm			
sedimentation	Volume of water	45 liters			
tank	Hydraulic retention time	3 hr.			
	$\begin{array}{c} \text{Length} \times \text{width} \times \\ \text{depth} \end{array}$	$50 \times 30 \times 30$ cm			
	Volume of water	45 liters			
Fonton tonk		Iron salts (Fe ⁺²)			
renton tank	Chemical additives	Hydrogen			
		peroxide (H ₂ O ₂)			
	Hydraulic retention time	60 minute			
	$\begin{array}{c} \text{Length} \times \text{width} \times \\ \text{depth} \end{array}$	$50 \times 30 \times 30$ cm			
Aeration tank	Volume of water	45 liters			
	Hydraulic retention time (HRT)	8 hr.			
	$\begin{array}{c} \text{Length} \times \text{width} \times \\ \text{depth} \end{array}$	$50 \times 30 \times 30$ cm			
Final setting	Volume of water	45 liters			
tank	Hydraulic retention time	2 h			
Filter	Filter media	Cotton			
Air blower	Rate of flow	420 L/h			

TABLE 2. shows the full description of the Laboratory

2.5 Description of the Experimental program

At first, optimization of the operation conditions of Fenton technology is an essential step. So, wastewater was fed into a primary sedimentation tank with a hydraulic retention time of 3 hours to settle all the suspended pollutants found in wastewater then passed into the Fenton tank to operate sets of experiments to determine the optimum dose of ferrous salt concentration, and H₂O₂ concentration. The hydraulic retention time needed for a reaction was 1 hour. The concentration of the used H_2O_2 is 50, 200, and 350 (mM per 1 Liter), the concentrations of ferric salt are 8, 15, and 22 (mM per 1 Liter). The Fenton process needs acidic conditions to operate so, H2so4 adjusts pH at a range between 2.5 to 6.5. After that, wastewater was fed into a final settling tank to settle the nonorganic substances produced from the organic substances. The scheme of the first trial is illustrated in Figure 1.



FIG 1. illustrates the scheme of first Experimental model

As shown in Figure 2, the second experimental trial was an application with the optimum doses of ferrous salt concentration, and H_2O_2 that is obtained from the first model. In the second experimental model, wastewater was fed into the primary sedimentation tank, and then fed into the Fenton tank with HRT of 1 hour, then, wastewater was passed to an aeration tank to perform perfect biodegradation of the remaining organic substances that are non-biodegradable in the Fenton tank with a hydraulic retention time of 8 hours. Finally, wastewater was passed to a filter of cotton to remove any nonorganic substances from the previous units.





FIG 2. illustrates the scheme of second Experimental model

3. RESULTS and DISCUSSION

3.1Characteristics of raw wastewater

TABLE 3. Characteristics of	Raw wastewater
Parameters	Concentrations
рН	8.4
Total suspended solids, TSS	320
Biological oxygen demand, BOD	274
Chemical oxygen demand, COD	525
Total nitrogen, TN	47
Total phosphorous, TP	6.7

From Table 3, it is noticed that the COD, BOD concentrations were 525 mg/l, and 274 mg/l, respectively which was matched with the average values reported by the other studies [16]. On the other hand, the concentrations of total nitrogen (TN) and total phosphorous (TP) were above the average values reported by other studies [17]. That may be due to the interaction between the domestic wastewater and the agricultural wastewater that may infiltrate into the sewage network. As known, the agricultural wastewater is concentrated with fertilizers which are a main source of nutrients [18].

3.2 The effect of PH on the performance of Fenton for organic Removal

The PH value of the effluent was adjusted by adding 5.5 ml, 6.5 ml, and 7.5 ml of concentrated sulfuric acid. Start the Fenton oxidation process by adding 8 milligrams (mM) of ferrous sulfate and 50 milligrams (mM) of hydrogen peroxide. Stir the mixture while it is happening. The COD value and Fenton oxidation effect were assessed using a sample. Using experiment 1, one might ascertain the initial PH value. Water sample 1 is the optimum, based on the experimental findings. Table 4 displays the optimal initial PH value of 4.5.

TABLE 4. shows the effect of pH varying on COD and TP removal efficiency

Parameters	5.5 ml of H ₂ SO ₄	6.5 ml of H ₂ SO ₄	7.5 ml of H ₂ SO ₄
starting PH value	6.5	4.5	2.5
Chemical oxygen demand, COD	228.9	230.5	269.3
COD removal efficiency	56.4%	56.1%	48.7%
Total phosphorous, TP	0.52	0.5	0.55
TP removal efficiency	89.4%	89%	88.1%

3.3 The effect of hydrogen peroxide on the performance of Fenton for organic Removal

8, 15, and 22 (mM per 1 Liter) of hydrogen peroxide (H_2O_2) were added to wastewater to determine the optimum dosage of hydrogen peroxide that achieved the highest COD, and TP removal efficiency. To initiate the Fenton oxidation reaction, 8 milligrams (mM) of ferric salt were added to one liter of water. The chemicals were mixed and stirred while the wastewater's pH was adjusted to 4.5. A sample was collected in order to assess the Fenton oxidation impact and determine the value of COD. Using experiment 2, the dosage of hydrogen peroxide could be ascertained. Table 5 displayed the outcomes of the trial. Based on the outcomes of the experiment, wastewater sample 1 removed COD and

TP with the desired efficiency, and 350 milligrams of hydrogen peroxide were used (1 liter).

TABLE 5.	shows the effect	of hydrogen	peroxide on	COD
	and TP rem	oval efficiend	cy	

Parameters	50mM of H2O2	200mM of H2O2	350mM of H2O2	
starting PH value	4.5			
Chemical oxygen demand, COD	257.3	247.8	236.3	
COD removal efficiency	51 %	53 %	55 %	
Total phosphorous, TP	0.14	0.14	0.14	
TP removal efficiency	97 %	97 %	97 %	

3.4 The effect of Ferric salt on the performance of Fenton for organic Removal

8, 15, and 22 (mM per 1 Liter) of Ferric salt (Fe2+) were added to wastewater to determine the optimum dosage of Ferric salt that achieved the highest COD, and TP removal efficiency. The pH of the wastewater was adjusted to 4.5 before adding 22 mM of H2O2 to initiate the Fenton oxidation process. The chemicals were mixed and stirred during the experiment. A sample was collected in order to assess the Fenton oxidation impact and determine the value of COD. Using experiment 3, the dosage of Fe2+ could be ascertained. Table 6 displayed the outcomes of the trial. The preferred COD and TP removal efficiencies were attained by Wastewater sample 1, and the dosage of ferric salt was 22 (mM per 1 Liter), based on the testing results.

TABLE 6. shows the effect of Ferric salt (Fe^{2+}) on CODand TP removal efficiency

Parameters	8mM of (Fe ²⁺)	15mM of(Fe ²⁺)	22mM of(Fe ²⁺)	
starting PH value	4.5			
Chemical oxygen demand, COD	391.65	306.6	263.55	
COD removal efficiency	25.4 %	41.6 %	49.8 %	
Total phosphorous, TP	0.6	0.42	0.19	
TP removal efficiency	87 %	91 %	96 %	

3.5 Optimization of the Fenton process using Design Expert Software

The Design Expert Software is frequently used for planning experiments that use computational and statistical methods to determine the best design response condition. Table 1 shows the three-level experiments that correspond to the Box–Behnken design and experimental plan used in _____

this investigation. Design-Expert Software carried out the Box–Behnken design (BBD). The goal of the tests was to see how pH, Fe+2, and H2O2 concentration affected phenol and COD reduction efficiency.

TABLE 7. shows the Modified Analysis of Variance
(ANOVA) test for the COD removal ratio.

Source	Sum of Square s	DF	Mean Square	F- value	p-value	
Model	1434.5 2	6	239.09	12.44	0.0004	signif icant
PH (A)	132.03	1	132.03	6.87	0.0256	
H ₂ O ₂ (B)	968.00	1	968.00	50.36	< 0.0001	
Ferrous salt (C)	258.78	1	258.78	13.46	0.0043	
(PH* H ₂ O ₂)	1.69	1	1.69	0.087 9	0.7729	
(PH* Fe ²⁺)	33.06	1	33.06	1.72	0.2190	
H ₂ O ₂ * Fe ²⁺)	40.96	1	40.96	2.13	0.1751	
$(PH* \\ H_2O_2* \\ Fe^{2+})$	0.0000	0				
Residu al	192.23	10	19.22			
Lack of Fit	191.56	6	31.93	191.1 8	< 0.0001	Signif icant
Pure Error	0.6680	4	0.1670			
Total	1626.7 5	16				

The inconsequential components were eliminated for simplicity's sake, and the following equations yielded a new regression model:

COD removal = 55.87+4.0625 PH+11 H_2O_2 + 5.6875 Fe^{2+} +0.649 PH* H_2O_2 -2.875 PH* Fe^{2+} -3.2 H_2O_2 * Fe^{2+}

For statistical significance, ANOVA was used to fit the model Equation. The F value and P-value can be used to assess the model's suitability. The P-value refers milli the model fits the experimental data. The range of an R^2 value is 0 to 1. A reasonable model fit requires an R^2 value greater than 0.80.



FIG 3 illustrates the relationship between each operation condition and the COD removal

Also, Figure 4 shows the interpolation between the PH, Fe^{+2} , and H_2O_2 concentration and its effect on the removal efficiency of organic substances in wastewater.



FIG 4 Model Graph exported from Design Expert software

3.6 Optimization of the Parameters of Fenton Process

Using design expert software and multiple response strategies, the optimal COD removal efficiencies were calculated. The desirability function method was also used to optimize various process parameter combinations. The researched variables pH, Fe+2, and H2O2 were considered to reach a maximal response for COD uptake rates to create ideal circumstances. The independent variables' optimum values were found here (PH=4.2, Fe⁺²= 21.9 mM per liter, H₂O₂= 335 mM per liter). At these conditions, removal percentages of the verification experiments are close to the predicted values with uptake rates of 72 % for COD as shown in Figure 5.



FIG 5 shows the optimum values of pH, Fe⁺², and H₂O₂concentrations



FIG 6 illustrates the predicated COD removal at optimum conditions

3.7 The Effect of interaction between aeration Tank and Fenton Technology

To maximize the efficiency of COD removal, an aeration tank was added to the second experimental model to reduce the remaining organic matter below desirable limitations suitable to reuse wastewater for irrigation purposes. The concentrations of COD, BOD, TSS, TP, and TN are illustrated in Table 8.

Parameters	Ra	After	Fento n tonk	Aeration	Filter
aU	W	4.5		7.4	7.4
рн	8.4	4.5	4.5	7.4	7.4
Total					
suspended	320	91	56.5	64	27
solids, TSS					
Biological					
oxygen	274	213.	102.2	17.9	14.0
demand,	274	7	102.5	17.0	14.9
BOD					
Chemical					
oxygen	525	425.	104 5	36.86	32.4
demand,	525	3	194.3	30.80	32.4
COD					
Total					
nitrogen,	47	36.8	28.7	15.9	12.1
TN					
Total					
phosphorou	6.7	5.1	1.36	1.14	1.12
s, TP					

TABLE 8. illustrates the physicochemical parameters after each treatment stage in mg/l

From the previous table, it was clear that Fenton is a very effective treatment technology as the initial COD and BOD concentrations are 525and 274 mg/l and after passing to the Fenton tank, COD and BOD concentrations reduced to 194.5 and 102.3 mg/l with removal efficiency up to 55%. According to other research, Fenton technology can remove COD with an efficiency of up to 70%, depending on the kind of wastewater, the operating environment, and the amount of H2O2 and ferric salt used [19].



FIG 7 illustrates Removal efficiencies achieved by each treatment stage

From Figure 8, it is noticed the availability of reuse the effluent from each treatment stage (agricultural drainage water and mixed wastewater) in irrigation purposes after comparing it with the allowable limitations of the Egyptian code [20]. The suitable group for the effluent characteristics of the research work is group D. The results revealed that the Egyptian code (ECP 501/2015) is highly compatible with the results of the research work, but there are some differences. These differences are allowable because (ECP 501/2015) prohibited the use of treated wastewater in irrigating raw vegetable crops. Finally, the acceptable chemical, physical, and agronomic properties of treated wastewater for agricultural applications in Egypt have to be more studied to make the best use of treated wastewater. On the other hand, the availability of reuse the effluent of BOD in irrigation purposes after comparing it with the allowable limitations of the Egyptian code [21].



FIG 8 shows a comparison between effluent from each stage and ECP

4. CONCLUSION

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From the results and discussions of the previous experiments carried out, the following conclusions can be taken:

- 1. For the Fenton process, the Box Behnken Experimental Design (BBD) was employed to identify the best removal efficiency for both COD and phenol.
- 2. The optimum conditions for pH, Fe^{+2} , H_2O_2 are 4.2, 21.9mM per liter, 335 mM for 71% COD removal.
- 3. Fenton Technology is a very efficient technology for wastewater treatment.

In further study for the Fenton process, the Box Behnken Experimental Design (BBD) will employed to identify the optimum removal efficiency for phenol.

ACKNOWLEDGMENTS

The authors want to thank the Laboratory of Environmental Engineering at Zagazig University's Faculty of Engineering and Beni-suef University's Faculty of Engineering since their contributions were invaluable throughout the experiments. We highlight their expertise, resources, and responsive support by presenting their essential role in promoting research.

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