



Short communication

Synthesis of α -Fe₂O₃/ZnO composites for photocatalytic degradation of pentachlorophenol under UV–vis light irradiation

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Abstract

A series of α -Fe₂O₃/ZnO composites with different α -Fe₂O₃ contents were successfully prepared by a simple and rapid method. The products were characterized by XRD, FESEM and UV–vis DRS. Photocatalytic activity of the as-prepared α -Fe₂O₃/ZnO composites was evaluated by degradation of persistent organic pollutant pentachlorophenol (PCP) in aqueous solution. Compared with pure ZnO, α -Fe₂O₃/ZnO composites show higher photocatalytic activity under UV–vis light irradiation. PCP is almost completely degraded by α -Fe₂O₃/ZnO composite with molar ratio 1:5 within 4 h.

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1. Introduction

Semiconductor photocatalysis has been attracting considerable attention for a long time because of its great potential to solve environmental problems [1]. Among the semiconductors employed, TiO₂ is universally recognized as the most photoactive catalyst. In fact ZnO is also an important semiconductor photocatalyst, because the wide direct band gap (3.37 eV) of ZnO is almost the same as that of TiO₂ (3.2 eV) and its photodegradation mechanism is similar to that of TiO₂ [2]. Despite the positive attributes of ZnO photocatalyst, however, there are some drawbacks associated with its use: (i) charge carrier recombination occurs very fast [3] and (ii) the large band gap does not allow the utilization of visible light [4]. One of the ways to overcome these problems is to modify ZnO by addition of another semiconductor. In the past several years, coupled semiconductors formed by ZnO and other metal oxides such as TiO₂, SnO₂, CuO, WO₃ and so on [5–8] have been reported.

Compared to single ZnO photocatalyst, the coupled semiconductor systems possess higher catalytic activity.

Hematite (α -Fe₂O₃) is the most thermodynamically-stable phase of iron oxide under ambient conditions with low cost, high resistance to photocorrosion and environment-friendly features. It can be driven by visible light up to 600 nm due to the narrow band gap of 2.2 eV, and has been confirmed to be an important member of visible-light-responsive semiconductor photocatalysts [9]. Although combinations of ZnO with Fe₂O₃ have been investigated in order to enhance the photocatalytic activity of ZnO on organic compound's photodegradation, most of the used synthetic methods are complicated and need templates and high temperature as well as special solvents and additives [10–12].

In this work, a series of α -Fe₂O₃/ZnO composites with different α -Fe₂O₃ contents were successfully prepared via a simple aqueous solution route using cheap reagents. Photocatalytic activity of the as-prepared α -Fe₂O₃/ZnO composites was evaluated by degradation of pentachlorophenol (PCP) in aqueous solution under UV–vis light irradiation. PCP is a widespread soil and water contaminant [13], which can produce mutations in animal (including human) cells and exhibit teratogenic and carcinogenic effects [14]. To the best

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of our knowledge, there is no similar report about α -Fe₂O₃/ZnO composites for photocatalytic degradation of PCP under simulated solar light.

2. Experimental

All of the chemicals were of analytical grade and used as received. Firstly, according to the molar ratio of α -Fe₂O₃ to ZnO, sodium hydroxide (6.0 mol L⁻¹) was added into some amount of ferric nitrate (1 mol L⁻¹) until pH 10 under vigorous stirring. Then the reaction solution was heated and refluxed for 1.5 h to obtain precursor solution A. At the same time, the pH of zinc nitrate (1 mol L⁻¹, 20 mL) was also adjusted to the desired value (10) with sodium hydroxide (4.0 mol L⁻¹) under vigorous stirring to get precursor solution B. Finally, precursor solution B was quickly added to precursor solution A, keeping the boiling state for 3 h. The hot solution was cooled to room temperature and then filtered. Products were collected and dried in air to constant weight.

X-ray diffraction (XRD) data were collected from powdered samples using a Bruker D8 Advance diffractometer with Cu K α radiation. Field emission scanning electron microscopy (FESEM) images were taken by using Hitachi S-4800. UV–vis diffuse reflectance spectra (UV–vis DRS) were recorded on a Hitachi UV-3010 spectrophotometer equipped with an integrating sphere.

Photocatalytic experiments were carried out in a 250 mL beaker. A total of 0.15 g photocatalyst powders was added into 100 mL of the PCP aqueous solution (10 mg L⁻¹, pH 9–10). The simulated solar light irradiation was provided by a 500 W xenon lamp. The distance between the solution and the lamp was about 10 cm. Before illumination, the suspension was stirred continuously in dark for 30 min to ensure adsorption equilibrium. Suspension containing PCP and powder catalyst, taken out at given intervals of time, was centrifugally separated and filtrated through a millipore filter to remove the catalyst. The kinetic photodecomposition process was monitored by measuring the residuary concentration of PCP with a UV–vis spectrophotometer at 220 nm [15].

3. Results and discussion

Fig. 1 shows the XRD patterns of pure ZnO and α -Fe₂O₃/ZnO composites with different mole ratios of α -Fe₂O₃ to ZnO. All the diffraction peaks can be well indexed to ZnO phase (JCPDS No. 36-1451) and α -Fe₂O₃ phase (JCPDS No. 33-0664), and no characteristic peaks from other impurities are detected. With the rising mole ratio of α -Fe₂O₃ to ZnO the peak intensity of α -Fe₂O₃ phase increases due to the growth of crystallites and enhancement of crystallization, while the peak intensity of ZnO phase decreases accordingly.

Fig. 2 presents FESEM images of pure ZnO and α -Fe₂O₃/ZnO composites with different mole ratios of α -Fe₂O₃ to ZnO. The pure ZnO particles are quasi-spherical or of spindle shape. The average diameter of the pseudo-spherical particles is 48 nm, while the diameter and the length of the spindle-shaped particles are about 0.11 μ m and 0.23 μ m respectively. When the mole

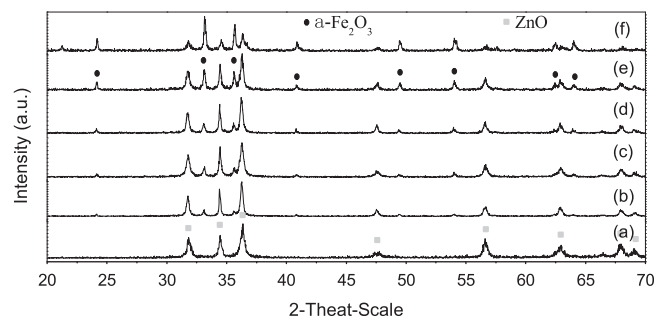


Fig. 1. XRD patterns of pure ZnO and α -Fe₂O₃/ZnO composites with different mole ratios of α -Fe₂O₃ to ZnO: (a) pure ZnO; (b) 1:9; (c) 1:7; (d) 1:5; (e) 1:3 and (f) 1:1.

ratio of α -Fe₂O₃ to ZnO is 1:9, α -Fe₂O₃/ZnO particles exhibit a well-defined 1D rod-like morphology with average diameter of 69 nm and length of 0.34 μ m (aspect ratio \sim 5). Although the average diameter of α -Fe₂O₃/ZnO (1:7) particles is similar to that of α -Fe₂O₃/ZnO (1:9) particles, their lengths are obviously shorter, in the range of 0.1–0.3 μ m. Both diameter and length of α -Fe₂O₃/ZnO (1:5) particles decrease dramatically, and their maximum values are 91 nm and 0.24 μ m respectively. With the continued increase in the mole ratio of α -Fe₂O₃ to ZnO, pseudo-spherical α -Fe₂O₃/ZnO particles with \sim 150 nm diameter present in the samples and become the main product when the mole ratio of α -Fe₂O₃ to ZnO reaches 1:1. It is apparent that the existence of the secondary oxide (α -Fe₂O₃) affects the particle size and morphology.

The UV–vis diffuse reflectance spectra of different samples are shown in Fig. 3. It can be seen that compared with pure ZnO, which can absorb only ultraviolet light, the adsorption edge of α -Fe₂O₃/ZnO composites exhibits red-shift, and the red-shift increases with the rise of α -Fe₂O₃ content. Moreover, when the mole ratios of α -Fe₂O₃ to ZnO reach 1:3 and 1:1, the samples show two-edge absorption, covering wavelengths from 200 to 800 nm. This result suggests that the α -Fe₂O₃/ZnO composites have potential for photocatalysis using the visible part of the spectrum.

Fig. 4 shows the degradation efficiencies of PCP over different samples. Blank study (absence of catalyst) was carried out as a background check. Without the photocatalyst, only 22% of PCP was degraded after 4 h, due to photolysis. In contrast, when catalysts were placed in the solution and UV–vis light was turned on, the degradation efficiency of PCP was much higher than that of direct photolysis. The α -Fe₂O₃/ZnO composites displayed obviously better catalytic activity than pure ZnO. However, it is noteworthy that the optimal mole ratio of α -Fe₂O₃ to ZnO was 1:5, not 1:3 or 1:1. PCP was almost completely degraded by α -Fe₂O₃/ZnO (1:5) within 4 h.

There are two main reasons why the photocatalytic activity of α -Fe₂O₃/ZnO composites improves greatly. First, the difference in energy levels between α -Fe₂O₃ and ZnO narrows the band gap of α -Fe₂O₃/ZnO composites, thus extends the optical absorption range and enhances the light utilization efficiency. Second, because the conduction band position of ZnO is higher than that of α -Fe₂O₃ and the ferric ions in α -Fe₂O₃ are easily

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