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# Photocatalytic degradation of C. I. Reactive Red 24 solution with $K_6SiW_{11}O_{39}Sn^{II}$

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**Abstract**

Environmental friendly materials, $K_6SiW_{11}O_{39}Sn$ (SiWSn), was synthesized. SiWSn photocatalytic decomposition of C. I. Reactive Red 24 (RR24) with the UV-lamp (253.7 nm, 20 W), Xenon lamp filtered less than 390 nm light (500 W) and sun light was investigated. The results showed that RR24 solution could be effectively decolorized with the SiWSn photocatalyst. The photocatalytic degradation efficiency of RR24 with SiWSn was affected by the initial concentration of RR2 solution, the amount of SiWSn and the photolysis time. It is demonstrated that the process of photodegradation of RR24 with SiWSn is a pesudo first-order reaction, which can be described by Langmuir-Hinshelwood equation. Hydroxyl radicals and holes are both the main oxidants in the photocatalytic reaction of RR24 with SiWSn.

**Key words**: photocatalytic degradation; C. I. Reactive Red 24; $K_6SiW_{11}O_{39}Sn^{II}$; reaction kinetics

## Introduction

Synthetic dyes have been widely used in printing and dyeing industry and other fields since 1850's. The amount of dyes produced in the world was estimated to be over 10,000 tons per year. It was assumed that a loss of 1%–2% in production and 1%–10% loss in use were fair estimate. Most of these dyes are toxic and potentially carcinogenic in nature and their removal from the industrial effluents is a major environmental problem (Forgacs et al., 2004). There are several methods used to decolorize the dye wastewater, these including the biodegradation, coagulation, adsorption, advanced oxidation process and the membrane process, among them, advanced oxidation process is a promising method to destroy the toxic dye thoroughly. Polyoxometalates (POM) is a kind of polyfunctional catalyst which can absorb 200–400 nm ultraviolet light. It can be dissolved in water and has strong oxidation when it is activated by absorbing photon. It is proved that POM can be used as photocatalyst for the degradation of organic pollutants (Hiski et al., 2001; Zhu et al., 2004). The development of photocatalysts that maximize the utilization of sunlight (comprised of 40% visible light) is the aim of many recent reports that investigate photon-driven reactions such as water splitting or degradation of organic compounds (Hernandez-Alonso et al., 2009). The most important advantage is the harnessing of an economical, eco-friendly, and abundantly available source of energy – solar energy.

In the present study, the environmental friendly materials, one of POM, $K_6SiW_{11}O_{39}Sn^{II}$(SiWSn), was synthesized. SiWSn is visible light response photocatalyst. The photocatalytic decomposition of C. I. Reactive Red 24 (RR24) with SiWSn radiated with the UV-light (253.7 nm, 20 W), Xenon lamp filtered less than 390 nm light (500 W) and sun light was investigated.

## 1 Materials and methods

### 1.1 Chemicals and instruments

SiWSn was synthesized according to the methods introduced by Deng and Yang (2007). Sodium hydroxide and perchloric acid were all analytical grade. RR24 was donated by State Key Laboratory of Fine Chemicals, Dalian University of Technology, China. Water used in the experiments was deionized and fully aerated by oxygen.

TENSOR27 Fourier transform infrared (FT-IR) spectrometer (Bruker, Germany), UV-2102PCS ultraviolet visible (UV-Vis) spectrophotometer (UNICO, USA), AL104 electronic analytical balance (Mettler Toledo), digital display acidity meter (Shanghai Tianda Instrument Co., Ltd., China), Quartz ultraviolet lamp (20 W, 253.7 nm, Dalian

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Qun Yi Special Light Source Factory, China), LSXS-500 xenon lamp (500 W, Zolix Instruments Co., Ltd., China), SCF-50S-39L filter (cutoff less than 390 nm light, Sigma Koki), were applied in the experiments.

### 1.2 Experimental methods

The UV-Vis spectrum and the maximum absorption wavelength in the visible region of RR24 aqueous solution were measured with UV-Vis spectrophotometer. A certain concentration of RR24 and a certain quantity catalyst SiWSn were put into the glass beaker. The first beaker was put on the rooftop of the No. 1 building at Dalian Jiaotong University, China (E121°54.399′ N38°34.286′, elevation 32 m) with the irradiation of the sun. The second beaker was put under the UV-lamp with 253.7 nm light, and the third beaker was put in front of Xenon lamp with a filter that filtered out all radiation below 390 nm. Samples were withdrawn at specific time intervals. Changes in the absorbance of RR24 were measured by the spectrophotometer at 536 nm. The decoloration rate (DC) was calculated according to Eq. (1):

$$\mathrm{DC} = \frac{A_0 - A}{A_0} \times 100\% \tag{1}$$

where, $A_0$ and $A$ are the absorbance of the initial RR24 solution and the RR24 solution after being irradiated for a certain time, respectively.

It is well-known that Lambert-Beer law is as follows:

$$A = \varepsilon \times l \times C \tag{2}$$

where, $A$ is the absorbance of a chemical solution, $\varepsilon$ (L/(mol·cm)) is the molar extinction coefficient of the chemical, $l$ (cm) is the optical length of colorimetric utensil and $C$ (mol/L) is the concentration of the solution.

According to Lambert-Beer law, the concentration of RR24 solution is proportional to its absorbance, which can be substituted by its absorbance in the photocatalytic degradation reaction kinetics study.

## 2 Results and discussion

### 2.1 Characterization of SiWSn

**Figure 1** shows the FT-IR spectrum and UV-Vis spectrum of SiWSn. From **Fig. 1a**, it can be seen that there are characteristic peaks during 500–1100 nm on the FT-IR spectrum of SiWSn, which are the characteristic peaks of Keggin-type polyoxometalates. From **Fig. 2b**, it can be seen that there is a characteristic peak at 250 nm on the UV-Vis spectrum of SiWSn, which is also the characteristic peak of Keggin-type polyoxometalates. The above results are consistent with literature (Deng and Yang, 2007).

### 2.2 UV-Vis spectrum of RR24 solution

**Figure 2** shows the UV-Vis spectrum of RR24 solution. The maximum absorption wavelength of RR24 solution in the visible region is 536 nm. Therefore, in the following experiments, the absorbances of RR24 solutions were all measured at 536 nm.

### 2.3 Blank experiments

The contrast experiments were carried out in two conditions: one with SiWSn (1.0 g/L) but no illumination, the

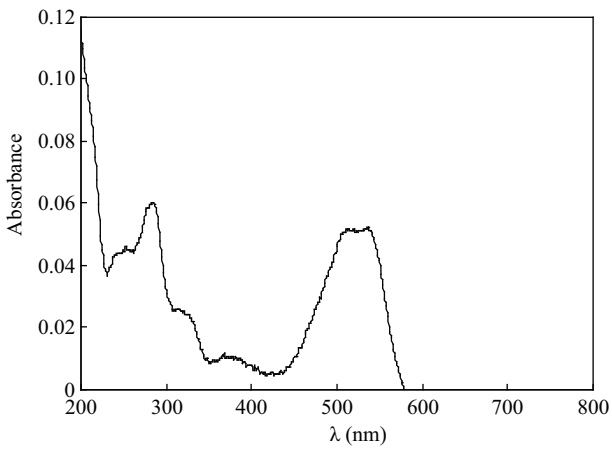


**Fig. 2** UV-Vis spectrum of RR24 solution.

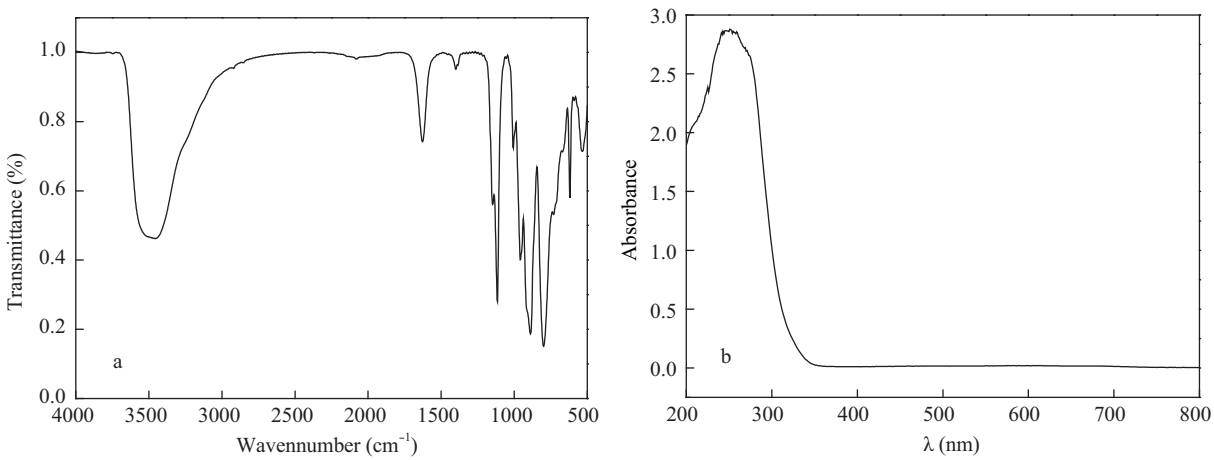


**Fig. 1** FT-IR spectrum (a) and UV-Vis spectrum (b) of SiWSn.

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other with illumination but no catalyst. The results showed that the concentration of RR24 solution (10, 20 mL) kept the same under the former condition, while in the later case changed little with UV-lamp (the decolorization rate of that was only 13.6%), and none with Xenon lamp and sunlight radiation. The blank tests proved the stability of RR24. Without illumination or photocatalysts, RR24 was kinetically stable.

### 2.4 Effect of photocatalysis time and catalyst concentration

The RR24 solution was irradiated under the sunlight. The reaction was measured in different interval photolysis time. **Figure 3** shows the decoloration rate of RR24 solution increased with photolysis time.

Six 50 mL glass beakers contained RR24 solution with different concentration catalyst was irradiated under the sunlight to investigated the effect of catalyst concentration (**Fig. 4**). The result indicates that the decoloration rate of RR24 increased with increasing catalyst concnetration from 0.25 to 1.0 g/L at first. When the catalyst concentration was above 1.0 g/L, the decoloration rate changed unobviously. When the concentration of the catalyst was changed from 1.0 to 2.0 g/L, the decoloration rate changed only 4.0%. According to the above results and considering the economic factor, the concnetration of catalyst 1.0 g/L was chosen in the following experiments.

### 2.5 Photocatalytic reaction kinetics with different light sources

Three 50 mL glass beakers contained RR24 solution and SiWSn catalyst were irradiated with UV-lamp (253.7 nm), Xenon lamp (cutoff less than 390 nm light) and sunlight, respectively.

It is well known that the reaction kinetics would follow the Langmuir-Hinshelwood (L-H) model for heterogeneous reaction system, for example $TiO_2$ photocatalsis system (Baran et al., 2008; Deng and Wu, 2003; Gao et al., 2002). Mylonas and Papaconstantinou (1996) and Turchi and Ollis (1990) also reported that the kinetics follows the L-H model for homogeneous reaction system.

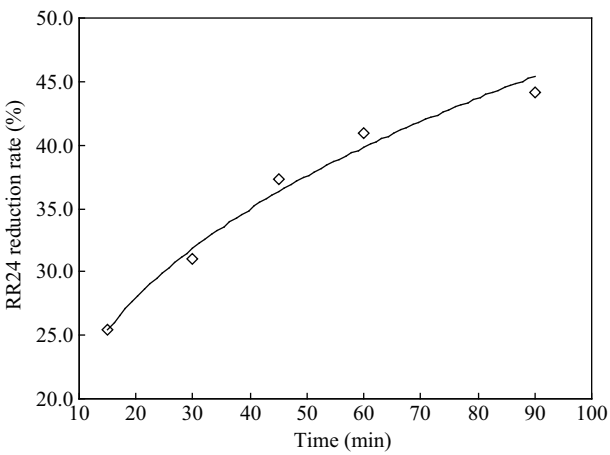


**Fig. 3** Effect of the photocatalysis time on RR24 degradation. Experimental condition: RR24 solution 6 mg/L 20 mL, initial pH 5, catalyst in solution 1.0 g/L, with sunlight irradiation.

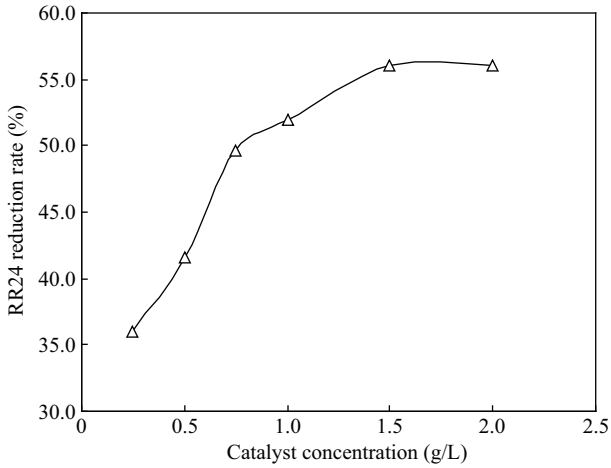


**Fig. 4** Effect of the catalyst concentration on RR24 degradation. Experimental condition: RR24 solution 5 mg/L 20 mL, initial pH 5, with sunlight irradiation 4.5 hr.

$$r = \frac{dC_t}{dt} = \frac{kKC_t}{1 + KC_t} \tag{3}$$

where, $r$ (mg/(L·min)) is photocatalytic reaction rate; $k$ (mg/(L·min)) is L-H rate constant; $K$ (L/mg) is the Langmuir adsorption constant of the RR24 in the photocatalytic reaction; and $C_t$ (mg/L) is the concentration of RR24 under the study.

**Figure 5** shows information on the kinetic of photocatalytic degradation of the RR24 solution with different light sources. The degradation rates fit a first-order model well, that is, the integral equation of $\ln(C_0/C_t) = K_{obs}t$ describes the tendency well, where, $C_0$ (mg/L) and $C_t$ (mg/L) are the concentration of RR24 solution at time 0 and photocatalytic reaction time $t$ (hr), respectively, and $K_{obs}$ ($hr^{-1}$) is the observed pseudo first-order rate

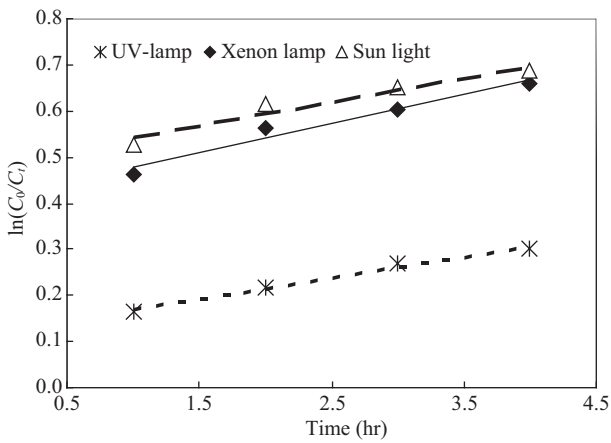


**Fig. 5** $\ln(C_0/C_t)$ of RR24 solution versus photocatalytic reaction time with different light sources. Experimental condition: RR24 solution 6 mg/L 20 mL, initial pH 5, catalyst in solution 1.0 g/L, irradiation 4 hr.

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**Table 1** Reaction kinetic equation and parameter for photolysis RR24 solution

| Light source | Reaction kinetic equation | $K_{obs}$ ($hr^{-1}$) | $t_{0.5}$(hr) | $R$ |
|---|---|---|---|---|
| UV-lamp | $\ln(C_0/C_t) = 0.0453t + 0.1241$ | 0.0453 | 15.3 | 0.9945 |
| Xenon lamp | $\ln(C_0/C_t) = 0.0.0629t + 0.4143$ | 0.0629 | 11.0 | 0.9828 |
| Sun light | $\ln(C_0/C_t) = 0.0513t + 04918$ | 0.0513 | 13.5 | 0.9705 |

constant. The slope of a liner plot of $\ln(C_0/C_t)$ versus time gives the apparent degradation rate constant. To make the further mathematic inferences clear, all the relating kinetic parameters, such as the reaction rate constants ($K_{obs}$), half-life ($t_{0.5}$), and interrelated coefficients ($R$) of the lines in **Fig. 5** are all presented in **Table 1**. From **Fig. 5** and **Table 1**, it can be concluded that the process of photodegradation of RR24 solution with SiWSn followed the first-order reaction kinetics model with studied three light sources.

### 2.6 Effect of initial concentration

Five 50 mL glass beakers contained RR24 solution with different initial concentrations were irradiated under the sunlight. The effect of the initial concentration on decoloration rate is shown in **Fig. 6**. The maximum decoloration rate was 52.0% when the initial concentration of RR24 solution was 5.0 mg/L. It is well-known that the photocatalytic reaction rate, $r$ (mg/(L·min)) can be described by the kinetic equation of Langmuir-Hinshelwood (Deng and Wu, 2003). When the initial concentration of the solution is low, $KC \ll 1$, then,

$$r = kKC = K'C \tag{4}$$

where, $C$ (mg/L) is the concentration of the solution; $k$ (mg/(L·min)) is the Langmuir rate constant; $K$ (L/mg) is the Langmuir adsorption constant and $K'(\text{min}^{-1})$ is the reaction rate constant. Equation (4) indicates that the reaction rate is postively correlated with the solution concentration, therefore, the decoloration rate of the dye solution increased with its initial concentration. However, increasement of the initial concentration of the dye solution resulted in darker colority of the solution, then the optical length of the incident light in the reaction solution became shorter, photocatalytic reaction could not occur in the center of the reactor very well, and as the amount of the catalyst and the photocatalysis time were fixed, the decoloration rate of the dye solution did not increase with its initial concentration all the time

### 2.7 Effect of OH radical and holes scavengers

Generally, there are the similar behavior of the photooxidation of organic compounds with POM and $TiO_2$ systems via a common oxidant OH· produced from the reaction of the excited states of both systems with water molecules (Kormali et al., 2007).

$$\text{M}\,(\text{h}^+ + \text{e}^-) + \text{H}_2\text{O} \rightarrow \text{M}^- + \text{H}^+ + \text{OH}\cdot \tag{5}$$

where, $M(h^+ + e^-)$ represents the excited state of POM or $TiO_2$.

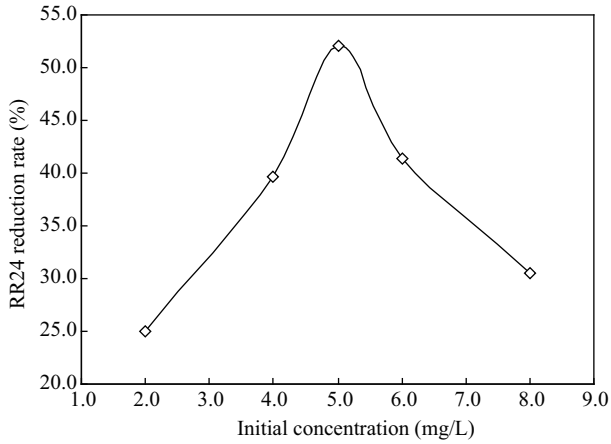


**Fig. 6** Effects of initial RR24 concentration on decoloration rate. Experimental condition: RR24 solution 20 mL, initial pH 5, catalyst in solution 1.0 g/L, with sunlight irradiation 4.5 hr.

In order to elucidate the main oxidant in the photocatalytic reaction with SiWSn, i.e. whether it is exercised via OH· or holes or both, KBr (1 mL, 1 g/L), KI (1 mL, 1 g/L) and isopropyl alcohol (1 mL, 0.01%, $V/V$) were added into RR24 solution (5 mg/L, 20 mL, initial pH = 5) with 1 g/L SiWSn catalyst, respectively. Then the above solutions and the RR24 solution (5 mg/L, 20 mL, initial pH = 5) with 1 g/L SiWSn catalyst were irradiated under the sunlight for 3 hr. Without any scavengers, the decoloration rates of RR24 solution was 40.6%. While in the present of KBr, isopropyl alcohol and KI the decoloration rate of RR24 solution were 33.8%, 6.0% and 14.3%, respectivly.

From the results, it can be seen that OH· scavengers KBr, isopropyl alcohol and holes scavenger KI all had effect on the decoloration rate of RR24 solution. It means that OH· and holes are both the main oxidants in the photocatalytic reaction of RR24 solution with SiWSn.

The mechanistic scheme suggested to take place during the photocatalytic decoloration of RR24 solution in the presence of SiWSn, can be generally summarized as follows:

$$\text{SiWSn} + h\nu \longleftrightarrow \text{SiWSn}\ (\text{h}^+ + \text{e}^-) \tag{6}$$

$$\text{SiWSn}\ (\text{h}^+ + \text{e}^-) + \text{H}_2\text{O} \longleftrightarrow \text{SiWSn}\ (\text{e}^-) + \text{OH}\cdot + \text{H}^+ \tag{7}$$

$$\text{OH}\cdot(\text{h}^+) + \text{RR24} \longrightarrow \text{Oxidation products} \tag{8}$$

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## 3 Conclusions

C. I. Reactive Red 24 solution can be decolored and degraded with the visible light response photocatalyst, SiWSn, under the UV-lamp, Xenon lamp and solar radiation. The decoloration rate of RR24 solution was affected by its initial concentration, the photolysis time and the amount of the photocatalyst. The process of photodegradation of RR24 solutions all followed the first-order reaction kinetic model with the above three kinds of light sources. The main oxidants in the photocatalytic reaction of RR24 solution with SiWSn are hydroxyl radicals and holes.

**Acknowledgments**

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