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Investigation on the degradation of acid fuchsin induced oxidation by $MgFe_2O_4$ under microwave irradiation

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ABSTRACT

The degradation of acid fuchsin solutions under microwave irradiation (MW) combined with the $MgFe_2O_4$ was studied in this work. The degradation process was investigated by UV–vis, FT-IR, TOC and ion chromatogram techniques. The results indicated that the degradation percentage of acid fuchsin could reach up to 99.78%, corresponding to 91.2% of TOC removal, and acid fuchsin had been degraded into harmless products under microwave irradiation time 1.5 min. The effects of pH, addition amount of catalysts on the efficiency of the degradation have been investigated. The mechanism of acid fuchsin degradation was also discussed. With the advantages of low cost and rapid processing, this novel MW/$MgFe_2O_4$ composite could gain promising application in the treatment of various dyestuff wastewaters on a large scale.

1. Introduction

Dyes and pigments are often used for imparting color to industrial products. There are approximately 10,000 different industrial dyes and pigments, and over 7×10^5 tons of these dyes are annually produced worldwide. Consequently, it is inevitable that dyes and pigments will be released into wastewater from various industrial branches. Dyes usually have complex aromatic molecular structures which make them more stable and more difficult to degrade. If not treated, these materials also induce certain health hazards and environmental pollution. Therefore, the treatment of various dye wastewaters has been regarded to be one of the main aims in controlling environmental pollution all over the world.

Many investigators have presented some methods for the removal of pollutants from wastewater, such as biological decoloration [1,2], chemical oxidations, adsorption [3–8], the photocatalytic degradation [9,10], the ultrasonic degradation [11,12], and the advanced oxidation process with photo-Fenton reaction [13,14]. But many problems were not avoided, such as enhanced foam formation and retarded biodegradation of the accompanying pollutants [15]. Using conventional techniques to eliminate the dyes may be difficult as they are especially refractory to oxidants. Adsorption methods cannot degrade pollutants but just transfer their phase [16]. The applications of the photocatalytic degradation and the ultrasonic degradation are also limited due to the related expensive operating costs and high energy requirements. Therefore, it is necessary to explore an effective process for the abatement of pollutants.

One of the most promising technologies is to utilize microwave-enhanced advanced oxidation procedure for the degradation of organic pollutants. Microwave irradiation technology has already been applied to industry, family, medical science and environmental organic pollution for polycyclic aromatic hydrocarbons [17]. Using solid materials as catalysts is of great interest and important in purifying waste waters containing phenol. Activated carbon [18,19], ferromagnetic metals [20–22], and the transition metal oxides [23–26] have been proved to be active in the catalytic reactions of the degradation of phenol, its derivatives and dyes.

In previous work, we have succeeded in developing the microwave enhanced catalytic degradation methods [27,28] by which malachite green and brilliant green can be completely degraded into harmless products within 2 min. So the microwave induced catalytic degradation method should be an efficient process for the treatment of dye compounds.

Recently, we have synthesized $MgFe_2O_4$ with the assistance of microwave irradiation. $MgFe_2O_4$ as magnetic particles can be easily separated and recovered by magnetic separation technology. Moreover, we also found that $MgFe_2O_4$ could quickly adsorb dyes and have high catalytic activity and adsorption capacity.

Based on the above reasons, the combination of the microwave irradiation technology and $MgFe_2O_4$ can promote the degradation of acid fuchsin and might be a possible path for practical application in the future.

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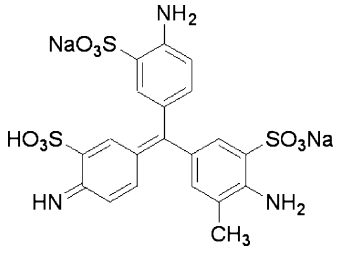
2. Experimental

2.1. Reagents and instruments

The initial concentration of acid fuchsin solution is 50 mg L^{-1}. $Fe(NO_3)_3$, $Mg(NO_3)_2$ and NaOH (2.5 mol L^{-1}) are of analytical grade (purchased from Shanghai Xinzhong Chemical Reagent Co., China). FT-IR spectra of $MgFe_2O_4$ were measured using FT-IR 5700 (Nicolet Company, USA). The X-ray diffraction (XRD) patterns of $MgFe_2O_4$ powders were recorded on Siemens D5000 Diffractometer (Germany). Thermal gravimetric analysis (TG/DTG) was carried out using a TA SDT Q600 TG system (USA), the rate of heating was maintained at 10 °C min^{-1}. The acid fuchsin solution was irradiated using NJL07-3 model MW apparatus (Jiequan Equipment Ltd., China), and MW output power ranged from 100 to 900 W. The pH of the acid fuchsin solution was measured by S-3C model pH-meter (China). Cary 5000 UV–vis–NIR (Varian, USA) was used to check the degradation efficiency of acid fuchsin. Total organic carbon (TOC) in the solutions was measured with a TOC-VSCN analyzer (Shimadzu Co., Japan) to investigate the mineralization degree of acid fuchsin. Ion chromatography (Dionex Company, USA) was used to inspect the degradation process of acid fuchsin. Molecular structure of acid fuchsin is as follows:



2.2. Preparation of $MgFe_2O_4$

$MgFe_2O_4$ was synthesized by chemical co-precipitation coupled with microwave method in an aqueous solution. The preparation of $MgFe_2O_4$ was performed as follows: some $Mg(NO_3)_2$ and $Fe(NO_3)_3$ salts with a molar ratio of 1:2 were dissolved in 200 mL deionized water. 2.5 mol L^{-1} NaOH was added dropwise to the solution until its pH around 11. A mixed solution was irradiated under microwave with output power of 200 W for 10 min. The precipitate was filtered, washed with deionized water, and dried in a muffle furnace at 110 °C for 24 h. Then as-prepared sample $MgFe_2O_4$ was calcined at 200, 300, 400, 500 and 600 °C for 3 h, respectively.

2.3. Adsorption experiment

The adsorption experiments were carried out using a series of 50 mL flasks containing 0.8% of $MgFe_2O_4$ and 25.0 mL of 50.0 mg L^{-1} acid fuchsin solution. After ultrasonic dispersion for 1 min, and the solutions were shaken, then the solid/liquid phases were separated by centrifuging at 3000 rpm for 5 min. The suspension was immediately analyzed for the determination of acid fuchsin concentration. The adsorption percentage (*Ads.*%) was calculated based on the following equation:

$$Ads.\% = \frac{C_0 - C_e}{C_0} \times 100 \quad (1)$$

where C_0 and C_e are the initial and the equilibrium concentration (mg L^{-1}) of acid fuchsin in solution, respectively.

Adsorption isotherm studies were carried out with initial concentrations of acid fuchsin varying between 300 and 1000 mg L^{-1}, the sorbent amount was kept constant and three different temperatures were applied (298, 313 and 333 K). The equilibrium adsorption capacity was calculated using the following expression:

$$q = \frac{V \times (C_0 - C_e)}{m} \quad (2)$$

where q is the adsorption capacity of $MgFe_2O_4$ (mg g^{-1}); V is the volume of the solution (L); m is the mass of added sorbent (g).

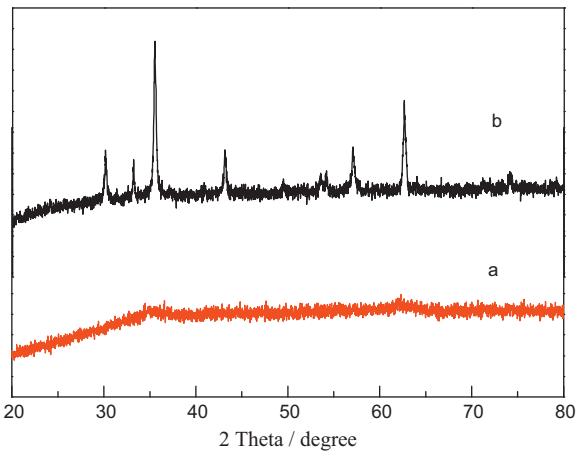


Fig. 1. XRD spectra of $MgFe_2O_4$: (a) 300 °C; (b) 500 °C.

2.4. Procedure

25 mL of acid fuchsin solution (50 mg L^{-1}) and 0.08% of $MgFe_2O_4$ were put into a glass reactor, and treated using the following methods: including MW method, MW combined with $MgFe_2O_4$ and $MgFe_2O_4$ (without MW), respectively. The output power of MW varied from 100 to 900 W. The samples were taken out periodically for the analysis of acid fuchsin degradation efficiency.

All UV–vis absorption spectra of acid fuchsin solutions were recorded in the wavelength range from 190 to 800 nm. The maximal absorbencies (at 546 nm) of 0–50 mg L^{-1} acid fuchsin solutions abide Lambert–Beer's law. The calibration curve of standard acid fuchsin solutions was used to estimate the degradation efficiency of acid fuchsin. The degradation percentage was calculated with the equation:

$$\text{Degradation percentage } (\%) = \frac{C_0 - C}{C_0} \times 100 \quad (3)$$

where C_0 is the initial concentration of acid fuchsin (mg L^{-1}) and C the concentration of acid fuchsin at time t.

In order to check up the mineralized degree of acid fuchsin, after MW/$MgFe_2O_4$ treatment, the inorganic ions of the solutions were determined by ion chromatography. The determination conditions of ionic chromatography were as follows: AS23 [Dionex] column (250 mm × 4 mm), 4.5 mmol L^{-1} Na_2CO_3/0.8 mmol L^{-1} $NaHCO_3$ eluent, 1.0 mL min^{-1} flow rate.

The total organic carbon (TOC) value of the acid fuchsin solutions was determined for investigating the degradation process of acid fuchsin after MW/$MgFe_2O_4$.

3. Results and discussion

3.1. Characterization of the prepared $MgFe_2O_4$

The XRD analysis on the prepared $MgFe_2O_4$ catalyst was carried out. It was found that diffraction peaks increased with calcining temperature increasing from 300 to 500 °C, but kept unchanged above 500 °C. So Fig. 1 only presents the XRD patterns of $MgFe_2O_4$ prepared at 300 and 500 °C. When the calcining temperature was

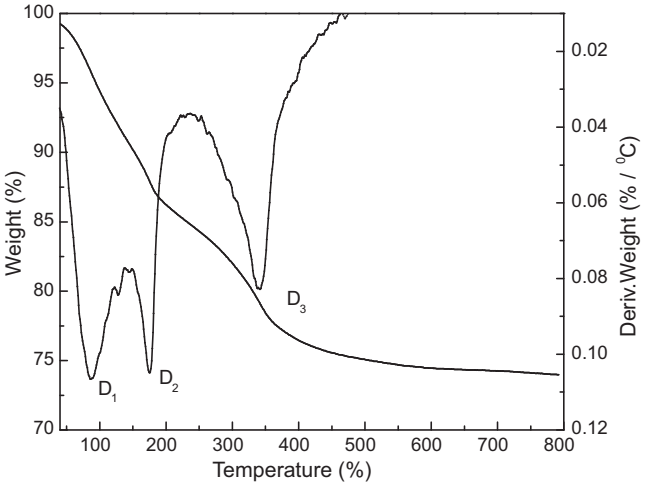


Fig. 2. TG/DTG profiles of the $MgFe_2O_4$ catalyst in a dynamic nitrogen environment.

above 500 °C, the spinel structure of $MgFe_2O_4$ was formed and displayed a better crystalline state. Moreover, it was found that $MgFe_2O_4$ particles prepared at 500 °C had the highest degradation ability on acid fuchsin.

Fig. 2 is the TG/DTG curves for the thermal decomposition of $MgFe_2O_4$ under nitrogen flow (100 mL min^{-1}). Three weight loss steps (assigned as D_1, D_2 and D_3) are observed on the TG curve, the DTG curve shows that the rapid weigh loss rate appeared in the range of 70–180 °C. Below 100 °C, the rapid weight loss (D_1 step) should be attributed to the desorbing of water from $MgFe_2O_4$ surface in heating process. The dehydration (removal hydroxy) from the process in which MgO and Fe_2O_3 were transferred into $MgFe_2O_4$ is accompanied in D_2 and D_3 steps. Above 500 °C, no obvious weigh loss occurred. The test results are in accord with the XRD results.

3.2. The UV–vis absorption spectra of acid fuchsin

In order to compare the degradation efficiency, the UV–vis absorption spectra of acid fuchsin solutions were recorded, and shown in Fig. 3. It could be seen from Fig. 3 that absorption peaks of acid fuchsin solution declined rapidly with MW combined with $MgFe_2O_4$ system (curve d). And the degradation efficiency for acid fuchsin was nearly 99.8% with MW/$MgFe_2O_4$ system within 1.5 min. It could also be found that the absorption peak (curve b) went down slightly under onefold microwave irradiation, which meant that only a few of acid fuchsin molecules were degraded, and the corresponding degradation ratio was about 12.4%. It can be concluded that there are synergistic effects of MW and $MgFe_2O_4$ on the degradation of acid fuchsin. Moreover, it could be also seen from Fig. 3 that the percentage of acid fuchsin adsorbed by $MgFe_2O_4$ exceeds 86% after 5 min. Although the removal ratio of acid fuchsin by $MgFe_2O_4$ is very high in short time, the only using adsorption method cannot degrade acid fuchsin into simple inorganic ions but just transfer their phase.

The degradation of acid fuchsin was also investigated with traditional heating as a reference. The test results show that acid fuchsin is a stable chemical compound, and cannot be decomposed at 100 °C (30 min). After traditional heating for 30 min, the absorption spectrum of acid fuchsin solution was also presented in Fig. 3 (complete overlapping with curve a).

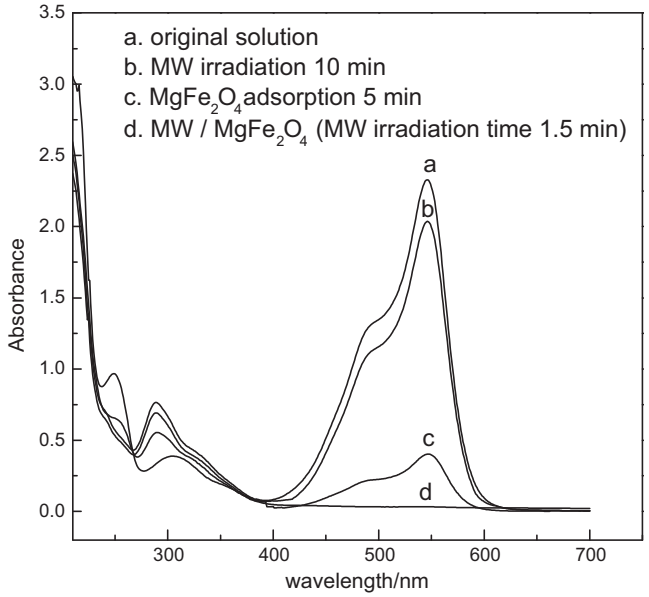


Fig. 3. The UV absorption spectrogram of 25 mL of 50 mg L^{-1} acid fuchsin solution; output power 300 W; 0.08% of $MgFe_2O_4$; (a) original solution (25 °C); (b) MW (MW irradiation time 10 min); (c) $MgFe_2O_4$ (adsorption time 5 min, 25 °C); (d) MW with $MgFe_2O_4$ (MW irradiation time 1.5 min).

3.3. Effect of microwave irradiation time on degradation of acid fuchsin

As shown in Fig. 4, the influence of microwave irradiation time on the degradation of acid fuchsin from 30 s to 10 min was reviewed in details. Nearly 100% degradation efficiency of acid fuchsin was achieved after 1.5 min in MW/$ZnFe_2O_4$ system. However, only 12% could be achieved within 10 min in MW system. Therefore, the MW/$MgFe_2O_4$ system has stronger degradation ability to acid fuchsin dye.

In addition, in order to infer the reaction kinetics of degradation process of acid fuchsin, curve fitting was performed by pseudo first-order and pseudo second-order reaction models based on the experimental data, respectively. The results showed that all calculated values deviated from linearity. Because of the disturbances of the degradation of the intermediates and the adsorption of acid

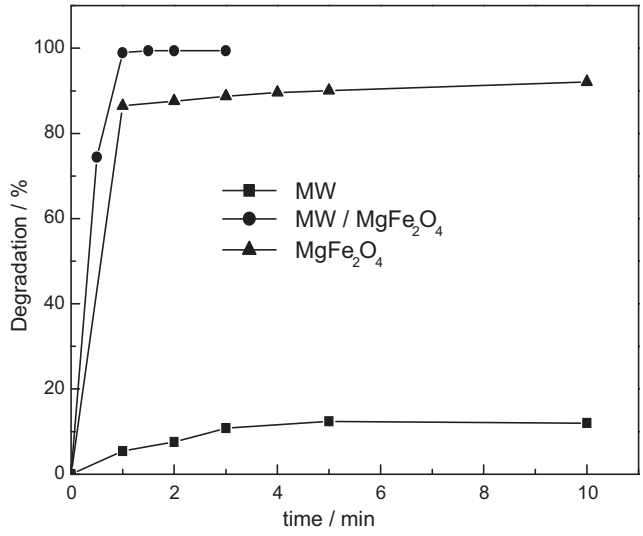


Fig. 4. Influence of acting times on degradation ratio; out power 300 W; 0.08% of $MgFe_2O_4$.

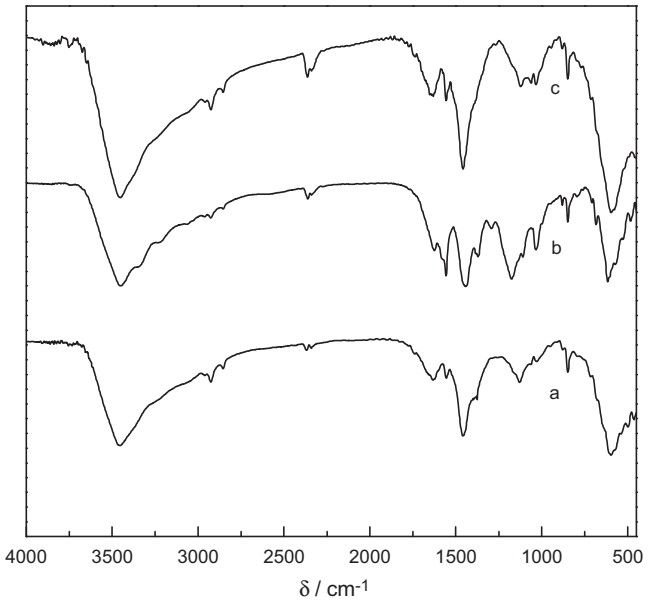


Fig. 5. The IR spectra of $MgFe_2O_4$; (a) $MgFe_2O_4$; (b) $MgFe_2O_4$ after acid fuchsin adsorption; (c) $MgFe_2O_4$ after MW degradation.

fuchsin on $MgFe_2O_4$, it is difficult to infer the exact degradation reaction kinetics.

3.4. FT-IR spectra of acid fuchsin on the surface of $MgFe_2O_4$ powder

To further track the degradation behaviors of acid fuchsin, the FT-IR spectra of $MgFe_2O_4$ were recorded (Fig. 5). It was found that the wave number for the main acid fuchsin function group is between 1750 and 750 cm^{-1}. It could be seen from Fig. 5 that the main function groups of acid fuchsin disappeared after MW degradation, demonstrating that acid fuchsin could be adsorbed by $MgFe_2O_4$ and degraded by MW. Based on the analysis of the above UV–vis and IR spectra, it can be concluded that only acid fuchsin molecules adsorbed on the surface of $MgFe_2O_4$, but also those in the solution were degraded by MW.

3.5. Adsorption property of $MgFe_2O_4$

Adsorption isotherms describe how solutes interact with sorbents. The equilibrium adsorption amount of acid fuchsin on $MgFe_2O_4$ as a function of the equilibrium concentration of acid fuchsin is depicted in Fig. 6. An increased adsorption is observed for acid fuchsin until saturation is attained.

Equilibrium sorption isotherms are often described by the Langmuir model:

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{bq_m} \quad (4)$$

where q_m is the maximum monolayer adsorption (mg g^{-1}), C_e is the equilibrium concentration of acid fuchsin, q_e is the amount of acid fuchsin adsorbed per unit weight of $MgFe_2O_4$ at equilibrium concentration (mg g^{-1}) and b is the Langmuir constant related to the affinity of binding sites (L mg^{-1}).

The Langmuir isothermal constants were determined from the plots of C_e/q_e against C_e at 298, 313 and 333 K, respectively. The obtained isothermal constants and the correlation coefficients are listed in Table 1. It is found that the adsorption of acid fuchsin on $MgFe_2O_4$ correlates well ($r > 0.99$) with the Langmuir equation in the concentration range studied. The maximum adsorption capacity of acid fuchsin on $MgFe_2O_4$ is 1.39, 1.41, and 1.43 g g^{-1} at 298, 313 and 333 K, respectively, indicating that $MgFe_2O_4$ as catalyst has very high adsorption capacity.

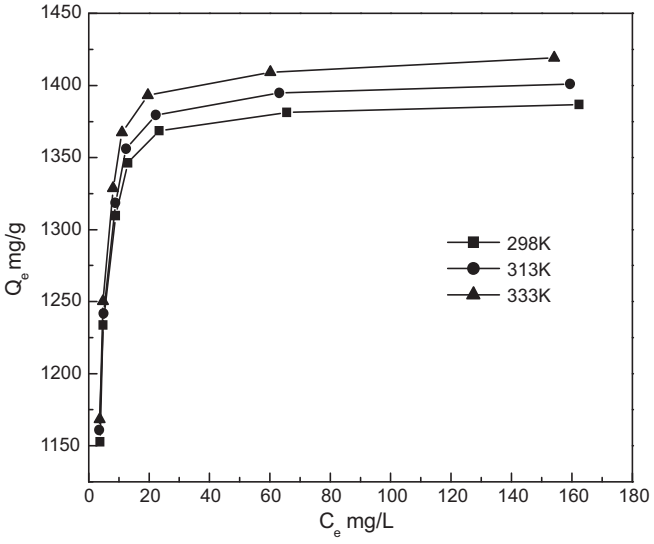


Fig. 6. Isotherm of acid fuchsin adsorption on $MgFe_2O_4$ at different temperatures (298 K, 313 K and 333 K); 0.08% of $MgFe_2O_4$; the initial acid fuchsin concentration range was 500–1000 mg L^{-1}.

Table 1
Langmuir isotherm constants and correlation coefficients at the different temperatures.

T (K)	Langmuir		
	b (L mg^{-1})	q_m (g g^{-1})	r
298	1.3957	1.39	0.9999
313	1.2967	1.41	0.9999
333	1.1937	1.43	0.9999

3.6. Effects of initial pH of the solution

The influences of different initial pH values ranging between 3.0 and 10.0 were also studied. Fig. 7 shows that the degradation ratio

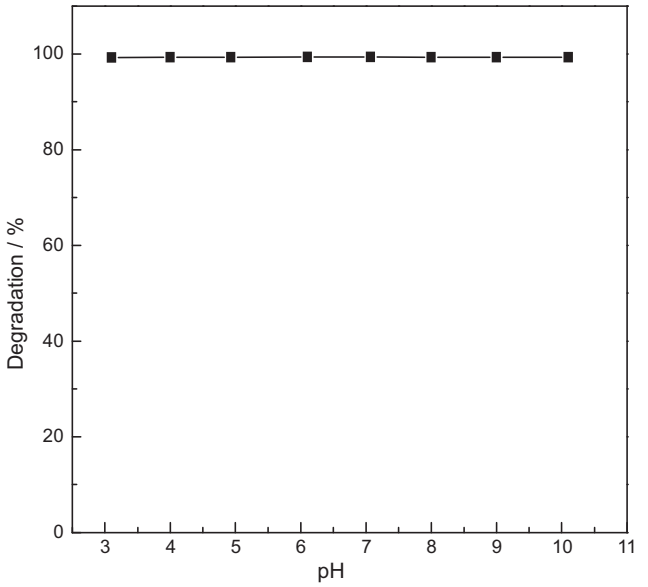


Fig. 7. Variation of degradation with different pH values; 25 mL of 50 mg L^{-1} acid fuchsin solution; MW output power 300 W; 0.08% of $MgFe_2O_4$; MW irradiation time 1.5 min.

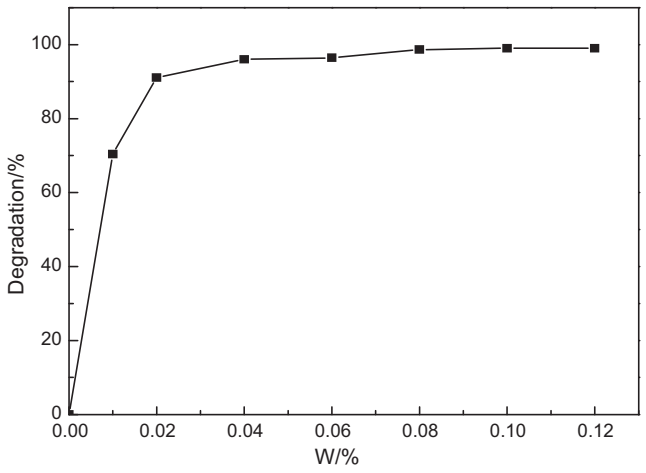


Fig. 8. Variation of degradation with different $MgFe_2O_4$ dosages; 25 mL of 50 mg L^{-1} acid fuchsin solution; MW output power 300 W; MW irradiation time 1.5 min.

of acid fuchsin almost keeps constant with increasing pH from 3.0 to 10. In general, the natural pH of acid fuchsin solution was close to 5.0. In this work the acid fuchsin solution without adjusting pH was popularly adopted.

3.7. Effect of microwave power

MW output power is regarded as the most important factor in the experiment, since the temperature that $MgFe_2O_4$ could reach was directly related to the power level. The investigated power levels were 200, 400, 500, 600 and 700 W, and other parameters remained constant, namely, acid fuchsin aqueous solution (50 mg L^{-1}) and 0.08% (w/w) $MgFe_2O_4$, irradiation time 1.5 min. The results showed that the degradation ratio of acid fuchsin was almost not increasing with the increase of MW output power. So the output power of 300 W was chosen throughout the experiment.

3.8. Effect of $MgFe_2O_4$ amount

The addition amount of the catalyst is one of the important parameters for the catalytic activity. The more addition amount of catalyst is used, the higher catalytic activity is obtained. So the effect of addition amount of $MgFe_2O_4$ powders was investigated in the range of 0.01–0.12% under MW irradiation for 2 min. The results were shown in Fig. 8. It can be found that the degradation ratio increases with the increase of catalyst addition amounts from 0.01% to 0.04%, while the degradation ratio did not change obviously at the higher concentrations (>0.08%). In this work, 0.08% was adopted as the optimal addition amount of $MgFe_2O_4$ catalyst.

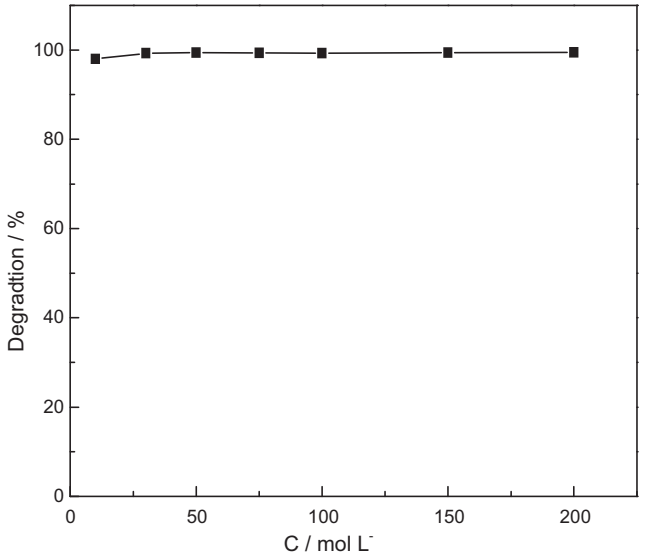


Fig. 9. The effect of initial acid fuchsin concentrations on the degradation efficiency: MW output power 300 W; MW irradiation time 1.5 min; mass fraction of $MgFe_2O_4$ 0.08%.

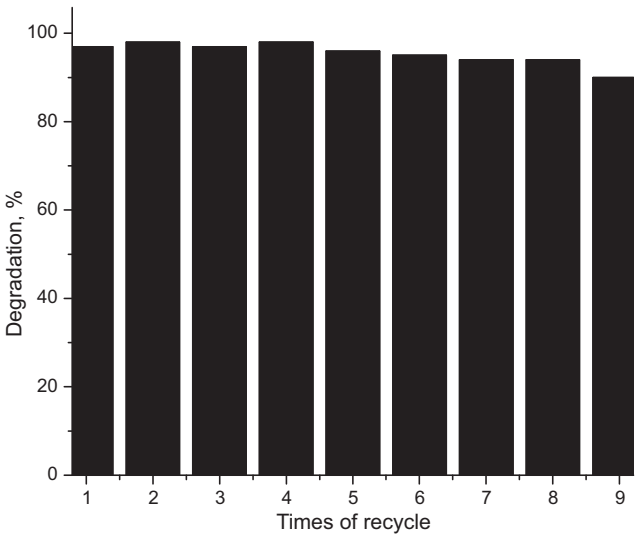


Fig. 10. Times of $MgFe_2O_4$ powder recycling use.

3.9. Influence of initial concentration on degradation of acid fuchsin

The effect of initial concentration of acid fuchsin on degradation was investigated in the concentration range of 20–200 mg L^{-1}. Fig. 9 shows that the degradation ratio of acid fuchsin was unchanged with the increase of the initial concentration. It is proved again that $MgFe_2O_4$ has highly adsorption capacity. So large numbers of acid fuchsin molecules in solution did not disturb the transmission and absorption of microwave energy, which cannot reduce the catalytic activity of $MgFe_2O_4$.

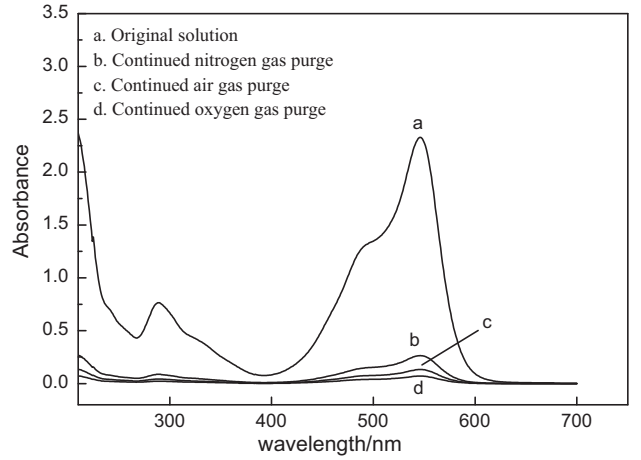


Fig. 11. The effects of bubbling gases on the efficiency degradation of acid fuchsin: 25 mL of 200 mg L^{-1} acid fuchsin solution; 400 W output power; 0.08% of $MgFe_2O_4$; MW irradiation time 3 min.

Table 2
Effect of various ions on the percent adsorption and degradation of acid fuchsin.

Dye	Anions (% adsorption)	Cations (% adsorption)	Anions (% degradation)	Cations (% degradation)
Acid fuchsin	Sulphate (96.60) Thiosulphate (96.28) Acetate (96.22) Phosphate (96.12) Carbonate (96.17)	Potassium (96.54) Nickel (95.63) Zinc (95.49)	Sulphate (99.49) Thiosulphate (99.54) Acetate (99.49) Phosphate (99.33) Carbonate (96.44)	Potassium (99.49) Nickel (96.33) Zinc (94.83)

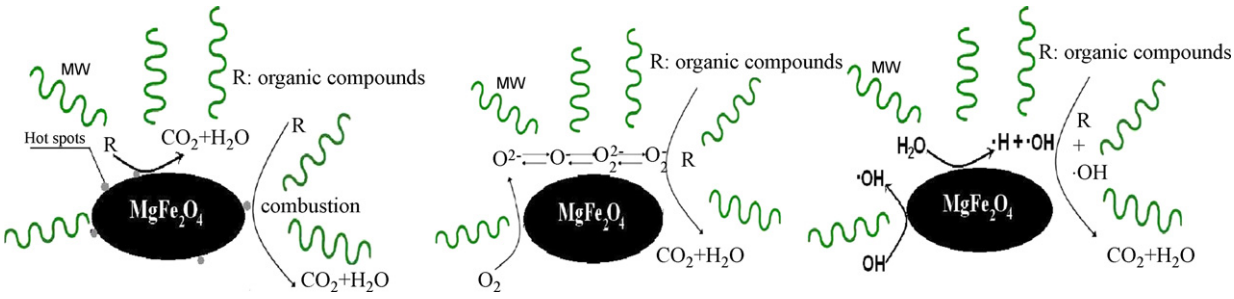


Fig. 12. The reaction pathway for acid fuchsin degradation by microwave-enhanced catalytic method over $MgFe_2O_4$.

3.10. Effect of various ions on degradation of acid fuchsin solutions

Since industrial effluents are always contained with various additives such as inorganic salts, it is necessary to study the effects of these ions on the adsorption property and the degradation ratio of acid fuchsin solutions. The adsorption and degradation of acid fuchsin in the presence of anions (added as sodium salts) and cations (added in the nitrate form) were performed. The concentrations of all these ions in solution were fixed at 1×10^{-3} mol L^{-1} in each case. The results were given in Table 2. It can be seen that the adsorption and degradation ratios of acid fuchsin are almost unchanged in the presence of anions and cations.

3.11. The reusability of magnetic powder $MgFe_2O_4$ as adsorbent

Nine degradation cycles were carried out to evaluate the reusability of $MgFe_2O_4$ powder as adsorbent/catalyst for the degradation of acid fuchsin (Fig. 10). It was found that $MgFe_2O_4$ could be used repeatedly more than 9 times and the high efficiency (94%) of the dye removal was maintained (Fig. 10). Analyses of FTIR for the reaction process in the ninth cycle did not show evident change as compared to that of the first cycle (curve c in Fig. 5). These experiments demonstrated that $MgFe_2O_4$ as adsorbent/catalyst could be used repeatedly over many cycles.

3.12. The mechanism of MW degradation acid fuchsin

$MgFe_2O_4$ as microwave absorbent can strongly absorb and transfer microwave energy. Under the microwave irradiation, $MgFe_2O_4$ particle surface can produce great amount of "hot spots" (their temperature would reach over 1200 °C [29]) which could lead to the more rapid oxidations and combustion of acid fuchsin molecules. The electrophilic oxygen ions (O_2^-, O^- and O^{2-}) that come from lattice oxygen on $MgFe_2O_4$ ferrite show high activity in catalytic reactions and can be donated to participate in the degradation of acid fuchsin and capture oxygen from air [26,30]. Fig. 11 shows that oxygen can increase the efficiency degradation of acid fuchsin. Meanwhile, with the assistance of microwave energy, the OH groups in the structure of $MgFe_2O_4$ will be transformed into •OH radicals which can oxidize acid fuchsin [23]. In addition, the microwave irradiation induces a rotation and a migration violently for the motion of polar molecules, resulting in a fast increase of the solution temperature due to friction. Also, the violent motion of polar substances can lead the molecules to a higher excited state through an increase of collision numbers between reactants, resulting in accelerating the rate of acid fuchsin degraded. It means that the simultaneous combination of microwave and catalysis can effectively degrade several kinds of intermediates produced in the course of acid fuchsin degradation, eventually driving the intermediates into CO_2, H_2O and mineral acids. On the basis of the results obtained above, the prepared $MgFe_2O_4$ may be considered to be good catalyst in microwave induced catalytic degradation process. Fig. 12 shows the reaction pathway for acid fuchsin degradation by microwave-enhanced catalytic degradation method over $MgFe_2O_4$.

In order to farther prove and explore the degradation process of acid fuchsin, the degradation products and TOC removal percentage were determined using ion chromatograph and TOC analyzer. The concentration of NO_2^-, NO_3^- and SO_4^{2-} were 4.8, 13.5 and 24.7 mg L^{-1}, respectively. TOC removal percentage was up to 91.2%. The results showed that the most of organic acid fuchsin molecules has been converted into the simple and innocuous inorganic NO_2^-, NO_3^-, SO_4^{2-} ions. The acid fuchsin solutions have been mineralized.

4. Conclusion

A novel and environmentally friendly process for the degradation of acid fuchsin has been successfully developed. $MgFe_2O_4$ was synthesized by the co-precipitation method with the assistance of microwave irradiation. The experiment demonstrated that $MgFe_2O_4$ could act as an efficient catalyst in the microwave degradation process. The catalytic activity of $MgFe_2O_4$ might derive from its strong microwave absorbing property and the role of the electrophilic oxygen, •OH group under microwave irradiation. Acid fuchsin was degraded into harmless products (CO_2, H_2O, NO_2^-, NO_3^- and SO_4^{2-}) by the microwave-enhanced catalytic degradation method, and the degradation percentage of acid fuchsin was about 99% in 1.5 min. Based on the efficient degradation of acid fuchsin and the reaction mechanisms, microwave induced catalytic degradation process with $MgFe_2O_4$ catalyst could be a potential promising technology to remove organic pollutants from waste waters.

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