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Graphitic carbon nitride-stabilized CdS@CoS nanorods: An efficient visible-light-driven photocatalyst for hydrogen evolution with enhanced photo-corrosion resistance

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ARTICLE INFO

Article history:

Received 25 May 2017

Received in revised form

7 September 2017

Accepted 11 September 2017

Available online xxx

Keywords:

Core-shell

Nanorods

CdS

G-C₃N₄

Photocatalytic hydrogen evolution

ABSTRACT

In this work, a series of highly efficient and noble metal-free Co_{0.2}Cd_{0.8}S/g-C₃N₄ nanocomposites were synthesized via a facile one-step hydrothermal strategy. The Co_{0.2}Cd_{0.8}S/g-C₃N₄ composites modified with 10 wt% g-C₃N₄ (CCNE10) exhibited the highest photocatalytic hydrogen evolution rate, which was 1.85-times as high as that of pure Co_{0.2}Cd_{0.8}S and 7.85-times higher than CdS. Furthermore, the CCNE10 catalyst was very stable in cycling hydrogen production, which is ascribed to the unique g-C₃N₄-modified CdS@CoS structure with fast photogenerated electron transport character.

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Introduction

Energy shortage and environmental pollution are urgent problems for society [1,2]. For example, pollution is released in the form of sulfur and carbon oxides during the use of traditional fossil fuels [3–5]. Therefore, it is a top priority to find a clean energy alternative to traditional energy sources. Of the

various renewable energy sources, solar production of clean hydrogen (H₂) fuels with high calorific values through photocatalytic water splitting is widely recognized as an ideal route to utilizing solar energy because both solar energy and water are highly abundant on earth [6,7].

To date, many efforts have been made in the area of hydrogen production. This started with Fujishima and Honda who split water via photocatalytic decomposition on a TiO₂

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<https://doi.org/10.1016/j.ijhydene.2017.09.075>

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semiconductor electrode in 1972 [8]. Ultraviolet light only accounts for 4% in the sunlight [9–12]. CdS has a narrow band gap of 2.4 eV. This is favorable for water splitting (valence band potential at 1.5 eV and conduction band potential at -0.87 eV). Thus, it has garnered much attention because it is a highly active photocatalyst for H_2 generation upon exposure to visible light [13–17].

However, in the absence of surface modifications, CdS has an efficiency far worse than theoretical levels. Thus, it suffers from severe light corrosion that limits its practical applications. Recently, to overcome these challenges, research into CdS-based metal sulfide semiconductor heterojunctions has been proposed. The results show that the photocatalytic activity of CdS can be significantly improved. Particularly, a series of core-shell structure photocatalysts with CdS as the core have outstanding performance in visible-light-driven hydrogen production. The Du research group reported several CdS-based core-shell catalysts, and the results indicate that after adding a shell, the hydrogen production rate is greatly improved [18–20]. However, the problem of light corrosion was not perfectly solved.

Recently, the Yu research group reported CdS/g- C_3N_4 core/shell nanowires by a combined solvothermal and chemisorption method. At the optimal loading content (2 wt%), the hydrogen production rate of CdS/g- C_3N_4 nanowires is up to $4152 \mu\text{mol h}^{-1} \text{g}^{-1}$ with an apparent quantum efficiency up to 4.3%. The stability of the CdS/g- C_3N_4 core/shell nanowires was also significantly improved [21]. This inspired the design and preparation of highly active CdS/g- C_3N_4 composites with ideal anti-corrosion performance under solar light irradiation. As a typical metal-free polymeric semiconductor, graphitic carbon nitride (g- C_3N_4) has attracted a great deal of attention due to its abundance, high thermal and chemical stability, and visible-light response [22–32]. However, the photocatalytic efficiency of pure g- C_3N_4 is limited by the high recombination rate of photo-generated electron–hole pairs and low surface area.

Of the various strategies to enhance the photoactivity of g- C_3N_4 , designing heterojunction composites is one of the most effective. The g- C_3N_4 -modified heterojunction structure quickly separates the electron–hole pairs leading to improved quantum efficiency. However, to the best of our knowledge, there are rare reports on the g- C_3N_4 modified core@shell CdS-based metal sulfide semiconductor heterojunctions towards highly efficient and stable hydrogen production from water splitting.

In this paper, Co was used to form metal sulfide heterojunction with CdS. The g- C_3N_4 nanosheets were then supported on $\text{Co}_{0.2}\text{Cd}_{0.8}\text{S}$ nanocomposites to further enhance the photostability of the metal sulfide heterojunction. We developed a facile one-step hydrothermal method to prepare a series of g- C_3N_4 sheet-supported $\text{Co}_{0.2}\text{Cd}_{0.8}\text{S}$ nanocomposites with different g- C_3N_4 contents. The visible-light-driven photocatalytic H_2 evolution performance and photostability of the $\text{Co}_{0.2}\text{Cd}_{0.8}\text{S/g-C}_3\text{N}_4$ composites was systematically investigated and compared to pristine $\text{Co}_{0.2}\text{Cd}_{0.8}\text{S}$ and g- C_3N_4 . Based on the experimental results, a possible mechanism of the enhanced photocatalytic activity and photostability of $\text{Co}_{0.2}\text{Cd}_{0.8}\text{S/g-C}_3\text{N}_4$ was also proposed.

Experimental

Preparation of $\text{Co}_{0.2}\text{Cd}_{0.8}\text{S/g-C}_3\text{N}_4$

The g- C_3N_4 powders were initially synthesized using a thermal polymerization method. Briefly, 10 g melamine was added into a crucible with a cover, and then transferred into a muffle furnace and heated to 823 K at a heating rate of 2 K per minute in air. The temperature was maintained at 823 K for 4 h. The yellow product, g- C_3N_4 , was collected and ground into powders.

The preliminary experiment (see Fig. S1 in Supporting information) shows that the prepared pristine $\text{Co}_x\text{Cd}_{1-x}\text{S}$ solid solution exhibits the highest photocatalytic activity at $x = 0.2$. Thus $\text{Co}_{0.2}\text{Cd}_{0.8}\text{S}$ solid solution was used as starting material in the following study. The $\text{Co}_{0.2}\text{Cd}_{0.8}\text{S/g-C}_3\text{N}_4$ composite catalysts were synthesized from a hydrothermal process. Firstly, 0.4982 g $\text{Co}(\text{AC})_2 \cdot 4\text{H}_2\text{O}$, 2.1322 g $\text{Cd}(\text{AC})_2 \cdot 2\text{H}_2\text{O}$ and 0.7612 g thiourea were dissolved into 15 mL deionized water to form a clear solution. A certain quantity of synthesized g- C_3N_4 was dispersed into 15 mL distilled water under sonication for 30 min 30 mL ethylenediamine and the g- C_3N_4 solution was added to the previous solution containing Co and Cd elements gradually with stirring. After stirred continuously for 1 h, the suspension was transferred to a 100 mL Teflon-lined autoclave, and heated at 473 K for 12 h [13]. The final products were rinsed repeatedly by ethanol and distilled water to remove impurity, and then dried at 333 K for 12 h under vacuum. For comparison, $\text{Co}_{0.2}\text{Cd}_{0.8}\text{S}$ was prepared as reference by the same hydrothermal method without g- C_3N_4 .

Characterization of photocatalysts

The crystal structures of the samples were identified by an X-ray diffractometer (XRD, Bruker/D8 Advance) with Cu $K\alpha$ radiation under such conditions: a power setting of 40 kV, 30 mA, and a scan rate of 2°min^{-1} . The morphologies and microstructures of the samples were analyzed by transmission electron microscopy (TEM, JEOL, JEM-2010). The X-ray photoelectron spectroscopy (XPS) measurements were carried out in an ESCALAB 250 XPS meter with Al $K\alpha$ monochromatization. Nitrogen adsorption–desorption isotherms were measured at 77 K on a BeiShiDe 3H-2000PS4 apparatus. The Brunauer–Emmett–Teller (BET) method was utilized to measure the specific surface areas and the pore size distributions. The UV–vis absorption spectra of the samples were recorded with a UV–vis spectrophotometer (Shimadzu, Japan, UV-2401) in the range of 200–800 nm and with BaSO_4 as the reflectance standard. Photoluminescence (PL) spectroscopy was performed on a Hitachi F-7000 spectrophotometer at room temperature. Fourier transform infrared (FT-IR) spectra were carried out on a Spectrum100 (PerkinElmer) FT-IR spectrometer within the wavenumber range of 400–4000 cm^{-1} . Transient photocurrent responses and the electrochemical impedance spectroscopy (EIS) measurements were performed on an electrochemical workstation (CHI 660E, Shanghai Chenhua Instrument Company, China).

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