Original Research

Optimizing Operating Conditions for Advanced Treatment of Landfill Leachate Using the Coagulation-Fenton Oxidation Method

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Abstract

High COD concentrations in effluent, high membrane loading, and frequent congestion on the membrane are three common shortcomings of treating landfill leachate using the biodegradation technique. Accordingly, advanced treatment of the bio-chemical effluent of landfill leachate by Fenton oxidation combined with coagulation technique were performed in this study. The experiment was designed with the response surface method (RSM) considering three factors, i.e., the dosages of polymeric ferric sulfate (PFS), $n(H_2O_2)/n(Fe^{2+})$, and H_2O_2 . Experimental results show that the fitting curve has high significance and sensitivity, and the prediction error of the COD removal rate is smaller than ±5%. The results also show that H_2O_2 has the greatest influence on COD removal rate, while PFS has the smallest. The optimal operating conditions for landfill leachate with a COD concentration of 2,000 mg/l are: the dosages of PFS (the concentration is 0.05g/ml), and H_2O_2 (30%) and $n(H_2O_2)/n(Fe^{2+})$ are 67 ml, 17 ml, and 6 respectively. The optimized COD removal rate can reach as high as 92.11%.

Keywords: fenton oxidation, coagulation, response surface analysis, landfill leachate

Introduction

As municipal solid waste increases annually, the leachate would have a significant impact on the steady running of the landfill [1-5]. The biological method is the main processing technique in most landfills and the membrane module is an advanced treatment technique. However, with the passage of time the accumulation of toxic and hazardous substances would lead to a decrease of biodegradability and processing capacity, increase membrane loading, and reduce treatment efficiency [67]. Physicochemical methods, including coagulation and Fenton-oxidation, are now applied widely in advanced biological processing or membrane pretreatment [8-10]. The organics and heavy metals in the leachate could be effectively reduced with the coagulation method, and the biodegradability of the refractory organics could be improved with the Fenton-oxidation method [11-12]. The stability of leachate quality in the effluent could be guaranteed with their combination. However, in the current combination of coagulation-Fenton oxidation, in which the two reactions are regarded as two independent processes [13], the dosage as well as the cost is high, leading to low economic benefit [14-16]. This study uses

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the coagulation-Fenton oxidation method between the biological treatment and the membrane treatment, which could reduce the dosage of reagents, optimize the effluent, and reduce the membrane load. Treating the coagulation-Fenton oxidation as one process and considering the effects of PFS on the Fenton process, the Box-Behnken experiments design (BBD) [17] test, and the response surface method (RSM) is used to optimize the operating conditions.

Materials and Methods

Characteristics of the Leachate

The leachate was collected from effluent of the biochemically treated leachate from a landfill in Nanning, Guangxi, and its characteristics are shown in Table 1. The origins of reagents in this study are shown in Table 2.

Testing Methods

Employing the software package Design-Expert (v. 8.0.5b), a three-factor (PFS(ml), $n(H_2O_2)/n(Fe^{2+})$, $H_2O_2(ml)$) three-level Box-Behnken experiments design (BBD) was performed with COD removal rate as the target response value. The response of the interaction of the factors to the COD removal rate is determined by fitting

Table 1. Characteristics of the leachate before processing.

Item	Value				
pH	8.00-9.00				
COD (mg/l)	2300-2500				
NH ₃ -N (mg/l)	1,300-1,600				
SO ₄ ²⁻ (mg/l)	90.05-92.10				
Cl ⁻ (mg/l)	1,400-1,800				
Alkalinity (mg/l)	4,015-4,428				
Chromaticity	512				

Tab	le	2.	Experiment	reagents.
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the response surface model to predict the best operating conditions for the landfill leachate treated with the Fenton oxidation-coagulation method.

The three-level values are -1 (lower level), 0 (middle level), and 1 (upper level). Single factor experimental results show that the optimized dosage of PFS(ml), $n(H_2O_2)/n(Fe^{2+})$, and $H_2O_2(ml)$ are 10-20 ml, 1-7, and 0.5-5 ml, respectively (Table 3).

The samples were divided into three groups according to the sequence numbers (1-6, 7-12, 13-17) and one group was tested at one time. For each test, 2.5 L landfill leachate was used and the pH value was adjusted to 5-6 with H₂SO₄ and NaOH solutions. After 450 ml leachate was added into six 500 ml beakers, respectively, PFS was added into each beaker and the dosage was set according to Table 4. Then the beakers were stirred slowly by a synchronous electrical stirrer for 20 min and standing for 1 h. Then 300 ml supernatant in each beaker was taken and added into another 500 ml beaker. The pH value was adjusted to 3.0 with H₂SO₄, FeSO₄•7H₂O, and H₂O₂, whose amounts were determined according to Table 4, added into the beakers, and then stirred again at medium speed for 5 h. When the reaction was over, the pH values of the samples in the six beakers were adjusted to 7.0. After standing, the supernatant was taken out and tested for COD with dichromate titration [18].

Results and Discussion

Regression Model Analysis

Response surface analysis was performed with Design-Expert and the experimental data were fitted with standard polynomials. The results are shown in Fig. 1, and the fitted polynomial is as follows:

$$Y(\%) = 67.242 + 0.077A + 3.374B + 3.524C-0.017AB + 0.029AC + 0.500BC + (5.600E-003)A2 - 0.457B2 - 0.649C2$$

(1)

As seen from Figs 1a) and b), the relative errors and residuals are both very small. Also, the residual data are

Reagent	Grade	Origin
30%H ₂ O ₂	GR	Chinese Medicine Group Chemical Reagent Co., Ltd
FeSO ₄ ·7H ₂ O	AP	Guangdong Engineering Technology Research Center For Chemical Reagent
PFS	AP	Tianjin Damao Chemical Reagent Company
Potassium dichromate	AP	Chengdu Kelong Chemical Reagent Company
$(\mathrm{NH}_4)_2\mathrm{Fe}(\mathrm{SO}_4)_2\cdot\mathrm{6H}_2\mathrm{O}$	AP	Guangdong Guanghua Science and Technology Co., LTD
H_2SO_4	AP	Guangdong Shantou Xilong Chemical Engineering Co., Ltd
$[SO_4]^{2-}$	AP	Shanghai Shenbo Chemical Engineering Co., Ltd
1,10-Phenanthroline monohydrate	AP	Tianjin Damao Chemical Reagent Company

Table 3. Independent variables and levels for the BBD design.

Fastar	Level value					
Factor	-1	0	1			
PFS X ₁ (ml)	10 15 20					
$n(H_2O_2)/n(Fe^{2+}) X_2$	1	4	7			
H ₂ O ₂ X ₃ (ml)	0.5	2.75	5			

distributed symmetrically along a line and they are dense in the middle while sparse at either end, indicating a normal distribution characteristic. The p value is 0.0682, which is not significant (>0.05), indicating that the fitted polynomial can be used in the following analysis instead of the real experimental data.

Table 5 shows the analysis of variance (ANOVA) results, with F and p values being parameters that indicate the significance level of the model. If p value is greater than 0.1, the influence of the factor on the response value is not significant, and vice versa [19-20]. We can see in Table 5 that for COD removal rate the linear terms of the independent variables B (p = 0.04 < 0.04), C (p = 0.0011 < 0.0011), the quadratic terms BC (p = 0.0498 < 0.0498), and B2 (p = 0.0212 < 0.05) are significant items. Thus we can conclude that in the advanced treatment of landfill leachate with Fenton oxidation-coagulation method, the

Table 4. Three-factor and three-level BBD design and experimental results

	Е	xperimental de	COD removal			
Number	PFS (ml)	$n(H_2O_2)/n(Fe^{2+})$	H_2O_2	rate (%)		
1	(111)		(111)	04.2		
	10	/	2.75	84.3		
2	20	7	2.75	87.6		
3	10	4	0.50	75.4		
4	20	4	5.00	91.6		
5	15	7	0.50	73.7		
6	10	1	2.75	76.6		
7	15	4	2.75	87.9		
8	15	4	2.75	83.9		
9	15	4	2.75	86.5		
10	20 4 0		0.50	76.1		
11	1 20 1 2.75		2.75	80.9		
12	15	4	2.75	87.8		
13	10	4	5.00	89.6		
14	15	4	2.75	85.5		
15	15	1	0.50	77.5		
16	15	7	5.00	87.1		
17	15	1	5.00	77.4		

factors influencing the COD removal are ordered as, H_2O_2 (ml) > n (H_2O_2)/n (Fe²⁺) > PFS (ml). Increasing the doses of H_2O_2 and increasing n(H_2O_2)/n (Fe²⁺) are more effective for the degradation of organic matter, while increasing PFS is not. The reason is that with the increase of n (H_2O_2)/n (Fe²⁺), more ·OH will be produced, which can increase the reaction rate, enhance the capacity of oxidation, degrade the organic matter efficiently, and greatly improve the removal rate of COD. While for PFS, which is a composite coagulation made up of elemental iron or iron oxide and other iron compounds, the PFS remaining in the water after coagulation has little effect on Fe²⁺ content.

Unlike p value, the greater the F value, the more significant the effects of the factor on the response value. In this ANOVA analysis, F = 6.36 and p < 0.0117, indicating that the model has a high significance in prediction of COD removal rate. The adjusted R² coefficient (adj) is 0.8910 (> 0.80) and the coefficient of variation (CV) is 3.44%, indicating that the model has a variation of 10.9%, which could be explained by the model. The signal-to-noise ratio is 8.003 (> 4), showing that the experimental results can be well represented by the model. The above indicators further indicate that the fitting is relatively good and can be used to analyze and predict treating landfill leachate with the Fenton oxidation-coagulation method.

Response Surface Analysis

Response surfaces graphs and contour plots between various factors and response values can be obtained according to the respective regression equations. The response surface graph is a three-dimensional curved surface of each factor, and the steeper the surface, the greater the influences of the factors on the response values. The contour plot reflects the interaction intensity of the various factors. The oval indicates a significant interaction between two factors while the circle indicates that the interaction intensity is not significant [21].

Effects of $n(H_2O_2)/n(Fe^{2+})$ and PFS on COD Removal Rate

The effects of $n(H_2O_2)/n(Fe^{2+})$ and PFS as well as the interaction on COD removal rate are shown in Fig. 2, where we can see that when $n(H_2O_2)/n(Fe^{2+})$ is between 1.00 and 5.00, the COD removal rate increases significantly with the increase of $n(H_2O_2)/n(Fe^{2+})$. When $n(H_2O_2)/n(Fe^{2+})$ is greater than 5.00, the COD removal rate decreases when $n(H_2O_2)/n(Fe^{2+})$ increases. This is because in the process of the Fenton reaction, the concentrations of H_2O_2 and Fe^{2+} will directly influence the oxidation of organic compounds. When Fe²⁺ is constant and in excess, the oxidation of organic matter will increase since the concentration of the hydroxyl radical participated in the reaction increases with the increase of $n(H_2O_2)/n(Fe^{2+})$. However, when the concentration of H₂O₂ is far higher than that of Fe²⁺, the hydroxyl radicals generated with the Fenton method would react with H₂O₂, produce



Fig. 1. The experimental results and the fitting line a), the distribution of the residuals for Yb).

 HO_2 , and trigger a series of reactions. In addition, Fe^{3+} will react with HO_2 and produce O_2H^+ , leading to a high consumption of H_2O_2 and a decrease of the COD removal rate [22]. The maximum removal rate of COD is obtained when $n(H_2O_2)/n(Fe^{2+})$ equals 5.00.

When the dosage of PFS increases from 10 ml to 20 ml gradually, the COD removal rate increases slowly but not significantly. The reason is that PFS is added in the coagulation stage, in which only a small amount of COD will be removed and part of the organic matters will be adsorbed and coagulated, leading to a small increase in the COD removal rate [23]. It can also be seen from Fig. 2 that

the influences of $n(H_2O_2)/n(Fe^{2+})$ on COD removal rate are more significant than PFS. The effects of the interaction between $n(H_2O_2)/n(Fe^{2+})$ and PFS on COD removal rate is insignificant. Therefore, in the laboratory it is an effective way to increase COD removal rate by increasing $n(H_2O_2)/n(Fe^{2+})$.

Effects of H₂O₂ and PFS on COD Removal Rate

The effects of H_2O_2 and PFS on COD removal rate are shown in Fig. 3, which shows that with the increase of

Source	Sum of square (SS)	Degree of freedom	Mean square	F value	p value
Model	465.33	9	51.70	6.36	0.0117
A-PFS	13.26	1	13.26	1.63	0.2422
$B-n(H_2O_2)/n(Fe^{2+})$	51.51	1	51.51	6.34	0.0400
C-H ₂ O ₂	231.12	1	231.12	28.43	0.0011
AB	0.25	1	0.25	0.031	0.8658
AC	0.42	1	0.42	0.052	0.8262
BC 45.56		1	45.56	5.61	0.0498
A^2	0.083	1	0.083	0.010	0.9226
B ²	71.12	1	71.12	8.75	0.0212
C ²	45.44	1	45.44	5.59	0.0500
Residual error	56.90	7	8.13		
Lack of fit	45.65	3	15.22	5.41	0.0682
Pure error	11.25	4	2.81		
Total error	522.23	16			

Table 5. ANOVA for COD removal rates.



Fig. 2. Effects of $n(H_2O_2)/n(Fe^{2+})$ a) and PFS b) as well as their interaction on COD removal rate.

H₂O₂ dosage, the COD removal rate increases significantly and the H₂O₂ surface is steeper. The maximum COD removal rate is obtained when the H₂O₂ dosage is 4.5 mL. But when the H_2O_2 dosage is greater than 4.5 ml, the COD removal rate decreases gradually. The reason is that when the amount of H₂O₂ is small, the corresponding amount of hydroxyl is small and the oxidation of organic matter is less, leading to a low COD removal rate. When H₂O₂ gradually increases, the hydroxyl will increase, leading to a high COD removal rate. When the amount of H_2O_2 is greater than 4.5 ml, Fe^{2+} will be oxidized to Fe^{3+} by the excessive H₂O₂, leading to a low effective utilization rate of H_2O_2 . Besides, Fe^{3+} would inhibit the production of $\cdot OH$ ^[24] and the COD removal rate decreases. Therefore, the changes of H₂O₂ will have significant influences on the COD removal rate. In the process of the Fenton reaction, the concentration of H2O2 will directly influence the oxidation of organic compounds. The oxidation of organic matters will increase with the increase of H₂O₂ dosage.

With the increase of PFS, the COD removal rate increases slowly, but not significantly, because with the increase of PFS, multi-charged polymeric ions promote the aggregation of leachate colloid, leading to an increase



Fig. 3. Effects of H_2O_2 a) and PFS b) as well as their interaction on COD removal rate.

in the COD removal rate. But excessive PFS re-stabilized the agglomerated colloid, leading to a decrease in the COD removal rate [25]. Therefore, increasing the COD removal rate by increasing the PFS dosage is not obvious [26], while increasing H_2O_2 is obvious. The effects of the interaction between H_2O_2 and PFS on COD removal rate is not significant. Therefore, in the laboratory it's an effective way to increase COD removal rate by increasing H_2O_2 .

Effects of H_2O_2 and $n(H_2O_2)/n(Fe^{2+})$ on COD Removal Rate

The effects of H_2O_2 and $n(H_2O_2)/n$ (Fe²⁺) as well as their interaction on the COD removal rate are shown in Fig. 4. As seen from Fig. 4, the COD removal rate firstly increases with the increase of H_2O_2 dosage, and then tends to be flat in the end. The reason is that with the increase of H_2O_2 , the hydroxyl radicals increase, the oxidation ability also gradually increases, and the degradation of organic matter increases gradually but significantly. However, when the dosage of H_2O_2 is greater than or equal to 4.5 ml, though the hydroxyls increase, some organic matter cannot be degraded by hydroxyl radical oxidation



Fig. 4. Effects of H_2O_2 a) and $n(H_2O_2)/n(Fe2+)$ b) as well as their interaction on COD removal rate.

and excessive H_2O_2 cannot increase the COD removal rate. The COD removal rate also increases firstly and then tends to be flat as $n(H_2O_2)/n$ (Fe²⁺) increases. This is because in the Fenton reaction process the hydroxyl radicals are produced by H_2O_2 reacting with Fe²⁺.

Although \cdot OH increased with the increase of Fe²⁺ and H₂O₂, the organic matter to be decomposed is limited. In addition, H₂O₂ is easy to decompose into H₂O and O₂ [24], so there is an optimal dosage of H₂O₂. Fig. 4 shows that the optimal dosage is 5.00. When n(H₂O₂)/n (Fe²⁺) is greater than 5.00, excessive H₂O₂ can react with hydroxyl radicals and generate HO₂, which has an lower oxidizability than the hydroxyl radicals, and thus the COD removal rate of won't increase unlimitedly with the increase of n(H₂O₂)/n(Fe²⁺)[27].

In summary, COD removal can be improved significantly by increasing H_2O_2 and $n(H_2O_2)/n(Fe^{2+})$ simultaneously. Fenton reaction is the main process of removing COD in landfill leachate, and H_2O_2 and Fe^{2+} are the producers of \cdot OH in Fenton reaction. Increasing H_2O_2 and $n(H_2O_2)/n(Fe^{2+})$ will result in an increase of \cdot OH and an obvious increase of COD removal rate. When H_2O_2 and $n(H_2O_2)/n(Fe^{2+})$ increases, the COD removal rate tends to be flat. The reason is that only part of the organic matter

in the landfill leachate could be oxidized and degraded by hydroxyl radicals.

Optimizing Operating Conditions

Optimization was carried out on the experimental operation parameters. The limits are PFS: 10-20 ml, $n(H_2O_2)/n(Fe^{2+})$: 1-7, H_2O_2 : 0.5-5 ml, and the response value is the maximum COD removal rate of the landfill leachate. The main factors that limit the commercialization of Fenton coagulation is the high cost and material corrosion caused by harsh reaction conditions. Therefore, the aim of this study is to effectively degrade COD in the shortest time using the smallest consumption of oxidant in the presence of PFS, $n(H_2O_2)/n(Fe^{2+})$, and H_2O_2 . Eleven optimized operating conditions have been obtained, which are shown in Table 6. As seen from Table 6, No.1 is the best operating condition as it has the minimum Fe²⁺ dosage. And the COD removal rate can reach up to 92.11% with PFS 20 ml, $n(H_2O_2)/n(Fe^{2+})$ 6, and H_2O_2 5ml.

Economic Analysis

The economic analysis of treating landfill leachate with the Fenton oxidation-coagulation method is shown in Table 6. The density of landfill leachate and H_2O_2 is 1 g/cm³. The unit prices of the reagents are as follows (Chinese Yuan/t, from Alibaba.com):

PFS: 1,550, H₂O₂: 1,400, FeSO₄·7H₂O: 350; FeCl₃: 3,200.

Table 6. Optimization of testing conditions.

Number	PFS ml	$n(H_2O_2)/n(Fe^{2+})$	H_2O_2 ml	COD removal rate (%)	
1	20	6.06	5	92.11	
2	20	6.11	5	92.11	
3	20	6.02	5	92.11	
4	20	5.99	5	92.11	
5	20	6.21	5	92.10	
6	20	6.09	4.97	92.09	
7	20	5.84	5	92.09	
8	20	6.36	5	92.07	
9	20	6.04	4.89	92.03	
10	20	6.09	4.88	92.03	
11	20	5.83	4.72	91.89	
12	20	5.74	4.72	91.87	
13	20	5.23	5	91.79	
14	20	6.38	4.71	91.78	
15	20	5.73	4.47	91.62	
16	20	6.16	4.35	91.39	

	Reference		1 Wang et al. (2009) [28]	oqi Zhou (2008) [29]	ı Sun,et al (2013) [30]	Zhijun Zhang,et al (2011) [31]	1g Guo et al. (2010) [32]	Amor et al. (2015) [33]	Moradi et al. (2014) [34]	/edrenne et al. (2012) [35	This study
			Xiaojui	Sha	Yihu		Jin-Sor	Carlos	Mahsa	Michel V	
		Cust (Yuan	2.22	2.55	4.38	44.44	36.16	9.23	8.95	3.41	31.43
	COD	Removal rate (%)	66.67	06	67.2	90.3	93.3	68	74.7	63.0	92.1
	ation process	$n(H_2O_2/Fe^{2+})$	1.2	1	2.86	4	2.45	48.89	2	114	9
	enton oxida	${\rm H_2O_2} \\ {\rm mmol/L}$	5.4	9	13.8	240	176	17.6	20	18.5	150
	F6	μd	т	'	2.5	·	3	3	3	3	3
ethod.	cess	Dosage mg/L	600	652	1,110	1,500	800	2,000	1,500	300	3,350
1-coagulation m	Coagulation pro	Coagulation reagent	PFS	PFS	PFS	FeSO_4	PFS	$FeCl_{3} \cdot 6H_{2}O$	FeCl ₃	$FeCl_{3} \cdot 6H_{2}O$	PFS
kidatior	•	Ηd	5	5	5.06	ı	5	5	7	·	5
g the Fenton or	Influent	COD (mg/L)	600-700	640	1,560	2,633	4,150	5,700	11,280±300	12,161 ±11	2,000
momic analysis of treating landfill leachate using		Treatment method	SBR+ Fenton oxidation-coagulation +BAF ^[28]	SBR+ Fenton oxidation-coagulation +SBR ^[29]	biological treatment + Fenton oxidation- coagulation ^[30]	Fenton oxidation-coagulation ^[31]	Fenton oxidation-coagulation +SBR [32]	Fenton oxidation-coagulation ^[33]	Fenton oxidation-coagulation ^[34]	Fenton oxidation-coagulation ^[35]	Biological treatment +coagulation + Fenton oxidation
Table 7. Ec.		Number	1	2	3	4	5	6	7	8	6

As seen from Table 7:

- 1. Treating landfill leachate with Fenton oxidationcoagulation method and the cost is greatly influenced by water quality. Great differences can be found in the landfill leachate with similar COD concentrations and different ages.
- 2. When the cost of the Fenton oxidation-coagulation method is low, the COD removal rate is usually not ideally high. The poor water quality can't satisfy the requirement, resulting in a high load of subsequent processing technique and high total operational cost.
- 3. Treating landfill leachate with a concentration of 2,000 mg/l with Fenton oxidation-coagulation method, the cost in this study reaches the lowest while the COD removal rate is above 90%. The increase of PFS leads to optimization of the coagulation effect and reduction of consumption of the Fenton reagent, as well as the treatment cost.

Conclusions

- 1. Treating landfill leachate using the Fenton oxidationcoagulation method and fitting the equation of COD removal rate is obtained by BBD tests as well as RSM with PFS, $n(H_2O_2)/n(Fe^{2+})$, and H_2O_2 as influencing factors. The fitting equation has a high significance and sensitivity and the predicted results are close to the real measured data. Compared with the experimental results, the errors in the prediction of COD removal rate is less than $\pm 5\%$.
- 2. Results of the ANONA analysis of the fitting equation show that factors influencing the COD removal rate are: $H_2O_2 > n(H_2O_2)/n(Fe^{2+}) > PFS$. The increase of H_2O_2 dosage and $n(H_2O_2)/n(Fe^{2+})$ is more effective in degradation of the organic matter, while the increase of PFS is not.
- 3. For biochemical effluent of landfill leachate with a concentration of around 2,000 mg/l, the optimum operating conditions for Fenton oxidation-coagulation are (for one liter landfill leachate): PFS-67 ml (0.05g/L), H₂O₂ (30%)-17 ml, and n(H₂O₂)/n(Fe²⁺)-6, and the COD removal rate can reach as high as 92.11%.

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