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Application of response surface method for Total organic carbon reduction in leachate treatment using Fenton process

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Abstract

Removal of the untreated landfill leachate can be a wellspring of risk to accepting waters. Hence, the treatment of landfill leachate is considered environmentally essential. Response surface methodology (RSM) was used in this study to obtain the best result from the treatment of landfill leachate, particularly the total organic carbon (TOC) removal. Various parameters such as the hydraulic retention time, pH, Fe^{2+} concentration and H_2O_2: Fe^{2+} ratio on the total organic carbon removal were investigated. Response surface methodology was connected to enhance the treatment procedure and characterise the parameters playing the most important role. It was evident that the underlying pH had the most noteworthy negative impact, while retention time had a positive effect. The Fe^{2+} concentration had a negative effect, and the ratio of H_2O_2: Fe^{2+} did not have a significant impact on the removal of organic carbon. Results showed that the optimum total organic carbon removal was observed at a retention time of 37.31 minutes, pH of 3, H_2O_2:Fe^{2+} ratio of 4.75, and Fe^{2+} concentration of 634.49 mg/L. The experimental values of the total organic carbon removal were validated through the response surface methodology and at the optimum settings, whereby up to 95.16% of total organic carbon removal was observed and thus confirmed the results of the developed model. A high R^2 value of 95.29% coefficient close to 1 confirmed some of the close similarities in the results of both experimental and model analyses. The generated empirical model can be used by landfill operators to estimate the removal of total organic carbon from the leachate by using Fenton treatment.

Keywords: Fenton process; Landfill leachate; Response surface methodology (RSM); Total organic carbon (TOC); Waste treatment

1. Introduction

The main challenges of municipal solid waste (MSW) management in many countries are waste disposal through traditional landfilling, among others. Landfills are considered as open space areas for dumping unrecovered waste disposal (Manaf, 2017; Selvam *et al.,* 2019). It is well reported that a majority of the MSWs are discarded in open dumps, and the landfill locations are causing issues to health and the ecology (Ghosh, 2019). According to Slazck *et al*. (2005), about 70% of MSW are disposed into landfills worldwide. As a result, if the MSW landfill sites are improperly managed, they can create harmful impacts, such as gaseous pollution and liquid emissions as leachate (Kamyab et al.,2015; Okoye and Elbeshbishy, 2019). Landfill leachate can be considered as one of the strongly polluted wastewaters (Mohajeri et *al.,* 2019). Due to the issues related to organic framework regulations, there is an emerging requirement in order to form an effective treatment process or enhance the current treatment procedures. The conventional methods of treating leachate, including adsorption, may not be effective because of the recalcitrant nature of contaminants (Bernal-Martínez *et al*., 2010; Colombo et *al*., 2019). Advanced oxidation processes (AOPs) is one of the effective technologies for wastewater treatment (Roudi *et al.,* 2018). AOPs take place by propelled oxidation, which can be affected by high decomposition capacity and rate (Müller *et al.,* 2015). Recently, a significant interest is gained for AOPs applicability for organic removal and the biodegradability improvement of leachate (Umar *et al.,* 2010 and Okoye and Elbeshbishy, 2019). Fenton's Reagent is considered as a suitable option in advanced oxidation process due to its resistance to be utilised in UV or ozone. Typically, hydrogen peroxide and ferrous ion complex are added to an aqueous system as the catalytic oxidation process (Benatti *et al.,* 2006). The toxic materials in the leachate may cause severe problems to both humans and the environment; thus, it is essential to monitor the concentration of certain parameters in leachate discharged from the landfill. Amongst the advanced oxidation processes, the Fenton process has been used extensively for leachate treatment. Studies have shown that this process has the capability to decrease organic contaminants and colour in leachate treatment. The process has some advantages, such as high organic matter removal, no toxic by-products, less energy requirement, and simple experiment (Roudi *et al.,* 2019)

The application of iron with hydrogen peroxide generates highly reactive hydroxyl radical, while reduces the chemical oxygen demand (COD), biological oxygen demand (BOD) and colour removal, as well as odour reduction, toxicity reduction, and improvement in biodegradability (Lopez *et al.,* 2004). Furthermore, the Fenton process has a few points of interest over other substance treatment techniques, such as high productivity, simple operation, and ability to treat a wide range of substances (Amiri and Sabour, 2014; Roudi *et al.,* 2018).

In order to gather knowledge on the relationships among parameters that require optimisation during the treatment of wastewater, Response Surface Methodology (RSM) is employed (Jasni et al., 2020). The method is beneficial to optimise parameters during the treatment of wastewater such as landfill leachate. The experimental design methods are useful to evaluate the parameters involved in the treatment with a minimum number of experiments. This will reduce the need for reagents and materials for experiments, which prompts an expansion in time and costs. Therefore, the main objective of this study is to reduce the total organic carbon (TOC) from the landfill leachate using RSM. Specifically, the main aim is to evaluate the effect of various parameters such as pH, retention time, H_2O_2: Fe^{2+} ratio, and Fe^{2+} concentration on the removal of TOC during landfill leachate treatment using the Fenton process. The RSM technique is connected to streamline the treatment procedure and decide the parameters playing the most important role.

2. Materials and Methods

2.1 Leachate Characteristics

The landfill leachate was taken from Jeram Landfill, Kuala Selangor, Malaysia. Before the experiments, debris and large particles were removed by using sieve analysis (gradation test with pore size 100 and 150 um), 0.45-μm Whatman Filter Glass paper, and a centrifuge device (Kubota 2420 model) from the leachate. This would reduce the effect on the oxidation reactions using Following are the characteristics of the landfill leachate: pH=7.5, COD=10,516 mg/L. TOC of samples was measured by TOC analyzer (Shimadzu-VCSH, Japan). Electrical conductivity was measured by EC meter (Hach-MM374). The total organic carbon (TOC) was measured with TOC Analyzer (TOC- Shimadzu).

2.2 Fenton experiments

The experiment was conducted at ambient temperature (25 ± 1°C) in a glass reactor using the jar-test. Around 300 ml of leachate was used, and the pH was adjusted to 3, 4.5, 6, 7.5, and 9 (Schoot instruments). The Fenton response was completed by the incorporation of powdered ferrous sulphate ($FeSO_4.7H_2O$) of varying concentrations (500, 750, 1000, 1250, 1500 mg/L) and different amounts of H_2O_2:Fe^{2+} (2, 4, 6, 8 and 10). They were blended for exactly 5 min to obtain a homogeneous suspension. Subsequently, the outlined measure for hydrogen peroxide mixture (H_2O_2, 30% w/w) was included, and the Fenton response was initiated. The samples were mixed in the jar-test at 250 rpm for 80 seconds, and at that point, they were gradually blended for the balanced retention time (5, 18.75, 32.5, 46.25, 60 min) at 50 rpm. The sample was allowed to precipitate for a period of 1 h. A full factorial design effect plot for the variable was prepared using Design-Expert software. To develop an estimate and demonstrate the associations between *k* plan factors, a full factorial approach was important to examine every conceivable mixture. These screening phases included four steps and 20 runs in each set of experiments (i.e. 80 total runs, designed using 2^k, where *k* is the variable number). Responses are generally measured using all combinations of levels of the experimental factors in a full factorial experiment. Moreover, fractional factorial designs are the ones that one or more combinations are excluded from them. In factor screening, fractional factorial designs are more useful since they decrease the run numbers (Dean *et al*., 2017). The experimental design is called 2^k full factorial. The selection of the full factorial design occurred based on the ratio of H_2O_2: Fe^{2+} (2, 5, 6 and 10) as the variable factor. The various Fe^{2+} concentrations were based on previous studies. Several runs were operated at each concentration as follows: Step 1: 500 mg/L Fe^{2+}, Step 2: 4000 mg/L Fe^{2+}, Step 3: 1000 mg/L Fe^{2+} and Step 4: 2000 mg/L Fe^{2+}.

2.3 Statistical model

RSM was used to determine the effective variables upon five responses, namely: retention time (A), Fe^{2+} concentration (B), pH (C), and H_2O_2:Fe^{2+} (D) ratio on TOC (Y_1). After determining which factors affected the responses (after accessing the whole responses from 80 runs, provided the effect plot graphs via R software, (version 3.2.2), the second step was initiated using the actual parameters and excluding the others. The new outline included 44 runs applying central composite design (CCD). The experimental data with the coded variables or the experimental values (A, B, C, D) and the responses (Y_1) were given. Each response was the retention time function (A), the concentration of Fe^{2+} (B), the pH (C), and ratio of H_2O_2: Fe^{2+} (D) in which the important terms of the first order (A, B, C, and D), the second-order (A^2, B^2, C^2, and D^2), and the interaction effects (AB, AC, AD, BC, BD, and CD) were involved. The variable levels were formulated to cover the varied estimated range of 5-60 mins for hydraulic retention time, 500-1500 mg/L for Fe^{2+} concentration, 3-9 for initial pH, and 2-10 for the ratio of H_2O_2:Fe^{2+}. The responses were for overall TOC removal (Y_1). In total, 44 tests were plotted using 2*k*, 20 replicates at the centre point, 16 factorial points, and 8 axial (star) points, where "k" is the variable number. The process had dependent variables with the elimination of efficiency of TOC (Y_1) serving as the output responses. Certain independent parameters are coded based on Equation 1 (Aslani *et al.,* 2016).

$$x_i = \frac{(Xt - X0)}{\Delta X} \tag{1}$$

The coded value that is dimensionless is called X_i, with the independent variable of the *i*th. The focal value of Xi is called X_0, and the value for the step change is called ΔX. The relationships between Y, the responses, and the X variables are attained by applying Equation 2, which is a quadratic model deemed a suitable (second-order) model. Moreover, a linear model was generated to forecast the responses in all investigational fields applied in order to estimate the correlation between TOC removal responses as Y and four independent variables (Aslani *et al.,* 2016).

$$Y = \beta_0 + \sum_{j=1}^{k} \beta_j X_j + \sum_{j=1}^{k} \beta_{jj} X_j^2 + \sum_{i=1}^{k-1} \sum_{j=2}^{k} \beta_{ij} X_i X_j + e \tag{2}$$

The process response-dependent variable is Y (TOC removal), the considered independent variables are X_i (A = Time; B = Fe^{2+} concentration; C = pH and D= H_2O_2:Fe^{2+} ratio), and the corresponding regression coefficients are b_i, b_{ii} and b_{ij}. The findings were analysed statistically through analysis of variance (ANOVA) by using the Minitab software. The appropriate value of this model was stated by the adjusted R^2 and coefficient of determination R^2. The consequence of quadratic and linear terms was assessed using F-tests. The ultimate subset of variables was carefully chosen based on a 95% level of assurance of the P-value. The prognostic capability of the planned model was designated by the prediction of the R^2 coefficient. These processes were carried out by the predicted residual error sum of squares (PRESS). The fitted polynomial equation was presented in contour and surface plots to demonstrate the relationships among the experimental level of each variable and the responses. The statistical model authorised all variables in the designed space. Therefore, a batch of Fenton experiments under favourable situations was achieved, and the findings were associated with the estimated values. The three-dimensional response designs and their outlines were organised using a custom program written in design expert software. It was used to indicate the relations among paired variables.

3. Results and Discussion

3.1 Preliminary study

In total, 80 runs were performed in the laboratory for the preliminary study. Experiments were carried out as per the design obtained by the full factorial method. The design was based on a variable ratio of H_2O_2:Fe^{2+}. Each Fe^{2+} concentration was assessed with a number of runs. As a consequence, this stage was able to determine the current variables and their sufficient range. Due to assessing variables and how each of them affects the response variables and also results of the outline were prepared based on the outcomes and were presented accordingly. Similar results were obtained by Borba *et al*. (2013), which optimised the photo-Fenton process for the removal of pollutants from the tannery industry by means of a complete factorial design. A factorial experimental design was applied to optimise the following parameters: initial pH, Fe^{2+}, and H_2O_2 concentrations.

3.2 Analytical techniques

The experimental data were obtained from 44 observations, which were normalised. The evaluated variables were hydraulic retention time, the concentration of Fe^{2+}, pH of leachate, and H_2O_2:Fe^{2+} ratio, which were respectively named as A, B, C, and D. Central Composite Design (CCD) was employed to identify the interaction between different factors (Shi *et al*., 2019). The independent variables had five levels including $-\alpha$, -1, 0 and +1, $+\alpha$. This range was identified due to pilot studies and previous research. According to Bianco *et al.* (2012) and Lak *et al.* (2012), such codes are largely employed in fitting regression models, and leads to the variables ranged between $-\alpha$ and $+\alpha$. The adopted configurations in each central composite design are summarised in Table 1.

Table 1: Coded variable of central composite experiments design

Run	Time (A)	Fe^{2+} (B)	pH (C)	H_2O_2:Fe^{2+} Ratio (D)	Time (min)	Fe^{2+} (mg/L)	pH	Ratio

1	1	−1	−1	−1	46.25	750	4.5	4
2	1	1	1	1	46.25	1250	7.5	8
3	0	0	0	0	32.5	1000	6	6
4	0	0	0	0	32.5	1000	6	6
5	0	0	0	0	32.5	1000	6	6
6	0	0	0	0	32.5	1000	6	6
7	0	0	0	0	32.5	1000	6	6
8	0	0	0	0	32.5	1000	6	6
9	0	0	0	0	32.5	1000	6	6
10	0	0	0	0	32.5	1000	6	6
11	−1	−1	1	−1	18.75	750	7.5	4
12	−1	−1	−1	1	18.75	750	4.5	8
1	0	0	0	0	32.5	1000	6	6
14	1	1	−1	−1	46.25	1250	4.5	4
15	0	0	0	0	32.5	1000	6	6
16	−1	1	1	1	18.75	1250	7.5	8
17	0	0	0	0	32.5	1000	6	6
18	0	0	0	0	32.5	1000	6	6
19	0	0	0	0	32.5	1000	6	6
20	1	1	1	−1	46.25	1250	7.5	4
21	0	0	0	0	32.5	1000	6	6
22	0	0	0	0	32.5	1000	6	6
23	1	1	−1	1	46.25	1250	4.5	8
24	−1	1	−1	−1	18.75	1250	4.5	4
25	−1	−1	1	1	18.75	750	7.5	8
26	−1	1	1	−1	18.75	1250	7.5	4
27	0	0	0	0	32.5	1000	6	6
28	1	−1	1	1	46.25	750	7.5	8
29	0	0	0	0	32.5	1000	6	6
30	1	−1	−1	1	46.25	750	4.5	8
31	−1	1	−1	1	18.75	1250	4.5	8
32	0	0	0	0	32.5	1000	6	6
33	0	0	0	0	32.5	1000	6	6
34	−1	−1	−1	−1	18.75	750	4.5	4
35	0	0	0	0	32.5	1000	6	6
36	1	−1	1	−1	46.25	750	7.5	4
37	0	−α	0	0	32.5	500	6	6
38	α	0	0	0	60	1000	6	6
39	0	α	0	0	32.5	1500	6	6
40	0	0	−α	0	32.5	1000	3	6
41	−α	0	0	0	5	1000	6	6
42	0	0	α	0	32.5	1000	9	6
43	0	0	0	−α	32.5	1000	6	2
44	0	0	0	α	32.5	1000	6	10

3.3 TOC removal

Table 2 shows the COD results, where the estimated findings (Y_1) are evaluated as a function of the Fe^{2+} concentration (B), the ratio of H_2O_2:Fe^{2+} (D), pH (C), and time for reaction (A). The results were analysed using ANOVA to evaluate the "goodness of fit." According to the results obtained, the maximum and minimum removal of TOC were 81.19% and 6.7%, respectively. As described by Roudi *et al.* (2018), chemical oxygen demand (COD) removal efficiency was 78.9% and 9.3%, respectively. Using the experimental results, an empirical formula was developed relating the response to the variables for TOC removal through the Fenton process. It was observed that the general TOC prediction error by RSM method was 2.8% compared to the experimental data. It was shown that the TOC removal obtained from the empirical formula and experimental data were in good agreement. This demonstrated that the proposed empirical formula was suitable for predicting TOC removal and provided reasonably good agreement. Colombo *et al.* (2019) stated that COD removal obtained only 87% by using the combination of photo-Fenton and biological processes for treating landfill leachate. Another report by Kang and Hwang (2000) indicated that the efficiency of hydrogen peroxide obtained from the removed COD values by oxidation in landfill leachate was observed to be about 45%.

Table 2: Experimental design and results for TOC removal

Run	Time (min.)	Fe^{2+} Con. (mg/L)	pH	H_2O_2: Fe^{2+} Ratio	TOC Remova l (%)	Predicted TOC Removal (%)	Error (%)
1	46.25	750	4.5	4	72.67	70.315	3.239
2	46.25	1250	7.5	8	11.71	5.946	49.216
3	32.5	1000	6	6	26.01	25.009	3.845
4	32.5	1000	6	6	25.58	25.009	2.229
5	32.5	1000	6	6	22.42	25.009	-11.551
6	32.5	1000	6	6	28.31	25.009	11.657
7	32.5	1000	6	6	34.83	25.009	28.194
8	32.5	1000	6	6	32.37	25.009	22.737
9	32.5	1000	6	6	24.12	25.009	-3.689
10	32.5	1000	6	6	22.46	25.009	-11.352
11	18.75	750	7.5	4	17.2	10.885	36.710
12	18.75	750	4.5	8	40.29	40.002	0.714
13	32.5	1000	6	6	25.36	25.009	1.380
14	46.25	1250	4.5	4	52.71	50.158	4.840
15	32.5	1000	6	6	25.95	25.009	3.623
16	18.75	1250	7.5	8	6.7	7.3741	-10.061
17	32.5	1000	6	6	23.47	25.009	-6.560
18	32.5	1000	6	6	25.26	25.009	0.990
19	32.5	1000	6	6	25.93	25.009	3.548
20	46.25	1250	7.5	4	15.78	14.388	8.820
21	32.5	1000	6	6	27.51	25.009	9.088
22	32.5	1000	6	6	24.34	25.009	-2.751

23	46.25	1250	4.5	8	29.24	33.874	-15.848
24	18.75	1250	4.5	4	27.75	27.356	1.419
25	18.75	750	7.5	8	17.95	17.354	3.317
26	18.75	1250	7.5	4	7.19	6.422	10.674
27	32.5	1000	6	6	22.94	25.009	-9.022
28	46.25	750	7.5	8	23.35	22.064	5.507
29	32.5	1000	6	6	24.5	25.009	-2.080
30	46.25	750	4.5	8	61.928	59.548	3.842
31	18.75	1250	4.5	8	18.39	20.464	-11.282
32	32.5	1000	6	6	27.32	25.009	8.456
33	32.5	1000	6	6	24.42	25.009	-2.415
34	18.75	750	4.5	4	38.76	41.376	-6.750
35	32.5	1000	6	6	31.23	25.009	19.917
36	46.25	750	7.5	4	30.21	24.988	17.284
37	32.5	500	6	6	33.63	37.921	-12.760
38	60	1000	6	6	29.3	33.838	-15.489
39	32.5	1500	6	6	9.66	7.783	19.423
40	32.5	1000	3	6	81.19	76.891	5.294
41	5	1000	6	6	8.45	6.326	25.129
42	32.5	1000	9	6	11.76	18.473	-57.087
43	32.5	1000	6	2	22.85	27.420	-20.001
44	32.5	1000	6	10	19.76	17.604	10.907

3.4 Regression models and statistical testing

An empirical relationship was developed to predict TOC removal based on all variables which could be used to make a quick assessment of TOC removal in landfill leachate when using the Fenton process. The empirical formula is as Equation 3.

$$Y(\text{TOC Removal}) = 6.88 \times A - 7.53 \times B - 14.6 \times C - 2.45 \times D - 1.53 \times (AB) - 3.71 \times (AC) - 2.35 \times (AD) + 2.39 \times (BC) - 1.38 \times (BD) + 1.96 \times (CD) - 1.23 \times A^2 - 0.054 \times B + 5.67 \times C^2 - 0.62 \times D^2 + 25.01 \quad (3)$$

Where A, B, C, and D are retention time, Fe^{2+}concentration, pH, and H_2O_2:Fe^{2+} ratio, respectively, and the response is Y (i.e. the predicted TOC removal percentage). The measured and predicted response data were obtained from Equation 3. The model was examined at 95% confidence level of the p-value. The value of the fit polynomial model was declared by the coefficient of determination of Adj R^2 and R^2, and Fisher's F-test verified the statistical significance (Umar *et al.,* 2011). In the case where landfill leachate degradation value was at a very low probability (p-value < 0.0001), the regression was proven to be statistically significant (F-value: 40.45). R^2 values were high, which showed the importance of the model and the second-order polynomial reliability. Moreover, any adequate precision more than 4 (28.724) confirmed that this model could be employed to plan the design space by the CCD. Table 3 displays that the F-value of 40.45 model was significant (*$p < 0.0001$*). Only 0.01% chance existed that such a "Model F-Value" might take place because of the element of noise. Therefore, A (time), B (Fe^{2+} concentration), C (pH), D (H_2O_2: Fe^{2+}ratio), AC (time: pH), AD (time: H_2O_2: Fe^{2+}ratio), BC (Fe^{2+} concentration: pH), and C^2 (pH) were the model terms with significant values. If the value is more than 0.1, it implies that they are not significant. Multiple insignificant model terms mean that the model decrease amends the model. The "Lack of Fit F-value" of 2.61 indicated that the lack of fit was, in fact, significant. There was a 3.73% probability that such "Lack of Fit F-value" might occur because of the element of noise. For statistical analysis, the coefficient of determination (R^2) is an important criterion for model evaluation (Razi and Athappilly, 2005).

Table 3: ANOVA test for the model of response surface quadratic

Source	Sum of Squares	*df*	Mean Square	F Value	P-value Prob> F	Remark
Model	9398.541	14	671.324	40.450	< 0.0001	Significant
A-Time	1135.310	1	1135.310	68.408	< 0.0001	Significant
B-Fe	1362.448	1	1362.448	82.094	< 0.0001	Significant
C-pH	5118.994	1	5118.994	308.447	< 0.0001	Significant
D-Ratio	144.511	1	144.511	8.707	0.0063	Significant
AB	37.662	1	37.662	2.269	0.1432	Insignificant
AC	220.136	1	220.136	13.264	0.0011	Significant
AD	88.228	1	88.228	5.316	0.0287	Significant
BC	91.336	1	91.336	5.503	0.0263	Significant
BD	30.437	1	30.437	1.834	0.1865	Insignificant
CD	61.512	1	61.512	3.706	0.0644	Significant
A^2	49.8006	1	49.8006	3.0007	0.0942	Insignificant
B^2	9.546	1	9.546	0.575	0.4545	Insignificant
C^2	1054.468	1	1054.468	63.537	< 0.0001	Significant
D^2	12.792	1	12.792	0.770	0.3874	Insignificant
Residual	464.688	28	16.596			
Lack of Fit	257.078	9	28.564	2.614	0.0373	Significant
Pure Error	207.609	19	10.926			
Cor Total	9865.555	43				

The R^2 coefficient (0.9529) offers the total response variation anticipated by this model as the ratio of the sum of squares because of regression compared to their total sum (Table 4). If the R^2 is as big as 95.29%, it shows that an agreement with the adjusted R^2 is essential (Joglekar and May, 1987). A big R^2 coefficient confirms a logical modification of the quadratic model to the experimental data. An adjusted R^2 of 92.93% was detected in this study, demonstrating that the regression model could explain the relationship between the independent variables and the response. The coefficients of determination of the model confirmed the statistical significance of the model. The values of R^2 were analysed to be 0.9529 for TOC of landfill leachate. The "Pred R-Squared" equal to 0.7773 was a satisfactory arrangement, with "Adj R-Squared" equal to 0.9293. "Adeq Precision" assessed the sign to the noise ratio. If the ratio is more than 4, it means it is favourable. The ratio of 28.724 implied a sufficient sign and the model could be employed to plan the design space.

Table 4: Regression analysis

Regression parameters	Magnitudes	Regression parameters	Magnitudes
Std. Dev.	4.07	R-Squared	0.952
Mean	27.56	Adj R-Squared	0.929
C.V. %	14.78	Pred R-Squared	0.777
PRESS	2196.57	Adeq Precision	28.724

Table 5 shows the dropped quadratic models in term of coded factors. The results demonstrated that all models were significant at the 95% level of confidence, with lower than 0.05 p-values. The Model F-value equal to 53.60 showed that it was significant. The probability of 0.01% showed that such a large "Model F-Value" might take place because of the element of noise. A "Prob > F" less than 0.05 specifies the significant model terms. Therefore, A, B, C, D, AC, AD, BC, CD, A^2, and C^2 were considered as significant model terms. "Lack of Fit F-value" equal to 2.45 indicated that the lack of fit was, in fact, significant. A probability of 3.71% showed that such a big "Lack of Fit F-value" might take place because of the element of noise. Variance analysis showed that for all responses, a second-order model fit the experimental data. A larger *t* value, together with a smaller *p*-value indicates that the parameter is of higher significance (Khuri *et al.,* 1997).

Table 5: ANOVA test for the response surface reduced quadratic model

Source	Sum of Squares	*df*	Mean Square	F Value	P-value Prob.> F	Remark
Model	9307.52	10	930.75	53.6	< 0.0001	significant
A-Time	1135.31	1	1135.31	65.38	< 0.0001	significant
B-Fe	1362.45	1	1362.45	78.46	< 0.0001	significant
C-pH	5118.99	1	5118.99	294.77	< 0.0001	significant
D-Ratio	144.51	1	144.51	8.32	0.007	significant
AC	220.14	1	220.14	12.68	0.0012	significant
AD	88.23	1	88.23	5.08	0.0312	significant
BC	91.34	1	91.34	5.26	0.0285	significant

CD	61.51	1	61.51	3.54	0.0689	significant
A^2	52.38	1	52.38	3.02	0.0921	significant
C^2	1044.52	1	1044.52	60.15	< 0.0001	significant
Residual	555.71	32	17.37			
Lack of Fit	348.1	13	26.78	2.45	0.0371	significant
Pure Error	207.61	19	10.93			
Cor Total	9865.56	43				

The "Pred R-Squared" equal to 0.8360 is an appropriate settlement with "Adj R-Squared" equal to 0.9261 (Table 6). "Adeq Precision" assessed the ratio of signal to the element of noise. A ratio of more than 4 was considered acceptable, while a ratio equal to 32.425 implied merely sufficient signal. This model may thus be employed to plan the design space. Figure 1 shows the predicted versus actual value plots for parameter removal.

Table 6: Regression analysis

Regression parameters	Magnitudes	Regression parameters	Magnitudes
SD	4.17	R-Squared	0.943
Mean	27.56	Adj R-Squared	0.926
C.V. %	15.12	Pred R-Squared	0.836
PRESS	1617.25	Adeq Precision	32.425

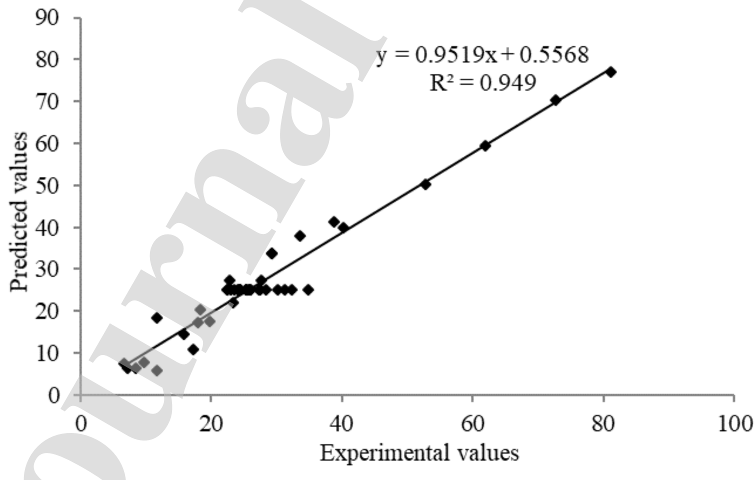


Figure 1: Percentage of the experimental and predicted TOC removal

The regularity of the data was checked through standard deviations of the actual values based on the predicted values. The normal probability plot subsequently revealed that the residuals followed the normal curve distribution. This is the main important assumption for analysing the sufficiency of a statistical model. Residuals are distributed normally if the plot points are on a straight line (Figure 2). The high connection between the experimental and predicted data showed slight differences. In order to approve if a certain model delivers a sufficient approximation of the actual system, the standard probability schemes of the studentised residuals and diagnostics are provided. Accordingly, the data can be measured as normally disseminated in the responses of selected models. As described by Larsen and McCleary (1972), partial residual plot shows the extent and direction of linearity while displaying the deviations from linearities, such as outliers, inhomogeneity of variance, and curvilinear relationships.

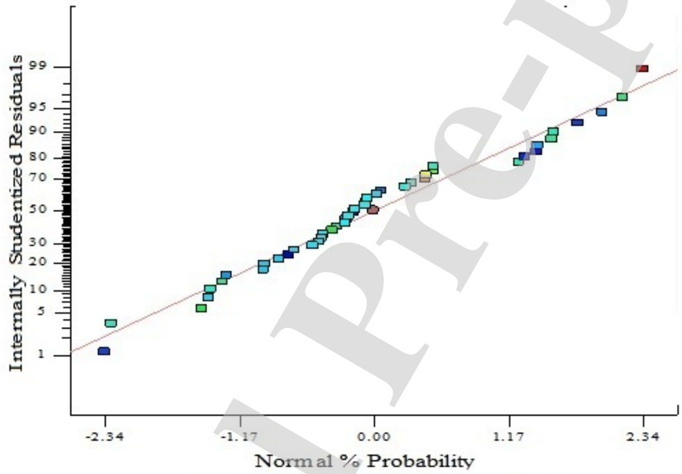


Figure 2: The studentised residuals and normal % probability

Figure 3 is a perturbation plot for TOC removal, which demonstrates the impact of all variables around the centre in the design space. It was evident that the pH (C) at the early stage had a high negative impact on overall entire TOC removal (Y_1). Therefore, if the corresponding response (Y) goes up, the effect factor (i.e. A, B, C and D) level is boosted, which is a positive effect. Meanwhile, if the corresponding response (Y) goes down when the level rises, it is a negative effect. According to the perturbation plot in Figure 3, it is evident that increasing the retention time (A) causes an increase in the TOC removal percentage. Therefore, the response time (A) had a positive effect on the TOC concentration. Furthermore, increasing the Fe^{2+} concentration (B) led to a slight reduction in TOC removal, thus indicating that Fe^{2+} concentration did not have a positive impact on the TOC removal efficiency, even as H_2O_2: Fe^{2+}ratio (D) did not have a significant effect on TOC removal. Besides, Ishak *et al*. (2018) showed that in terms of organic content, 76.9 and 72.6% of COD and TOC removal were achieved respectively using coagulation-flocculation by landfill leachate treatment.

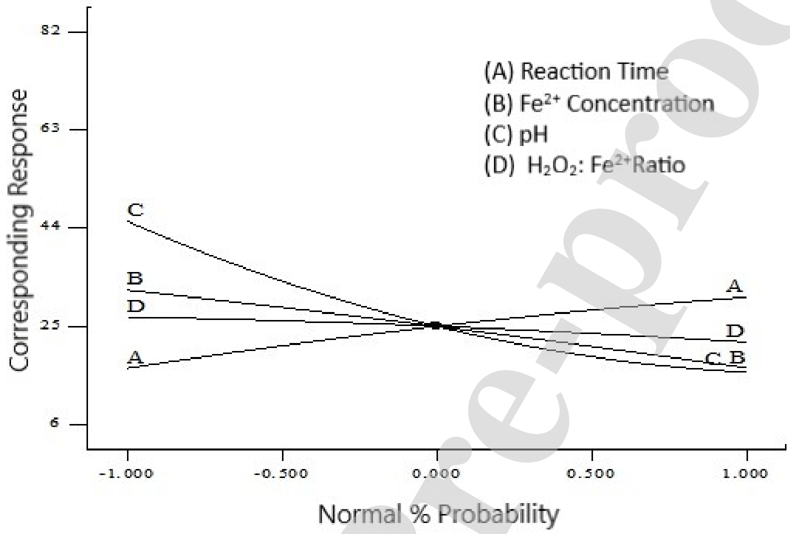


Figure 3: Perturbation plot of TOC removal: (A) Retention Time, (B) Fe^{2+} Concentration, (C) pH and (D) H_2O_2: Fe^{2+}ratio

3.5 Response Surface Model Analysis and Contour Plots

Figures 4 (a) and (b) demonstrate the hydraulic retention time effect in the Fenton process on the removal of TOC. When the retention time increased, the efficiency of TOC removal also went up. Moreover, the effectiveness of Fenton for TOC removal decreased with increasing the concentration of Fe^{2+}. The maximum concentration of Fe^{2+} for the removal of TOC was 1000 mg/L. An additional raise of concentration of Fe^{2+} caused the decline of TOC elimination productivity. Higher Fe^{2+} concentration might cause the OH self-inhibition of radicals by Fe^{2+} ions and reduce the rate of degradation in contaminants. Regarding the Fenton process efficiency for TOC reduction as a role of pH, at low pH, the TOC reduction efficiency was high. Meanwhile, by raising the pH to 3, the removal efficiency decreased (Figures 4(c) and (d)). After this point, TOC removal dropped. The solution pH in the Fenton process played a significant impact. The removal efficiency rapidly raised when the initial pH reached 3 but decreased again at pH > 3. The reason is that the detached form of hydrogen peroxide (HO^{2-}) reacts with hydroxyl radicals more than two orders of magnitude faster than hydrogen peroxide. The percentage of TOC removal increased by increasing the retention time: the percentage of TOC removal dropped within less retention time. Wu *et al.* (2004) reported that by using leachate with pH 8.1, the mass flow of 40 mg O_3 min^{-1}, and reaction time of 30 min had obtained removal rates of 15% of total organic carbon.

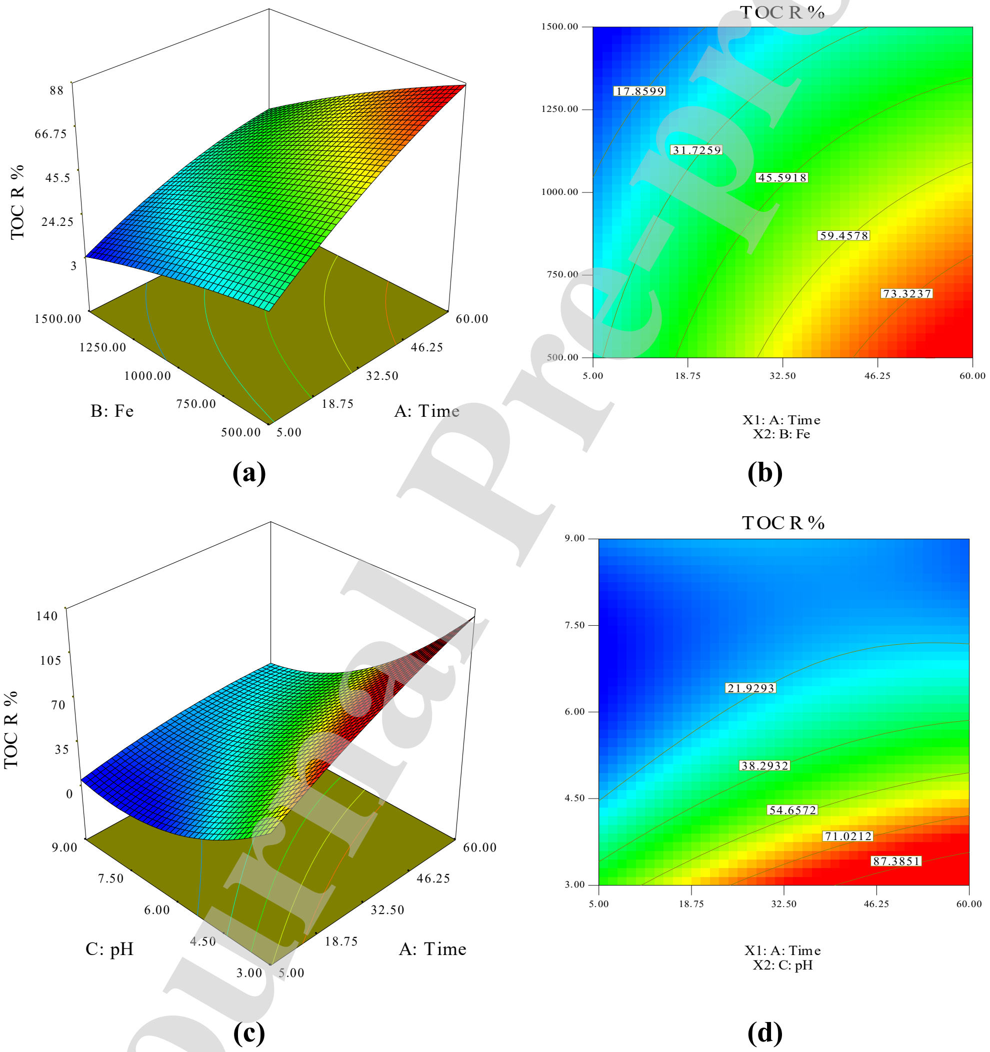
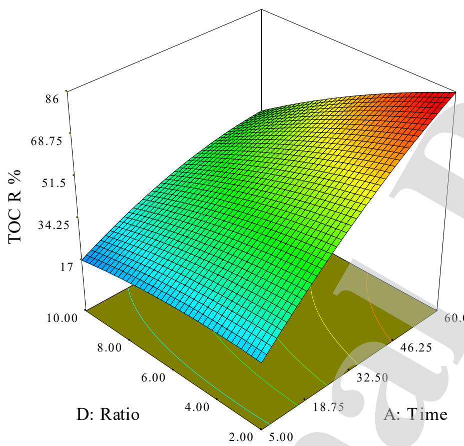


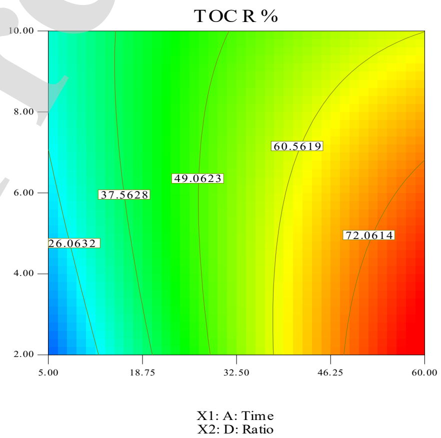
Figure 4: RSM analysis and contour plots, a) and b) Function of time and Fe^{2+} concentration on TOC removal; c) and d) Function of pH and time on TOC removal

The effect of the H_2O_2:Fe^{2+} ratio on TOC removal with different H_2O_2:Fe^{2+} ratios within a range between 2 and 10 are presented in Figures 5(a) and (b). Meanwhile, Figures 5(c) and

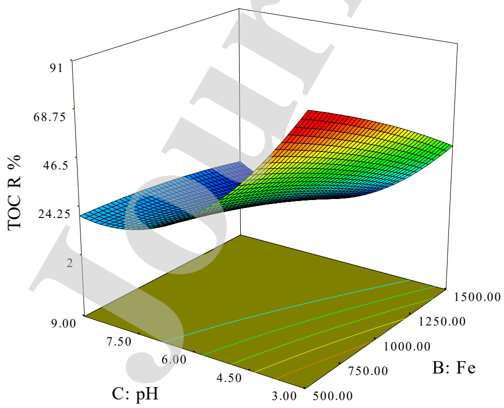
(d) show that TOC removal was raised with the decrease of the ratio of H_2O_2:Fe^{2+}. The findings also displayed that removal efficiencies improved with the rise of the ratio of H_2O_2:Fe^{2+}. With a low H_2O_2:Fe^{2+} ratio, the rate of the reaction tracks the second pseudo-order kinetics to the stoichiometry ratio of 2Fe (II) ≅ H_2O_2. However, with the growth of the ratio of H_2O_2:Fe^{2+}, the reaction kinetics approaches zero-order (Lunar *et al.,* 2000; Atmaca, 2009). At high ratios in H_2O_2:Fe^{2+}, this structure changes, and the reaction become independent of hydrogen peroxide (Zhang *et al.,* 2005). To examine the initial pH, which was an effect on TOC removal efficiency, Fenton treatment was done by changing the initial pH from 3 to 9. As shown in Figures 5(c) and (d), the highest TOC removal efficiency is attained with an initial pH of 3. The optimal pH for Fenton treatment has been reported as pH 3 (Panizza and Cerisola, 2009). At higher pH, more Fe^{3+} results in $Fe(OH)_3$ precipitation that can decline the active sites on the cathode for making H_2O_2 (Wang *et al.,* 2008).



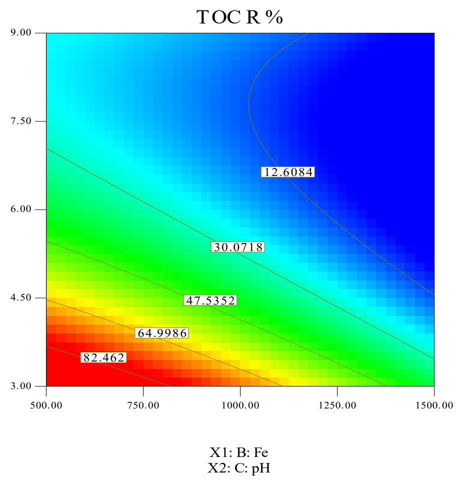
(a)



(b)



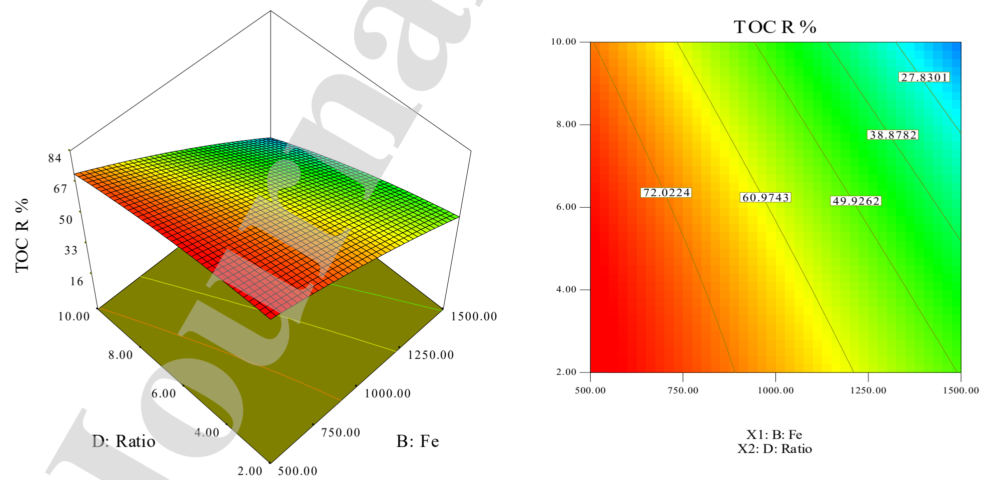
(c)



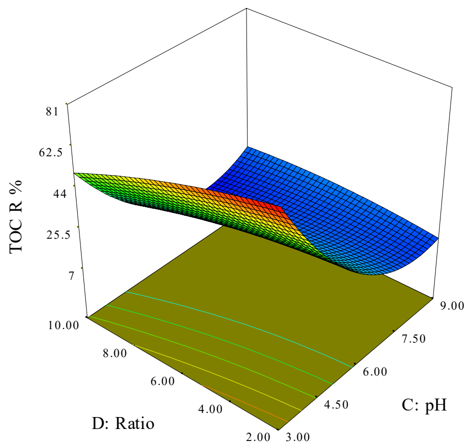
(d)

Figure 5: RSM analysis and contour plots, a) and b) Function of ratio and time on TOC removal; c) and d) Function of pH and concentration of Fe^{2+} on TOC removal

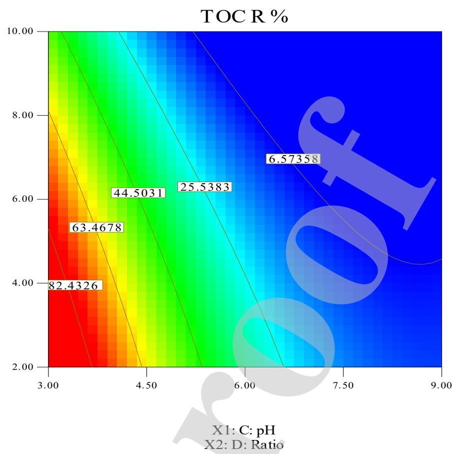
This made a little decline in the TOC removal efficiency at pH 4 and 5. The least TOC removal efficiency occurred at a pH of 2. According to Deng (2007) and Mohajeri *et al.* (2010), the oxidation of organic compounds at the landfill leachate faced the inhibition of Fenton reactions at a very acidic pH. Therefore, by decreasing the pH (acidic range), TOC removal will be improved. With regards to Fe^{2+}, by decreasing the Fe^{2+}concentration, the reduction of TOC percentage will be increased. In order to analyse the optimum ratio of H_2O_2:Fe^{2+}, six different H_2O_2:Fe^{2+} ratios were tested. It showed that TOC removal efficiencies were raised with higher ratios of H_2O_2:Fe^{2+} to the molar ratio of 6 (Figures 6(a) and (b)). Additional growth in H_2O_2:Fe^{2+} ratio over 6 led to less effective enhancements of removal. As pH rises, iron precipitates as $Fe(OH)_3$ and H_2O_2 are decomposed to oxygen. Alternatively, when the pH is lower than 3, inorganic carbon can also be detached, thus limiting its $OH^{\bullet}$ scavenging activity, raising the solubility of iron, and improving the efficiency of the process (Figures 6(c) and (d)). Decreasing the pH increased the removal of TOC. The designed RSM recognised pH of 2.8 as the ideal value of the Fenton treatment in landfill leachate (Kiwi *et al.,* 1993). Decreasing the H_2O_2:Fe^{2+}ratio resulted in an enhanced percentage of TOC removal, but an additional increase in ratio reduced the efficiency of TOC removal.



(a) **(b)**



(c)



(d)

Figure 6: RSM analysis and contour plots, a) and b) Function of $H_2O_2:Fe^{2+}$ ratio and concentration of Fe^{2+} on TOC removal; c) and d) Function of $H_2O_2:Fe^{2+}$ ratio and pH on TOC removal

3.6 Response optimisation and validation of the experimental model

The term optimisation described the highest performance achievable to TOC removal at the desired operation conditions (i.e. Fe^{2+} concentration, $H_2O_2:Fe^{2+}$ ratio, retention time, and pH). To achieve the maximum COD removal (100%), the following optimum values were used: retention time = 37.31 min, $H_2O_2:Fe^{2+}$ ratio = 4.75, pH = 3 and Fe^{2+} concentrations = 634.49 mg/L. To validate the model prediction, the TOC removal efficacy was experimentally obtained in ideal functional situations. From the experimental study, 95.16% TOC removal was obtained in the ideal situations and established the results of the developed model. Residual H_2O_2 may also inhibit subsequent biological treatment (Deng and Englehardt, 2006). The low sludge production in the photo-Fenton process can be further allied to residual iron reuse, as presented by Kattel *et al.* (2016) in reference to the low cost of final residue deposition. Zhang *et al.* (2006) also exhibited COD removal from landfill leachate at only 65% when hydrogen peroxide alone was applied, with the presence of ferrous ion greatly improved the COD removal.

4. Conclusion

The RSM results obtained in this study showed the crucial impacts of four operating variables (i.e. retention time, $H_2O_2:Fe^{2+}$ ratio, Fe^{2+} concentration, and pH) on the TOC removal. The highest TOC decrease of 81.1 % was experimentally observed at optimum values, namely: retention time= 37.31 min, pH= 3, H_2O_2:Fe^{2+} ratio of 4.75, and Fe^{2+} concentration of 634.49 mg/L. The variance analysis showed that the coefficient of determination value was high ($R^2 = 0.949$), with P values showed as <0.0001, thus indicating the model was very significant. The empirical modelling technique developed in this research can be used to model other complex systems, including other kinds of wastewater.

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Conflict of interest

The author declares that there is no conflict of interests regarding the publication of this manuscript. In addition, ethical issues including plagiarism, informed consent, misconduct, data fabrication and/or falsification, double publication and/or submission, and redundancy have been ultimately observed by the authors.

Abbreviations

ANOVA	analyse of variance
AOPs	Advanced oxidation processes
BOD	Biological oxygen demand
CCD	Central composite design
COD	Chemical oxygen demand
Eq	Equation
$Fe(OH)_3$	Ferric hydroxide
Fe^{2+}	Ferrous ion
Fe^{3+}	Ferric ion
$FeSO_4.7H_2O$	Ferrous sulphateheptahydrate
H_2O_2	Hydrogen peroxide
HO^{2-}	Hydrogen peroxide
mg/L	Milligrams per litre
ml	Millilitre
MSW	Municipal solid waste
OH	Hydroxyl radical

pH	Potential of hydrogen
PRESS	Predicted residual error sum of squares
R^2	R-squared
RSM	Response surface methodology
Std. Dev	Standard deviation
TOC	Total organic carbon
X	Variables
Y	Responses

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Research Highlights

- Landfill leachate by Fenton processes could remove of TOC
- Fenton process is an effective pretreatment method for removing organic pollutants
- Removal process for the studied parameters were obtained based on RSM

Credit authorship contribution statement:

Anita Maslahati Roudi: Formal analysis, Data curation, Writing - original draft. Hesam Kamyab: Formal analysis, Data curation, Writing - original draft. Shreeshivadasan Chelliapan: Formal analysis, Data curation, Writing - original draft. Veeramuthu Ashokkumar: Formal analysis, Data curation, Writing - original draft. Ashok Kumar: Formal analysis, Data curation, Writing - original draft. Krishna Kumar Yadav: Formal analysis, Writing - original draft. Neha Gupta: Formal analysis, Writing - original draft.

Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☒The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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