Photocatalytic degradation and kinetics of Orange G using nano-sized Sn(IV)/TiO$_2$/AC photocatalyst

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Abstract

Sn(IV) doped and nano-sized TiO$_2$ immobilized on active carbon (AC) (Sn(IV)/TiO$_2$/AC) were prepared by the sol–gel and dip-calcination method. An azo dye, Orange G (OG), was used as a model compound to study its photocatalytic activity in a fluidized bed photoreactor. The addition of Sn(IV) on TiO$_2$ could greatly improve the activity of TiO$_2$, and the optimal amount of tin was 2.5 at.%. The effects of calcination temperature, pH value, the initial hydrogen peroxide concentration ([H$_2$O$_2$]$_0$), the catalyst amount ([TiO$_2$]), the initial OG concentration ([dye]$_0$) and co-existing negative ions on the photocatalytic activity of Sn(IV)/TiO$_2$/AC were studied. The optimal conditions were as follows: pH 2.00, [H$_2$O$_2$]$_0$ = 1.5 mL/L, [dye]$_0$ = 50 mg/L, [TiO$_2$] = 12.5 g/L, when the 300 W high pressure mercury light was used as the light source. Under these conditions, the degradation efficiency of OG reached 99.1% after 60 min reaction. The kinetics of the OG degradation was also analyzed. The results showed that the kinetics of this reaction fit the Langmiur–Hinshelwood kinetics model well and the absorption of OG on the Sn(IV)/TiO$_2$/AC surface was the controlling step in the whole degradation process. In addition, the catalyst, liquid and gas were separated effectively, and the integrative process of reaction and separation was achieved during the experiment.
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Keywords: Sn(IV)/TiO$_2$/AC; Orange G; Photocatalytic degradation; Kinetics; Langmiur–Hinshelwood model

1. Introduction

The widespread presence of organic dyes in industrial wastewater results in a potentially serious environmental problem. Especially azo dyes, which are non-biodegradable, toxic and potentially carcinogenic in nature, are widely used [1]. The conventional treatment processes, including biological oxidation, chemical and physical–chemical treatments usually cause secondary pollutions. Therefore, novel, efficient and low cost technology in purification of azo dyes wastewater is in urgently need.

In recent years, photocatalytic reaction sensitized by TiO$_2$ has attracted extensive interests as a potential way to treat wastewater [2–4]. In many applications, TiO$_2$ exhibits its unique photochemical properties, and moreover, it is photostable, non-selective, and non-toxic in treating any toxic organic materials [5]. However, there are some drawbacks in using titanium oxide powder as catalyst, such as low quantum efficiency, difficult separation, and catalyst agglomerate, etc. Several attempts have been performed to improve the photocatalytic activity of TiO$_2$ in recent years. An effective method is the addition of some metal ions or their oxides to TiO$_2$ [6–8]. Besides, a large number of researches have shown that the catalyst can be effectively separated if TiO$_2$ was immobilized on some rigid support [9,10]. Due to the singular characteristics of active carbon (AC), the study of TiO$_2$ coatings on it has deserved much attention. Herein, Sn(IV)/TiO$_2$/AC were prepared by the sol–gel and dip-calcination method. Its photocatalytic activity was evaluated by degradation of a typical kind of azo dye Orange G (OG), whose structure is shown in Fig. 1. The effects of various parameters on the photocatalytic activity of Sn(IV)/TiO$_2$/AC were studied and the kinetics of OG degradation was also analyzed.

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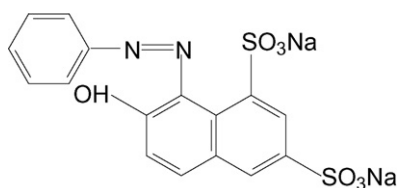*E-mail address:* sunjh@henannu.edu.cn (J. Sun).

Fig. 1. The structure of Orange G.

2. Experimental

2.1. Preparation of Sn(IV)/TiO₂/AC

The Sn(IV)/TiO₂/AC were prepared by the dip-coating technique with TiO₂ sol–gel. The procedure for the preparation of samples is given below: mixing tetra-*n*-butyl titanate with ethanol, and nitric acid to obtain pH 2. Then a small quantity of acetylacetone was used as a peptization agent and stabilizer. Furthermore, the solution was modified by 4 mL tin tetrachloride solution. This caused a hydrolysis and condensation reaction, which resulted in a doped and transparent gel. After that, AC was added. The diameter of AC is in the range of 0.45–0.9 mm, which was activated by nitric acid before using. After stirring and stabilization for 0.5 h, the samples were dried at 100 °C for 0.5 h and then calcinated at different temperatures (from 450 to 600 °C) for 2 h. The temperature of resistance furnace was raised to the desired temperature at a heating rate of 5 °C/min. In order to avoid shelling of TiO₂, the photocatalysts were all calcinated for 2 h between two coatings. After three dip coating/heat treatment, the photocatalysts were cleaned ultrasonically and air-dried at 100 °C and then the Sn(IV)/TiO₂/AC were obtained.

2.2. Photocatalytic activity

At room temperature, all the experiments were performed in a fluidized bed photoreactor of 2000 mL (shown in Fig. 2)
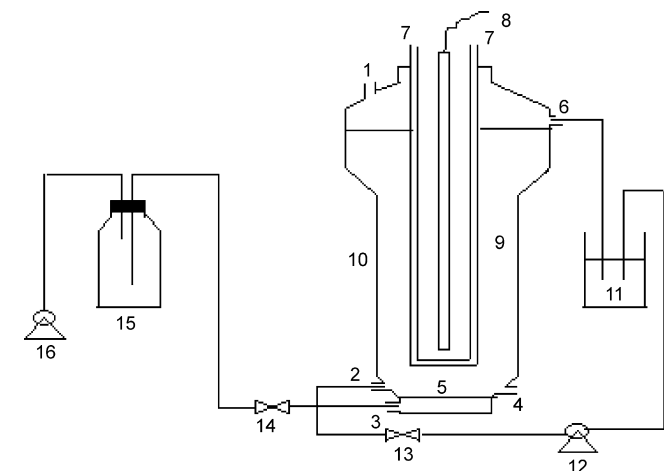

Fig. 2. Fluidized bed photoreactor. (1) Blow vent; (2) infall; (3) gasinlet; (4) discharge outlet; (5) aeration board; (6) outfall; (7) cooling water; (8) high pressure mercury lamp; (9) quartz tube; (10) water storage; (11) feed tank; (12) peristaltic pump; (13) liquid flow meter; (14) gas flow meter; (15) buffering bottle; (16) air compressor.

in volume, fitted with a high-pressure mercury lamp of 300 W (Yaming Company, Shanghai, the strongest emission at 365 nm). The initial volume of OG dye solution was 2500 mL, and pH was adjusted using dilute nitric acid or sodium hydroxide, which were measured by a pH meter (PHS-3C). The solution was added to the reactor and the feeding tank, respectively. Then the Sn(IV)/TiO₂/AC (which had achieved adsorption equilibrium in the OG solution) and H₂O₂ were also added to the reactor, and stirred with air at a flow rate of 0.2 m³/h. The solution was circulated continuously with a pump (BT00-300M Longer Peristaltic Pump) at a flow rate of 0.3 L/min. During the experiment, the catalyst, liquid and gas were separated effectively, and the integrative process of reaction and separation was achieved. Therefore, the samples, taken out from the outlet of reactor periodically, were analyzed directly without further purification. Each experiment was replicated three times or more.

2.3. Analytical methods

The UV–vis spectra of dye were recorded from 200 to 800 nm using a UV–vis spectrophotometer (Lambda 17, Perkin-Elmer) with a spectrometric quartz cell (1 cm path length). The maximum absorbance wavelength (λ_{max}) of OG is 476 nm. Therefore, the concentration of the dye in the reaction mixture at different reaction times were determined by measuring the absorption intensity at $\lambda_{max} = 476$ nm and a calibration curve. The photodegradation efficiency of OG was defined as follows:

$$\text{photodegradation efficiency}\,(\%) = \left(1 - \frac{C_t}{C_0}\right) \times 100\% \qquad (1)$$

where the C_0 is the initial concentration of OG, and C_t is the concentration of OG at reaction time t (min).

3. Results and discussion

3.1. Effect of calcination temperature on photocatalytic activity of Sn(IV)/TiO₂/AC

The temperature for calcination of TiO₂ has a significant effect on its activity, which has been reported in many other papers [11]. In order to find the optimal calcination temperature for Sn(IV)/TiO₂/AC preparation, the photocatalysts calcinated at different temperatures in the range of 450–600 °C were used to degrade OG. The results are illustrated in Fig. 3, which indicates that the photocatalytic activity of Sn(IV)/TiO₂/AC was significantly influenced by the calcination temperature and that the optimal calcination temperature was 550 °C. In addition, X-ray diffraction (XRD, Bruker D8X) was employed to examine the crystalline structure and phase of the catalysts. XRD patterns of Sn(IV)/TiO₂/AC are shown in Fig. 4. It can be seen that the Sn(IV)/TiO₂/AC which was calcinated at 550 °C contained both anatase and rutile phases of TiO₂, which may explain the higher photocatalytic activity for the degradation of OG. A communication [12] has shown that the photocatalyst consisting of anatase and rutile mixing TiO₂ has the higher photocatalytic activity. This is maybe due to the fact that each effective particle in the TiO₂ catalyst consists of an anatase core and a thin rutile
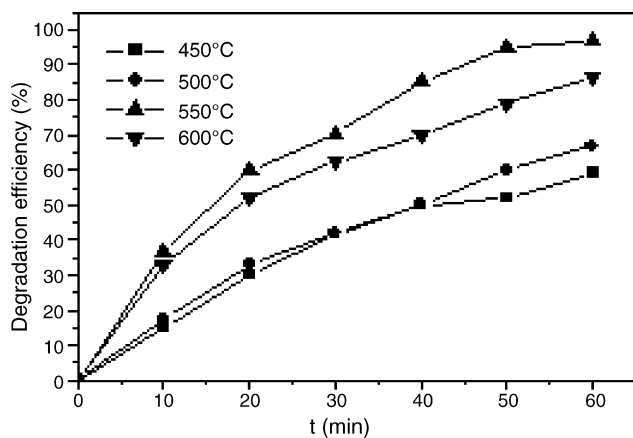
Fig. 3. The effect of calcined temperature on the degradation of OG (pH 2.00, $[H_2O_2]_0 = 1.5$ mL/L, $[dye]_0 = 50$ mg/L, [2.5 at.% Sn(IV)/TiO$_2$] = 10 g/L).

Fig. 5. The effect of different dosage of Sn(IV) on the degradation of OG (pH 2.00, $[H_2O_2]_0 = 1.5$ mL/L, $[dye]_0 = 50$ mg/L, [Sn(IV)/TiO$_2$] = 10 g/L).

cover layer, and that the holes photogenerated in the anatase core are effectively transferred to the rutile layer. So more holes are available for reaction with hydroxyl ions to form hydroxyl radicals. From XRD patterns of Sn(IV)/TiO$_2$/AC, we can also see that rutile TiO$_2$ was present at low calcination temperature of 500 °C. Theoretically, the transition temperature of crystal TiO$_2$ from anatase to rutile is 610–915 °C. The patterns indicated that the presence of Sn(IV) can be in favor of the formation of anatase and rutile TiO$_2$ at low temperature. The possible explanation of temperature decrease may be that the transition temperature of crystal form of TiO$_2$ descended with decreasing melting point of the metal oxide, and that the melting point of SnO$_2$ is below that of TiO$_2$ [13]. Therefore, rutile TiO$_2$ is formed at low temperature. Interestingly, the peak of SnO$_2$ could not be seen from the XRD patterns of catalysts, which may be attributed to the low dosage of Sn(IV). The crystalline particle size of the photocatalyst was derived from the Scherrer equation and the particles of TiO$_2$ were all about 15 nm [14].

Considering the above results, the optimal calcination temperature for Sn(IV)/TiO$_2$/AC preparation were kept constant at 550 °C for all the following experiments.

3.2. Optimum dosage of Sn(IV)

To elucidate the effect of the addition of Sn(IV) on the activity of TiO$_2$, a series of experiments was conducted with different
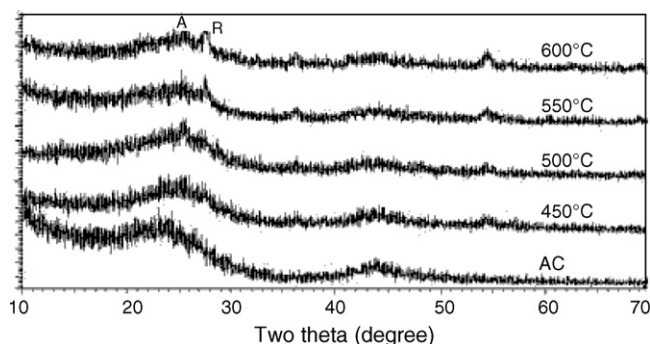

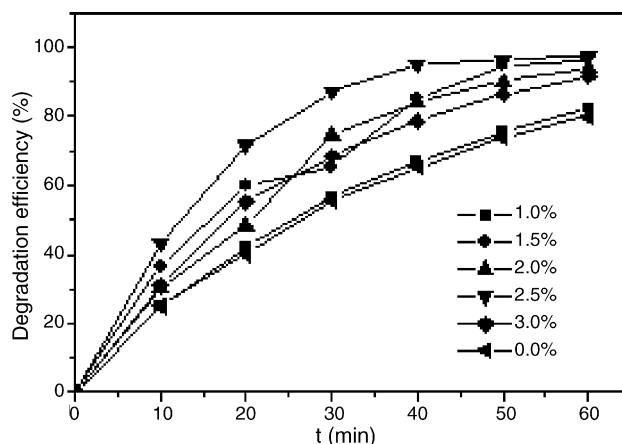Fig. 4. XRD patterns of 2.5 at.% Sn(IV)/TiO$_2$/AC at different temperature for 2 h.

doped dosages of Sn(IV) from 0 to 3.0 at.%. Fig. 5 showed the effect of Sn(IV) dosage on the photodegradation of OG. The addition of Sn(IV) could effectively improve the photocatalytic activity of TiO$_2$, and Sn(IV)/TiO$_2$/AC all had the higher catalytic activity than pure TiO$_2$/AC. When the amount of tin reached 2.5 at.%, the Sn(IV)/TiO$_2$/AC showed the highest photocatalytic activity.

The improvement of catalytic activity depended on several factors. On the one hand, in the course of OG photodegradation by pure TiO$_2$, the amounts of photogenerated electrons and holes were relatively few because of the high recombination ratio of them. The energy gap of TiO$_2$ (3.2 eV) is lower than that of SnO$_2$ (3.8 eV), and hence SnO$_2$ has higher ability of capturing electrons. This enhances the separation of photogenerated electrons and holes, leaving holes available for oxidation of OG. On the other hand, Sn(IV)/TiO$_2$/AC calcinated at 550 °C is advantageous for separation of electrons and holes because it consists of anatase and rutile, and this catalyst has a much larger specific surface area, which can improve the photocatalytic activity of TiO$_2$. But excessive amount of Sn(IV) in the catalyst can induce the recombination of photogenerated electrons and holes, and result in the lower photocatalytic activity of TiO$_2$.

3.3. Effect of pH value

The pH value of OG solution has significant influence on the photocatalytic activity of samples, which controls the production rate of hydroxyl radical. It is also an important operational variable in the actual wastewater treatment. In order to find the optimal pH of reaction mixture for the photodegradation of OG, a series of experiments was conducted at different pH values of 1.0, 2.0, 4.0, 8.0 and 12.0, etc. The results are illustrated in Fig. 6. Increase in pH of OG solution from 1.0 to 2.0 led to the enhanced efficiency in the degradation of OG. Further increase in pH from 2.0 to 12.0 had negative effect on the efficiency in OG degradation. The results indicated that the photodegradation of OG was most efficient in acidic solution and that the optimal pH was observed at a pH of about 2.0.
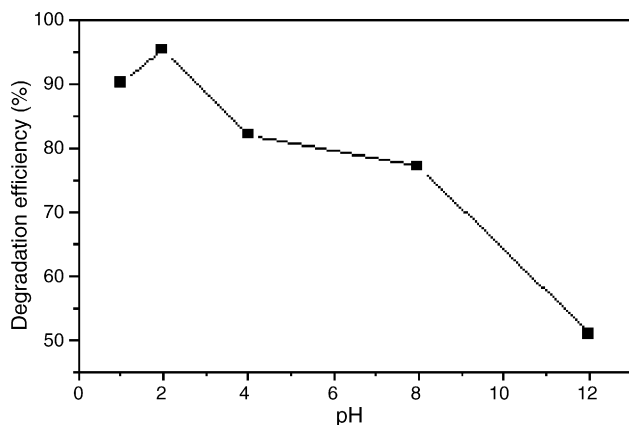
Fig. 6. The effect of pH value on the degradation of OG. ($[H_2O_2]_0 = 1.5$ mL/L, $[dye]_0 = 50$ mg/L, [2.5 at.% Sn(IV)/TiO$_2$] = 10 g/L).
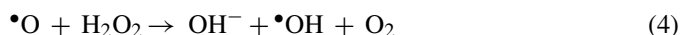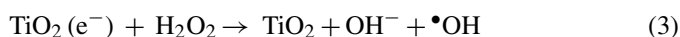
According to the potentiometric equation of TiO$_2$ (Eq. (2)) [15]:

$$E_{VB} = 3.15 - 0.05\,pH \qquad (2)$$

The lower the pH value, the higher the E_{VB}, and TiO$_2$ had higher activity in oxidating organic pollutants accordingly. At lower pH value than 2.0 the degradation efficiency also decreased. This is due to the following reason: in OG dye, the azo linkage (–N=N–) is particularly susceptible to electrophilic attack by $^\bullet$OH radical. But at low pH the concentration of H$^+$ is in excess and H$^+$ ions interact with the azo linkage decreasing the electron densities at the azo group. Consequently, the reactivity of $^\bullet$OH radical by the electrophilic mechanism decreased [16]. Besides, with the rise in the pH value, the –OH on the surface of TiO$_2$ could be replaced by –ONa, and the catalytic activity of TiO$_2$ decreased accordingly [17]. Thus, 2.0 was the optimum pH value for the photodegradation of the azo dye, as Zielińska et al. [18] had obtained through their experiment.

3.4. Effect of H$_2$O$_2$ concentration

Hydrogen peroxide plays the role of an oxidizing agent in photocatalytic processes. The photocatalytic degradation of OG was conducted at different hydrogen peroxide concentrations, the results of which are given in Fig. 7. The results indicated that the rate of degradation of OG increased with increasing H$_2$O$_2$ concentration up to 1.5 mL/L, but above it, there was a decrease in the degradation efficiency. Similar results have been reported in other articles [19,20]. This is because hydrogen peroxide inhibits the electron–hole recombination and could act as an alternative electron acceptor to oxygen at its low concentration, according to the Eqs. (3) and (4) [21]:

$$TiO_2\,(e^-) + H_2O_2 \rightarrow TiO_2 + OH^- + {}^\bullet OH \qquad (3)$$

$${}^\bullet O + H_2O_2 \rightarrow OH^- + {}^\bullet OH + O_2 \qquad (4)$$

But at high concentration of hydrogen peroxide, the very reaction $^\bullet$OH radical could be consumed by H$_2$O$_2$ and generates less reactive $^\bullet$OOH radical (Eq. (5)) [22]:
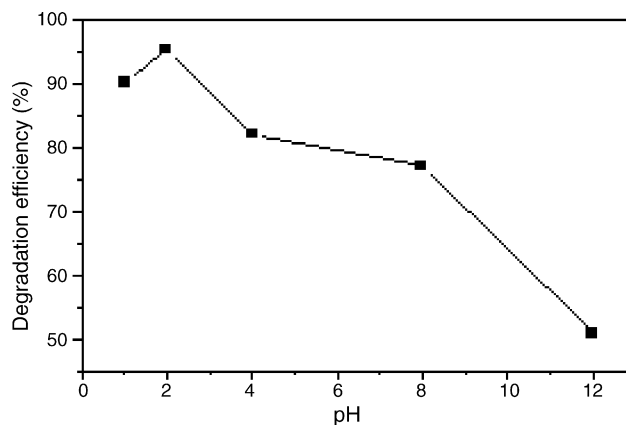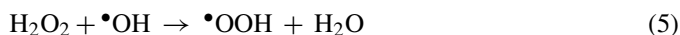
$$H_2O_2 + {}^\bullet OH \rightarrow {}^\bullet OOH + H_2O \qquad (5)$$

Fig. 7. The effect of H$_2$O$_2$ concentration on the degradation of OG (pH 2.00, $[dye]_0 = 50$ mg/L, [2.5 at.% Sn(IV)/TiO$_2$] = 10 g/L).

Thus, in the condition of excess of H$_2$O$_2$, it would become a scavenger of $^\bullet$OH radical and lead to the decrease in the degradation efficiency of OG [23].

Therefore in order to keep the high photocatalytic activity of TiO$_2$, it was necessary to choose the optimal H$_2$O$_2$ concentration and the optimal H$_2$O$_2$ concentration of this experiment was 1.5 mL/L.

3.5. Effect of catalyst amount

Photocatalyst amount is another critical parameter to the degradation efficiency. In order to determine the effect of catalyst amount on the degradation of OG, a series of experiments was conducted with varying catalyst amount from 5 to 15 g/L. The results are illustrated in Fig. 8. As the photocatalyst amount increased from 5 to 12.5 g/L the degradation efficiency of OG enhanced, and reached the maximum of 99.1% after 60 min treatment, when the catalyst amount was 12.5 g/L. This is due to the increase in the catalyst amount, which contributed to the increase in the number of photons absorbed and also the number of the dye molecules adsorbed [16]. Then the increase in the catalyst amount beyond 12.5 g/L had negative effect on the degradation efficiency of OG. Light blocking by excessive cat-
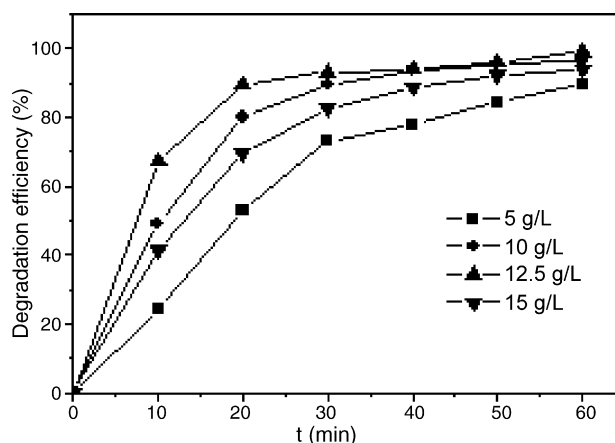

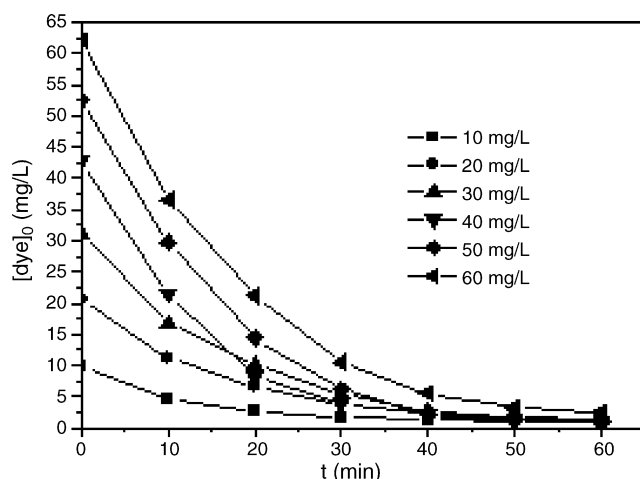Fig. 8. The effect of catalyst amount on the degradation of OG (pH 2.00, $[H_2O_2]_0 = 1.5$ mL/L, $[dye]_0 = 50$ mg/L).

Fig. 9. The relationship between different initial concentrations of OG and time (pH 2.00, $[H_2O_2]_0 = 1.5$ mL/L, $[Sn(IV)/TiO_2] = 10$ g/L).

Fig. 10. The relationship between $1/r_0$ and $1/C_0$ at different initial concentrations of OG.

alyst may account for the decreased degradation efficiency of OG solution [21]. The excessive of catalyst prevented the illumination of TiO_2. Hence, $^\bullet OH$ radicals, a primary oxidant in the photocatalytic system, decreased and the degradation efficiency of OG reduced accordingly.

3.6. Kinetics analysis

In this section, a series of OG solution were prepared, and the initial concentrations of OG were about 10, 20, 30, 40, 50, and 60 mg/L, respectively. The relationship between the concentration of OG and the illumination time catalyzed by Sn(IV)-doped TiO_2 are plotted in Fig. 9. Based on the removal curves, the degradation reaction rate constants and initial reaction r_0 were calculated and are summarized in Table 1.

In this reaction, $1/r_0$ were plotted versus $1/C_0$ based on the data from Table 1. As shown in Fig. 10, $1/r_0$ correlated to $1/C_0$ well, so the degradation of OG catalyzed by $Sn(IV)/TiO_2/AC$ fitted with the Langmiur–Hinshelwood (L–H) kinetics model, which is commonly expressed as Eq. (6) [11,24–26]:

$$r_0 = -\frac{dc}{dt} = \frac{kK_A C_0}{1 + K_A C_0} \tag{6}$$

where r_0 is the initial photocatalytic degradation rate (mg L^{-1} min^{-1}), C_0 the initial concentration of OG (mg L^{-1}), k the apparent reaction rate constant (mg L^{-1} min^{-1}), and K_A is the adsorption equilibrium constant (L mg^{-1}).

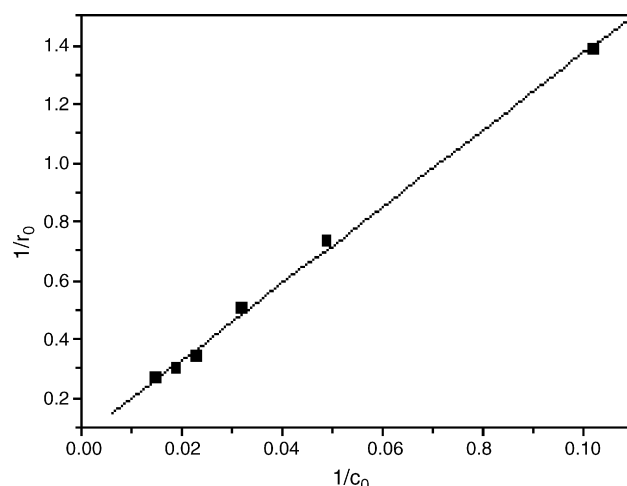Linearization of Eq. (6) gives the relationship (Eq. (7)):

$$\frac{1}{r_0} = \frac{1}{kK_A}\frac{1}{C_0} + \frac{1}{k} \tag{7}$$

According to the Langmiur–Hinshelwood (L–H) kinetics formula and the formula fitted in Fig. 10 a straight line was obtained with an intercept of $1/k$ and $1/(kK_A)$, from which k and K_A were estimated at 15.4 (mg L^{-1} min^{-1}) and 4.95×10^{-3} (L mg^{-1}). The regression line had a correlation coefficient of 0.9991. The results, $k > K_A$ (shown in Table 1), suggested that a surface reaction, where the dyes were absorbed, was the controlling step of the process.

3.7. Effect of co-existing negative ions

As we all know, there are large amounts of negative ions, such as SO_4^{2-}, $H_2PO_4^-$ and so on in wastewater of textile and dyestuff industries, whose effects on photodegradation of OG cannot be neglected. So Na_2SO_4 and NaH_2PO_4 were chosen to change the concentrations of SO_4^{2-} and $H_2PO_4^-$ in OG solution because the effect of Na^+ could be negligible [27]. As shown in Figs. 11 and 12, the presence of SO_4^{2-} and $H_2PO_4^-$ reduced the degradation efficiency of OG, and the elevated concentrations of SO_4^{2-} and $H_2PO_4^-$ caused an obvious decrease in the degradation efficiency of OG. The above results also showed that $H_2PO_4^-$ had more significant effect in the ion concentration range of experiment. This could be explained by

Table 1
The parameters of photocatalytic reaction at different initial concentration of OG

Catalyst	Initial OG concentration, C_0 (mg/L)	Reaction rate, k_1 (min^{-1})	Initial reaction rate, r_0 (mg/L/min)	Removal rate (%)
	9.8	0.03219	0.72	87.7
	20.6	0.03688	1.30	91.0
	31.0	0.04671	1.90	94.2
Sn(IV) + TiO$_2$/AC	42.7	0.05860	2.92	96.4
	52.5	0.07220	3.53	98.1
	65.0	0.05312	3.75	95.6
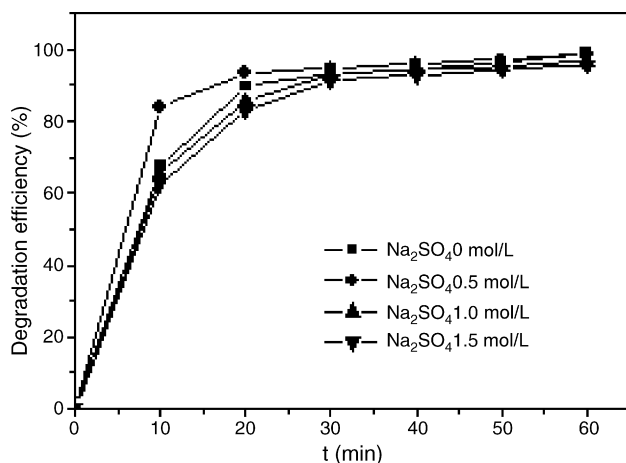
Fig. 11. The effect of SO_4^{2-} on the degradation of OG (pH 2.00, $[H_2O_2]_0 = 1.5$ mL/L, $[dye]_0 = 50$ mg/L, $[Sn(IV)/TiO_2] = 12.5$ g/L).
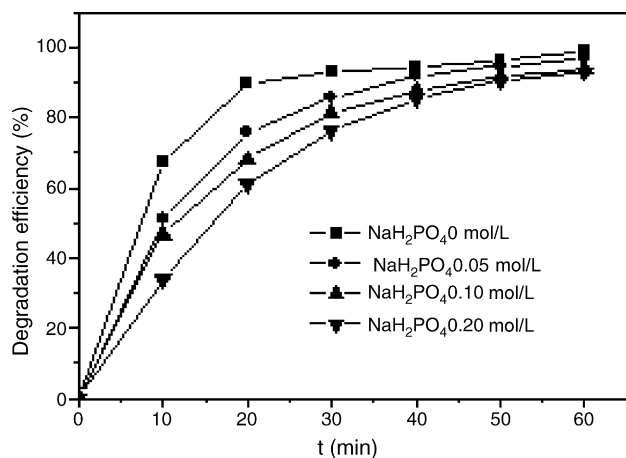
Fig. 12. The effect of $H_2PO_4^-$ on the degradation of OG (pH 2.00, $[H_2O_2]_0 = 1.5$ mL/L, $[dye]_0 = 50$ mg/L, $[Sn(IV)/TiO_2] = 12.5$ g/L).

negative ions adsorbed on the surface of catalyst, resulting in the decreased amount of adsorbed OG. In addition, as shown above, the adsorption of dyes was the controlling step of the process. Consequently, the degradation efficiency of OG was reduced accordingly. We concluded that SO_4^{2-} and $H_2PO_4^-$ had negative effects on degradation of OG, and this assumption warrants further experiments for confirmation.

4. Conclusions

The results of the present investigation conclude that the photocatalytic activity of Sn(IV)/TiO$_2$/AC is improved by the addition of Sn(IV), and that the optimal calcination temperature of this photocatalyst is 550 °C, the optimal dopant concentration of Sn(IV) is 2.5 at.%. It has been found that the solution pH, the H$_2$O$_2$ concentration, the photocatalyst amount, the initial dye concentration and the co-existing negative ions are the main factors that have strong influences on the photodegrada-

tion of OG. The optimal operation parameters for this reaction were 1.5 mL/L $[H_2O_2]_0$, 12.5 g/L [2.5 at.% Sn(IV)/TiO$_2$] for 50 mg/L $[dye]_0$ at an initial pH of 2.0 with a 300 W high pressure mercury light irradiated. Under these conditions, 99.1% degradation efficiency of dye in aqueous solution was achieved after 60 min reaction. The photocatalytic degradation kinetics of OG on Sn(IV)/TiO$_2$/AC immobilized membranes fits the Langmiur–Hinshelwood kinetics model well. The results, that $k > K_A$, suggested that a surface reaction, where the dyes were absorbed, was the controlling step of the process. Besides the influence of co-existing negative irons in OG solutions, such as SO_4^{2-} and $H_2PO_4^-$, cannot be neglected. Both SO_4^{2-} and $H_2PO_4^-$ have negative effects on the degradation of OG, and $H_2PO_4^-$ has more significant effect.

Acknowledgement

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