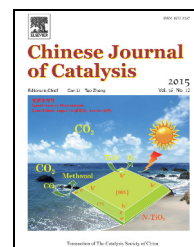


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Article (Special Issue on Photocatalysis)

Characterization of V_2O_5/MoO_3 composite photocatalysts prepared via electrospinning and their photodegradation activity for dimethyl phthalate

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ARTICLE INFO

Article history:

Received 26 August 2015

Accepted 29 October 2015

Published 20 December 2015

Keywords:

Electrospinning

Vanadium pentoxide

Molybdenum trioxide

Composites

Photodegradation activity

Dimethyl phthalate

ABSTRACT

Vanadium pentoxide (V_2O_5)/molybdenum trioxide (MoO_3) composites with different molar ratios of vanadium (V) to molybdenum (Mo) were synthesized via a simple electrospinning technique. The photocatalytic activity of the composites were evaluated by their ability to photodegrade methylene blue and dimethyl phthalate (DMP) under visible-light irradiation. Compared with pure V_2O_5 and MoO_3 , the V_2O_5/MoO_3 composites showed enhanced visible-light photocatalytic activity because of a V 3d impurity energy level and the formation of heterostructures at the interface between V_2O_5 and MoO_3 . The optimal molar ratio of V to Mo in the V_2O_5/MoO_3 composites was found to be around 1/2. Furthermore, high-performance liquid chromatographic monitoring revealed that phthalic acid was the main intermediate in the photocatalytic degradation process of DMP.

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1. Introduction

Dimethyl phthalate (DMP) is widely used as an additive in the manufacture of plastics, polyvinyl acetates and cellulose even though it is an endocrine-disrupting chemical and has great potential to interfere with the hormonal control systems of humans. With the manufacture and use of DMP, it is inevitably discharged into the environment [1–3]. It is important to effectively remove DMP from aqueous systems to minimize its adverse effects. An attractive method to remove DMP is photocatalytic treatment using solar radiation in the presence of a photocatalyst. In this respect, a semiconductor photocatalyst

that is inexpensive and stable under ambient conditions is required for use in clean technology.

In recent years, applications of semiconductors in the elimination of environmental pollutants from aquatic solutions have been attracting increasing attention because of their high physicochemical stability and photocatalytic performance [4,5]. Semiconductors like titanium dioxide (TiO_2) [6–9], molybdenum trioxide (MoO_3) [10–13], vanadium pentoxide (V_2O_5) [14–16], tungsten oxide (WO_3) [17–19], cerium oxide (CeO_2) [20], zinc oxide (ZnO) [21], and cadmium sulfide (CdS) [22,23], are widely used as photocatalysts. However, a common drawback of these photocatalysts is the fast recombination of pho-

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This work was supported by the National Natural Science Foundation of China (21373120, 21471022), the Development of Science and Technology Plan of Jilin Province, China (20101549, 20130102001JC), Program for Changjiang Scholars and Innovative Research Team in University (PCSIRT13022) of China, and the Program of Jilin Provincial Education Department (2013130, 2013146).

DOI: 10.1016/S1872-2067(15)61002-6 | <http://www.sciencedirect.com/science/journal/18722067> | Chin. J. Catal., Vol. 36, No. 12, December 2015

photogenerated electron-hole pairs in them, which lowers their photocatalytic efficiency. Several methodologies have been used to decrease the recombination rate of photogenerated electron-hole pairs and to enhance the photocatalytic efficiency of semiconductor photocatalysts, such as doping with metals (gold, vanadium, tungsten, strontium) [24–27] or nonmetals (carbon, nitrogen, sulfur) [28–30], surface modification [31,32], and combination with another semiconductor [33,34]. In our previous work, we used the semiconductor compound MoO_3 as a dopant to improve the photocatalytic performance of V_2O_5 and obtained the predicted improvement in performance [35]. It is well known that heterostructures formed by integrating two or more semiconductors can theoretically improve photocatalytic efficiency because the photogenerated electrons can migrate from a semiconductor with a higher conduction-band (CB) minimum to another with a lower CB minimum.

Besides being coupled with other semiconductors to improve the photocatalytic performance of the resulting composites [36,37], MoO_3 has also been widely used because of its unique structure and chemical properties [10]. What will happen when MoO_3 is used as the host and V_2O_5 as the dopant? In the present contribution, we explore the influence of a molar ratio of V to Mo, $n(\text{V})/n(\text{Mo})$ on the structure and photocatalytic activity of the resulting $\text{V}_2\text{O}_5/\text{MoO}_3$ composites. The $\text{V}_2\text{O}_5/\text{MoO}_3$ composites are fabricated via electrospinning, a useful technique for the preparation of composites with controllable hierarchical features [38]. The effect of $n(\text{V})/n(\text{Mo})$ on the structure, morphology, surface properties, and optical absorption of the composites, as well as their application in the degradation of DMP are analyzed.

2. Experimental

2.1. Preparation of catalysts

All reagents were of analytical grade and used as purchased from commercial suppliers without further purification. $\text{V}_2\text{O}_5/\text{MoO}_3$ composites with different $n(\text{V})/n(\text{Mo})$ were synthesized by the following electrospinning and calcination processes. Polyvinyl pyrrolidone (PVP, 1 g) was dissolved in ethanol (9 mL) and stirred for 10 h to form solution (A). Ammonium molybdate tetrahydrate $((\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}, 0.4618 \text{ g})$ and ammonium metavanadate $(\text{NH}_4\text{VO}_3, 0.0382 \text{ g})$ ($n(\text{V})/n(\text{Mo}) = 1/8$) were dissolved in 50% ethanol solution (4 mL) and then stirred for 10 h to form solution (B). Solution (B) was added to solution (A), and then the resulting mixture was stirred for 10 h at room temperature. A viscous gel of PVP/ $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}/\text{NH}_4\text{VO}_3$ formed during stirring. The as-obtained gel was transferred to a syringe, and a piece of copper wire connected to a high-voltage generator was inserted into the gel. A direct current voltage of 16 kV was applied for electrospinning. A piece of flat aluminum foil was placed 16 cm under the tip of the syringe to collect the composite fibers. The obtained composite fibers were calcined at a heating rate of $1^\circ\text{C}/\text{min}$ and held for 3 h at 300, 400 or 500 $^\circ\text{C}$ in air. Composites with $n(\text{V})/n(\text{Mo}) = 1/8, 1/6, 1/4, 1/2$ and $1/1$, which are denoted as VM-8, VM-6, VM-4, VM-2 and VM-1, respectively, were

prepared under the same conditions. For comparison, pure MoO_3 and V_2O_5 photocatalysts were also prepared under the same conditions.

2.2. Characterization

The crystal structure of samples was determined by X-ray diffraction (XRD) patterns recorded on a Rigaku D/Max-IIB diffractometer with $\text{Cu } K_\alpha$ ($\lambda = 0.15405 \text{ nm}$) radiation at a scan rate of $4^\circ/\text{min}$ in the range of $2\theta = 10^\circ\text{--}80^\circ$. The morphology of samples was observed by field-emission scanning electron microscopy (FE-SEM, FEI-Philips XL-30) and transmission electron microscopy (TEM, Philips T20ST). The specific surface areas (SSA) of samples were measured at liquid N_2 temperature using the Brunauer-Emmett-Teller method (BET, JW-K). X-ray photoelectron spectra (XPS) were recorded on an ESCALAB-MKII photoelectron spectrometer with $\text{Al } K_\alpha$ (1468.6 eV) radiation as the excitation source. Ultraviolet-Visible diffuse reflectance spectra (UV-Vis DRS) were obtained by a Lambda 900 UV-Vis-NIR spectrophotometer (Perkin-Elmer). The identification of degradation intermediates of DMP was performed by high-performance liquid chromatography-tandem mass spectrometry (OA_SPE Waters Xevo TQ_S).

2.3. Photocatalysis experiments

Photocatalysis experiments using the composites were performed in a self-assembled photoreactor at room temperature. The photoreactor used a 500-W high-pressure xenon lamp as a visible-light source ($\lambda > 420 \text{ nm}$), which was surrounded by a water-cooling quartz jacket and with a UV cutoff filter. For the photocatalytic degradation of methylene blue (MB) solution, MB aqueous solution (10 mg/L, 100 mL) and catalyst (50 mg) were stirred in the dark for ca. 30 min to let the catalyst disperse completely and establish an adsorption-desorption equilibrium between MB and catalyst. At given time intervals, 10-mL aliquots of the reaction mixture were withdrawn and centrifuged; the absorption of the resulting clear solutions at $\lambda_{\text{max}} = 664 \text{ nm}$ was examined using a UV-Vis 756B spectrophotometer. Photocatalytic degradation of DMP solution by the catalysts was tested under similar conditions. DMP solution (40 mg/L, 60 mL) and catalyst (100 mg) were stirred in the dark for ca. 30 min. Every hour, a 10-mL aliquot of the reaction mixture was withdrawn and centrifuged; the absorption of the clear solution at $\lambda_{\text{max}} = 230 \text{ nm}$ was detected by a UV-Vis 756B spectrophotometer. As control experiments, the photocatalytic degradation of both substrates was also tested in the absence of catalyst.

3. Results and discussion

3.1. Structure and morphology

The crystalline phases of pure MoO_3 , V_2O_5 , and the $\text{V}_2\text{O}_5/\text{MoO}_3$ composites were analyzed by XRD. As shown in Fig. 1(a), all of peaks observed in the XRD patterns of the composites can be indexed to orthorhombic MoO_3 (JCPDS 05-0508)

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