

Accelerated degradation of orange G over a wide pH range in the presence of FeVO₄

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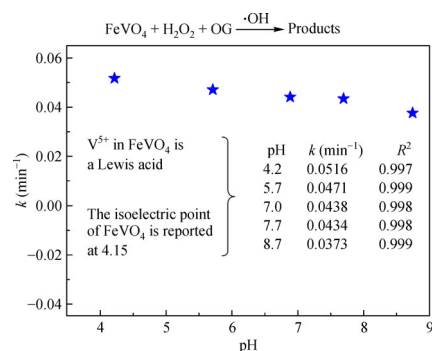
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HIGHLIGHTS

- The applicability of FeVO₄ extended the optimum pH range for heterogeneous Fenton process towards neutral conditions.
- The datas for the removal of OG in FeVO₄ systems conform to the Langmuir–Hinshelwood model.
- The irradiation of FeVO₄ by visible light significantly increases the degradation rate of OG due to the enhanced rates of the iron and vanadium cycles.

GRAPHIC ABSTRACT



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ABSTRACT

In this study, FeVO₄ was prepared and used as Fenton-like catalyst to degrade orange G (OG) dye. The removal of OG in an aqueous solution containing 0.5 g·L⁻¹ FeVO₄ and 15 mmol·L⁻¹ hydrogen peroxide at pH 7.0 reached 93.2%. Similar rates were achieved at pH 5.7 ($k = 0.0471 \text{ min}^{-1}$), pH 7.0 ($k = 0.0438 \text{ min}^{-1}$), and pH 7.7 ($k = 0.0434 \text{ min}^{-1}$). The FeVO₄ catalyst successfully overcomes the problem faced in the heterogeneous Fenton process, i.e., the narrow working pH range. The data for the removal of OG in FeVO₄ systems containing H₂O₂ conform to the Langmuir–Hinshelwood model ($R^2 = 0.9988$), indicating that adsorption and surface reaction are the two basic mechanisms for OG removal in the FeVO₄–H₂O₂ system. Furthermore, the irradiation of FeVO₄ by visible light significantly increases the degradation rate of OG, which is attributed to the enhanced rates of the iron cycles and vanadium cycles.

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1 Introduction

Advanced oxidation processes (AOPs) have become the focus of increasing research over the past two decades due

to their ability to destroy toxic and biologically refractory organic contaminants in aqueous solutions [1]. Otherwise known as Fenton system, such technologies generate reactive oxygen species, primarily in the form of the highly reactive and nonselective hydroxyl radical ($\bullet\text{OH}$), which can oxidize organic pollutants. Aqueous mixtures of ferrous iron (Fe^{II}) and hydrogen peroxide (H₂O₂) are common present-day AOPs [2,3]. However, the inherent setbacks of the homogeneous Fenton process, such as the difficulty in catalyst recovery, high sensitivity to pH, and

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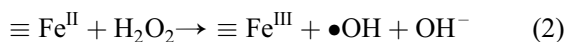
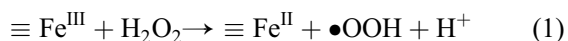
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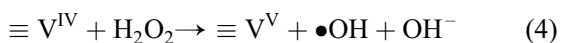
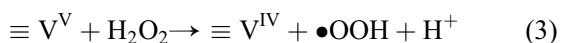
formation of ferric sludge, have limited the development of heterogeneous catalysts [4–7]. In particular, the optimum pH value for Fenton oxidations is usually reported in the acidic range near pH 3 and the necessity to acidify the reaction medium limits the applicability of the Fenton process in environmental technology [8].

Owing to the limitations of the homogeneous Fenton method when used to treat municipal wastewater, heterogeneous Fenton and Fenton-like catalysts, such as α -Fe₂O₃, α - and γ -FeOOH [9–11], Fe₃O₄ [12,13], and FeVO₄ [14], have been studied extensively.

The reaction mechanism between iron-containing materials and H₂O₂ is described by the following reactions [15].



Furthermore, there are anions that combine with iron cation (e.g., V) to form heterogeneous Fenton-like catalysts [14]. Moreover, hydroxyl radicals are produced by catalytic reactions that involve V and H₂O₂ [16,17]. These reactions are summarized below.



In recent years, because of the mass production and wide use of dyes, the treatment of dye wastewater has attracted attention. This study highlights the degradation efficiency of azo dyes, particularly orange G (OG), which is widely used in the textile, pulp, and paper industries [18]. Although there are data on the degradation of azo dyes, little data exists on the kinetics and operation factors of degradation systems. In this study, the detailed investigation of the degradation of OG in the presence of FeVO₄ is discussed with the aim of extending the pH range of Fenton-like process toward neutral conditions. The reaction rates are determined under various operating conditions and the kinetic parameters are expressed in terms of a pseudo-first-order equation. In addition, the role of visible light is investigated.

2 Materials and methods

2.1 Materials

Analytical-grade ammonium metavanadate, ferric nitrate 9-hydrate, and hydrogen peroxide (30%) were obtained from Sigma-Aldrich. Orange G was purchased from Hengrun Dyestuff Chemical Co. Ltd. (Guangzhou, China) and used without purification. All other reagents were from Sigma-Aldrich. The aqueous solutions used in the degradation reactions were prepared with deionized water obtained from a Millipore Milli-Q system. The stock solution of OG

(200 mg·L⁻¹) was prepared with distilled water. The H₂O₂ solution (100 mmol·L⁻¹) was prepared fresh.

2.2 Catalyst preparation

FeVO₄ was prepared following Ref [19]. Briefly, a 0.26 M iron nitrate solution was quickly mixed with a 4.27 mmol·L⁻¹ ammonium metavanadate solution under continuous stirring and the mixture was maintained at 75°C for 1 h in a temperature-controlled water bath. The yellow precipitate was then washed with deionized water until the pH of the supernate turned neutral. Then, the precipitate was washed by acetone several times. Finally, the precipitate was air-dried and homogenized.

2.3 Experimental procedure

The batch degradation experiments were performed in a 250 mL beaker. Reaction mixtures were obtained by taking an appropriate amount of the OG stock solution and FeVO₄, and were magnetically stirred for 30 min to complete the adsorption of OG on FeVO₄. The final volume of the reaction mixture was 100 mL. The reaction was initiated when H₂O₂ was added to the solution. HCl and NaOH (1 mol·L⁻¹) were used to adjust the initial pH value of solution before adding FeVO₄. Continuous mixing was maintained throughout the experiments. At intermittent reaction times, a portion of the reacted solution was withdrawn and filtered for analysis. In all the experiments, the temperature of the solution was maintained at 25.0°C±0.5°C. For the irradiation experiments, a solar simulator (CHF-XM35-500W) equipped with a 500 W Xe arc lamp (OSRAM, Germany) and an ultraviolet (UV)-cut filter was used as the visible light source. The reaction solutions were kept well mixed with a magnetic stirrer.

The samples for the total organic carbon (TOC) analysis were mixed with excess sodium azide to quench the radicals.

2.4 Determination of OG and TOC

The OG concentrations were determined using a UV–vis spectrophotometer (PerkinElmer, Lambda 20) with maximum absorption wavelength at 495 nm, and the TOC was measured using a TOC analyzer (Shimadzu TOC-5000A).

The analyses were repeated three times and the averages were then considered.

3 Results and discussion

3.1 Effect of FeVO₄ on the degradation of OG

In the absence of FeVO₄, OG was stable in solution containing H₂O₂. Then, OG was rapidly degraded at pH of

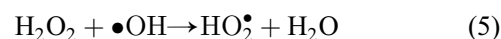
7.0±0.1 by the presence of both FeVO₄ and H₂O₂. Figure 1(a) shows the OG degradation vs the FeVO₄ concentration from 0.1 g·L⁻¹ to 2.0 g·L⁻¹ at pH 7.0±0.1 and initial concentrations of 20 mg·L⁻¹ OG and 15 mmol·L⁻¹ H₂O₂. The degradation of OG can be approximately modeled by pseudo-first-order kinetics. The curves $\ln(C_0/C) = kt$, fitted to the data by linear regression, are showed in the inset of Fig. 1(a). In the equation, k is the rate constant, t is time, and C_0 and C are the concentrations of OG at 0 min or t min, respectively. It can be seen from Fig. 1(a) that with increasing FeVO₄ concentration, the decolorization rate of OG increased and reached a plateau at FeVO₄ concentration of 1.0 g·L⁻¹. The corresponding pseudo-first-order rate constants (k) are 0.0094, 0.0222, 0.0438, 0.0695, and 0.0611 min⁻¹, respectively. As expected, when the amount of catalyst increases, the OG removal rate increases, which is attributed to the increased number of active sites for H₂O₂ decomposition and adsorption of organic compounds [20]. When catalyst loading exceeds a certain amount, many of the originally generated radicals are deactivated because of collisions, and the excess ground state FeVO₄ may cause scavenger effects [21,22]. Thus, the degradation rate of OG did not increase when the FeVO₄ concentration increased from 1.0 g·L⁻¹ to 2.0 g·L⁻¹. Although the optimal FeVO₄ concentration is 1.0 g·L⁻¹, experiments were also conducted at a concentration of 0.5 g·L⁻¹.

To evaluate the mineralization during the catalytic oxidation of OG, the TOC was monitored, as shown in Fig. 1(b). In terms of the TOC removal, as with the OG decolorization, a better performance is reached when the FeVO₄ concentration is 1.0 g·L⁻¹. In addition, after reaction for 1 h, more than 90% of OG and less than 50% TOC were removed when the concentration of FeVO₄ exceeded 0.5 g·L⁻¹ (Fig. 1(b)). Moreover, after reaction for 2 and 3 h with the solution containing 0.5 g·L⁻¹ FeVO₄, the TOC removal was 57.4% and 64.0% (data not shown),

respectively, indicating that the OG intermediates were long-lived and the mineralization rate was slow.

3.2 Effect of H₂O₂ concentration on the degradation of OG

The effect of H₂O₂ concentration on the OG degradation in the FeVO₄-H₂O₂ system at pH 7.0±0.1 is shown in Fig. 2(a). The degradation rate of OG in solutions containing 0.5 g·L⁻¹ FeVO₄ increases with increasing H₂O₂ concentration. For reaction time of 1 h, H₂O₂ concentrations of 5 mmol·L⁻¹, 10 mmol·L⁻¹, 15 mmol·L⁻¹, 25 mmol·L⁻¹, and 40 mmol·L⁻¹ correspond to 75.4%, 81.0%, 93.2%, 99.3%, and 99.3% OG removal, respectively. As seen from reactions (1)–(4), H₂O₂ can react with both iron species ($\equiv\text{Fe}^{\text{III}}/\text{Fe}^{\text{II}}$) and vanadate species ($\equiv\text{V}^{\text{V}}/\text{V}^{\text{IV}}$) to generate $\bullet\text{OOH}$ and $\bullet\text{OH}$. Increasing H₂O₂ concentration would benefit for increasing the rate of $\bullet\text{OH}$ formation through the above two pathways or more. Consequently, above 90% OG was degraded after adding 15 mmol·L⁻¹ H₂O₂. However, $\bullet\text{OH}$ is consumed by the excess H₂O₂ (reaction (5)). In this study, the optimum initial concentration of H₂O₂ is 15 mmol·L⁻¹.



The 1 h treatment of OG by FeVO₄-H₂O₂ systems removes 44.0%, 46.4%, and 47.8% TOC from neutral wastewater at a concentration of 15 mmol·L⁻¹, 25 mmol·L⁻¹, and 40 mmol·L⁻¹ H₂O₂, respectively (Fig. 2(b)). The mineralization of OG continued to occur by prolonging the reaction time. At the irradiation time of 3 h, TOC reduction of 70.8% was achieved in a neutral solution with 40 mmol·L⁻¹ H₂O₂ and 0.5 g·L⁻¹ FeVO₄.

3.3 Effect of initial OG concentration

A set of Fenton-like experiments at pH 7.0±0.1 was performed in an aqueous suspension with 0.5 g·L⁻¹

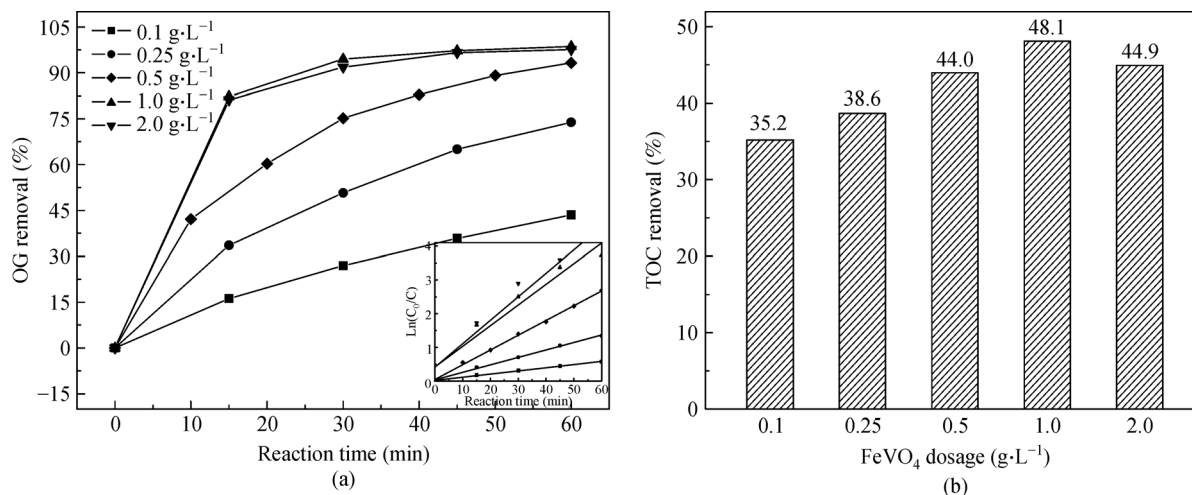


Fig. 1 (a) Orange G (OG) removal vs time (b) total organic carbon (TOC) removal vs FeVO₄ concentration after 1 h reaction. $[\text{OG}]_0 = 20 \text{ mg}\cdot\text{L}^{-1}$, $[\text{H}_2\text{O}_2]_0 = 15 \text{ mmol}\cdot\text{L}^{-1}$, and $\text{pH} = 7.0\pm 0.1$

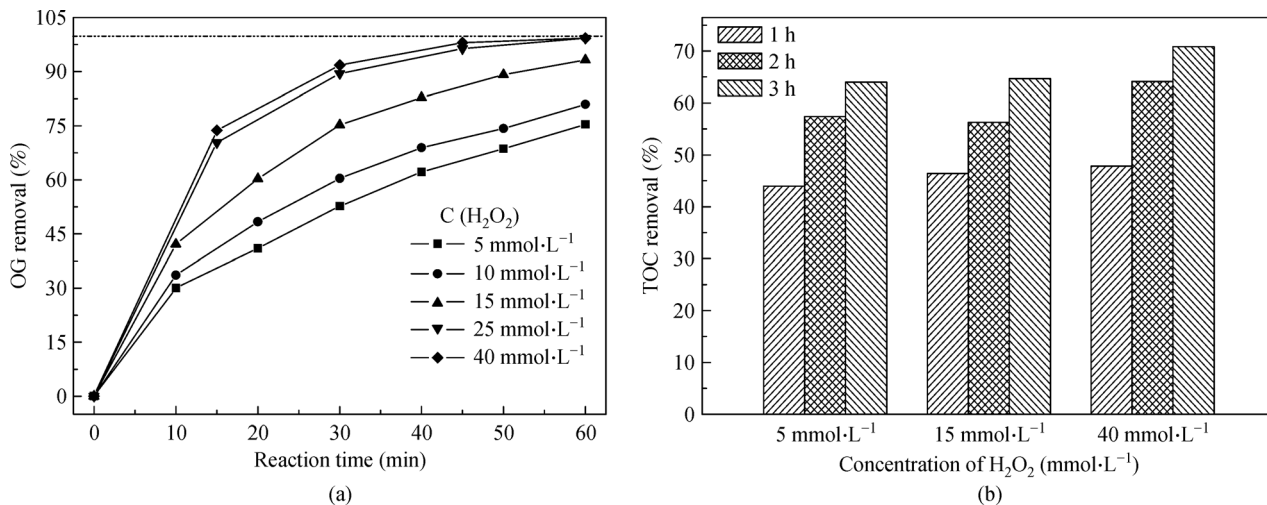


Fig. 2 (a) OG removal vs time and (b) TOC removal vs H₂O₂ concentration after reaction for 1 h, 2h and 3h. [OG]₀ = 20 mg·L⁻¹, [FeVO₄]₀ = 0.5 g·L⁻¹, and pH = 7.0±0.1

FeVO₄, 15 mmol·L⁻¹ H₂O₂ and different initial OG concentrations, i.e., 5, 10, 20, 35, and 50 mg·L⁻¹. Figure 3 (a) shows that the degradation rate constant decreases with increasing initial concentration. At high initial concentration, two factors restrict the degradation of OG. First, the increased amount of OG may occupy many FeVO₄ active sites, which suppresses the generation of the oxidants and lowers the degradation rate. Second, the high OG concentration means more intermediate products would react with the radicals instead of OG. Hence, the degradation of OG decreases with increasing initial OG concentration.

The kinetics of the catalytic oxidation of organic compounds can be successfully modeled using the Langmuir–Hinshelwood (L–H) equation to describe the correlation between the degradation rate constants and initial concentrations [21,23,24],

$$k = k_{\text{ini}} \frac{K_{\text{OG}}}{1 + K_{\text{OG}}C_0}, \quad (6)$$

where

$$\frac{1}{k} = \frac{1}{k_{\text{ini}}}C_0 + \frac{1}{k_{\text{ini}}K_{\text{OG}}}, \quad (7)$$

and k is the pseudo-first-order rate constant (min⁻¹), k_{ini} is the intrinsic reaction rate constant (mg·L⁻¹·min⁻¹), and K_{OG} is the L–H adsorption constant of OG over the FeVO₄ surface (mg⁻¹·L) in aqueous solutions. The plot of $1/k$ vs [OG]₀ gives the slope ($1/k_{\text{ini}}$) and intercept ($1/k_{\text{ini}}K_{\text{OG}}$) (Fig. 3(b)). A good linear correlation ($R^2 = 0.9988$) between $1/k$ and [OG]₀ was obtained, indicating that despite the poor adsorption of OG on the FeVO₄ surface, the reaction of OG with the surface OH radicals are important [25]. From the intercept and slope, the k_{ini} and

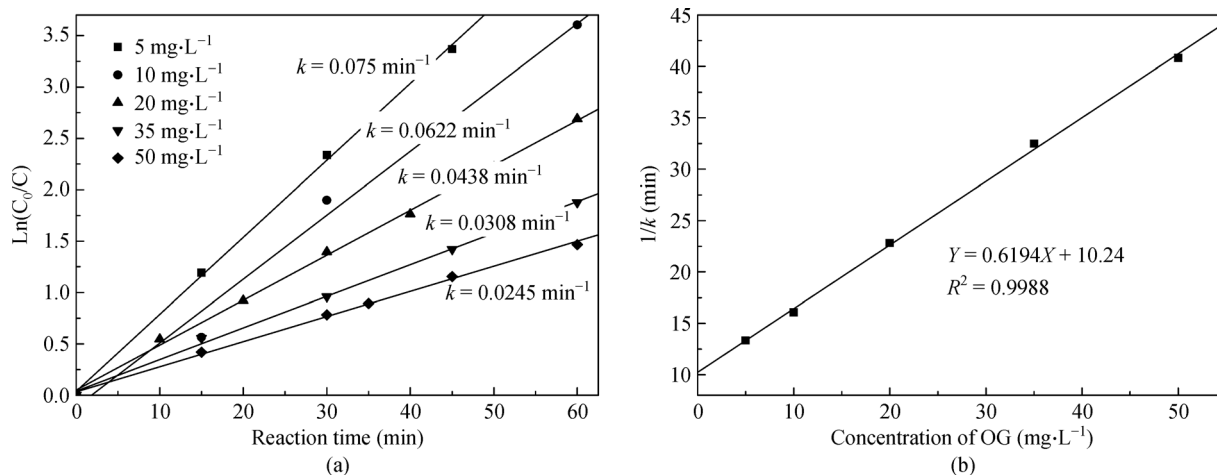


Fig. 3 The effect of initial OG concentrations: (a) Pseudo-first-order kinetics; (b) reciprocal of the pseudo-first-order rate constant vs the initial concentration of OG. [FeVO₄]₀ = 0.5 g·L⁻¹, [H₂O₂]₀ = 15 mmol·L⁻¹, and pH = 7.0±0.1

K_{OG} values for OG are calculated at $1.614 \text{ mg}\cdot\text{L}^{-1}\cdot\text{min}^{-1}$ and $0.0605 \text{ mg}^{-1}\cdot\text{L}$, respectively.

3.4 Effect of pH on the degradation of OG

pH is an important parameter that affects Fenton-like reactions. To observe the effect of pH on the degradation of OG, several experiments were conducted in the same solution but at different pH of 4.2–11.0 and the results are shown in Fig. 4(a). The corresponding pseudo-first-order rate constants (k) are shown in Fig. 4(b). The degradation of OG depends on pH, and with increasing pH, k decreases gradually. Fenton-like systems have excellent degradation rates of wastewater with acidic pH; however, at circum-neutral pH, the precipitation of $\text{Fe}_2\text{O}_3\cdot n\text{H}_2\text{O}$ inhibits the recycling of $\text{Fe}^{\text{III}}/\text{Fe}^{\text{II}}$ [8].

As shown in Fig. 4(b), the degradation of OG by $\text{FeVO}_4/\text{H}_2\text{O}_2$ systems at pH 5.7 ($k = 0.0471 \text{ min}^{-1}$) and pH 7.7 ($k = 0.0434 \text{ min}^{-1}$) is similar to that of the original system (pH 7.0, $k = 0.0438 \text{ min}^{-1}$) without pH adjustment, suggesting that the pH range was extended by the FeVO_4 catalyst. This is also supported by the k values of 0.0373 min^{-1} , 0.0284 min^{-1} , and 0.0259 min^{-1} for systems with pH 8.7, 9.2, and 9.8, respectively. This is attributed to V^{5+} in FeVO_4 being a Lewis acid that adsorbs OH^- in solution [26]. For better understanding the change of solution pH, the determination of pH value were carried out before and after the addition of FeVO_4 . As expected, the pH of the system decreased after adding FeVO_4 . For example, pH decreased from 7.0, 7.7, and 8.7 to 4.3, 5.2, and 5.4, respectively, after stirring the FeVO_4 solutions for 30 min. Undoubtedly, the decrease in the solution pH favors the degradation of pollutants in Fenton-like systems. On the other hand, the isoelectric point of FeVO_4 is reported at 4.15 [14], which is lower than that of $\alpha\text{-Fe}_2\text{O}_3$ (5.2–8.6), FeOOH (6.7 ± 0.2) and Fe_3O_4 (6.5 ± 0.2) [27]. Thus, compared with other iron oxides, FeVO_4 has a low isoelectric point, which means the adsorption of negatively

charge on FeVO_4 surface would occur over pH 4.15. Therefore, the pH application range of FeVO_4 was wide from 4.0 to 9.0 (Fig. 4(b)). The enhanced reactivity in neutral solution displayed by FeVO_4 may allow it to be the better candidate for heterogeneous Fenton catalysts than other iron catalysts such as $\alpha\text{-Fe}_2\text{O}_3$, FeOOH and Fe_3O_4 .

3.5 Visible light irradiation and the degradation of OG

It has been reported the band gap of FeVO_4 was between 2.05 and 2.7 eV [28–30] which means FeVO_4 can be a visible light photocatalyst. To evaluate the photocatalytic activity of FeVO_4 , the photodegradation of OG under visible light with and without FeVO_4 was investigated. Figure 5 shows the OG removal as a function of irradiation time. No significant change after 1 h irradiation was observed in the OG solution either with or without H_2O_2 , suggesting that the loss of OG owing to the visible light irradiation without catalyst can be ignored. By adding $0.5 \text{ g}\cdot\text{L}^{-1}$ FeVO_4 , obvious photodegradation was observed with 97.8% OG removal after irradiation for 30 min. For comparison, in an identical experiment in the dark, only 75.2% OG was removed. These results suggest that visible light enhances the photocatalytic activity of FeVO_4 in the presence of H_2O_2 .

The recycling of $\equiv\text{Fe}^{\text{III}}$ from $\equiv\text{Fe}^{\text{II}}$ by reaction (2) and $\equiv\text{V}^{\text{V}}$ from $\equiv\text{V}^{\text{IV}}$ by reaction (4) are several orders of magnitude slower than reaction (1) and reaction (3), respectively. Reaction (2) and reaction (4) are considered as the rate-limiting steps of the iron cycling and vanadium cycling, respectively. The presence of visible light increases the rates of reactions (2) and (4) [31–33] and simultaneously accelerates the regeneration of Fe^{II} and V^{IV} . As a result, the $\text{Fe}^{\text{III}}/\text{Fe}^{\text{II}}$ and $\text{V}^{\text{V}}/\text{V}^{\text{IV}}$ cycles are well maintained in the $\text{FeVO}_4\text{-H}_2\text{O}_2$ system and $\bullet\text{OH}$ are produced faster, which meaning that the role of incident light increases significantly the H_2O_2 efficiency in producing $\bullet\text{OH}$ according to reactions (1)–(4). Conse-

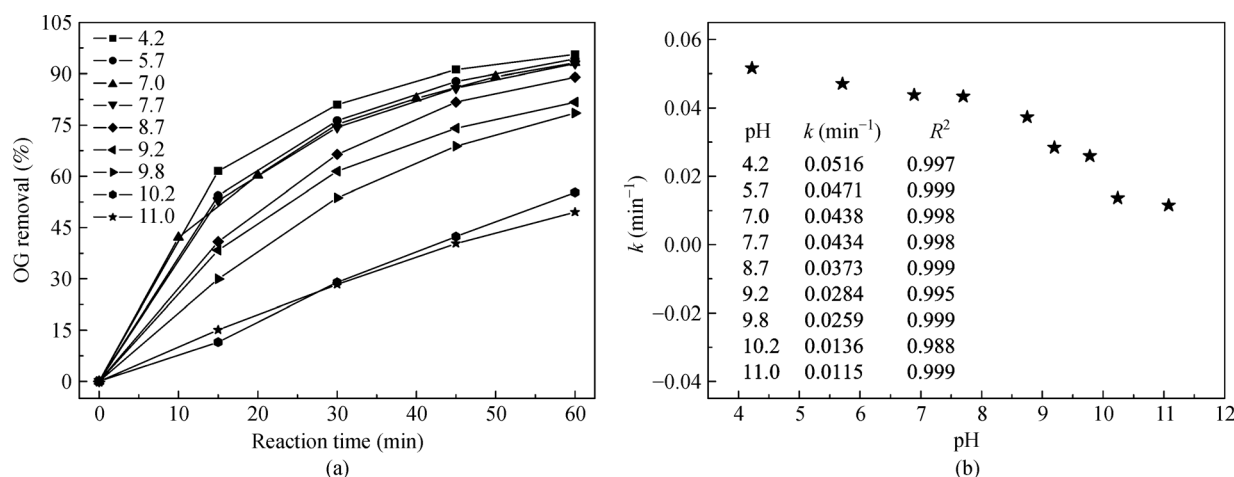


Fig. 4 Effect of pH on the degradation of OG (a) and k vs pH (b). $[\text{FeVO}_4]_0 = 0.5 \text{ g}\cdot\text{L}^{-1}$, $[\text{H}_2\text{O}_2]_0 = 15 \text{ mmol}\cdot\text{L}^{-1}$, and $[\text{OG}]_0 = 20 \text{ mg}\cdot\text{L}^{-1}$

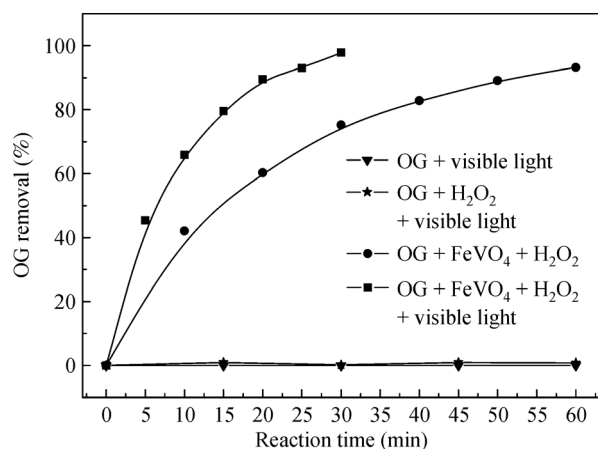


Fig. 5 Effect of visible light irradiation on the degradation of OG. $[OG]_0 = 20 \text{ mg} \cdot \text{L}^{-1}$, $[\text{FeVO}_4]_0 = 0.5 \text{ g} \cdot \text{L}^{-1}$, and $[\text{H}_2\text{O}_2]_0 = 15 \text{ mmol} \cdot \text{L}^{-1}$

quently, the oxidative capacity of the $\text{FeVO}_4\text{-H}_2\text{O}_2$ system greatly improves, and the decolorization rate of OG increases owing to the irradiation with visible light.

4 Conclusions

The degradation of OG in neutral solutions was investigated considering the FeVO_4 and H_2O_2 concentrations, and initial concentration of OG solutions. In the $\text{FeVO}_4\text{-H}_2\text{O}_2$ system, $\bullet\text{OH}$ is the most important reactive species for the degradation of OG. The OG degradation in the $\text{FeVO}_4\text{-H}_2\text{O}_2$ system follows first-order reaction kinetics and conforms to the Langmuir–Hinshelwood model ($R^2 = 0.9988$). FeVO_4 was successfully applied to the treatment of wastewater at circumneutral pH. The irradiation with visible light greatly enhanced the rate of oxidation of OG in the $\text{FeVO}_4\text{-H}_2\text{O}_2$ system at pH 7.0, which is attributed to the enhanced rates of the iron and vanadium cycles as FeVO_4 absorbed light.

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