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Facile synthesis of nano-TiO₂/stellerite composite with efficient photocatalytic degradation of phenol

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ABSTRACT

In this paper, we report a kind of nano-TiO₂/stellerite composite with enhanced photoactivity, which was synthesized by a typical homogeneous precipitation method followed by a calcination crystallization process using natural stellerite as support. The as-prepared composites were characterized via X-ray diffraction (XRD), scanning electron microscopy (SEM), nitrogen adsorption-desorption, high resolution transmission electron microscopy (HRTEM) and X-ray photoelectron spectroscopy (XPS). The results showed that TiO₂ loading amounts and calcination temperatures had significant influence on the adsorption and photocatalytic degradation properties of phenol. Moreover, it was indicated that the TiO₂ nanoparticles (NPs) with smaller grain size (around 12.0 nm) and narrower size distributions were uniformly deposited on the surface of stellerite as a layer of film. Compared with commercial P25, the received composite exhibited more superior photocatalytic degradation performance towards phenol. The enhanced photocatalytic degradation performance should result from the better dispersibility of TiO₂ NPs and higher separation efficiency of photogenerated electron-hole pairs. This work may set foundation for the practical application of this new composite photocatalyst in the field of wastewater treatment.

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1. Introduction

With the rapid development of economy and industry, the types and quantities of wastewater containing phenol are gradually increasing, which are mainly produced in papermaking, petrochemical, pharmaceutical, biotechnology and dye industries. Water pollution and deterioration of ecological environment have become increasingly prominent and threaten human lives greatly. Phenol, a kind of familiar phenolic compounds and basic organic raw materials of chemical industry, is one of the most common organic pollutants. It possesses stable structure and high toxicity, more importantly, which is not easy to be degraded and can cause serious harm to human [1]. Therefore, seeking an economic, efficient and new technology or method to degrade such pollutants has become a research hotspot in the field of environmental treatment.

Nano-TiO₂ photocatalytic degradation technology, as a green processing technology for the environmental pollutants, has been widely concerned due to its advantages including low energy

consumption in treatment, thorough degradation of pollutant and no secondary pollution. Hence, it has a great application prospect in the field of environmental protection [2,3]. However, TiO₂ NPs have many drawbacks in the practical application process, such as easy aggregation, difficult separation and recovery [4,5], and higher production costs. The previous studies had demonstrated that taking natural porous minerals as photocatalyst carriers, like kaolinite [6], opal [7], montmorillonite [8], diatomite [9], attapulgite [10], sepiolite [11], expanded perlite [12] and zeolite [13–16], is an effective way to solve these above issues of single TiO₂ NPs [17–19]. Among these various supporting minerals, zeolite seem to be an attractive choice due to its rich pore structure, superior adsorption properties and ion exchange capability [20,21]. However, in the previous research, most of TiO₂/zeolite composites were prepared using the artificial zeolite as the support, or simply physical mixing of TiO₂ and zeolite [22–25]. In recent years, natural zeolites, such as stellerite, have been employed as a potential substitute for the artificial ones, due to their large reserves, less chemical pollution and low cost in the practical application. As far as we know, few papers paid attention to the construction of nano-TiO₂/stellerite composite. Furthermore, the effect of preparation conditions on the photocatalytic perfor-

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mance of nano-TiO₂/stellerite composite has not been reported hitherto.

In this paper, stellerite, a typical natural zeolite, was selected and employed as the support of nano-TiO₂. And the nano-TiO₂/stellerite composite was prepared under different preparation conditions via homogeneous precipitation method followed by a calcination process. The influence of TiO₂ loading amounts and calcination temperatures on structural characteristics and photodegradation performance of composites was also discussed in detail based on the experimental results. Moreover, the possible degradation mechanism was also investigated.

2. Experimental

2.1. Preparation of nano-TiO₂/stellerite composite

In this work, the natural stellerite (Guangxi, China) was used as the catalyst support. The main chemical compositions are listed in Table 1. All chemicals were analytical grade without any further purification. Deionized water was used throughout all experiments in the research. The nano-TiO₂/stellerite composite was prepared as follows. Firstly, 8 g of stellerite and 240 mL of deionized water was mixed and stirred for 30 min to form suspension. Secondly, different amounts of TiOSO₄ solution (1 M) were added in the suspension. Subsequently, the powdery urea were directly added according to the mole ratio of TiOSO₄/urea = 1:2, and then the mixture continue to react for 2 h at 75 °C in water bath. The resulted products were transformed to the precursors of composites by filtering, washing and drying at 105 °C. Finally, various nano-TiO₂/stellerite composites were obtained after calcining at different temperatures for 2 h. The samples and stellerite were denoted as TS-X%-Y°C and S respectively, where the X and Y represented loading amounts (wt.%, the mass ratio of TiO₂ to stellerite) and calcination temperatures, respectively. The pure TiO₂ power was also prepared as reference without support in the same way, which was labeled as T-X%-Y°C.

2.2. Characterization

To detect the chemical composition of stellerite, the XRF-1800 X-ray fluorescence spectrometer was adopted. X-ray diffraction technique was used to analyse phase composition of samples through adopting the D8 ADVANCE X-ray power diffractometer (Bruker, Germany) at a scanning speed of 4°/min from 5° to 80° with Cu K α radiation; The S-4800 scanning electron microscope (SEM) equipped with an energy-dispersive spectrometer (EDS) was used to analyze the surface morphology and elements constitution of samples. The Brunauer–Emmett–Teller (BET) surface area (S_{BET}) and pore structural parameters were obtained through nitrogen adsorption–desorption isotherm and the Barret–Joyner–Halender (BJH) method using the JW-BK nitrogen adsorption apparatus (China). Internal structure morphology and crystal lattice fringes of the sample were further investigated by HRTEM (Philips Tecnai G2 F20). Additionally, surface elements and the chemical states were determined and analyzed quantitatively through X-ray photoelectron spectroscopy (XPS) on a Thermo Escalab 250Xi X-ray photoelectron spectrometer adopting Al K α radiation

(15 kV, 150 W). The peak of C1s at 284.8 eV was used to calibrate binding energies of the elements.

2.3. Adsorption and photocatalytic performances

The adsorption and photocatalytic properties of composites were evaluated using phenol solution as the target pollutant in a photochemical reactor (Shanghai Bilon Instruments Co. Ltd.). In each experiment, 0.2 g of catalysts were added into 100 mL of phenol solution (20 mg/L). After ultrasonic dispersion for 5 min, the suspension was stirred for 1 h in the dark condition to obtain the adsorption-desorption equilibrium on the surface of photocatalysts. Then the photocatalytic reaction was carried out under the irradiation of the UV light source (400 W mercury lamp). At the preset time interval, 4 mL of the resulted supernatant was taken and centrifuged, and then the absorbance of phenol solution was measured through adopting 4-amino antipyrine spectrophotometry. The degradation rate of phenol solution (DR) was calculated by the following formula:

$$\text{DR} = (C_0 - C_t)/C_0 \times 100\% \quad (1)$$

In the above equation, C_0 represents the initial concentration of phenol solution and C_t is the concentration at time t .

In addition, to inspect the relationship between dark adsorption property and photocatalysis under the UV light, the equilibrium adsorption capacity (Q_e) of different samples were calculated by the following formula:

$$Q_e = V(C_0 - C_e)/M \times 100\% \quad (2)$$

where V is the volume of phenol solution, C_0 and C_e refer to the concentration of phenol solution at initial time and after reaching adsorption equilibrium respectively, and M is the quality of the sample to be tested.

In this paper, the kinetics of photocatalytic reaction present a linear relationship. The values of k_{app} were obtained by the slope of the line plotting and fitting with $\ln(C_0/C_t) \sim t$, where C_0 and C_t are the concentration of phenol solution at the beginning time of the reaction and at given time t , respectively.

3. Results and discussion

3.1. Effect of TiO₂ loading amounts

The XRD analysis results of TS-X%-450°C are shown in Fig. 1. It can be seen that the characteristic peaks of stellerite (JCPDS 25-0124) in sample stellerite raw. All samples show the peaks of quartz (JCPDS 78-1252). However, the diffraction peaks of stellerite in TS-X%-450°C disappear, which can be explained that the thermal dehydration of stellerite occurs when the composites are calcined under the relatively high temperature [26,27]. This means that stellerite structure has been destroyed during the calcination process. Besides, In addition, TS-X%-450°C have distinct peaks relating to anatase phase TiO₂ (JCPDS 21-1272) at $2\theta = 25.8^\circ$ (1 0 1), 38.4° (0 0 4) and 48.6° (2 0 0). It verified that TiO₂ NPs had indeed been loaded on stellerite. Furthermore, the intensities of these peaks gradually enhance, while the peak shape become more and more sharper with the increase of TiO₂ loading amount. The average crystallite size of TiO₂ was calculated by Debye-Scherrer Eq. (3).

Table 1
Main chemical compositions of stellerite (wt.%).

Sample	SiO ₂	Al ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	Fe ₂ O ₃	L.O.I. ^a
Stellerite	59.28	12.05	9.87	0.32	0.14	0.08	0.09	18.17

^a Loss on ignition.

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