



Full Length Article

Fullerene (C₆₀)/CdS nanocomposite with enhanced photocatalytic activity and stabilityQiang Cai^a, Zhuofeng Hu^{b,**}, Qian Zhang^a, Boyuan Li^a, Zhurui Shen^{a,*}^a Key Laboratory of Advanced Ceramics and Machining Technology, Ministry of Education, School of Materials Science and Engineering, Tianjin University, Tianjin 300072, PR China^b Department of Chemistry, The Chinese University of Hong Kong, Shatin, New Territories, Hong Kong, PR China

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ABSTRACT

Herein, the fullerene (C₆₀)/CdS nanocomposite has been fabricated by a facile one-pot hydrothermal method. Its photocatalytic hydrogen (H₂) evolution rate and degradation efficiency of Rhodamine B (Rh B) are evaluated under visible light irradiation ($\lambda \geq 420$ nm). The content of C₆₀ has been changed from 0.4 wt% to 8 wt%, and the optimal value for photocatalytic activity is determined to be 0.4 wt%. The H₂ evolution rate over this optimal sample reaches 1.73 mmol h⁻¹ g⁻¹ and its apparent degradation rate of Rh B is 0.089 min⁻¹ (degradation efficiency of 97% within 40 min), which is 2.3 times and 1.5 times compared to that of pure CdS reference. Moreover, the photocorrosion of CdS in composite is effectively suppressed, and its photocatalytic activity can be well maintained after three recycles (97.8% retaining for composite vs. 84.4% retaining for CdS). Then, the enhanced photocatalytic activity and stability of C₆₀/CdS nanocomposite are further studied by spectroscopic and electrochemical methods. Results show that the C₆₀ species covering on the surface of CdS can efficiently accelerate the separation and transfer of photoexcited charge carriers, which can improve its activity, and reduce the photocorrosion of CdS.

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1. Introduction

Nowadays, environmental and energy crisis has become one of the most severe global problems, and tremendous efforts have been dedicated in environmental remediation and sustainable energy production over the decades [1–3]. Among kinds of solutions, photocatalysis is very promising due to its simplicity, low cost and direct utilization of solar energy [4–7]. Therefore, many excellent photocatalysts have been developed, such as TiO₂ [8–10], g-C₃N₄ [11–13], MoS₂ [14,15] and CdS [16–18]. Amongst, hexagonal cadmium sulfide (CdS) has been widely studied due to its suitable bandgap of 2.45 eV (the conduction band minimum is -0.97 V vs. NHE and valence band maximum is +1.48 V vs. NHE), which matches well with the visible region of solar light [19–23]. Furthermore, its conduction band (CB) edge position is more negative than that of most studied semiconductors [24]. Thus, CdS is a fas-

inating visible-light-driven photocatalyst. For example, Bao et al. [25] have prepared nanoporous CdS by a two-step aqueous route with a high hydrogen yield of about 4.1 mmol h⁻¹ (0.15 g) under visible light irradiation.

However, there are two main disadvantages still limiting further applications of CdS-based photocatalysts. One is the high recombination rate of photoinduced electron-hole pairs, which restricts the photocatalytic activity of CdS [26,27]. The other is its inherent photocorrosion problem [28,29], in which the S²⁻ would be easily oxidized by photogenerated holes. To address these problems, several methods have been developed. Besides the external doping [30,31] and loading of co-catalysts [32–34], choosing carbon as a modified material has received much attention recently. Photocatalyst combining carbon-based materials, such as carbon nanotubes (CNTs) [35–37], graphene oxide (GO) [38–42] and amorphous carbon [43,44], could effectively enhance the photocatalytic activity of CdS.

Fullerene (C₆₀) represents a third allotrope of carbon with unique properties owing to its special delocalized conjugated structures [45]. C₆₀ is well-known for its excellent electron affinities, which is efficient for electron transfer [46]. When combining C₆₀ with a semiconductor photocatalyst, it will bring an improved photoinduced charge separation and transfer, thereby enhancing the photocatalytic activity [47–49]. For example, Chai et al. [50] have

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fabricated $C_{60}/g-C_3N_4$ composite with excellent photocatalytic activity by a simple adsorption approach. The photocatalytic degradation efficiency of Rh B over the $C_{60}/g-C_3N_4$ composite shows a *c.a.* 40% increasing after introducing of 1 wt% C_{60} . More importantly, C_{60} molecules can work as the photocorrosion inhibitors for other semiconductor *e.g.* ZnO [51]. Therefore, it is very interesting to develop a C_{60} enhanced CdS composite toward the high photocatalytic activity and stability. Nevertheless, to the best of our knowledge, there are hardly any reports about this topic.

Herein, we report a facile one-pot hydrothermal preparation of C_{60}/CdS nanocomposite. The photocatalytic performance including H_2 evolution rate, degradation efficiency of Rh B and photostability of the C_{60}/CdS nanocomposites are investigated under visible light irradiation ($\lambda \geq 420$ nm). The quantity of C_{60} has been changed from 0.4 wt% to 8 wt%, and the optimal value for photocatalytic activity is determined to be 0.4 wt%. The H_2 evolution rate over this optimal sample reaches $1.73 \text{ mmol h}^{-1} \text{ g}^{-1}$ and its apparent degradation rate of Rh B is 0.089 min^{-1} (degradation efficiency of 97% within 40 min), which is 2.3 times and 1.5 times compared to that of pure CdS reference, respectively. Moreover, the photocorrosion of CdS in composite is effectively suppressed, and its photocatalytic activity can be well maintained after three recycles (97.8% retaining for composite vs. 84.4% retaining for CdS). Then, the enhanced photocatalytic activity and stability of C_{60}/CdS nanocomposite are further studied by spectroscopic and electrochemical methods. Results show that the C_{60} species covering on the surface of CdS can efficiently accelerate the separation and transfer of photoexcited charge carriers, which can improve its activity, and reduce the photocorrosion of CdS. This novel C_{60}/CdS nanocomposite exhibits great potential in water splitting and environmental remediation.

2. Experimental

2.1. Materials

$Cd(CH_3COO)_2 \cdot 2H_2O$ (99.9%) and C_{60} (99.9%) were purchased from Aladdin Reagent Company (Shanghai, China). L-cysteine ($C_3H_7NO_2S$, 99%) was obtained from Xima Chemical Reagent Company (Tianjin, China). Deionized (DI) water used in all experiments is from local sources.

2.2. Treatment of fullerenes (C_{60})

The purification and surface functionalization of C_{60} was firstly conducted before using in nanocomposite for better dispersity. Typically, 50 mg raw C_{60} was refluxed in 150 mL of concentrated nitric acid at 140°C for 4 h. Then, the product was washed with DI water several times until pH = 7. After this treatment, the product was dried in an oven at 80°C overnight.

2.3. Preparation of C_{60}/CdS nanocomposites

C_{60}/CdS nanocomposites were synthesized by a simple hydrothermal reaction. Typically, 5 mmol of $Cd(CH_3COO)_2 \cdot 2H_2O$, 10 mmol of L-cysteine and a varying amount of C_{60} were dissolved in 20 mL of DI water to form a homogeneous suspension by vigorous stirring for 1 h. Subsequently, the suspension was then transferred to a 30 mL Teflon-lined stainless steel autoclave and kept at 200°C for 10 h. After the reaction, the autoclave was cooled to room temperature, and then the yellow precipitates were filtered and washed with DI water to remove soluble species before dried in a vacuum oven at 70°C overnight. In order to investigate the effect of C_{60} content on the photocatalytic of the C_{60}/CdS nanocomposites, the weight percentage of C_{60} was varied from 0 to 8 (0, 0.4, 0.8, 4, 8 wt%).

The obtained samples were labeled as CdS, 0.4 C_{60}/CdS , 0.8 C_{60}/CdS , 4 C_{60}/CdS , 8 C_{60}/CdS , respectively.

2.4. Characterization

The X-ray diffraction (XRD) patterns were obtained using a D8 Advanced X-ray diffractometer (Brooklyn, Germany) at 40 kV and 40 mA with Cu K α radiation with a scan rate of $0.02^\circ 2\theta \text{ s}^{-1}$. Scanning electron microscopy (SEM) images were collected on an S-4800 field emission SEM (FESEM, Hitachi, Japan). Transmission electron microscopy (TEM) and high-resolution transmission electron microscopy (HRTEM) analyses were conducted with a JEM-2100F electron microscope (JEOL, Japan) using a 200 kV accelerating voltage. The Fourier transform infrared spectra (FT-IR) of the samples were recorded on a TENSOR27 FT-IR spectrometer. The X-ray photoelectron spectroscopy (XPS) analysis were performed on a Kratos Axis Ultra DLD X-ray photoelectron spectrometer using Mg K α radiation as the excitation source. The contaminant carbon (C1s = 284.6 eV) was chosen as a reference for calibration of the binding energies. The Brunauer-Emmett-Teller (BET) surface area was determined by a multipoint BET method using adsorption data in the relative pressure (P/P_0) range of 0.05–0.3. The UV–vis diffuse reflectance spectra (220–800 nm) were recorded using a UV–vis spectrophotometer (UV2550, Shimadzu, Japan). BaSO₄ was the reference sample. Photoluminescence (PL) spectra were measured using a fluorescence spectrophotometer (Fluorolog-3, HORIBA Scientific, America) with an excitation wavelength of 320 nm. The concentration of released Cd²⁺ in the reaction solution was carried out by inductively coupled plasma mass spectrometry (ICP-MS) (7700 \times , Agilent, America).

2.5. Photocatalytic hydrogen evolution

Typically, the hydrogen evolution experiments were performed using a Pyrex reaction cell connected to a closed gas circulation and evacuation system. In a typical experiment, 25 mg of the photocatalyst was dispersed with constant stirring in a 50 mL aqueous solution containing 10 vol% lactic acid and 1 wt% Pt. After being purged with argon for 30 min to remove dissolved air, the solution was irradiated by a 300 W xenon lamp with a 420 nm cutoff filter. The amount of hydrogen generated from photocatalytic reaction was analyzed by a gas chromatograph (Techcomp GC 7900) equipped with a TCD detector.

2.6. Photocatalytic degradation of Rh B measurements

The photocatalytic activities of C_{60}/CdS nanocomposites (or CdS reference) were evaluated by degradation of Rh B aqueous solution under visible light irradiation. For a typical test, 20 mg of as-prepared photocatalyst was added into 20 mL of 10 ppm of Rh B solution. The solution was placed under a 300 W xenon lamp with a 420 nm cutoff filter providing visible light irradiation. The suspension was magnetically stirred in the dark for 30 min to reach an adsorption-desorption equilibrium before turning on light. During the photocatalytic process, 3 mL of the reaction solution was centrifuged to separate the photocatalyst and Rh B solution after irradiation for a certain period. The concentration of Rh B solution was tested using UV–vis spectrometer according to the absorbance at 554 nm. The recycling experiment was conducted after every 40 min of photocatalytic degradation. Then the separated photocatalyst was obtained by centrifuging, washing with DI water, and drying in oven at 70°C overnight. Then it is reused for the next run to test the recycle ability.

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