

Preparation of Fe₃O₄/TiO₂ composite and its application in photocatalysis of organic pollutants

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Titanium iron ore is used as a raw material to prepare titanium dioxide and ferrous chloride by hydrochloric acid leaching method, and the titanium dioxide is coated on the magnetic Fe₃O₄ to form Fe₃O₄/TiO₂ composite. Through the design of a single variable test (temperature, added dose of Fe₃O₄ colloidal solution), eight Fe₃O₄/TiO₂ composites were prepared under different conditions. The different photocatalytic performances of the prepared Fe₃O₄/TiO₂ composites were investigated, and the effect of phosphotungstic acid addition on the performance of Fe₃O₄/TiO₂ composite was also examined. X-ray diffraction (XRD), X-ray energy dispersive spectrometry (EDX), scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FT-IR) and titration were used for characterization of the composites. In addition, recycling of the catalyst was explored. The results showed that the optimal conditions for preparation of Fe₃O₄/TiO₂ composite were: temperature below 80 °C, addition of 4 mL Fe₃O₄ colloidal solution. The prepared Fe₃O₄/TiO₂ composite had the highest removal efficiency of RhB. The photocatalysis under ultraviolet (UV) light is better than under sun light, with the highest removal rate of 98.3% and apparent rate constant *k* of 1.1 h⁻¹. TiO₂ was successfully coated on the Fe₃O₄ surface, as confirmed by EDX, SEM and FT-IR, and the Fe₃O₄/TiO₂ composite was of high purity. The addition of phosphotungstic acid did not improve the performance of the Fe₃O₄/TiO₂ composite, further study is needed.

Keywords: Ilmenite, Fe₃O₄/TiO₂ composite, Photocatalysis

INTRODUCTION

Research on nano-magnetic particles is being developed for over 30 years, owing to their advantages such as small size, strong surface plasticity and magnetism at room temperature [1-3]. The use of nano-magnetic particles coated on inorganic materials with photo-catalytic activity, such as titanium dioxide, etc., has become the focus in recent research [4-6]. Photocatalyst TiO₂ is nontoxic, harmless, less corrosive to the equipment, but the recycling process is difficult to conduct, so that preparation of Fe₃O₄/TiO₂ composite photocatalyst easy for recycling is important for the treatment of sewage [7-9]. However, there are several factors that may affect the catalytic ability of the nano-magnetic particles [10-12]. In order to degrade methyl orange, Yoon *et al.* [13] utilized TiO₂ which was surface-modified by phosphotungstic acid (H₃PW₁₂O₄₀, HPW) aqueous solution. It was found that the catalytic activity of the system was significantly improved and the improvement was attributed to the results of HPW and TiO₂ composite photoexcitation. Otherwise, Ozer *et al.* [14] reported that the HPW-modified TiO₂ has a very high degradation activity for dichlorobenzene and they attributed the increase in activity to the synergistic effect between polyoxometalates (POM) and TiO₂.

Ilmenite (FeTiO₃) is widely distributed in large amounts in China, and is mainly used for the production of titanium dioxide and artificial rutile, iron has not been well used. Thus, developing a new technology to make comprehensive utilization of the various elements in minerals has become an inevitable trend of mineral use.

In order to improve the utilization rate of ilmenite, Fe₃O₄ was prepared by the extraction of FeCl₂ and TiO₂ from ilmenite, and TiO₂ was coated on the magnetic Fe₃O₄ particles under the optimal conditions to prepare Fe₃O₄/TiO₂ composite photocatalyst. The effect of Fe₃O₄/TiO₂ composite photocatalyst on the degradation of rhodamine was studied. The photocatalytic activity of Fe₃O₄/TiO₂ composite modified by phosphotungstic acid was investigated and compared with that of the not modified composite; the recovery of the catalyst was also investigated.

EXPERIMENTAL DETAILS

Experimental apparatus and reagents

X-ray powder diffractometer (XRD, Bruker, D8-ADVANCE, Germany), scanning electron microscope (SEM, Quanta FEG 250, USA), X-ray energy dispersive spectrometer (EDX, Quanta FEG 250, USA), ultraviolet spectrophotometer (Shimadzu UV-2550PC, Japan), infrared spectrometer (Thermo Nicolet IS10, USA)

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Ilmenite (provided by Zhejiang Normal University, the chemical composition is shown in Table 1), Rhodamine B (analytical grade, Shanghai San Aisi Reagent Co., Ltd.)

Table 1. Chemical composition of the ilmenite (mass ratio %)

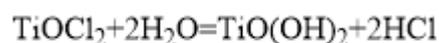
Material composition	TiO ₂	FeTiO ₃	Al ₂ O ₃	SiO ₂
Content	10.76	76.25	3.14	9.85

Preparation of catalyst

Extraction of FeCl₂ from ilmenite. Add 30 g of ilmenite and pour 90 g of concentrated hydrochloric acid (1:3 (m/m) of ilmenite and concentrated hydrochloric acid) into a 250 mL three-necked flask. The mixture was stirred in an oil bath at 80°C for 4 h to obtain a brown substance. The resulting ilmenite acid solution (filtrate) was filtered while hot and allowed to stay at room temperature for a period of time and then cooled in an ice-salt bath at -18°C for 12 h. FeCl₂ • 4H₂O crystals would precipitate. FeCl₂ • 4H₂O crystals and filtrate were separated by filtration. The main reaction equation is as follows:



Extraction of TiO₂ from ilmenite. The filtrate obtained as described above was stirred at 105°C for 5 h, poured into a beaker, cooled at room temperature for a period of time, then filtered to obtain white solid metatitanic acid. 3.0M hydrochloric acid was added to the metatitanic acid, and incubated in a water bath at 60°C for 2 h. The mixture was suction-filtered, washed with 1.0M sodium hydroxide solution, then twice with 1.0M hydrochloric acid, and finally washed three times with distilled water. The washed metatitanic acid was put into a muffle furnace at 500°C for 3 h to remove water, then TiO₂ solid was obtained. The main reaction equations are as follows:



Preparation of Fe₃O₄. Add 30 mL of distilled water to a mixture of 5.96 g of FeCl₂ • 4H₂O and 16.22 g of FeCl₃ • 6H₂O (n(Fe²⁺)/n(Fe³⁺)=1:2), heat in a water bath at 50°C, and stir to dissolve it. Then drop ammonia, quickly at first, and slowly at the end, adjust the pH of the reaction solution to 11, allow aging after the reaction, and finally wash, centrifugate the resulting product several times, and separate to get pure Fe₃O₄ colloidal solution.

Preparation and optimization of Fe₃O₄/TiO₂. A 10% TiO₂ solution was prepared and an appropriate amount of Fe₃O₄ colloidal solution was added to the solution. The reaction mixture was stirred for 2 h at a certain temperature in an oil bath. After completion of the reaction, the solid was filtered and washed with ethanol and distilled water and dried in a vacuum oven at 50°C for 3 h [11].

Four temperatures (40 °C, 60 °C, 80 °C and 100 °C) and different amounts of Fe₃O₄ colloidal solution (4 mL, 6 mL and 8 mL), modified or not by phosphotungstic acid, were selected to explore the optimal preparation conditions of the composite. Fe₃O₄/TiO₂ composites were labeled as catalysts No. 1-8 according to the different preparation conditions (see Table 2).

Table 2. Preparation of Fe₃O₄/TiO₂ composite under different conditions

No.	Reaction temperature (°C)	Amount of Fe ₃ O ₄ colloidal solution (mL)
1#	60	4
2#	40	4
3#	80	4
4#	100	4
5#	80	4
6#	80	6
7#	80	8
8# (+HPW)	80	4

Characterization of Fe₃O₄/TiO₂ composite

X-ray diffraction (XRD) analysis. The experimental conditions for XRD were: copper target, tube voltage 40 kV, tube current 40 mA, scanning step size 0.02 degrees, scanning angle 10-80°.

Fourier transform infrared spectroscopy (FT-IR). In this experiment, the sample preparation method for FT-IR was: mix the sample evenly with KBr of spectral purity in a ratio of 1: 100 before test.

Scanning electron microscopy (SEM) analysis. The conditions for this experiment were as follows: measured voltage of 15 kV, the sample should be tested after drying treatment.

X-ray energy dispersive spectrometry (EDX) analysis. Each element has its own X-ray characteristic wavelength, the magnitude of the characteristic wavelength depends on the energy released during the energy level transition energy ΔE. In this experiment, EDX was combined with SEM for analysis of the surface composition of the sample.

Photocatalytic properties of Fe₃O₄/TiO₂ composite

Photocatalytic properties. Add 50 mL of the 1×10⁻⁵M rhodamine B solution (shown in Figure 1)

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to 50, 100, 150 and 200 mg of the prepared catalyst, respectively, use ultrasound dispersion, allow adsorption equilibrium to be reached in a dark room for 30 min, exposed to ultraviolet light or solar light, respectively. The samples were taken every half hour and the supernatant was analysed after centrifugation. The absorbance was measured at 554 nm by an ultraviolet-visible spectrophotometer, and the changes in RhB concentration were observed. The photodegradation rate (Y) was calculated by the following formula:

$$Y = \frac{A_0 - A}{A_0} \times 100\% = \frac{C_0 - C}{C_0} \times 100\%$$

where C₀ is the initial concentration of RhB, C is the concentration of RhB at a certain point in the course of catalytic reaction, A₀ is the absorbance of RhB with concentration of C₀, and A is the absorbance of RhB of concentration C.

Dynamics of the photocatalytic degradation of rhodamine by Fe₃O₄/TiO₂ composite was studied and the photocatalytic activity of the catalyst was evaluated by the apparent rate constant k. According to the results, the effect of catalyst dosage on the degradation of rhodamine B was compared, the optimal dosage was selected and the effects of ultraviolet light (254 nm) and sunlight on photocatalytic degradation of rhodamine were compared.

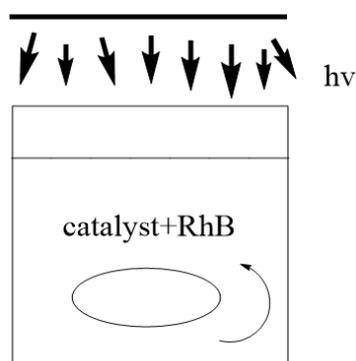


Fig. 1. Schematic diagram of photocatalytic reaction

Catalyst recovery application

The Fe₃O₄/TiO₂ composite in the rhodamine B solution was allowed to settle down by a permanent magnet base, and the upper layer solution was removed. The remaining Fe₃O₄/TiO₂ composite was washed with acetone and deionized water and vacuum-dried for recovery.

RESULTS AND DISCUSSION

Preparation and characterization of Fe₃O₄/TiO₂ composite photocatalyst

In this paper, the preparation of Fe₃O₄/TiO₂ composite was based on the extraction of FeCl₂ and

TiO₂ from ilmenite. XRD, FT-IR, SEM and EXD were used to characterize the structure, surface functional groups, composition and morphology of the photocatalyst.

The 1# and 3# Fe₃O₄/TiO₂ composites displayed better photocatalytic properties. The XRD spectrum is shown in Figure 2.

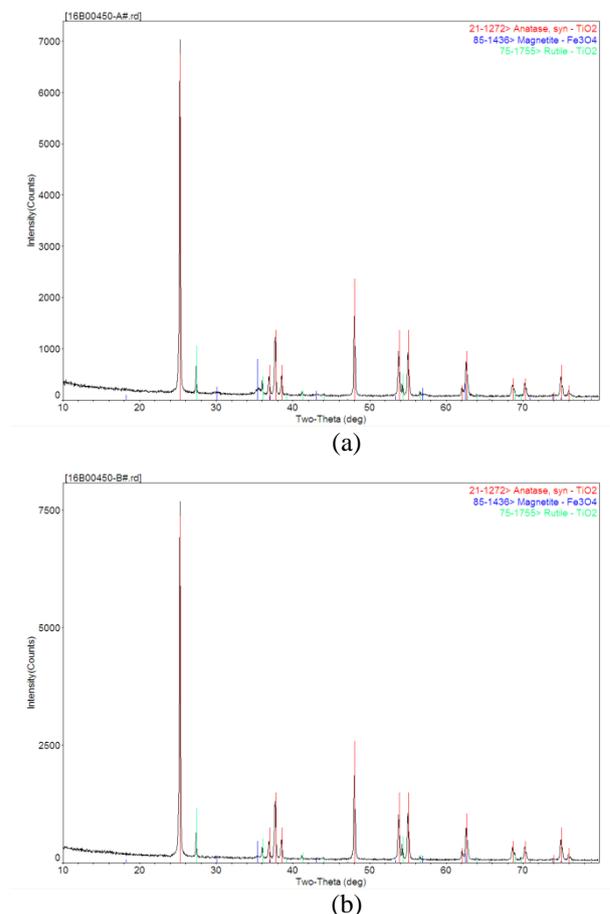


Fig. 2. XRD spectrum of 1# (a) and 3# (b) of Fe₃O₄/TiO₂ composite

Several main diffraction peaks marked by red lines correspond to (101), (004), (200), (105), (211), (116) crystal surfaces of TiO₂. The characteristic peak values agree well with the data of the standard card (JCPDS) NO.21-1272. Strong diffraction of TiO₂ corresponding to (101), (004) and (200) appeared, which confirmed that the of 1# and 3# catalysts include anatase-TiO₂. There are several diffractive peaks at 2θ=30.3°, 35.7°, 53.8°, 57.2° and 62.9° corresponding to the characteristics of FCC (face centered-cubic) Fe₃O₄ crystal (220), (311), (400), (422), (511) and (400). It means that both 1# and 3# catalysts contain Fe₃O₄ particles belonging to cubic crystal. There is no other impurity peak in the picture, which shows that the product is pure. There are several sharp diffractive peaks at 2θ=27.4°, 36.2°, 41.4° and 54.3°. They correspond to (101), (004), (200), (105), (211), (116) crystal surfaces of TiO₂. The characteristic

peak values agree well with the data of the standard card (JCPDS) NO.75-1755., which confirmed that the 1# and 3# catalysts both include rutile-TiO₂. The results showed that the 1# and 3# catalysts are composed of face-centered cubic Fe₃O₄, rutile-TiO₂ and anatase-TiO₂.

The infrared spectrum of Fe₃O₄/TiO₂ composite is shown in Figure 3. The position of the characteristic absorption peak is basically consistent with the report [15,16]. The absorption peak at 3416 cm⁻¹ corresponds to the stretching vibration peak of a hydroxyl group on the surface of the Fe₃O₄ particles. The peak at 1637 cm⁻¹ is the bending vibration between the H-O-H bonds. This was attributed to the absorption of water molecules on the surface of Fe₃O₄/TiO₂ magnetic composite, which was related to the small amount of adsorbed water in the composite. The absorption peak near 638 cm⁻¹ is caused by the stretching vibration of crystal TiO₂ and the Ti-O bond on the surface.

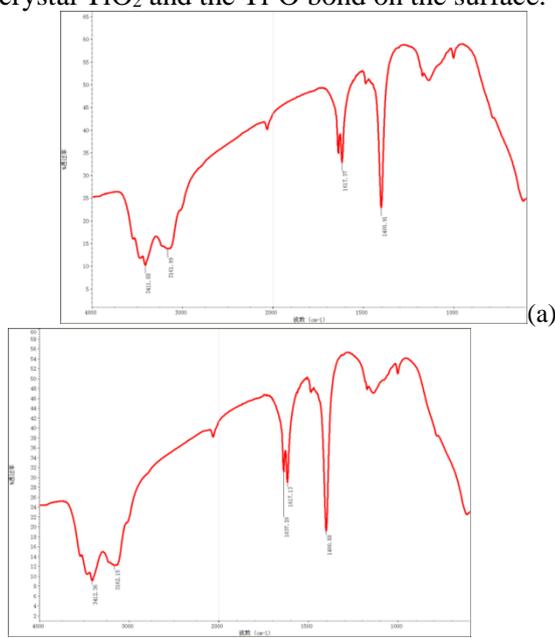


Fig. 3. FT-IR spectra of 1# (a) and 3# (b) of Fe₃O₄/TiO₂ composite

Figure 4 shows the SEM spectra of Fe₃O₄/TiO₂ composite at different magnifications. The SEM spectrum of 1# of Fe₃O₄/TiO₂ composite is presented in Figs. 4(a)-(d). The conditions for preparation of 1# were: 60°C with addition of 4 mL of Fe₃O₄ colloidal solution and reaction time 2 h. In Figs. 4(e)-(h) the SEM spectrum of 3# of Fe₃O₄/TiO₂ composite is shown. The conditions for preparation of 3# were: 80°C with addition of 4 mL of Fe₃O₄ colloidal solution and reaction time 2 h. Figure 4 shows that the prepared 1# and 3# of Fe₃O₄/TiO₂ composite have cluster structure. The magnetic properties of Fe₃O₄ make the Fe₃O₄/TiO₂ composite agglomerate together. The particle

diameters of the composite are about 50-120 nm. There are small particles on the surface, which should be of TiO₂ coated on the surface of Fe₃O₄. The particle diameters of Fe₃O₄ are about 25-40 nm, and of the TiO₂ nanoparticles - about 5 nm. Judging from the particle size, there are 1-3 layers of nano-TiO₂ coated on the surface of the magnetic Fe₃O₄[1]. The surface composition of the sample was analyzed by both EDX (Figure 5) and SEM.

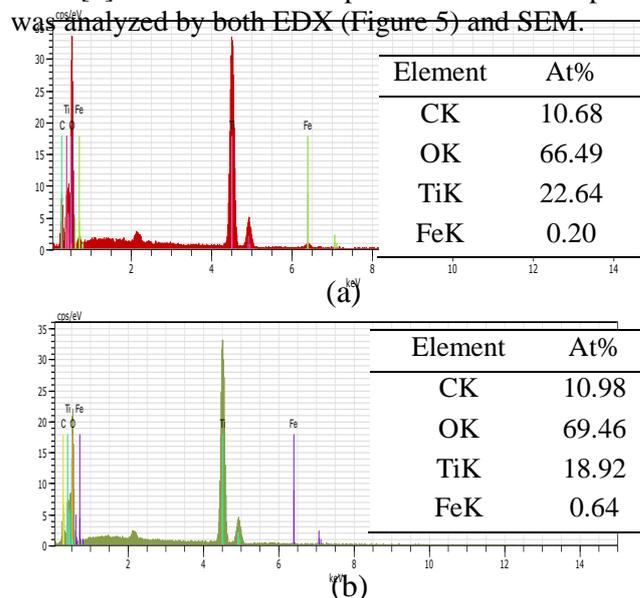


Fig. 5. EDX spectrum of 1# (a) and 3# (b) of Fe₃O₄/TiO₂ composite

Figure 5 shows the composition of both 1#(a) and 3#(b) catalysts. The major components of the surface of both catalysts are O, Ti and Fe. Among them, C is graphite during sample analysis and the content of Ti atoms is much higher than of Fe, which indicated that TiO₂ in 1# and 3# is situated on the Fe₃O₄ surface.

Effect of Fe₃O₄/TiO₂ addition on removal efficiency of RhB

Samples of 50, 100, 150 and 200 mg of the prepared catalyst were added to 50 mL of RhB solution of initial concentration of 1×10⁻⁵M and were subjected to photocatalytic experiments under sunlight. The results are shown in Figure 6.

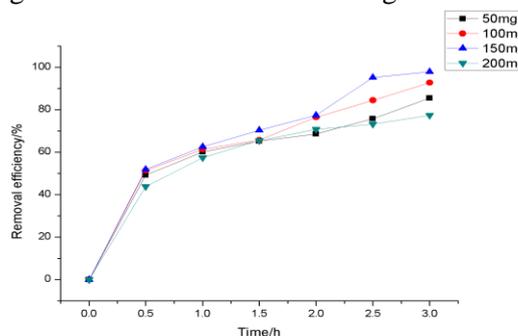


Fig. 6. Effect of Fe₃O₄/TiO₂ composite addition on removal efficiency of RhB.

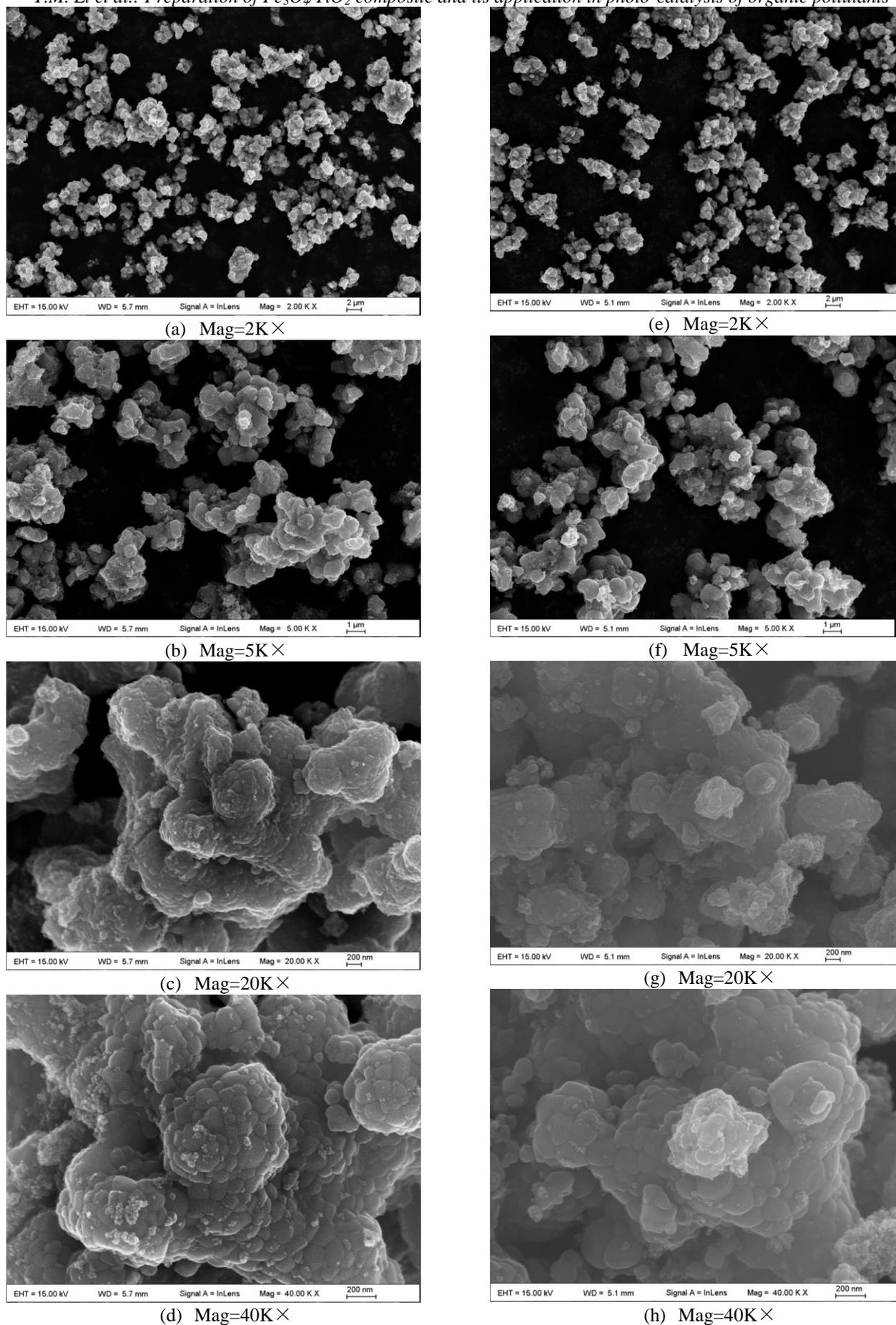


Fig. 4. SEM spectrum of 1# (a-d) and 3# (e-h) of Fe_3O_4/TiO_2 composite

The best addition of catalyst is 150 mg. After 3 h of photocatalytic reaction, the removal efficiency of RhB was 98.1% by adding 150 mg of catalyst. The removal efficiency of RhB was 75.9% and 85.6% with 200 mg and 50 mg of catalyst, respectively.

Effect of preparation temperature for Fe_3O_4/TiO_2 on removal efficiency of RhB

The effects of different preparation temperatures (40°C, 60°C, 80°C and 100°C) of the Fe_3O_4/TiO_2 catalysts on the removal efficiency of RhB were investigated under UV and sunlight. The results are shown in Figure 7. The results showed that the highest degradation efficiency of Fe_3O_4/TiO_2 catalyst prepared at 80°C is 92.2% and 89.0%, under UV light and sunlight, respectively. The degradation efficiency of Fe_3O_4/TiO_2 catalyst

prepared at 100°C was 75.9% and 72.8%, respectively.

Effect of Fe_3O_4 addition for Fe_3O_4/TiO_2 composite on removal efficiency of RhB

The effect of Fe_3O_4 colloidal solution (4 mL, 6 mL and 8 mL) for Fe_3O_4/TiO_2 catalyst on the removal efficiency of RhB was also investigated under UV and sunlight. The results are shown in Figure 8. The highest degradation efficiency of RhB by the Fe_3O_4/TiO_2 catalyst was achieved by adding 4 mL of Fe_3O_4 colloidal solution, with 98.1% and 98.3%, respectively, under UV light and sunlight. The degradation efficiency of the Fe_3O_4/TiO_2 catalyst prepared by adding 8 mL of Fe_3O_4 colloidal solution was 80.8% and 76.9%, respectively. Therefore, addition of 4 mL of Fe_3O_4 colloidal solution was considered as optimal.

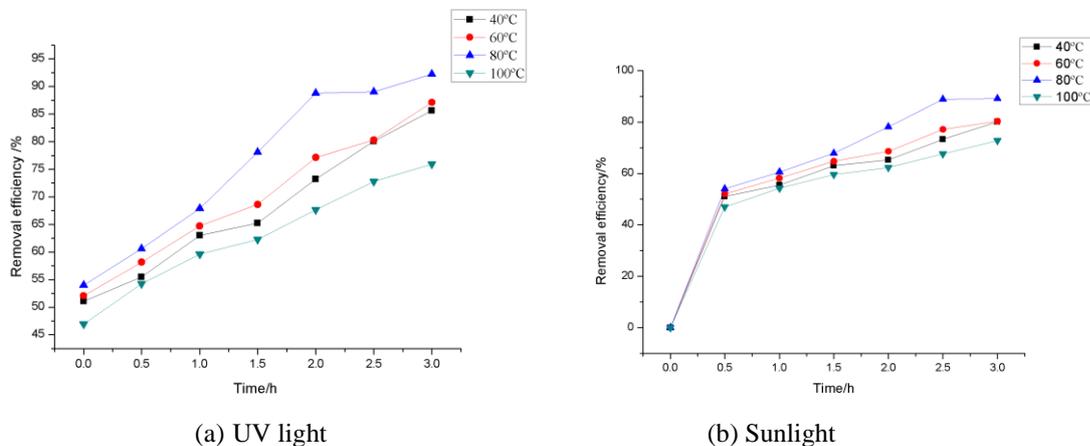


Fig. 7. Effects of preparation temperature of Fe_3O_4/TiO_2 composite on removal efficiency of RhB under (a) UV light and (b) sunlight

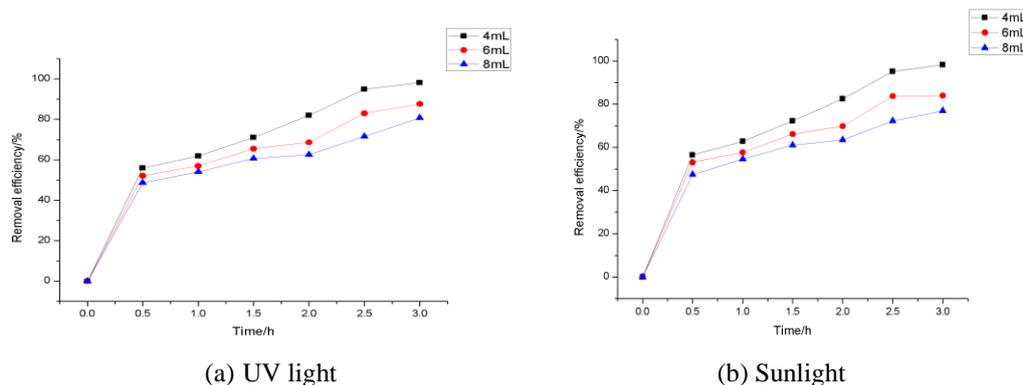


Fig. 8. Effects of Fe_3O_4 addition for Fe_3O_4/TiO_2 composite on removal efficiency of RhB under (a) UV light and (b) sunlight

Effect of light source for Fe_3O_4/TiO_2 composite on removal efficiency of RhB

The effects of ultraviolet light and sunlight for Fe_3O_4/TiO_2 catalyst on the removal efficiency of RhB were compared. Fe_3O_4/TiO_2 catalyst was

prepared by the optimal conditions. The average of the degradation efficiency is shown in Figure 9. The photocatalysis under ultraviolet (UV) is better than under sun light, 95.1% and 93.7%, respectively, with the highest removal rate of 98.1% and 98.3%, respectively.

The effect of HPW-modified catalyst on the removal efficiency of RhB was investigated by using HPW impregnation method to improve Fe₃O₄/TiO₂ composite photocatalyst. The results are shown in Figure 10. The degradation efficiency of Fe₃O₄/TiO₂ catalyst prepared by adding HPW did not improve the performance of the Fe₃O₄/TiO₂ composite, which was 26.5% and 23.8%, respectively, much lower than those under the optimal conditions. Further study is needed to reveal the mechanism.

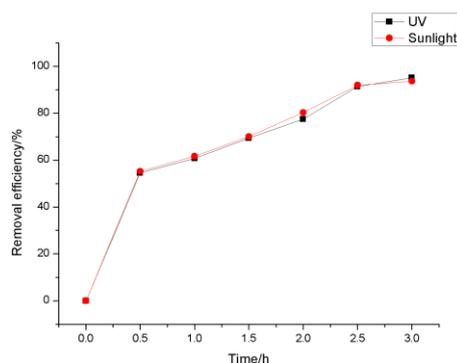


Fig. 9. Effects of light source on removal efficiency of RhB by Fe₃O₄/TiO₂ composite under UV light and sunlight

Photocatalytic degradation of RhB by Fe₃O₄/TiO₂

Ozer *et al.* [16] reported that for the POM composite photocatalyst, the photocatalytic oxidation reaction follows the Langmuir-Hinshelwood (L-H) kinetic equation:

$$r = -\frac{dC}{dt} = \frac{k_r K_a C_0}{1 + K_a C_t}$$

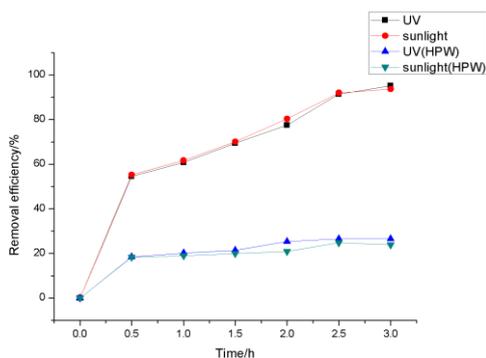


Fig. 10. Effects of phosphotungstic acid addition to Fe₃O₄/TiO₂ composite on removal efficiency of RhB under UV light and sunlight

In the equation, *r* is the initial apparent reaction rate (mg·L⁻¹·h⁻¹), *C_t* is the concentration of reactants at time *t* (mg·L⁻¹), *C₀* is the initial concentration of the reactants (mg·L⁻¹), *K_a* is the L-H equilibrium adsorption constant (L·mg⁻¹), *K_r* is the reaction rate

constant (mg·L⁻¹·h⁻¹). The degradation reaction was of first order and when *KC₀* << 1, the kinetic equation can be simplified as follows:

$$\ln \frac{C_0}{C} = k_r K_a t = kt$$

In the equation, *K* (h⁻¹) is the apparent first-order reaction rate constant.

In this experiment, 150 mg addition of Fe₃O₄/TiO₂ catalyst was used to study the reaction kinetics as a function of reactant concentration. The activity difference of the catalyst prepared under different conditions was compared by calculating the apparent rate constant *k*. The results are shown in Table 3. Six different concentrations of RhB standard solution were prepared. The standard curve was as shown in Figure 11, the correlation coefficient was 0.9982.

The data in Table 3 show that the apparent rate constants of the 3 # catalyst on the photodegradation process of RhB are the largest ones, 1.0646 and 1.0851 h⁻¹, respectively, whether under ultraviolet light or sunlight. 3 # catalyst was prepared at 80 °C, by adding 4mL of Fe₃O₄ colloidal solution, consistent with the best conditions.

Table 3. Comparison of apparent rate constants (*k*) for Fe₃O₄/TiO₂ composite under different light source

	1#	2#	3#	4#
UV light	0.5369	0.5019	1.0646	0.3900
Sunlight	0.3036	0.4344	1.0851	0.3026
	5#	6#	7#	8#
UV light	0.7256	0.5908	0.4373	0.0710
Sunlight	0.6619	0.5448	0.4081	0.3053

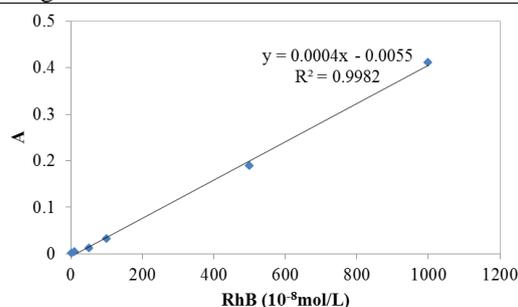


Fig. 11. Standard curve for RhB

Table 4. Effects of Fe₃O₄/TiO₂ composite recycling on removal efficiency of RhB

	First recycling	Second recycling	Third recycling
Removal efficiency	90%	81%	75%

Recovery of Fe₃O₄/TiO₂ composite catalyst

The effect of Fe₃O₄/TiO₂ composite recycling on removal efficiency was also investigated. It was found that the removal efficiency decreased from

98% to 75% after 3 cycles of recovery (Table 4). The best recycling strategy should include 2 cycles.

CONCLUSIONS

The average efficiency of degradation of RhB by Fe₃O₄/TiO₂ composite prepared from ilmenite under UV light is better than under sunlight, and the apparent rate constant *k* of 1.1 h⁻¹. The amount of catalyst, the preparation temperature of the catalyst and the amount of Fe₃O₄ colloidal solution for the preparation of the catalyst, all have effects on the photodegradability. The optimal conditions for preparation of Fe₃O₄/TiO₂ composite are: 80°C, with addition of 4 mL of Fe₃O₄ colloidal solution. The addition of phosphotungstic acid did not improve the performance of the Fe₃O₄/TiO₂ composite.

TiO₂ was successfully coated on the Fe₃O₄ surface as confirmed by EDX, SEM and FT-IR, and the Fe₃O₄/TiO₂ composite was of high purity. It is easy to use the magnetic property to recycle, the best recycling strategy should be 2 cycles, with degradation efficiency up to 81%.

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Получаване на Fe₃O₄/TiO₂ композит и приложението му за фотокатализ на органични замърсители

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(Резюме)

Титаново-желязна руда е използвана като суровина за получаване на титанов диоксид и железен (II) оксид чрез излужване със солна киселина. Титановият диоксид е нанесен върху магнитния Fe₃O₄ с образуване на Fe₃O₄/TiO₂ композит. Осем Fe₃O₄/TiO₂ композита са получени при различна температура и количество на колоидния разтвор на Fe₃O₄. Изследвани са фотокаталитичните свойства на композитите, както и влиянието на добавка от фосфофолфрамова киселина. Композитите са охарактеризирани чрез рентгенова дифракция (XRD), енергийно дисперсивна рентгенова спектроскопия (EDX), сканираща електронна микроскопия (SEM), Fourier трансформираща инфрачервена спектроскопия (FT-IR) и титруване. Изследвани са възможностите за рециклиране на катализатора. Оптималните условия за получаване на Fe₃O₄/TiO₂ композит са: температура под 80°C и добавка от 4 mL Fe₃O₄ колоиден разтвор. Така полученият Fe₃O₄/TiO₂ композит има най-висока ефективност за отстраняване на родамин В. Фотокатализът е по-ефективен при облъчване с UV светлина, отколкото със слънчева светлина. Максималната степен на извличане е 98.3%, а привидната скоростна константа *k* е 1.1 h⁻¹. TiO₂ е нанесен успешно на повърхността на Fe₃O₄, което е потвърдено чрез EDX, SEM и FT-IR, като Fe₃O₄/TiO₂ композитът е с висока чистота. Добавянето на фосфофолфрамова киселина не води до подобряване на неговите свойства.