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Facile fabrication of novel red phosphorus-CdS composite photocatalysts for H₂ evolution under visible light irradiation

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ABSTRACT

Novel red phosphorus-CdS (P-CdS) composite photocatalysts were successfully prepared through a facile and fast precipitation method. The samples were then characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS), Brunauer–Emmett–Teller (BET), ultraviolet–visible diffuse reflectance spectroscopy (UV–Vis DRS) and photoluminescence emission spectroscopy (PL). The photocatalytic activity was evaluated by H₂ generation in the aqueous solution containing Na₂S/Na₂SO₃ as hole scavengers under visible light irradiation ($\lambda \geq 400$ nm). The results show the optimal P molar ratio was 10 mol% and the corresponding H₂ production rate reached 923 $\mu\text{mol h}^{-1} \text{g}^{-1}$, which is about 2.53 times higher than that of CdS sample. Meanwhile, the composite photocatalysts show favorable photostability. The enhanced photocatalytic activity can be attributed to the highly effective charge separation on P-CdS hybrids as confirmed by PL result. Also, a possible photocatalytic mechanism of the P-CdS composites is proposed and discussed in detail.

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Introduction

As the world facing serious energy challenge, the development of clean and renewable energy technologies has become increasingly urgent worldwide [1]. H₂ energy is broadly considered as an ideal power candidate for the future, which possesses huge potentials in solving energy crisis and environmental contamination caused by the consumption of traditional fossil fuels. Photocatalytic H₂ evolution from water

on semiconductors by solar irradiation has received considerable attention due to its potential applications in H₂ economy [2–6]. In order to utilize more of the visible light (~43% of the solar spectrum), designing and developing highly efficient visible-light-active semiconductor photocatalysts with narrow band gaps is of vital significance.

Among various nanostructured semiconductors, CdS photocatalyst has attracted much more attention for water splitting due to its band gap that suits well with the solar spectrum and appropriate band positions for reducing water

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to H₂. However, it is unstable and exhibits very low photocatalytic activity because of the rapid recombination rate of photogenerated electron–hole pairs, the fast backward reaction between H₂ and O₂, and the large H₂ production overpotential as well [7]. To suppress the recombination of photogenerated charge carriers and enhance its chemical stability, great efforts have been made, such as loading different cocatalysts [8–14], modulating the shape and morphology of CdS [13,15] and coupling CdS with other semiconductors [16–22]. Wang et al. [17] synthesized ZnO/CdS heterostructures by a method of wet chemistry, and the H₂ evolution of 1 wt% Pt-loaded (ZnO)₁/CdS_{0.2} reached 3870 μmol h⁻¹ g⁻¹. Zhang et al. [20] prepared NiS nanoparticle (NP) modified CdS nanorod (NR) p–n junction photocatalysts through a simple two-step hydrothermal method. The optimal NiS loading content was determined to be 5 mol%, and the corresponding H₂ production rate reached 1131 μmol h⁻¹ g⁻¹. Wang et al. [21] synthesized layered CdS/ZnS catalyst film using stepped chemical bath deposition method. The greatest H₂ production rate over the CdS/ZnS/Ru film in the formic acid solution reached 123 μmol/m²·h under visible light and 135 mmol/m²·h under the simulated solar light. Nevertheless, these studies mainly focused on the enhanced photocatalytic H₂ generation performance of transition-metal compounds hybridized CdS, which may result in potential metallic secondary pollution.

More recently, P (in this article, if no special instruction, red phosphorus are noted as P), as an elemental semiconductor, has received much attention since its first report by Wang et al. [23]. P is one of the most versatile elements and exhibits wide absorption range in the visible light. Furthermore, the conduction band (CB) of CdS is more negative than that of P, while the valence band (VB) of P is more positive than that of CdS. When combine them to form composite photocatalysts, the charge recombination can be effectively suppressed, which benefits the photocatalytic performance. However, to the best of our knowledge, there are few reports on the synthesis and application of CdS coupled with elemental P for H₂ evolution via photocatalytic water splitting.

In this paper, we present the first example of a P-hybridized CdS (P-CdS) photocatalyst prepared by a precipitation method. Compared with CdS, P-CdS composites exhibited a better photocatalytic performance and excellent photostability. It is postulated that the enhanced photocatalytic performance of the P-CdS photocatalyst is arising from the high separation efficiency of photoinduced electron–hole pairs. The structure between CdS and P as well as the mechanism of enhanced photocatalytic activity were systematically investigated.

Experimental

Catalyst preparation

All analytical grade reagents, including P, Cd(CH₃COO)₂·2H₂O, Na₂S·9H₂O and NaOH were used directly without any treatment. P-CdS composite photocatalysts were synthesized by a precipitation method. Typically, first of all, commercial P was ground slightly into fine, homogeneous powder and then

crystallized at 400 °C for 2 h in Ar atmosphere with a ramp rate of 10 °C min⁻¹. Then, 0.2 g of NaOH and 0.007 g of pretreated P were mixed with 50 mL distilled water. Next, 0.534 g of Cd(CH₃COO)₂·2H₂O dissolved in 20 mL of distilled water was slowly added dropwise into the above aqueous solution under vigorous stirring. Afterwards, 20 mL 0.1 M Na₂S aqueous solution was added into the mixture drop by drop under stirring at room temperature to form a kind of yellow precipitate, followed by continuous stirring for additional 1 h. The P-CdS precipitates were washed with distilled water and ethanol several times. Finally, the obtained yellow products were dried at 70 °C for 24 h generating target P-CdS composite with 10% mole ratio of P (noted as P_{0.1}-CdS). The P-CdS samples with different mole ratios of P (5, 8, 10, 15 and 20 mol%) were synthesized in the same way and the samples were labeled as P_{0.05}-CdS, P_{0.08}-CdS, P_{0.1}-CdS, P_{0.15}-CdS and P_{0.2}-CdS, respectively. For comparison purpose, P and CdS sample were also prepared under the identical conditions, respectively.

Characterization

XRD patterns of samples were obtained by a rotating-anode diffractometer with Cu–Kα radiation (D8 Advance, Germany). UV–Vis spectra were collected by using a UV–Vis spectrophotometer (U-3010, Hitachi, Japan). PL spectra were measured on a fluorescence spectrophotometer (F-4500, Hitachi, Japan). TEM observations were conducted with a 204B electron microscope (FEI, TecnaiF20, USA) operating at an accelerating voltage of 200 kV. The XPS (USA) measurements were done on a PHI QUANTUM (USA) using Al Kα radiation. All binding energies were referred to C 1 s peak at 284.8 eV. Specific surface areas were determined using the BET (Brunauer–Emmett–Teller) method based on N₂ adsorption/desorption at 77 K. The BJH (Barret–Joyner–Halenda) method was applied to get the pore size distributions for the catalysts. N₂ adsorption desorption isotherms were measured in a Micromeritics ASAP 2020M apparatus (USA).

Photocatalytic activity measurement

Photocatalytic activity of the samples was determined by the splitting of water under visible light irradiation. The photocatalytic H₂ production reaction was measured using a 300-W Xenon lamp (PLS-SXE-300UV, Beijing Trusttech Co. Ltd., China) as light source through a UV cutoff filter to obtain the visible light irradiation (λ ≥ 400 nm). Experiments were conducted at ambient temperature as follows: 50 mg of photocatalysts was added to 100 mL 0.1 M Na₂S and 0.1 M Na₂SO₃ aqueous solution. 1 wt% Pt as co-catalyst was carried out by dissolving H₂PtCl₆ into the suspension directly, followed by stirring and irradiation (300-W Xenon lamp) for 1 h at ambient condition to reduce the Pt species. A vacuum pump was used to form a vacuum environment inside the reactor. The reaction cell was linked to a closed gas circulation system and the gases evolved were analyzed with an on-line gas chromatograph adapted with a TCD detector (GC9800, N₂ carrier, TDX-01 carbon molecular sieve column).

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