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Original Research Paper

Synthesis of Bentonite-Supported Fe₂O₃-Doped TiO₂ superstructures for highly promoted photocatalytic activity and recyclability

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ABSTRACT

A magnetic recyclable Bentonite-Supported Fe₂O₃-Doped TiO₂ (Fe₂O₃-B-TiO₂) photocatalyst with high visible light photocatalytic activity was synthesized by a facile sol-gel method. The samples thus synthesized were characterized by multiple techniques, including the scanning electron microscopy, transmission electron microscopy, powder X-ray diffraction, X-ray photoelectron spectroscopy, fourier transform infrared spectroscopy, X-ray fluorescence, vibrating sample magnetometer, electron paramagnetic resonance and ultraviolet–visible diffuse reflectance spectroscopy. The results confirmed the magnetic Fe₂O₃-B-TiO₂ photocatalysts were synthesized successfully. The photocatalytic activity was evaluated by the degradation of methyl blue (MB) under the irradiation of visible light. The Fe₂O₃-B-TiO₂ exhibited the best photocatalytic activity among all the samples. Almost all of the MB was degraded by Fe₂O₃-B-TiO₂ within 120 min. The enhancement of photocatalytic activity could be attributed to the better optical absorption properties, stronger oxidation ability by hydroxyl radicals as well as the enhanced dispersion of TiO₂ nanoparticles. In addition, the newly synthesized Fe₂O₃-B-TiO₂ can be collected by magnetic separation easily and maintained high photocatalytic efficiency for several time reuses. Our findings pave a way to design recyclable Fe₂O₃-B-TiO₂ photocatalysts for photodegradation of organic pollutants in aqueous solution.

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1. Introduction

Titanium dioxide (TiO₂) as a semiconductor photocatalyst has been widely used in the degradation of various pollutants because of its fine electronic properties, high photocatalytic activity and chemical stability [1,2]. However, anatase TiO₂ imposes limitation due to its large band gap structure (3.2 eV). Only UV light can be utilized for realizing the photoelectric reaction [3,4]. In addition, it is difficult to separate the TiO₂ nanoparticles from reaction system for recycling use [5]. Therefore, it is essential to improve the photocatalytic activity and solve the recyclability issues of TiO₂ nanoparticles. Several methods have been employed for improving the photocatalytic activity, such as tuning of reactive facets and doping with impurities.

The synthesizing of modified TiO₂ photocatalysts has attracted much attention as an effective method to improve the photocatalytic efficiency. The environmental friendly material of Fe₂O₃

was widely used to improve the photocatalytic efficiency of TiO₂ because of its stability, low cost and high visible light adsorption capacity [6]. Many studies showed that the photocatalytic activity could be greatly improved by modified TiO₂ with α -Fe₂O₃ [7,8]. This result could be attributed to the fact that a dopant energy level was formed by α -Fe₂O₃ [9].

Moreover, magnetic TiO₂ catalysts, which synthesized by doping the TiO₂ with Fe₃O₄, were manipulated to solve the recyclability issue. However, the synthesizing process of Fe₃O₄-TiO₂ was complicated [10–12]. Very recently, the fabrication of magnetic γ -Fe₂O₃ nanomaterials shed a new light on designing recyclable catalysts due to its magnetism [13]. Generally speaking, the γ -Fe₂O₃ was synthesized via the calcination of Fe₃O₄ in low temperature. Subsequently, the γ -Fe₂O₃ particles were doped into TiO₂ system to fabricate the magnetic TiO₂ photocatalysts [14,15]. However, such method can't resolve the aggregation problem of TiO₂ nanomaterials. For this reason, the clay mineral of bentonite, with thermal stability and strong cations exchange ability, attracted much attention as a supporter to prevent the aggregation of photocatalysts [16–19].

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In this study, the Bentonite-Supported Fe_2O_3 -Doped TiO_2 photocatalysts (Fe_2O_3 -B- TiO_2) were successfully synthesized via a facile sol-gel method. The photocatalytic activity of the photocatalysts was measured by the degradation of methyl blue (MB) under the visible light irradiation. The morphologies, components, surface electronic states as well as the optical absorption properties of these samples were well measured. The results showed that the Fe_2O_3 -B- TiO_2 photocatalysts exhibited not only the enhanced photocatalytic activity, but also the excellent recyclability. The possible photocatalytic mechanism was also evaluated.

2. Experimental section

2.1. Materials

Tetrabutyl titanate (TBOT), ethyl alcohol, ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$), acetic acid, hydrochloric acid and Na-bentonite were purchased from Sinopharm Chemical Reagents Company, China. All the chemicals used in this experiment were analytical grade without further purification.

2.2. Fabrication of photocatalysts

Pure TiO_2 and bentonite supported TiO_2 were synthesized by sol-gel methods. In a typical synthesis, 20 mL tetrabutyl titanate, 60 mL absolute ethanol and 2 mL acetic acid were firstly mixed in a three-necked flask. Secondly, 0.6 mL HCl, 20 mL ethanol and 8 mL ultrapure water (18.3 M Ω cm, 25 °C) were mixed and added dropwise into the above mixed solution under vigorously stirring. Thirdly, 1 g Na-bentonite was added into the mixtures and stirred continuously for 1 h to synthesize the bentonite loaded TiO_2 . Finally, the solutions were kept in room temperature for 24 h and dried at 80 °C for 12 h to form the xerogel.

The magnetic photocatalysts were fabricated during the calcining process of xerogel. Fig. 1 shows the fabrication process of magnetic photocatalysts. Firstly, FeCl_3 solution was prepared by adding 40 g $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ into 50 mL water. Secondly, 4 g of above xerogel was put into 20 mL FeCl_3 solution. Thirdly, the solution was stirred for 2 h and heated for 4 h at 70 °C in air to form the red brown precipitates. The fourth, the precipitates were calcined at 600 °C for 2 h in N_2 atmosphere. Finally, the calcined samples were collected and washed by deionized water for three times.

2.3. Characterization of photocatalysts

The morphologies and micro structures of modified magnetic TiO_2 catalysts were characterized by various methods including scanning electron microscopy (SEM, JSM-6460LV, JEOL, Japan) and transmission electron microscope (TEM, JEM-2100, JEOL, Japan). The weight percentages of component in as-prepared catalysts were measured by X-ray fluorescence spectrometry (XRF, XRF-1800, Shimadzu, Japan). The crystalline structures of photocatalysts were determined by X-ray diffraction (XRD, Rigaku D/Max 2500, Japan). The surface electronic states and the chemical compositions of the catalysts were characterized by X-ray photoelectron spectroscopy (XPS, ESCALAB 250 Xi, Thermo Scientific, U.S.A.). Fourier transform infrared spectra were recorded on a FT-IR instrument (Spectrum 100 FT-IR spectrometer, PerkinElmer, U.S.A.). The UV-Vis absorption spectra of the catalysts were recorded on TU-1901 spectrophotometer. The specific surface areas of the composites were measured using an AuPosorb IQ2 nitrogen adsorption/desorption apparatus (Quantachrome, U.S.A.). Electron paramagnetic resonance (EPR) spectra were recorded with a Bruker E580 CW spectrometer at 295 K, operating with a microwave frequency of 9.7 GHz. The hydroxyl radicals were captured by DMPO in water. The magnetism of catalysts was measured by vibrating sample magnetometer (VSM-7307, Lake Shore Cryotronics, Westerville, U.S.A.).

2.4. Photocatalytic activity measurements

The photocatalytic activities of the samples were evaluated by the degradation of methyl blue (MB) in batch experiments under the irradiation of visible light (500 W xenon lamp) with a UV cut-off filter. Firstly, 0.2 g of photocatalysts were added in a reaction vessel containing 200 mL MB aqueous solution at a concentration of 10 mg/L. Prior to irradiation, the suspension was vibrated in dark for 30 min to attain adsorption-desorption equilibrium of MB. Then, the solution was illuminated by visible light for 120 min with continuously shaking. During the photodegradation reactions, MB solution (2 mL) was collected from the batch reactors at a certain time intervals and filtered immediately through a Millipore filter (pore size, 0.22 μm). Finally, the MB concentration of the supernatant was determined by a UV-Vis spectrometer (UV-1750, Shimadzu, Japan).

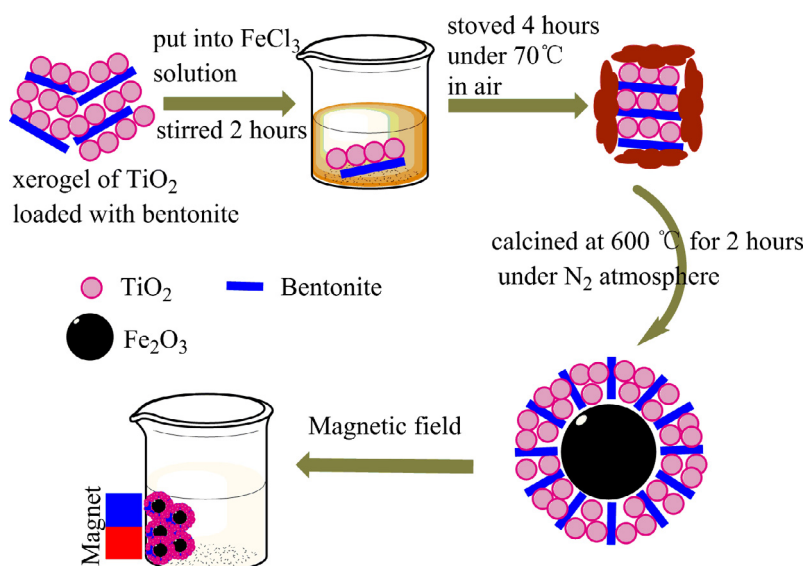


Fig. 1. Schematic illustration for the synthesis of Fe_2O_3 -B- TiO_2 .

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