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Removement of thiocyanate from industrial wastewater by microwave-Fenton oxidation method

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Abstract

The microwave radiation oxidation process, Fenton as catalytic agent, was used to remove the thiocyanate from the industrial wastewater. The effects of microwave power, radiation time, pH and the feeding in ways of catalyst on the degradation rate of synthetic wastewater were investigated using the microwave radiation oxidation process by orthogonal experiment. The results show Fenton catalyst ratio was 1:20, the microwave radiation power was 900 W, the microwave radiation time was 7 min and the value of pH was 3. Under the optimum conditions, the removal of KSCN can reach over 90%. The apparent kinetics of removal was studied, which conformed to kinetics first-class reaction. In short, for the thiocyanate from the industrial wastewater, microwave-Fenton oxidation method is feasible and effective.

Key words: microwave technology; fenton reagent; thiocyanate; orthogonal experiment

Introduction

Thiocyanate (SCN^-) is a linear, electronegative polyatomic ion (Hughes, 1975). Due to some of its rather unique properties, thiocyanate is used in a variety of industrial processes such as industrial and mineral wastewater, especially in coking wastewater, in which thiocyanate is the highest concentrations (ranged between 100 and 1500 mg/L) of inorganic pollutant concentrations (Sharma et al., 2002). Thiocyanate is one of the important substances cause struma (Ermans et al., 1979; Astwood, 1943). Thiocyanate also has some toxic effects, which include respiration problems or can even provoke human death while the formation of toxic gases from contact with acids (Vicente and Díaz, 2003). It is very difficult to remove thiocyanate using traditional high-pressure hydrolysis method. Hung and Pavlostathis (1997) investigated biological denitrification ammonia nitrogen wastewater in the presence of thiocyanate, partial inhibition of nitrification was observed and attributed to thiocyanate. Therefore, it is very important and necessary to develop new methods for the pretreatment desulfurization waste solution to reduce the content of cyanide-containing pollutants and then carry out biological denitrification.

In recent years, microwave (MW) radiation has attracted a great deal of attention due to superior performance in wastewater treatment (Li et al., 2010; Lin et al., 2009a, 2009b). Fenton method has been universally studied and applied in the field of wastewater treatment because the method's merits are fast reaction, easy control and auto-generation flocculation (Lin and Lo, 1997; Ruppert and Bauer, 1993). Using MW-Fenton reagent to oxidative thiocyanate and transforming it into HCN. Then using the high-pressure hydrolysis method for further treatment after absorbed by lye, which can ensure the safety of operation process. In short, this method could solve over high cyanide-containing in industrial wastewater once and for all, and remission the pressure of biological denitrification, is of great theoretical significance and potential of the application.

As a result of high colourity, complicated composition and more interference factors in industrial wastewater, KSCN has been simulated to experimental treatments reference to the thiocyanate contents in industrial wastewater. In order to explore the optimum condition of the removal of thiocyanate from wastewater by MW-Fenton oxidation, MW radiation power, MW radiation time, the value of pH and the feeding in ways of catalyst were investigated, the effect law of various factors on KSCN removal was found. Conditions were also optimized through orthogonal experiments, which provide a theoretical basis and operational guidance in the actual industrial wastewater treatment process.

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1 Materials and methods

1.1 Materials

MW reactor (900 W, 2450 MHz, University of Science and Technology Liaoning) with different power setting was used as MW source. pH meter (pHSJ-5, INESA Scientific Instrument Co., Ltd.) was used as the measure of pH. Potassium thiocyanate (99.5%, analytical reagent, Tianjin Kermel Chemical Reagent Development Center, China) was used as the source of thiocyanate. Deionized water was used for the preparation of solutions. All other reagents were above analytical grade.

1.2 Methods

During the MW treatment process, the devices have atmospheric condensation cooler, but the volume of wastewater slightly decreased due to evaporation of water. At the end of the experiment, cooled the wastewater to room temperature, deionized water was added into the reactor in order to maintain the same initial volume of the wastewater. The measurement method of thiocyanate was used by indirect iodometry.

2 Results and discussion

2.1 Effect of microwave radiation power on KSCN removal

Taking 100 mL KSCN standard samples, the adding amount of Fe^{2+} was 1.6 mmol, H_2O_2 was 32 mmol, under the MW radiation time of 7 min, change MW radiation power and determine the KSCN removal. The effect of MW radiation power on the KSCN removal was investigated. **Figure 1** illustrates the removal of KSCN at different MW radiation power. It can be seen that the removal was increased with the increasing of MW radiation power. That is mainly because of more energy in unit area was absorbed with the increasing of MW radiation time, made more heat generated, induced more impetuous and rapid molecular motion. This benefited the degradation of KSCN. Meanwhile, the increase of ·OH from reaction between Fe^{2+} and H_2O_2 with increased with radiation power, which have strong oxidation capability and is able to increase the removal of KSCN. Therefore, 900 W was considered to be the optimal radiation power.

2.2 Effect of MW radiation time on KSCN removal

Taking 100 mL KSCN standard samples, the adding amount of Fe^{2+} was 1.6 mmol, H_2O_2 was 32 mmol, under the MW power of 900 W, change MW radiation time and determine the KSCN removal. The effect of MW radiation time on the KSCN removal was investigated. **Figure 2** illustrates the removal of KSCN at different MW radiation time. The removal of KSCN increased with radiation time and attained to the optimum of 91.3% after 7 min. In addition, longer MW radiation time have few contribute to the removal of KSCN. That might due to the increase of ·OH from reaction between Fe^{2+} and H_2O_2 with increased with radiation time, which have strong oxidation capability and is able to increase the removal of KSCN. However, ·OH gradually decreased with consumption of H_2O_2, which makes the removal of KSCN increase few even unchanged. Considering the removal efficiency and the economic factor, 7 min was considered to be the optimal radiation time.

2.3 Effect of pH on KSCN removal

Taking 100 mL KSCN standard samples, the adding amount of Fe^{2+} was 1.6 mmol, H_2O_2 was 32 mmol, under the MW power of 900 W and radiation time 7 min, change the value of pH and determine the KSCN removal. Taking into account the Fenton reagent has a relatively good treatment effect under acidic conditions, so the experiment pH was changed from 1 to 5.

The effect of the value of pH on the KSCN removal

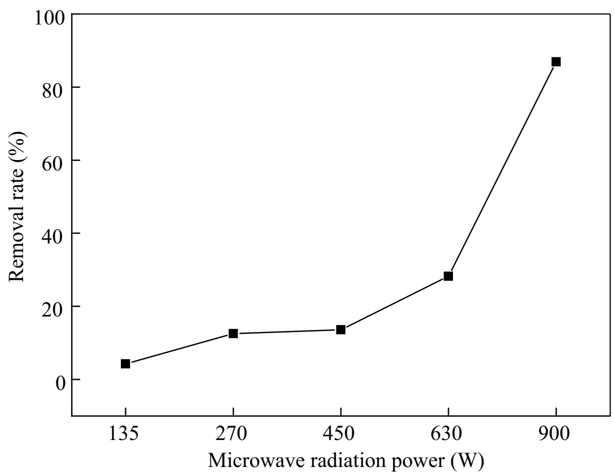

Fig. 1 Effect of MW radiation power on the KSCN removal ratio.

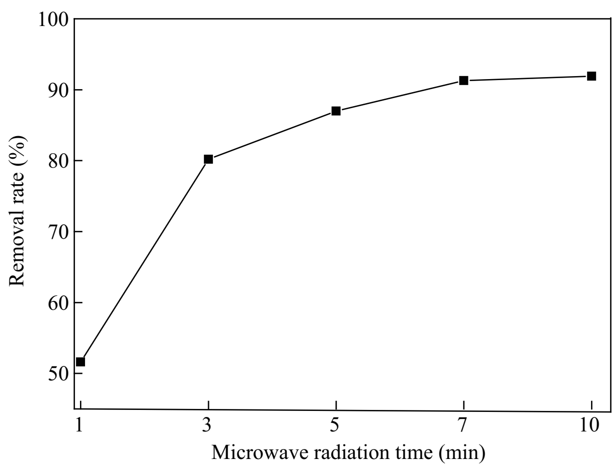

Fig. 2 Effect of MW radiation time on the KSCN removal ratio.

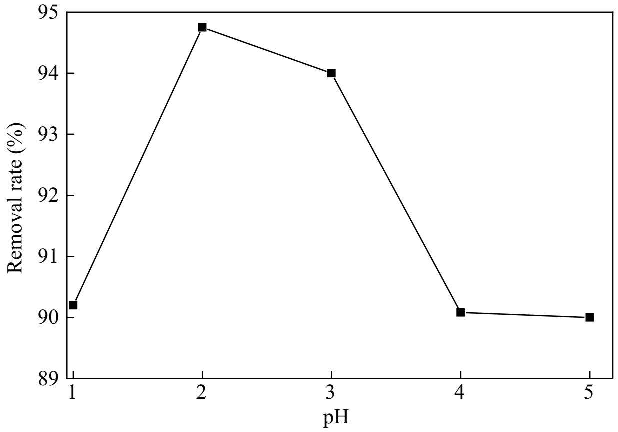

Fig. 3 Effect of pH on the KSCN removal ratio.

was investigated. **Figure 3** demonstrates that the removal of KSCN at different value of pH. When the value of pH change from 1 to 5, KSCN removal first increase and then drop with the increase of pH. The optimal pH was found to be 2, which resulted in 94.75% KSCN removal. When the pH was 3, there is higher removal. Then decrease when the pH was 4 and 5. The reasons are mainly the OH-produced by the reaction between Fe^{2+} and H_2O_2 as the following reaction:

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

At high pH goes, the formation of ·OH, which can oxidative most organic compounds, was unstable and easily decomposed. However, at low pH have some inhibitory effects, makes it difficult to formation of ·OH, then decrease the removal rate. Therefore, taking account into the economic factors, we chose pH was 3 as the optimal value.

2.4 Effects by the feeding in ways of catalyst on KSCN removal

Taking 100 mL KSCN standard samples, the adding amount of Fe^{2+} (Fe^{2+} is catalyst in Fenton oxidation) was 1.6 mmol, under the MW power of 900 W and radiation time 7 min, change the feeding in ways of catalyst and determine the KSCN removal. One way was once adding Fe^{2+}. Another way was continuous adding Fe^{2+}, that is to say, slowly and evenly adding Fe^{2+} in 7 min. The effect by the feeding in ways of catalyst on the KSCN removal was investigated.

Table 1 Effects by the feeding in ways of catalyst on the removal

Feeding in the ways of catalyst	Pre-treatment concentrations (g/L)	Post-treatment concentrations (g/L)	Removal rate (%)
Once adding	4.9573	0.4015	91.90
Continuous adding	4.9573	0.3718	92.50

2.5 Orthogonal experiments

According to the method of single factor analysis, molar ratio of Fe^{2+}/H_2O_2, n(Fe^{2+}) and the value of pH were investigated. The optimum technological parameters have been determined by orthogonal experiment. The orthogonal test of factors and standard levels are listed in **Table 2**. **Table 3** shows the Orthogonal regression experiment

It could be seen from **Table 3** that the effect order on removal from high to low is B > A > C > D, that is to say, B > A > C, so the optimum conditions was A3B3C3. Then the **Table 4** can be obtained by using variance analysis ($\alpha = 0.05$) of the result.

It could be learned from the data of F values in **Table 4** that effect order is consistent between the variance analysis and intuitive analysis. Among them, n(Fe^{2+}) is the most remarkable for the effect of KSCN removal

2.6 Verification experiment

To check the reproducibility of the optimum conditions, added one test of optimum condition. The result of removal was 94.6%. That is to say, under this condition, the result was better than orthogonal prediction experiments. In view of the industry cost consideration, it is appropriate to reduce the amount of catalyst for cost savings under a

Table 2 Orthogonal test of three factors and three standard levels

Level	Molar ratio of Fe^{2+}/H_2O_2 A	n(Fe^{2+})(mmol) B	pH C
1	1:10	8	2.0
2	1:30	32	4.0
3	1:20	16	3.0

Table 3 Orthogonal regression experiment

No.	A	B	C	D	Removal (%)
1	1	1	1	1	39.90
2	1	2	2	2	70.00
3	1	3	3	3	91.10
4	2	1	2	3	78.10
5	2	2	3	1	91.90
6	2	3	1	2	86.95
7	3	1	3	2	86.40
8	3	2	1	3	87.15
9	3	3	2	1	92.90
Ij	67.00	68.13	71.33	74.90	
IIj	85.65	83.02	80.33	81.12	
IIIj	88.82	90.32	89.80	85.45	
Rj	21.82	22.19	18.47	10.55	

Table 4 The table of variance analysis

Source of variance	Sum of square	Degree of freedom	Mean square	Fetest	Significant
A	766.904	2	383.452	4.545	**
B	833.817	2	416.909	4.942	***
C	511.636	2	255.818	3.032	*
D	168.727	2	84.364	1.000	
Error	168.727	2	84.364		

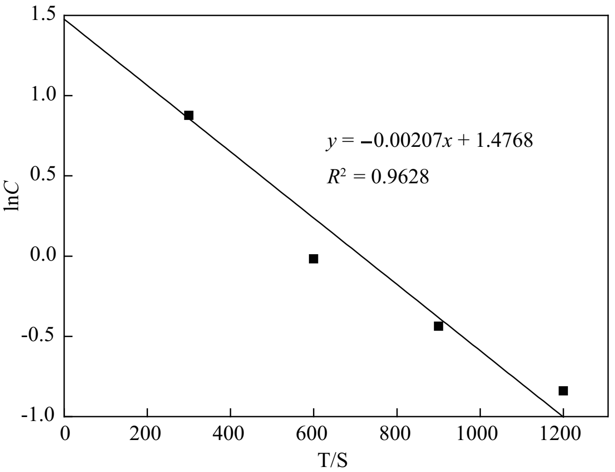

Fig. 4 Apparent reaction kinetics using MW-Fenton removal KSCN.

better removal.

2.7 Apparent kinetics

Taken 100 mL KSCN standard samples, the adding amount of Fe^{2+} was 1.6 mmol, H_2O_2 was 32 mmol, under the MW power of 900 W, change MW radiation time and determine the concentrations of KSCN. Using MW radiation time as abscissa and the logarithms of concentrations of KSCN as ordinate, then does simple linear regressions. **Figure 4** shows that there was a good linear relation between the logarithms of concentrations of KSCN and MW radiation time, approximately obeyed first-order reaction. The reaction kinetics equation:

$\ln C = -0.00207t + 1.4768$

the thermodynamics factors: R^2 = 0.9628, that is the relationship between the logarithms of concentrations of KSCN and MW radiation time is direct ratio. It is indicates that the apparent kinetics was first-order reaction, with reaction rate constant $k = 0.22207 \text{sec}^{-1}$ and half life $t_{1/2}$ = 335 sec.

3 Conclusions

(1) The effects of MW radiation power, MW radiation time, pH and the feeding in ways of catalyst on the degradation rate of synthetic wastewater were examined. The result reveals that with the increase of MW radiation power and radiation time, the removal was increased. However, KSCN removal first increase and then drop with the increased of pH. There is no difference in the feeding in ways of catalyst.

(2) Through orthogonal experiments, the optimum technical conditions of the condensation were obtained as follows: Fenton catalyst ratio was 1:20, the MW radiation power was 900 W, the MW radiation time was 7 min and the value of pH was 3.

(3) Under the orthogonal optimum condition, the removal of KSCN can reach to 94.6 %.

(4) The reaction approximately obeyed first-order reaction. The reaction kinetics equation: $\ln C = -0.00207t + 1.4768$, the thermodynamics factors: R^2 = 0.9628, with the reaction rate constant $k = 0.\ 22207 \text{sec}^{-1}$ and half life $t_{1/2}$ = 335 sec.

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