

Modeling Fenton Treatment for Landfill Leachate with the Focus on Initial COD and Sludge Generation

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Abstract:

There are different methods to treat landfill leachates among which the Fenton process as a pretreatment physiochemical method is used in this paper to improve the BOD to COD ratio. While the center of attention of most previous studies has been the removal of organic pollutants, the amount of generated sludge is normally neglected which is an important element. Therefore, in this study, two other factors (SIR and ORSR) and COD removal rate, are evaluated to consider the sludge generation factor as well. Meanwhile, the initial COD concentration of the leachate is a changeable factor depending on the site, region, climate, and internal reactions that are examined in this research. For the design of the experiment, central composite design was used to minimize the required tests and see the interaction between variables. Therefore, input variables were pH, $[\text{H}_2\text{O}_2]/[\text{Fe}^{2+}]$, Fe^{2+} dosage, and initial COD concentration while COD removal, SIR, and ORSR were seen as output targets. The interesting result is that the initial concentration of COD was not a determining factor for COD removal rate, SIR, and ORSR. Although low pH values were favorable for the COD removal rate, high pH values led to better SIR and ORSR quantities, which indicated the lower usage of chemicals for pH adjustments. For low (favorable) SIR, the amount of Fe dosage must be restricted to generate lower sludge. For better (higher) ORSR values, high pH and $[\text{H}_2\text{O}_2]/[\text{Fe}^{2+}]$ quantities were suggested. The formulas proposed for each output target could pave the way for more accurate predictions of the results.

1. Introduction

Environmental protection practices are becoming more important than ever before in different areas [1]. One practical approach to managing urban solid waste is to dispose in sanitary landfills [2]. However, the most important concern with landfills is the uncontrolled leakage of leachate that could contaminate water streams. Unfortunately, leachates in different landfill sites or different spots in one specific landfill site possess characteristics that vary remarkably because of several affecting factors, namely: elapsed time, landfill site depth,

hydrology of the site, temperature, and moisture content [3]. The main pollutants in leachates include high concentrations of ammoniacal nitrogen [4], high dosages of recalcitrant compounds [5], and low BOD to COD ratio [6]. There are mainly two specific approaches to leachate treatment: biological and physicochemical [7]. Among these methods, membrane filtration [8][9], activated carbon, ozonation, and Fenton processes have been more prevalent [10]. Nevertheless, the Fenton process including conventional Fenton, photo-Fenton, and electro-Fenton have gained more popularity in recent years [11]. The main reasons for this popularity include effective biodegradability enhancement, high efficiency in removal, simplicity of operation, and flexibility in terms of influent types [12].

The benefit of the conventional Fenton process is that it is simple and takes advantage of the presence of the reagent solution called hydrogen peroxide (H_2O_2) and an iron

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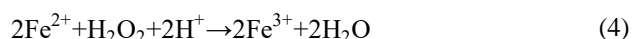
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catalyst (usually FeSO₄). This advanced oxidation process (AOP) method has been used frequently in treating pharmaceuticals, petroleum refineries, cosmetics, pesticides, and dye wastewater. This method takes benefit of the formation of highly reactive hydroxyl radicals (OH[•]) and oxidizes complicated organic substances (RH) that form stable ferric-hydroxo complexes [10]:



The summary of equations 1 to 3 could be expressed as Eq. (4) [6]:



Unfortunately, the focus of most studies conducted on the treatment of leachate via the use of the Fenton process was on the removal of COD [13] while the quantity of sludge produced in the process and the amount of Fe²⁺ used in the experiment should also be of significant importance [14]. The Fenton reaction is indeed efficient and popular, but the volume of sludge generated, the hazards with the sludge, the relatively high cost of chemicals, and changeable operational conditions make it a baffling method for users [12]. Once the optimum condition can be predicted by a model and the definitions for efficiency change from only a high organic removal rate to low values of sludge plus a high removal rate, these complications may be resolved [15]. Meanwhile, most studies in the literature have concentrated on the effective parameters of the Fenton reaction for leachate treatment while the impact of the type of leachate with different initial COD values has not been thoroughly investigated [16]. Different COD values may require different operational factors such as H₂O₂ and Fe²⁺. Developing a model that could predict the COD removal rate as well as an index for the generated sludge could help experts to have a better prediction and understanding of operative conditions.

When it comes to the design of experiments, the response surface method is believed to be an effective help to minimize the test numbers and examine the interactions between different factors [17]. In conventional studies, one factor is measured while others are kept constant; however, the interaction between elements cannot be fully seen [18]. One practical method is the use of central composite design (CCD) which is a popular fractional factorial design [19]. In this design of the experiment method, first-order and second-order terms could be estimated [20]. This test design enables users to run the minimum possible experiments [21] while the interaction of different factors is simultaneously

seen [22]. In addition, introducing a multivariate formula to represent the function of the Fenton process could shed light on researchers to have logical predictions of the efficiency of Fenton processes [23].

The research gap that should be covered is that initial COD concentration rarely has been considered as an effective factor. Moreover, most studies have prioritized COD reduction alone, with a limited focus on sludge management metrics like SIR and ORSR. To determine the amount of the generated sludge, sludge to iron ratio (SIR) and organic removal to sludge ratio (ORSR) are used to assess the efficiency of the Fenton process for organic removal and sludge generation [24]. Hence, in this paper, the Fenton reaction is used for the treatment of the leachate from the Kahrizak site in the south of Tehran Iran. Four affecting factors include pH, [H₂O₂]/[Fe²⁺] mole ratio, [Fe²⁺] dosage, and initial COD concentration while the removal rate of COD, SIR, and ORSR are introduced as output targets. To achieve a better understanding of the process, a multivariate regression is proposed to achieve a math formula and a prediction for the results.

2. Material and Method

2.1 Experimental procedure

The collected samples were prepared from the Kahrizak landfill site in Tehran Iran. They were manually collected from ponds of leachates on the Kahrizak site in clean and sterilized polyethylene containers which were taken to the laboratory and kept in the fridge at 4°C. The important characteristics of the leachate (mean pH and mean initial COD) are mentioned in Table 1.

Table 1: Mean values of pH and initial COD of the collected leachate

Parameter (unit)	Sample A	Sample B	Sample C	Sample D	Sample E
pH	8.1	7.7	7.4	7.5	7.3
COD (mg/l)	1155	4265	9560	14765	18275

Chemicals used in this research were prepared by Merc Company in Germany. The pH of solution was measured via the use of WTW SERIES, pH730 instrument while Lovibond spectrophotometer was used to measure COD using medium and high range vials for COD measurement. All laboratory tests were performed at room temperature and atmospheric pressure. One-liter glasses conserved from the infiltration of UV were used to run experiments. Notably, sufficient space was considered to avert the overflow of foaming resulting from the initial stages of the reaction. At the beginning of the experiment, the adjustment of the initial pH was done using 1 M sulfuric acid and 10 M sodium

hydroxide solution. Then, the beaker was filled with 400 ml of the leachate sample and the mixing procedure started. A Jar-test device was used for rapid mixing at 175 rpm. Next, the predetermined amount of powered $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ was added to the beaker and mixed for 5 minutes to reach a homogeneous solution. Thereafter, the required amount of hydrogen peroxide solution (H_2O_2 , 30% w/w) was added to the mixture and the Fenton reaction started. The reaction continued until the designated time was passed (90 minutes for all experiments), then to adjust the pH to around 7, NaOH was added. Next, the solution of 10M NaOH was used dropwise to the pH of 8, and the solution was mixed slowly at 30 rpm for 20 minutes for flocculation. Thereafter, a 100 ml sample of the final solution was dispensed to a graduated cylinder to determine the volume of the sludge. It should be noted that samples were warmed in a 50°C water bath for 30 minutes to remove the residual H_2O_2 in the solution as it may interfere with the COD analysis. The sedimentation took 60 minutes and the height of the sludge was recorded. The COD of the solution was examined from the supernatant of the beaker.

2.2 Test Design Procedure

Test design software is developed to minimize the number of laboratory tests and make sure that an adequate amount of information is provided to be examined for the lack of fit. Central composite design (CCD) is an effective method that provides a reasonable estimation of the nonlinearity of the dependent variable and allows for maximum information with minimum experimental results, leading to fewer experiments to predict quadratic terms in the second-order model. For this purpose, the Design Expert Software (version 11) was applied to the experimental design. In this research, four influencing factors include pH, $[\text{H}_2\text{O}_2]/[\text{Fe}^{2+}]$ ratio, $[\text{Fe}^{2+}]$ and initial COD concentration while COD removal rate, sludge to iron ratio (SIR), and organic removal to sludge ratio (ORSR) were chosen as targets. Table 2 shows the factor levels for the design of experiments.

Table 2: Factor levels for the design of experiments

Test variable (unit)	Symbol	Coded values				
		-2	-1	0	+1	+2
Initial pH	X ₁	2	3	4	5	6
$[\text{H}_2\text{O}_2]/[\text{Fe}^{2+}]$ mole ratio	X ₂	4	8	12	16	20
$[\text{Fe}^{2+}]$ (mM)	X ₃	80	120	160	200	240
Initial COD (mg/l)	X ₄	1155	4265	9560	14765	18275

To the best of our knowledge, most studies have considered COD or TOC removal as the most crucial response in the

Fenton process. Nevertheless, the efficiency of the COD removal rate does not consider the amount of generated sludge or the quantity of added ferrous iron to the experiment. Therefore, SIR (the ratio of sludge to the amount of iron compound) and ORSR (the ratio of organic pollutant removal to the quantity of the generated sludge) are examined in this paper as other important targets proposed by Amiri and Sabour [24]:

$$\text{SIR} = \frac{\text{produced sludge volume (l)}}{\text{added ferrous iron (mole)}} \quad (5)$$

$$\text{ORSR} = \frac{\text{overall COD removal (g)}}{\text{produced sludge volume (l)}} \quad (6)$$

Table 3 illustrates the details of the CCD test design.

Table 3: The proposed test design

Test	Real Values			
	pH	$[\text{H}_2\text{O}_2]/[\text{Fe}^{2+}]$ (mole ratio)	$[\text{Fe}^{2+}]$ (mM)	Initial COD (mg/l)
1	4	12	160	18275
2	5	8	120	4265
3	3	16	200	4265
4	4	20	160	9560
5	3	16	120	4265
6	5	8	120	14765
7	5	16	200	4265
8	3	16	120	14765
9	5	8	200	14765
10	2	12	160	9560
11	4	12	160	1155
12	4	12	160	9560
13	3	16	200	14765
14	3	8	200	14765
15	5	8	200	4265
16	4	12	160	9560
17	5	16	120	14765
18	4	4	160	9560
19	4	12	80	9560
20	4	12	160	9560
21	5	16	200	14765
22	4	12	240	9560
23	3	8	200	4265
24	5	16	120	4265
25	3	8	120	4265
26	4	12	160	9560

27	4	12	160	9560
28	6	12	160	9560
29	4	12	160	9560
30	3	8	200	14765

This test design produces a matrix that examines both linear and quadratic impacts across varying levels of each factor. The design of experiments included the following parts: (a) sixteen tests of the two-level factorial design (b) eight tests at the star points and (c) one central point and five repetitions for it to calculate the test error and any potential effects of a curvature in the response surfaces.

2.3 Multivariate Regression

In general, multivariate regression analysis is applied to derive predictive relationships between independent variables and output targets. Multivariate multiple regression is a tool to model multiple responses (known as dependent variables) while a single set of independent (predictor) variables is used; i.e. four factors mentioned above (X_1 to X_4) can affect the value of COD removal, SIR, and ORSR. For this purpose, a built-in data analysis tool was used. This tool can propose a formula for the prediction of the process and reveals important features of the correlation such as the fitting quality of the model (correlation coefficient R^2) and the importance of linear terms as F-values. The schematic relation in the type of regression is shown in Equation 7.

$$y = \alpha_0 + \alpha_1 X_1 + \alpha_2 X_2 + \alpha_3 X_3 + \alpha_4 X_4 \quad (7)$$

3. Result and Discussion

3.1 Multivariate Regression

In this article, COD removal is considered as a key factor to improve the BOD_5/COD ratio. The higher value of the BOD_5/COD ratio may indicate better biodegradability and potential for landfill site rehabilitation. Therefore, COD removal efficiency was measured as a valuable tool to evaluate the overall pollution level as well as the organic content of water. The results of COD removal are shown in Fig. 1 and Fig. 2. According to the figures COD removal efficiency decreases with increasing pH (X_1), indicating that lower pH levels are preferable. It is understood that this factor was not effective up to pH equal to 4, but then had a major impact. This observation aligns with previous studies [25] [26] indicating that the Fenton process operates most effectively at acidic pH levels. COD removal increased with higher values of X_2 and X_3 ($[H_2O_2]/[Fe^{2+}]$ and $[Fe^{2+}]$). In other words, when more Fe^{2+} is used and the ratio of H_2O_2 to Fe^{2+} is high enough, COD removal efficiency is high. This is previously mentioned by other researchers as well [24].

However, the influence of initial COD concentration is not noticeable, and with the increase in this concentration, the COD removal rate fluctuates slightly. The peaks that COD removal shows are related to Fe^{2+} values greater than 200 mM, which is not in contrast with previous studies (literature suggests the dosage over 150 mM) [27]. One interesting conclusion that could be achieved is that high values of pH mean lower chemical usage for pH adjustment, so if the adequate dosage of Fe is maintained, the removal efficiency of COD will be satisfactory.

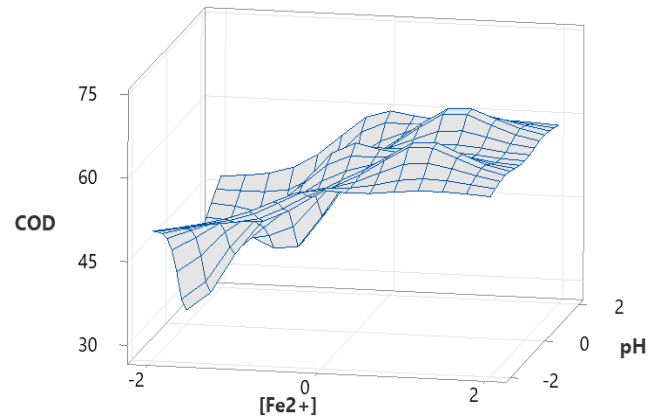


Fig. 1: Surface plot of COD as a function of X_1 and X_3

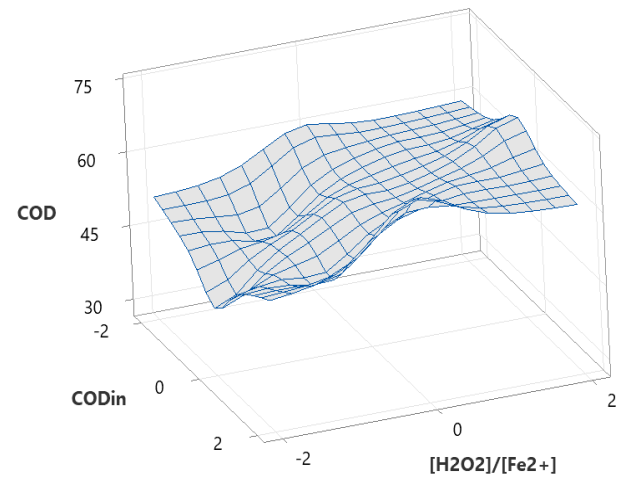


Fig. 2: Surface plot of COD as a function of X_2 and X_4

To achieve a relation that shows the influence of the independent variable (X_1 to X_4), a multivariate regression is suggested with the help of the regression tool in the Data Analysis section (Excel). Equation 8 shows the COD removal rate as a function of X_1 to X_4 . In this formula, X_1 represents pH, X_2 represents $\frac{H_2O_2}{Fe^{2+}}$, X_3 represents Fe^{2+} , and X_4 represents the initial COD of the leachate while Y_1 indicates the rate of COD removal. Table 4 represents the regression statistics for COD removal efficiency.

$$Y_1 = -1.19X_1 + 1.89X_2 + 0.22X_3 \quad (8)$$

Table 4: The regression statistics for COD removal rate

Multiple R	R Square	Adjusted R Square	Standard Error	Observations
0.992	0.984	0.943	7.790	30

Equation (8) indicates that the initial concentration of COD is not a determining factor and its impact is completely negligible which is quite interesting. Meanwhile, since the correlation coefficient (R^2) is around 99% and the multiple R is above 99%, the relation can create an image for prediction of results.

Table 5 presents the ANOVA results for the COD removal rate regression model. The f-value can indicate the statistical significance of the regression equation as a whole. Since the F-value exceeds 4 (around 357), this model is statistically significant, indicating a robust predictive capability for COD removal under varying conditions. The value for significance-F should be less than 0.05, which is completely suitable in this model. Introducing such models can facilitate the initial predictions of the process for future studies to have a clearer imagination of the reaction and affecting factors.

Table 5: The ANOVA results for COD removal rate

ANOVA					
	df	SS	MS	F	Significance F
Regression	4	96269.7	24067.4	356.6	3.2E-22
Residual	26	1577.9	60.7		
Total	30	47847.7			

3.2 SIR

An important consideration in the Fenton process is the quantity of sludge generated. It is important from the following aspects: a) waste management approaches, b) limited landfill space, c) environmental aspects, d) operational efficiency, and e) regulatory compliance. The Sludge Index Ratio (SIR) serves as a key criterion for assessing sludge production. Figures 3 and 4 represent the results of SIR versus different variables. As illustrated in Figure 4, an increase in pH is associated with a reduction in SIR, which is desirable for minimizing sludge production. Lower sludge production occurs at near-neutral pH levels, which also reduces the chemical demand for pH adjustment. However, it must be noted that lower values of pH were crucial for high COD removal. This highlights the importance of considering different factors when deciding favorable operating conditions. The H_2O_2/Fe^{2+} ratio (X_2) initially decreases SIR, after which the reduction rate stabilizes. Optimal SIR results are achieved when X_2 is approximately 12, balancing minimal chemical use with effective sludge control. Higher Fe^{2+} dosages (X_3)

correspond to increased SIR values, suggesting that Fe^{2+} dosing should be carefully managed to limit sludge generation. The effect of X_4 was not remarkable (similar to COD removal) which shows that the initial concentration of COD is not an important factor in the operational perspective. The practical result here is that high values of pH and low values of Fe could lead to minimum amounts of SIR. These observations were mostly seen in previous studies [28][26].

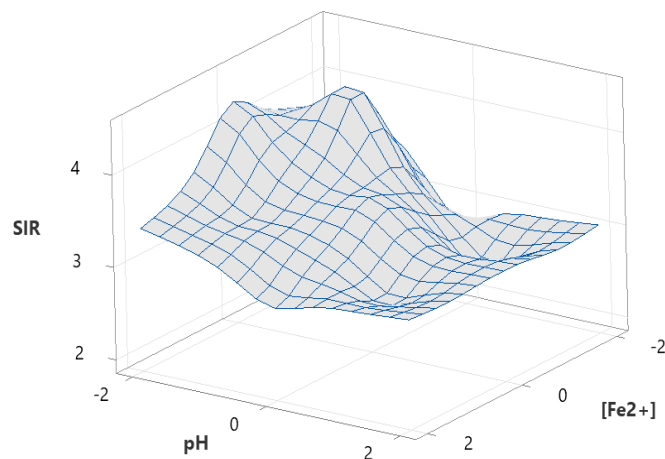


Fig. 3: Surface plot of SIR as a function of X_1 and X_3

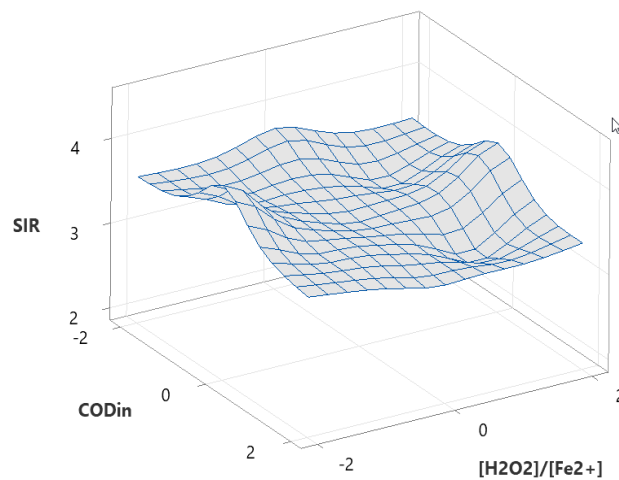


Fig. 4: Surface plot of SIR as a function of X_2 and X_4

A model is also presented to evaluate the impact of independent variable (X_1 to X_4) on SIR. Equation 9 shows the SIR as a function of X_1 to X_4 . In this formula, X_1 represents pH, X_2 represents $\frac{H_2O_2}{Fe^{2+}}$, X_3 represents Fe^{2+} , and X_4 represents the concentration of input COD while Y_2 indicates SIR. The formula confirms that X_4 (initial COD concentration) is not an effective parameter in SIR measurement. Table 6 represents the regression statistics for SIR.

$$Y_2 = +0.07X_1 + 0.07X_2 + 0.01X_3 \tag{9}$$

Table 6: The regression statistics for SIR

Multiple R	R Square	Adjusted R Square	Standard Error	Observations
0.964	0.930	0.883	0.969	30

According to Table 6, the correlation coefficient (R^2) is 0.93 and the multiple R is almost 0.96, indicating that the regression model for SIR provides a strong predictive framework, and the standard error is within the acceptable range (less than 2%).

Table 7 presents the ANOVA results for the SIR model, with an F-value of approximately 87, which is well above the threshold of 4, demonstrating the model's statistical significance. When the significance-F is less than 0.05, the model is meaningful; i.e. the null hypothesis is rejected, and input variables can predict output ones. Moreover, a value greater than 4 (around 87) shows that the model is statistically significant.

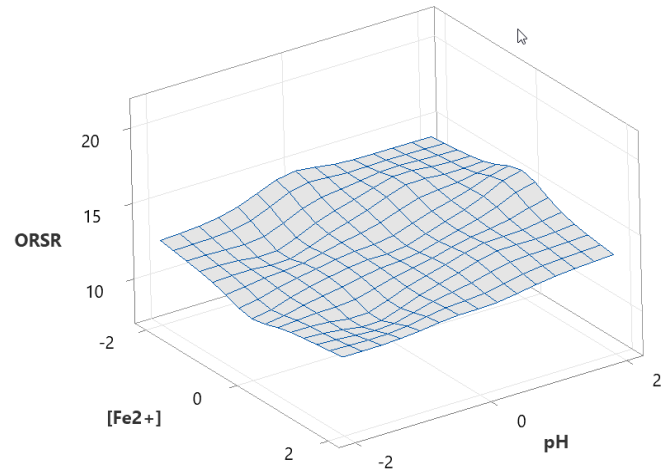
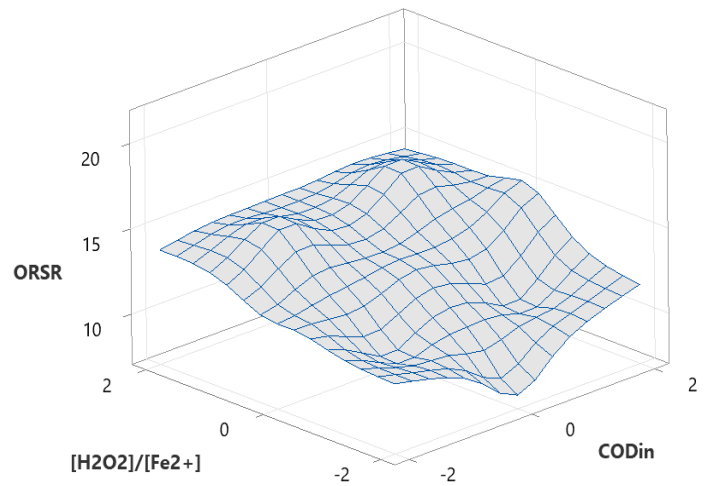
Table 7: The ANOVA results for SIR

ANOVA					
	df	SS	MS	F	Significance F
Regression	4	325.5	81.4	86.6	2.8E-14
Residual	26	24.4	0.9		
Total	30	349.9			

3.3 ORSR

One helpful criterion to consider for COD removal rate and sludge generation at the same time is ORSR. Achieving optimal values in ORSR is crucial for maximizing COD removal while minimizing sludge production. Figures 5 and 6 illustrate ORSR versus X_1 to X_4 variables. The philosophy that dictates the necessity of this criterion is that only high removal of efficiency of organic pollutants cannot be ideal, and the impact of sludge produced is also important. As the amount of pH and $\frac{H_2O_2}{Fe^{2+}}$ increased, the value of ORSR improved proportionally. Conversely, variations in Fe^{2+} dosage (X_3) and initial COD concentration (X_4) do not yield a similar positive effect on ORSR. This could be attributed to the significant role of oxidation and the insignificant role of coagulation. In fact, the meaningful presence of H_2O_2 led to the domination of oxidation (therefore high COD removal) while the role of coagulation is minimized. Therefore, high amounts of pH and $\frac{H_2O_2}{Fe^{2+}}$ are recommended. Other researchers have reached the same conclusion as well [24]. On the other hand, the role of Fe dosage and initial concentration of COD are not determining factors here. The key result is that elevated pH values enhance operational efficiency by reducing the need for pH adjustment chemicals, while still achieving satisfactory COD removal

and minimizing sludge generation. In this criterion (similar to COD and SIR), the impact of initial COD is not considerable, which could show that the Fenton reaction is not dependent on the initial concentration of pollutant at least at these examined values.

**Fig. 5:** Surface plot of ORSR as a function of X_1 and X_3 **Fig. 6:** Surface plot of ORSR as a function of X_2 and X_4

Like other parts of this research, a formula is introduced to assess the impact of independent variable (X_1 to X_4) on ORSR. Equation 10 shows the ORSR as a function of X_1 to X_4 . In this formula, X_1 represents pH, X_2 represents $\frac{H_2O_2}{Fe^{2+}}$, X_3 represents Fe^{2+} , and X_4 represents initial COD concentration while Y_3 indicates ORSR. The formula confirms that X_4 (initial COD concentration) is not an effective parameter in ORSR measurement. The results of regression statistics for ORSR are presented in Table 8.

$$Y_3 = +1.34X_1 + 0.64X_2 - 0.07X_3 \quad (10)$$

According to Table 8, the correlation coefficient (R^2) is about 0.99 and the multiple R is over 0.99, so the results can be predicted, and the standard error (1.5) is within the acceptable range (less than 2%).

Table 8: The regression statistics for ORSR

Multiple R	R Square	Adjusted R Square	Standard Error	Observations
0.995	0.989	0.949	1.453	30

Table 9 illustrates the ANOVA results of the ORSR formula. The f-value is approximately 620, which is greater than 4, and indicates that the model is statistically significant. Significance-F also indicates the predictability of the formula.

Table 9: The ANOVA results for ORSR

	ANOVA				
	df	SS	MS	F	Significance F
Regression	4	5232.1	1308.0	619.6	1.3E-24
Residual	26	54.9	2.1		
Total	30	5286.9			

4. Conclusion

The Fenton process as a physical/chemical pretreatment method, was applied to landfill leachates in Iran. Most studies in the literature have indeed investigated only the removal of organic contaminants, but this paper also emphasizes sludge generation as a critical factor alongside COD removal. In the experimental design of the procedure, pH, $[H_2O_2]/[Fe^{2+}]$, Fe^{2+} dosage, and initial COD concentration were considered as input variables whereas COD removal, SIR and ORSR were assumed as output targets. The following results were observed in this research:

1. The correlation coefficients were 0.93, 0.98, and 0.98 for Y_1 , Y_2 , and Y_3 which indicates the predictability of the models. Meanwhile, the quantities of F-value and standard error were all within the acceptable range.
2. Although low pH values were suggested for better removal of organic pollutants, in terms of applicability and financial management, high pH amounts were more pleasant. More importantly, SIR and ORSR were more favorable in high dosages of pH which makes a practical result for less effort of pH adjustment.
3. The higher ratio of H_2O_2 to Fe^{2+} led to better results of COD removal, SIR and ORSR; however, the restriction of Fe^{2+} dosage was more appropriate.
4. The initial concentration of COD of the leachate was not a meaningful element whether in final COD removal or SIR and ORSR; therefore, the results obtained here could be generalized for initial CODs ranging from 1155 mg/l to 18275 mg/l.

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