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Optimization of COD, apparent color, and turbidity reductions of landfill leachate by Fenton reagent

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ABSTRACT

The efficiency of Fenton reagent ($Fe^{2+} + H_2O_2 + H^+$) for the leachate generated from Pulau Burung sanitary landfill site, located in Malaysia, was evaluated with the objectives of chemical oxygen demand (COD), apparent color, and turbidity reductions. Response surface methodology was used to study the effect of hydrogen peroxide (as oxidant) and ferrous sulfate (as catalyst) on COD, apparent colour, and turbidity reductions of sanitary landfill leachate. A central composite design in two factors [hydrogen peroxide dosage (mg/L) and ferrous sulfate dosage (g/L)] was employed to build a model as well as to perform optimization of the Fenton process. The optimum results for COD, apparent color, and turbidity reductions were 68, 81, and 86%, respectively, setting [H_2O_2] at 747.8 mg/L and [$FeSO_4$] at 12.8 g/L.

Keywords: Central composite design; Fenton; Landfill leachate; Response surface methodology

1. Introduction

Landfill leachate in municipal solid waste landfills is a contaminated liquid that contains a number of dissolved or suspended materials [1]. Over 200 individual organic compounds have been identified in leachate including cyclic and bicyclic compounds, aromatic hydrocarbons, and many others within a wide concentration range from less than 1 μg/L to several hundred μg/L [2]. Although landfill leachate has been proved to be toxic and recalcitrant, landfilling still remains one of the main methods for municipal and industrial solid waste disposal [3].

Many factors affect the municipal landfill leachate characterization such as landfill age, local climate and weather, disposal methods etc. Based on leachate characterization, several studies have been investigated on leachate treatment including coagulation–flocculation, chemical precipitation, ammonium stripping, membrane filtration, biological treatment methods, etc. Although biological treatments are the most common and cost-effective methods of waste water treatment, it should be considered that high amount of chemical oxygen demand (COD), biochemical oxygen demand (BOD_5), and low biodegradability (in terms of BOD_5/COD ratio) in leachate characterization makes it as a refractory pollutant and classifies it as a resistant wastewater for any treatment (include biological treatment methods). In this case, to achieve better yield in order to enhance the leachate biodegradability, many researchers have worked on various treatment methods [4–17].

One of the most used pretreatment methods is advanced oxidation processes (AOP). Traditionally, AOP involve the generation of the hydroxyl radicals

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(·OH) which has a very high oxidation potential and is able to oxidize almost all organic pollutants [18]. Hydroxyl radicals are highly reactive species which are extremely strong oxidants resulting in several reactions including synergistic action of two oxidants (O_3, H_2O_2), a catalyst and an oxidant (Fe^{++}, H_2O_2), a photo catalyst and an oxidant (TiO_2, H_2O_2) or irradiation plus oxidation (UV, O_3, H_2O_2) [18].

One common feature of such system is the high demand of electrical energy for devices such as ozonizers, UV lamps, etc. and as a consequence, rather high treatment costs [3]. Among the mentioned AOP systems, Fenton process is cheap and effective enough to have preference over similar treatment methods. Fenton reagent is a method in which a mixture of [$FeSO_4$/H_2O_2] under acidic condition produces (·OH) radicals and oxidizes and decomposes organic compounds. Considering other advantages of the Fenton reagent (Fe^{++}, H_2O_2, H^+), it could be mentioned that there is no mass transfer limitation due to its homogeneous catalytic nature, there is no form of energy involved as catalyst, and the process is technologically simple [3].

Fenton's reagent is defined as the catalytic generation of hydroxyl radicals (**.**OH) resulting from the chain reactions between ferrous ion and hydrogen peroxide. Oxidation of organic compounds (RH) by Fenton's reagent can proceed by the following chain reactions [19]:

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + {}^{\cdot}OH \tag{1}$$

$$Fe^{2+} + {}^{\cdot}OH \rightarrow Fe^{3+} + OH^- \tag{2}$$

$$RH + OH \rightarrow H_2O + R^{\cdot} \tag{3}$$

$$R^{\cdot} + Fe^{3+} \rightarrow R^+ + Fe^{2+} \tag{4}$$

Many researchers have worked on Fenton process within a wide range of [Fe^{++}]/[H_2O_2] ratios. In a study on a landfill leachate with 1,500 mg/L of initial COD, 200 mg/L H_2O_2 and 300 mg/L Fe^{++} (Fe^{++}/H_2O_2: 1.5) have been used and 70% COD reduction is reported [20]. On the other hand, in another research on raw leachate with 10,540 mg/L initial COD concentration, 60% COD reduction is reported by using 10,000 mg/L H_2O_2 and 830 mg/L Fe^{++} (Fe^{++}/H_2O_2: 0.083) [3]. Electro-Fenton technique and response surface methodology (RSM) were applied in a research in order to determine the optimal experimental conditions for E-Fenton process and the maximum COD and color removals achieved were 94.04 and 95.83% [21].

The present work was carried out to evaluate the effect of Fenton process on landfill leachate generated from Pulau Burung landfill, located in Penang, Malaysia. The objective of this study was to optimize the oxidant [H_2O_2] and catalyst [$FeSO_4$] in order to achieve highest COD reduction, apparent color removal, and turbidity reduction by implementing RSM through central composite design (CCD).

2. Materials and methods

Samples were taken from Pulau Burong landfill site, located in Penang. Pulau Burung landfill site is classified as a semi-aerobic landfill site in which the generated and collected leachate is aerated by the air pumps installed at the bottom of the collecting ponds and hence, generates mature and stabilized leachate type. Leachate sample was placed in high density polyethylene containers (volume of 40 L), transported to the laboratory, and was immediately stored in the refrigerator at 4°C as stated in the American Public Health Association (APHA, 1992) preservation techniques to minimize any changes in properties and characteristics of the samples [22].

Analytical grade chemical reagents were used in the study. Ferrous sulfate (FeSO4, 7 H_2O) and sodium hydroxide (NaOH) supplied by Systerm, sulfuric acid (H_2SO_4, 97%), and hydrogen peroxide (H_2O_2, 30%), disodiumhydrogen phosphate heptahydrate ($Na_2HPO_4{\cdot}7H_2O$, >98), di-kaliumhydrogen phosphate, (K_2HPO_4, reagent grade) supplied by Merck, magnesium sulfate heptahydrate ($Na_2HPO_4{\cdot}7H_2O$, 99%) supplied by Bio-Lab, calcium chloride anhydrous ($CaCl_2$, ~97%) supplied by Sigma, Iron (III) chloride anhydrous ($FeCl_3{\cdot}6H_2O$ ~97%) supplied by Fluka, potassium dichromate ($K_2Cr_2O_7$, MW:294.19 g/mol) by R&M and silver sulfate (Ag_2SO_4 MW: 311.79) supplied by QReC.

The apparent color and COD were measured by using HACH DR/120 portable data-logging spectrophotometer (reported in units Pt/Co and mg/L, respectively). The apparent color unit calibrated based on APHA recommended standard of one colour unit being equal to 1 mg/L platinum as chloroplatinate ion. COD was measured using dichromate reactor design colorimetric, United State Environmental Protection Agency (USEPA) approved. BOD_5 was measured according to the standard method for the examination of water and wastewater using YSI 5100 dissolved oxygen meter. pH was measured by using HACH Senion pH meter. Turbidity was measured by using HACH 2100 turbidity meter (reported in N.T.U).

Leachate characterization was carried out instantly within two days for pH, COD, apparent colour, turbidity, and BOD_5. All analysis procedures were performed according to standard methods and HACH method (modification of APHA standard method as approved by USEPA. Table 1 shows the characterization of Pulau Burung landfill leachate and based on leachate characterization, it can be considered a mature leachate with a BOD_5/COD ratio of 0.063. Hydrogen peroxide (H_2O_2, 30%) supplied by Merck OHG was used as an oxidant and $FeSO_4$ supplied by SYSTERM chemicals was used as a catalyst. Primarily, based on the literature review and according to the studies on the same samples [13 and 21], initial pH was fixed at 3.0, then predetermined dosage of $FeSO_4$ was added to samples and were stirred for three minutes in rapid mixing phase (200 rpm), then related H_2O_2 dosage were added to the samples and stirred for another three minutes in the same rapid mixing stage. Thereafter, the samples were stirred for one hour in slow mixing (50 rpm) and finally, one hour settling time was set to allow the generated sludge and particles to settle down.

Based on jar-test procedure, first, the various dosages of $FeSO_4$ (3, 6, 9, 12, 15, and 18 g/L) were examined at a fixed arbitrary H_2O_2 dosage (500 mg/L). After selecting the best $FeSO_4$ dosage – the dosage that gives the highest COD reduction– various dosages of H_2O_2 (200, 400, 600, 800, 1,000, 1,200, 1,400, 1,600, 1,800, 2,000, 2,200, and 2,400 mg/L) were examined. Based on results, the highest COD reduction percentage (60%) in related hydrogen peroxide (800 mg/L) and ferrous sulfate dosages (15 g/L) were selected for optimization.

3. Experimental design

RSM is a collection of mathematical and statistical techniques for empirical model building. The objective of RSM is to optimize a response (output variables) which is influenced by several independent variables (input variables). This method is used for a careful design of experiments, which is a series of tests called runs in which changes are made in the input variables in order to identify the reasons for changes in the output response. The most extensive applications of RSM are in industrial fields where several input variables potentially influence some performance measure or quality characteristic of the product or process. This performance measure or quality characteristic is called the response [23]. RSM also specifies the relationships between one or more measured responses and the essential controllable input factors [24]. When all independent variables are measurable, controllable, and continuous in the experiments, with negligible error, the response surface can be expressed by

$$y = f(x_1, x_2, \ldots, x_k) \tag{5}$$

where k is the number of independent variables. To optimize the response "y", it is necessary to find an appropriate approximation for the true functional relationship between the independent variables and the response surface. Usually, a second-order polynomial Eq. (6) is used in RSM.

$$y = \beta_0 + \sum_{i=1}^{2} \beta_i x_i + \sum_{i=1}^{2} \beta_{ii} x_i^2 + \sum \sum_{i<j} \beta_{ij} x_i x_j \tag{6}$$

where $\beta_0, \beta_i, \beta_{ii}$, and β_{ij} are regression coefficients, and x_i and x_j are the coded variables.

A CCD with two independent variables was used for optimization and 13 runs were required to cover all possible combinations of factors levels. Data were collected from 2^2 factorial augmented by five (5) center points and four extra points (axial points or star points) as shown in Table 2. Experiments were run in random order to minimize the effects of unexpected variability in the observed responses.

Table 1
Landfill leachate characterization

Parameters	Average
pH	8.3
COD (mg/L)	3,511
BOD_5 (mg/L)	206
BOD_5/COD	0.06
Apparent colour (Pt.Co.)	6,019
Turbidity (N.T.U)	113

4. Result and discussion

The experimental range for each independent variable was based on the preliminary trials, H_2O_2 dosage levels were selected in the range of (200–800) mg/L and $FeSO_4$ dosage levels were (6–15) g/L.

The results of 13-run CCD in the two variables, H_2O_2 and $FeSO_4$ dosage and three responses, COD, apparent color, and turbidity reductions are given in Table 2. The percentage of COD, apparent color, and turbidity reductions were studied in their specified levels, H_2O_2 dosage (200–800) mg/L and $FeSO_4$ dosage (6–15) g/L.

RSM was used to evaluate the effect of H_2O_2 and $FeSO_4$ dosage on the COD, apparent color, and turbidity reductions, then a model was built in order to describe the behavior of each response in order to

Table 2
CCD in natural and coded variables with the experimental data

Natural variable		Coded variable		Responses (%)			Predicted value (%)		
ξ_1	ξ_2	H_2O_2	$FeSO_4$	COD	Apparent colour	Turbidity	COD	Apparent colour	Turbidity
200	6	−1	−1	45.00	53.80	−41.70	36.36	47.84	−78.98
800	6	1	−1	44.80	59.70	−15.40	46.04	63.01	−12.59
200	15	−1	1	56.60	70.20	47.10	55.16	66.00	19.94
800	15	1	1	62.70	75.90	81.60	64.84	81.16	86.33
75.74	10.5	−1.41	0	35.40	47.60	−79.40	41.83	62.03	−39.72
924.26	10.5	1.41	0	58.60	82.30	65.40	55.52	83.48	54.17
500	4.14	0	−1.41	34.70	41.70	−88.30	39.24	43.41	−69.82
500	16.86	0	1.41	67.00	70.00	60.10	65.81	69.09	70.07
500	10.5	0	0	68.00	77.40	60.50	66.38	72.76	56.48
500	10.5	0	0	65.80	75.00	52.60	66.38	72.76	56.48
500	10.5	0	0	66.10	74.40	53.00	66.38	72.76	56.48
500	10.5	0	0	66.10	76.60	59.60	66.38	72.76	56.48
500	10.5	0	0	65.90	75.20	56.70	66.38	72.76	56.48

optimize the process by finding the best setting of H_2O_2 and $FeSO_4$ dosage that maximize COD, apparent color, and turbidity reductions. Second-order model for COD reduction, apparent color removal and turbidity reduction in terms of coded variable are given by Eqs. (7)–(9), respectively:

$$y_1 = +66.38 + 4.84x_1 + 9.40x_2 - 8.85x_1^2 - 6.93x_2^2 \tag{7}$$

$$y_2 = +72.76 + 7.58x_1 + 9.08x_2 - 8.25x_2^2 \tag{8}$$

$$y_3 = +56.48 + 33.20x_1 + 49.46x_2 - 24.63x_1^2 - 28.18x_2^2 \tag{9}$$

where y_1, $y_{2,}$ and y_3 represent the percentage of COD, apparent color, and turbidity reduction, respectively, and x_1 and x_2 represent $[H_2O_2]$ and $[FeSO_4]$, respectively.

The second-order regression models obtained for COD, apparent color, and turbidity reductions are satisfied since the values of the coefficient of determination (R^2) are high and close to 1. The values of R^2 for COD, apparent color and turbidity reductions models are 0.913, 0.818, and 0.895, respectively. When the value of R^2 for COD is 0.913, it means that 0.913 of total variation is explained by the model and 0.087 of total variation is unexplained. Similarly, when the value of R^2 for apparent color is 0.818 of total variation is explained by the model, 0.117 of total variation is unexplained. This indicates that 0.82–0.91 of the total variation is explained by the model and only (0.09–0.18) of the total variation is unexplained.

The 3-D response surface for COD, apparent color, and turbidity reductions are given in Figs. 1–3 showing the effect of $FeSO_4$ and H_2O_2 dosage on COD, apparent color, and turbidity reductions, respectively. A positive sign for the regression coefficient in the fitted model indicates the ability of the factor to increase the response while the negative sign indicates the ability of a factor to decrease the response. It can be seen that all responses exhibited a clear peak suggesting that the optimum condition for maximum COD, apparent color, and turbidity reductions are well inside the design boundary.

Figs. 1–3 show a decrease in responses reduction efficiency. This could be due to the fact that during

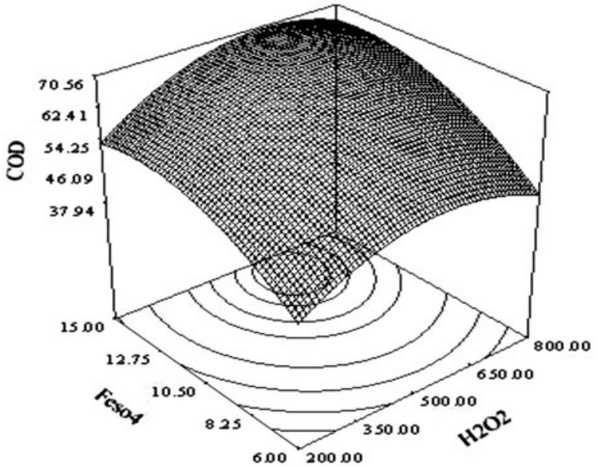

Fig. 1. 3D response surface for COD reduction as a function of H_2O_2 and $FeSO_{4.}$

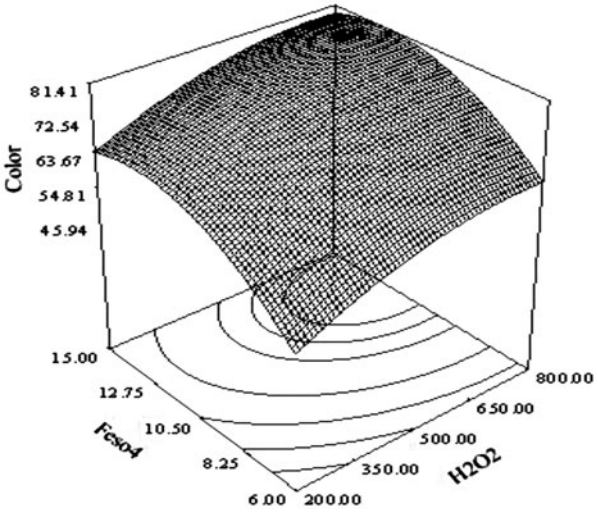

Fig. 2. 3D response surface for apparent color removal as a function of H_2O_2 and $FeSO_4$.

oxidation process, these organic compounds are less stabilized and more ready for degradation through oxidation. These compounds are oxidized first and the remaining compounds are refractory complexes and resistant to oxidation. The results of the analysis of variance (ANOVA) for the quadratic models summarize the analysis of each response and show significant model terms. Table 3 shows the ANOVA for COD, apparent color, and turbidity reductions.

The analysis of variance revealed that a second-order model adequately fitted the experimental data for all responses. For COD, apparent color and turbidity reductions, it could be seen that linear and quadratic effect of $[H_2O_2]$ (x_1) and $[FeSO_4]$ (x_2) are significant, except the quadratic effect of $[H_2O_2]$ for apparent color (all related *P*-values shown in Table 3 are less than 0.05). The interaction between $[H_2O_2]$ and $[FeSO_4]$ (×) shows insignificant effect for all responses (related *P*-values > 0.05).

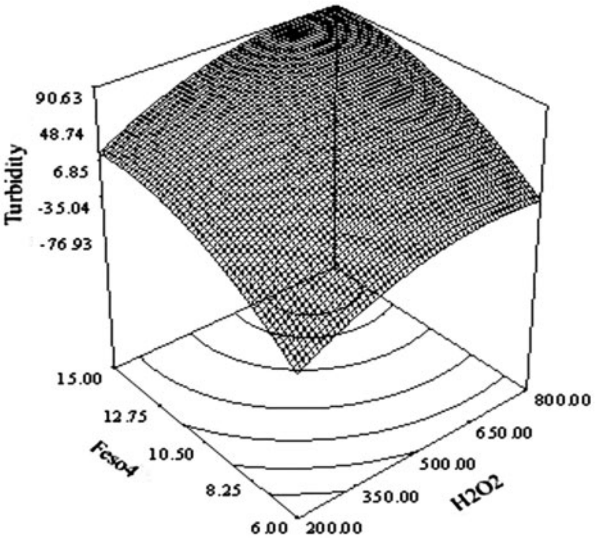

Fig. 3. 3D response surface for turbidity reduction as a function of H_2O_2 and $FeSO_4$.

Results reveal that higher concentration of $[FeSO_4]$ yields higher efficiency on responses reductions until maximum reductions called optimum conditions are achieved and then a decrease in efficiency is observed. Same behavior for $[H_2O_2]$ was detected meaning that as far as $[H_2O_2]$ concentration was increased, COD, apparent color and turbidity reductions increased as well and reached up to maximum reduction, optimum condition, and then beyond that, responses reductions started to slow down (decrease). However, when $[H_2O_2]/[Fe^{2+}] \geqslant 2.0$, sludge settling was hindered by the oxygen bubbling produced from the decomposition of excess hydrogen peroxide which leads to the side reactions (10) and (11) [19,25, 26].

One of the deterrent factors in Fenton process, which causes the observed descent in process efficiency is due to hydroxyl scavenging role of iron ion against the produced OH (by H_2O_2) during reaction 10, which describes that by increasing the $FeSO_4$ dosage, the available additional iron ions try to oxidize themselves through the reaction below

$$Fe^{2+} + {}^{\cdot}OH \rightarrow Fe^{3+} + OH^{-} \qquad (10)$$

Incidentally, another hydroxyl radicals scavenging is done by hydrogen peroxide itself as shown in reaction 11.

$$H_2O_2 + {}^{\cdot}OH \rightarrow H_2O + {}^{\cdot}O_2H \quad [3] \qquad (11)$$

5. Optimization of the experiment

Based on the above discussion, it is better to run an optimization study to find out the optimal operating conditions for H_2O_2 concentration and $FeSO_4$. In fact, once the model was developed and checked for adequacy, the optimization criteria was set to find out the optimum operating conditions. Maximum reduction for all responses was achieved at 12.8 g/L of $FeSO_4$ and 747.8 mg/L of H_2O_2 concentration. To validate the optimum combination of the process variables, confirmatory experiments were carried out. The selected combinations of the two variables resulted in 68% COD reduction, 81% apparent color removal, and 86% turbidity reduction.

Table 3
ANOVA for COD, apparent color and turbidity reductions

Source	Sum of squares	DF	Mean Square	*F*-Value	*P*-Value
COD					
Model	1,674.79	4	418.70	21.07	0.0003
H_2O_2	187.31	1	187.31	9.43	0.0153
$FeSO_4$	706.49	1	706.49	35.56	0.0003
$H_2O_2 \times H_2O_2$	545.16	1	545.16	27.44	0.0008
$FeSO_4 \times FeSO_4$	333.85	1	333.85	16.80	0.0034
Residual	158.95	8	19.87		
Total	1,833.75	12			
Apparent color					
Model	1601.58	3	533.86	13.52	0.0011
H_2O_2	460.15	1	460.15	11.66	0.0077
FeSO4	659.25	1	659.25	16.70	0.0027
$FeSO_4 \times FeSO_4$	482.18	1	482.18	12.21	0.0068
Residual	355.30	9	39.48		
Total	1,956.88	12			
Turbidity					
Model	37,015.75	4	9,253.94	17.01	0.0006
H_2O_2	8,816.47	1	8,816.47	16.21	0.0038
$FeSO_4$	19,569.27	1	19,569.27	35.97	0.0003
$H_2O_2 \times H_2O_2$	4,219.23	1	4,219.23	7.76	0.0237
$FeSO_4 \times FeSO_4$	5,523.28	1	5,523.28	10.15	0.0129
Residual	4,352.35	8	544.04		
Total	41,368.10	12			

6. Conclusion

The effects of $[H_2O_2]$ and $[FeSO_4]$ on the Fenton process using RSM, were evaluated in a batch reactor. It was proved that a second-order model adequately fitted the experimental data for all responses. For COD, apparent color, and turbidity reductions, it could be seen that linear and quadratic effect of $[H_2O_2]$ and $[FeSO_4]$ are significant, except the quadratic effect of $[H_2O_2]$ for apparent color. It could be concluded that optimal condition for Fenton process is at 747.8 mg/L of $[H_2O_2]$ and 12.8 g/L of [FeSO4] for COD, apparent color, and turbidity reductions. The combination of two factors (H_2O_2 and $FeSO_4$ dosage) resulted in 68, 81, and 86% reductions for COD, apparent color, and turbidity, respectively. The responses reduction efficiency decreases beyond the optimal conditions.

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