Optimization of 4-chlorophenol decomposition by H_2O_2 activate catalytic magnetic iron oxide an activated carbon carrier

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ABSTRACT

Wastewater from chemical plants that produce pesticides always carries a large amount of organic matter that is difficult to decompose. One of them is the compound 4-chlorophenol, which has difficult-to-decompose properties, is durable in the environment, and is also listed in the group of substances that are likely to cause cancer in humans. In this study, the 4-chlorophenol compound was treated with heterogeneous Fenton by H_2O_2 activated by magnetic iron oxide nanoparticles on activated carbon. Magnetic iron oxide nanoparticles were successfully synthesized and mounted on activated carbon 10-15 nm in size with material surface morphological parameters such as specific surface area 330.28 cm^2/g; total pore volume 0.16 cm^3/g; magnetization 8.19 emu/g. The optimization of the 4-chlorophenols decomposition reaction with pH parameters, the content of catalytic materials, and the initial concentration of 4-chlorophenol is carried out using the Box-Behnken Design. The results showed that the removal efficiency of 4-chlorophenol was 96.5% achieved with optimum parameters pH 2.9; catalytic concentration 0.32 g/L; initial concentration of 4-chlorophenol 92.3 mg/L. The results of the study show the efficiency of the decomposition of an organic compound using magnetic activated carbon.

Keywords: Magnetic activated carbon; 4-chlorophenol; Advanced oxidation processes.

1. INTRODUCTION

Wastewater is contaminated by 4-chlorophenol organic compounds with non-biodegradable characteristics and is difficult to thoroughly treat with conventional methods [1]. Therefore, a suitable method is required to treat this source of contamination completely. The Advanced oxidation process is suitable for the strong oxidation capacity of the hydroxyl radical (•OH) produced in the reaction and the post-reaction product consists only of CO_2 and H_2O [2]. There have been many studies of high-performance treatment of organic compounds with the advanced oxidation process. Chen R and colleagues (2015) [3] treated 4-chlorophenol with magnetic nanoparticles with different pH values. Results showed the highest 4-chlorophenol removal at pH 5. In addition, the surface reaction mechanism explaining the degradation of 4-chlorophenol has been investigated in detail by combined oxidation and adsorption. Gan Q and colleagues (2020) [4] studied the treatment of 4-chlorophenol using sludge and a Fenton agent. The results showed that 4-chlorophenol was completely decomposed after 120 minutes of reaction, with influencing factors such as pH, initial concentration of 4-chlorophenol, catalyst concentration, and concentration of peroxide. Chen Y and colleagues (2020) [5] studied the optimization of 4-chlorophenol electrochemical treatment using the response surface method. The highest 4-chlorophenol removal was 96.13% with optimization parameters: concentration of Na_2SO_4 2 g/L, electrode plate distance of 2 cm, current intensity of 2 A, and particle electrode dosage of 14 g. These studies have all achieved high treatment efficiency, but investigating the adsorption process before the oxidation reaction takes place is often overlooked because the carrier does not have a large surface area. The use of adsorbent materials, together with advanced oxidation processes, is an effective technology

for the treatment of organic pollutants. Heterogeneous catalysis can completely process difficult-to-decompose organic compounds, but the solution after the reaction has the dissolution of metal ions. It is necessary to apply a carrier material with a large specific surface area to overcome this disadvantage (the specific surface area from 300 – 2500 m^2/g). Activated carbon with a large surface area helps the recovery of the iron involved in the reaction on the surface of the carrier to avoid dissolution into the post-treatment solution compared to other materials.

This study focuses on the synthesis of magnetically activated carbon materials by co-precipitation in anaerobic environments. 4-chlorophenol treatment was investigated as an oxidation reaction combining adsorption with influencing factors: pH of the solution, ratio concentration of H_2O_2 and 4-chlorophenol, catalyst concentration and initial concentration of 4-chlorophenol. The response surface methodology was used to optimize 4-chlorophenol treatment.

2. EXPERIMENT

2.1. Chemicals

Chemicals used to synthesize magnetic activated carbon include activated carbon (0.5 – 1.0 mm particle size powder - China), iron II chloride tetrahydrate ($FeCl_2.4H_2O$ 99.5% - China), iron III chloride hexahydrate ($FeCl_3.6H_2O$ 99.7% - China), ammonia solution (NH_3 28-30% - China), acid hydrochloric (HCl 36.5% - China), ethanol (C_2H_5OH 99% - China), sodium hydroxide (NaOH 98% - China), deionized water (DI).

2.2. Preparation of magnetic activated carbon

First, treat the activated carbon source with 0.1 M HCl to remove ash, dust, and impurities. Prepare 150 mL of a mixture of $FeCl_2$ and $FeCl_3$ iron salts in a molar ratio of 1/2, add activated carbon to the mixture (ratio of Fe_3O_4 equal to 20% of the mass of activated carbon), stirring the mixture with a magnetic stirrer heated at 70-80 °C [6] and proceed under anaerobic conditions by using nitrogen gas flow to expel all air in the reaction mixture. The mixture was precipitated with 20 mL of 28-30% NH_3 solution, and the mixture was stirred for another 20 min. After the reaction, the mixture was allowed to age at room temperature for 24 hours. The resulting solid mixture was washed with ethanol and deionized water several times until the washing water had a neutral pH, and then the resulting solid was brought into the oven at 70 °C to a constant mass [7].

2.3. Characterization of magnetic activated carbon

Surface morphological characteristics, properties, and composition of materials are analyzed by modern methods such as: Transmission electron microscopy – TEM (JEM-2100F, Japan), X-ray diffraction – XRD (D2 Phaser, Bruker, Germany), energy dispersive X-ray – EDX (Shimadzu EDX-LE, Japan), vibrating sample magnetometer – VSM (8600 Series VSM, LakeShore, United Kingdom), specific surface area and pore size distribution (BET/BJH) was analyzed from an N_2 adsorption/desorption at 77.35 K [7].

2.4. Experimental design method

To study the removal efficiency of 4-chlorophenol by adsorption process combining oxidation (After equilibrium adsorption, H_2O_2 is added to the mixture to trigger the 4-chlorophenol decomposition reaction that takes place) with influencing factors: initial catalyst dosage of 0.2 – 0.4 g/L, 200 mL of 4-chlorophenol solution to be treated with an initial concentration of 50 – 150 mg/L, pH of the solution with a value of 2 – 7 (The pH of the solutions was adjusted with HCl 0.1 mol/L and NaOH 0.1 mol/L), the duration of the treatment process and initial concentration ratio of H_2O_2 and 4-chlorophenol [H_2O_2]/[4-CP]. To remove the volatilized portion of 4-chlorophenol in the experiments, the white sample was run in parallel in all experiments, followed by an analysis of 4-chlorophenol content by high-performance liquid chromatography equipped with a C18 column (4.66×150 mm; 5.0 µm) and a photodiode array detector (PDA). The mobile phase was a

binary mixture of methanol and water (70%/30%, v/v) at a flow rate of 1 mL/min [8]. The response surface method (Box-Behnken design) is applied to optimize the regression evaluation of the target function as a removal of 4-chlorophenol. The statistical calculations were conducted on the Design Expert 11 software [17].

3. RESULTS AND DISCUSSION

3.1. Characteristics of the magnetic activated carbon

The analysis results of morphological characteristics, surface properties and composition of magnetic activated carbon Fe_3O_4/AC materials are shown in Fig. 1.

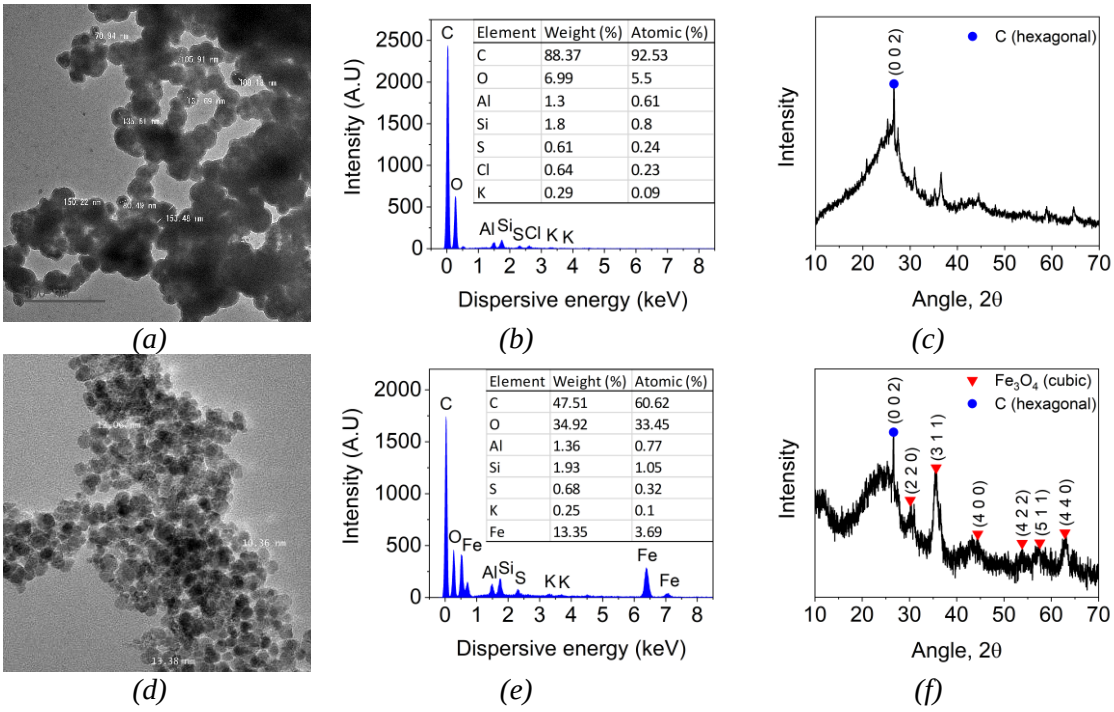
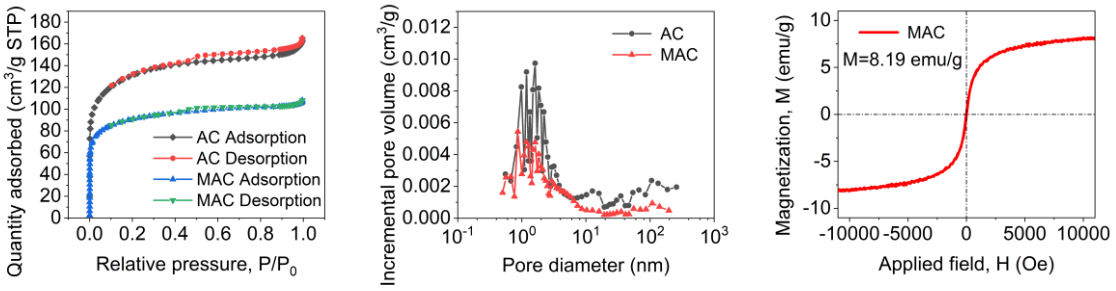


Figure 1. *Characterization: (a) TEM micrograph; (b) EDX spectrum; (c) XRD spectrum of activated carbon; (d) TEM micrograph; (e) EDX spectrum; (f) XRD spectrum of magnetic activated carbon.*

From Fig. 1, it can be seen that magnetic iron oxide nanoparticles have been successfully synthesized with an average size of 10-15 nm mounted on the surface of activated carbon. From the XRD spectrograph, it can be seen that the magnetic iron oxide nanoparticle is synthesized with a cubic crystal configuration with an edge size of 8.37 Å mounted on the hexagonal structure of graphite crystals with a profile of P63/MMC (materials studio) [16]. The diffraction peaks show miller faces characteristic of magnetic iron oxide (220), (311), (400), (422), (511), (440) along with the face (002) characteristic of hexagonal structure of carbon (ICSD collection code 084611) [9]. The elemental components present in the pre and post-denaturation coal samples were also tested by EDX spectroscopy with the composition of magnetic activated carbon consisting mainly of elements C, O, and Fe compared to activated carbon with the main component being element C (accounting for 88.37% of the mass).

The analysis results also show that the charcoal surface structure before and after denaturation is mainly in the form of micropores (with characteristic isotherm adsorption langmuir form type IV) [10]. The specific surface area of magnetic activated carbon reaches 330.28 m^2/g with an average pore size of 1.56 nm (table 1). The material sample also super convergence paramagnetic properties with a saturation magnetization value of 8.19 emu/g.



(a) *(b)* *(c)*

Figure 2. *Surface property and magnetization (a) nitrogen adsorption-desorption isotherm, (b) pore size distribution, (c) vibrating sample magnetometry.*

Table 1. *Pore structure properties.*

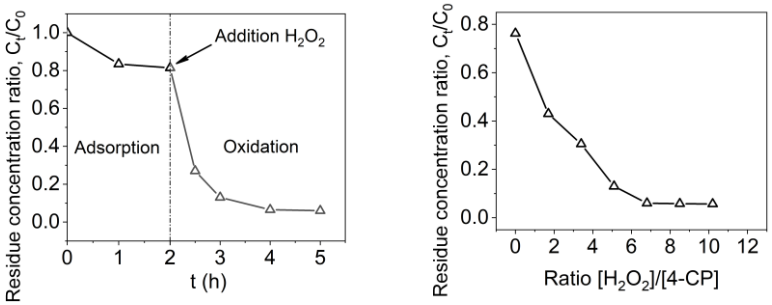
Adsorbent	**Specific surface area (m^2/g)**	**Specific surface micropore area (m^2/g)**	**Average pore diameter (nm)**	**Pore volume (cm^3/g)**
Activated carbon	469.89	283.55	1.86	0.25
Magnetic activated carbon	330.28	219.13	1.56	0.16

With a large specific surface area (> 300 m^2/g), when studying the decomposition process of 4-chlorophenol for magnetic activated carbon, it is necessary to consider the influence of surface area by studying the combined oxidation and adsorption of magnetic activated carbon [11].

3.2. Survey domain experiment design

3.2.1. Effect of time, the initial concentration ratio of peroxide and 4-chlorophenol

The results of the study on the effect of reaction time and initial concentration ratio of H_2O_2 and 4-chlorophenol are shown in Fig. 3.



(a) (b)

Figure 3. *Effect of (a) time of reaction and (b) initial concentration ratio [H_2O_2]/[4-CP].*

The results of Fig. 3 indicate that adsorption reaches equilibrium after 120 min of reaction and after 180 min of near oxidation of decomposition 4-chlorophenol reaches a saturated state. In terms of the effect of peroxide, with ratio concentration [H_2O_2]/[4-CP] = 6.8, oxidation reaches a saturated state. Therefore, these values are used to study the domain of the experimental design of subsequent factors.

3.2.2. Effect of catalyst dosage, pH of the solution and initial concentration of 4-chlorophenol

Define the domain of experiment design with influencing factors: catalyst dosage, m (g/L), pH of the solution, initial concentration of 4-chlorophenol, and C_0 (mg/L).

The study of the effect of catalyst dosage (pH = 3, C_0 = 100 mg/L) showed that the optimal amount of catalyst for processing is 0.3 g/L, the amount of catalyst is too large, leading to the formation of an iron precipitate, which consumes hydroxyl activating radicals (•OH), leading to a decrease in processing efficiency. Conversely, too small a catalytic content will not provide sufficient amounts of Fe^{2+} catalysts through which the amount of hydroxyl radical (•OH) also decreases [12]. The study of the effect of the pH of the solution (m = 0.3 g/L, C_0 = 100 mg/L) showed that a pH value of 3 was optimal for the treatment process. The pH value is too low to prevent the reduction of Fe^{3+} to Fe^{2+} (reaction 2, 3) due to the chemical balance shifting in the opposite direction, the pH is too high, which will consume hydroxyl radicals (•OH) during treatment (reaction 1) reverse displacement equilibrium is a disadvantageous direction for oxidation.

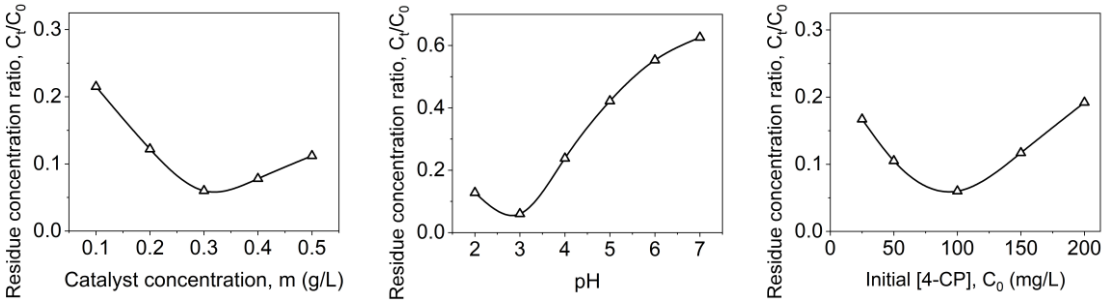


Figure 4. *Define the domain of experiment design with influencing factors (a) catalyst dosage, (b) pH of the solution (c) initial concentration of 4-chlorophenol.*

$$Fe^{2+} + H_2O_2 \longrightarrow Fe^{3+} + OH^- + \cdot OH \quad (1)$$

$$Fe^{3+} + H_2O_2 \longrightarrow Fe^{2+} + H^+ + \cdot HO_2 \quad (2)$$

$$Fe^{3+} + \cdot HO_2 \longrightarrow Fe^{2+} + H^+ + O_2 \quad (3)$$

The study of the effect of the initial concentration of 4-chlorophenol (m = 0.3 g/L, pH = 3) resulted in the optimal value of 4-chlorophenol content equal to 100 mg/L. At high concentrations, the tendency to create higher intermediate by-products hinders the degradation potential of 4-chlorophenol, which is detrimental to processing. Also, low concentrations of hydroxyl oxidation radicals (•OH) are not sufficient agents to be able to fully oxidize instead they can react oxidally with Fe^{2+} ions for a greater rate of reaction due to the excess amount of iron ions in this case compared to the amount of 4-chlorophenol to be processed [12].

From the above studies, the planning domain of the main influencing factors was found to be catalyst dosage, m (0.2-0.4 g/L); pH of the solution (2-4); initial concentration of 4-chlorophenol, C_0 (50-100 mg/L). These values were selected to research to optimize the processing efficiency of 4-chlorophenol.

3.3. Optimization of 4-chlorophenol oxidation using response surface method

The implementation plan is calculated with influencing factors: catalyst dosage, m (g/L); pH of the solution; initial concentration of 4-chlorophenol, C_0 (mg/L); with the target function of removal (%) of the oxidation process. The planning value domain of the influencing factors is shown in table 2.

Table 2. *Experimental range and levels in the Box-Behnken Design.*

Variables	Symbol	-1 (low value)	0 (mid value)	+1 (high value)
m (g/L)	A	0.2	0.3	0.4
pH	B	2	3	4
C_0 (mg/L)	C	50	100	150

The results of the ANOVA analysis (table 3) show the correlation between theory and experiment. The p-value is intended to determine the probability of identifying incompatible elements beyond the assumptions made. The results obtained with p-value of the model less than 0.0001 show that the second-order model is meaningful, the variables in the model with p-value > 0.05 are not statistically significant, and the reduction of these variables can improve the model. The f-value of the chosen model is 43.28, indicating a critical model, of which there is only a 0.01% chance of such a large f-value occurring due to noise. The lack of fit f-value of 4.41 implies there is a 9.29% chance that a lack of fit f-value this large could occur due to noise. This value of less than 10% proves that the model is fully compatible with the experiment [13].

Table 3. *ANOVA for quadratic model.*

Source	**Sum of squares**	**df**	**Mean square**	**f-value**	**p-value**	
Model	1774.95	9	197.22	43.28	< 0.0001	**significant**
A: m	38.28	1	38.28	8.4	0.023	
B: pH	75.65	1	75.65	16.6	0.0047	
C: C_0	19.53	1	19.53	4.29	0.0772	
AB	3.06	1	3.06	0.672	0.4394	
AC	0.04	1	0.04	0.0088	0.928	
BC	0.7225	1	0.7225	0.1585	0.7024	
A^2	1039.84	1	1039.84	228.17	< 0.0001	
B^2	363.39	1	363.39	79.74	< 0.0001	
C^2	99.66	1	99.66	21.87	0.0023	
Residual	31.9	7	4.56			
Lack of fit	24.49	3	8.16	4.41	0.0929	**not significant**
Pure error	7.41	4	1.85			
Cor total	1806.86	16				

R^2 coefficient of 0.9823 shows the high compatibility of the real variable model with the regression equation. The model sets the Adjusted R^2 value to 0.9596 to adjust the number of indicators if the additional indicators do not increase. The Predicted R^2 value of the model achieved is 0.7767, and the difference value between Adjusted R^2 and Predicted R^2 is less than 0.2, indicating the high reliability of the model. Adeq Precision measures the signal-to-noise ratio. A ratio greater than 4 is desirable. A ratio of 17.952 indicates an adequate signal. This model can be used to navigate the design space [14].

In addition, the mean and coefficient of variation of the measured values are 82.02 and 2.6%. The dispersion of the data set evaluated by the standard deviation with a value of 2.13 characterizes a large variation when changing the value of the influencing factors, which shows that the experimental data are reliable [15].

Table 4. *Fit statistics.*

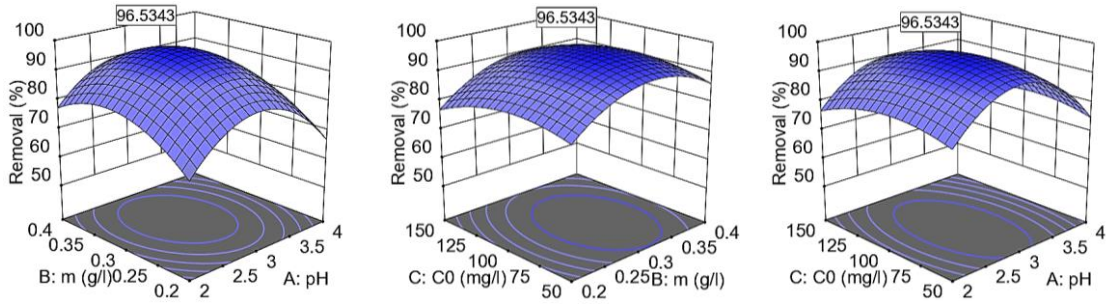
Parameter	**Value**
Standard deviation	2.13
Mean	82.02
Coefficient of variation (%)	2.6
R^2	0.9823
Adjusted R^2	0.9596
Predicted R^2	0.7767
Adeq precision	17.9519

The final equation in terms of coded factors.

$$Y = 96.08 - 2.19A + 3.08B - 15.72A^2 - 9.29B^2 - 4.86C^2 \quad (4)$$

Where A: pH of the solution; B: Catalyst dosage, m (g/L); C: Initial concentration of 4-chlorophenol, C_0 (mg/L).

The regression equation of the target function (Eq. 1) gives experimental prediction results according to the levels of each encoded factor. This equation is significant for evaluating the mutual impact between factors through the coefficients of the parameters.



(a) *(b)* *(c)*

Figure 5. *Response surface of the factor pairs (a) pH and m, (b) m and C_0, (c) C_0 and pH.*

Optimization results from Design Expert 11 software gave the highest 4-chlorophenol processing efficiency of 96.5%, with factors at optimal values: pH of solution equal to 2.9; catalyst dosage, m = 0.32 g/L; initial concentration of 4-chlorophenol, C_0 = 92.3 mg/L. This result was similar to the previous 4-chlorophenol decomposition studies by advanced oxidation process (table 5). From the above results, it shows that 4-chlorophenol treatment efficiency of magnetic activated carbon. The response surface at the optimization values of each element is shown in Fig. 5.

Table 5. *Comparison of 4-chlorophenol decomposition of Fe_3O_4/AC with other catalysts.*

Catalyst	Removal 4-chlorophenol	References
Fe_3O_4/AC	96.5%	This study
Fe_3O_4/GO	90.1%	[18]
Zn-CNTs-Fe_3O_4	98.8%	[19]
Fe/Cu/γ-Al_2O_3	99.4%	[20]

Experimental testing of 4-chlorophenol treatment efficiency against theoretical calculations was performed with 3 experimental tests (table 6). The error results between the theoretical and experimental calculation models are insignificant (< 3%), showing the high reliability of the model when applying the 4-chlorophenol processing performance calculation and there is little organic intermediate produced during treatment.

Table 6. *Optimization result.*

Numbers	Factors			Removal (%)	
	A: pH	B: m (g/L)	C: C_0 (mg/L)	Theory	Experimental
1	2.9	0.32	92.3	96.5	93.8
2	2.9	0.32	92.3	96.5	94.7
3	2.9	0.32	92.3	96.5	95.4

4. CONCLUSIONS

The research has successfully synthesized magnetically activated carbon materials by the co-precipitation method. Materials obtained by modern research methods have demonstrated the existence of Fe_3O_4 (the crystal structure characterized by miller planes by XRD analysis) on activated carbon surfaces with a size of 10-15 nm, the cubic crystal configuration with an edge

size of 8.37 Å has a saturation magnetization of 8.19 emu/g showing the superparamagnetic nature of the material. Research on optimizing the processing capacity of 4-chlorophenol for magnetic activated carbon has been carried out using the second-order experimental planning method. The results indicated that the 4-chlorophenol processing capacity achieved the highest efficiency by 96.5% with optimal conditions: catalyst dosage, m = 0.32 g/L; pH of solution 2.9; initial concentration of 4-chlorophenol, C_0 = 92.3 mg/L. This result shows the effective treatment of the persistent organic matter of magnetic-activated carbon materials.

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TÓM TẮT

Tối ưu hóa quá trình phân hủy 4-chlorophenol bằng H_2O_2 kích hoạt oxit sắt từ tính xúc tác trên chất mang than hoạt tính

Nước thải tại các nhà máy hóa chất sản xuất thuốc trừ sâu luôn mang theo một lượng lớn chất hữu cơ khó phân hủy. Một trong số đó là hợp chất 4-chlorophenol có đặc tính khó phân hủy, bền trong môi trường và còn được liệt vào nhóm chất có khả năng gây ung thư cho con người. Trong nghiên cứu này, hợp chất 4-chlorophenol được xử lý bởi phương pháp fenton dị thể với H_2O_2 hoạt hóa bằng các hạt nano oxit sắt từ trên than hoạt tính. Kết quả chỉ ra rằng, đã tổng hợp thành công hạt nano oxit sắt từ gắn trên than hoạt tính có kích thước 10-15 nm với các thông số hình thái bề mặt vật liệu như diện tích bề mặt riêng 330,28 cm^2/g; tổng thể tích lỗ rỗng 0,16 cm^3/g; độ từ hóa 8,19 emu/g. Việc tối ưu hóa phản ứng phân hủy 4-chlororphenol với các thông số pH, hàm lượng chất xúc tác, nồng độ ban đầu của 4-chlorophenol được thực hiện bằng kế hoạch Box-Behnken. Kết quả cho thấy hiệu suất loại bỏ 4-chlorophenol đạt 96,5% với thông số tối ưu pH 2,9; nồng độ xúc tác 0,32 g/L; nồng độ ban đầu 4-chlorophenol 92,3 mg/L. Kết quả của nghiên cứu cho thấy hiệu quả phân hủy hợp chất hữu cơ bằng than hoạt tính từ tính.

Từ khoá: Than hoạt tính từ tính; 4-chlorophenol; Oxy hóa bậc cao.