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Applied Surface Science

journal homepage: www.elsevier.com/locate/apsusc

Full Length Article

Inhibited photocorrosion and improved photocatalytic H_2 -evolution activity of CdS photocatalyst by molybdate ions

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1. Introduction

Based on the ever-increasing energy shortage, hydrogen as a renewable and clean energy has attracted great attention to effectively relieve the human dependence on fossil fuels and resolve the serious energy crisis [1–[5\].](#page--1-0) In all hydrogen production technologies, photocatalytic $H₂$ evolution based on semiconductors is one of low-cost and ideal means of hydrogen production [6–[19\]](#page--1-1). Among various semiconductor materials, CdS with a band gap of 2.4 eV has a strong reduction potential $(-0.51 \text{ V} \text{ vs. SHE})$ satisfying the requirements for photocatalytic H_2 production $[20-25]$ $[20-25]$. However, naked CdS generally shows low H2-production efficiency and weak stability because of the fast recombination of photogenerated electrons and holes and the serious photocorrosion during photocatalytic reactions [\[26,27\].](#page--1-3) To improve the H2-production efficiency of CdS, all kinds of methods have been developed, such as controlling its morphology [28–[32\]](#page--1-4), coupling between CdS and other semiconductors [33–[38\],](#page--1-5) doping elements [\[39,40\]](#page--1-6) and modifying by cocatalysts [\[41](#page--1-7)-48]. However, the photocorrosion of CdS photocatalyst under light irradiation during H_2 -evolution process limits its broad range of application. The main reason can

be ascribed to the fact that the photogenerated holes on the valence band (VB) of the CdS are accumulated and subsequently induced the fast oxidation of S^{2-} ions on the CdS surface to generate S⁰ phase, leading to relatively low photostability [\[27,49\].](#page--1-8) Consequently, it is the main task to develop new ideas to solve photocorrosion and further promote photocatalytic H₂-production efficiency of CdS.

Recently, extensive efforts have been widely focused on inhibiting the photocorrosion and enhancing the performance of CdS [50–[52\].](#page--1-9) For example, Wang et al. [\[50\]](#page--1-9) reported that the improved photocorrosion resistibility of CdS electrode by covering with impermeable graphene layers could effectively prevent the contact with water upon visiblelight exposure. Ai et al. [\[51\]](#page--1-10) proved that the suitable phase junction between cubic phase and hexagonal phase of CdS can resolve the photocorrosion of CdS in terms of band engineering theory. The prepared samples kept high stability and their optimal photocatalytic activity ca. 60 times as high as that of the single phase samples. Moreover, Ning et al. [\[52\]](#page--1-11) prepared stable CdS nanoparticles surface-modified by chemical inert Al₂O₃ shell as a stable oxide matrix photostability of CdS during water splitting. On the basis of the above strategies, the stability and activity of CdS have been enhanced to some degree. However, the

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<https://doi.org/10.1016/j.apsusc.2018.08.125>

Received 21 July 2018; Received in revised form 11 August 2018; Accepted 17 August 2018 Available online 23 August 2018 0169-4332/ © 2018 Elsevier B.V. All rights reserved.

available and effective methods are still very scarce and the mechanism of inhibition photocorrosion and improved H_2 -evolution performance remains currently unclear. Therefore, it is very necessary to explore novel strategy and reasonable mechanism to promote the photostability and H_2 -production efficiency of CdS.

In this paper, molybdate ions were injected into the CdS suspension in lactic acid solution as sacrificial agent (the sample was denoted as CdS(M)). The results indicated that the photocorrosion of CdS was obviously restrained and the H2-evolution efficiency of CdS was simultaneously enhanced. Based on the above experimental results, a possible mechanism was suggested to explain the improved photostability and H_2 -production performance of CdS by molybdate ions. This research may provide new insights to develop efficient and stable CdS-based photocatalysts.

2. Experimental details

2.1. Synthesis of CdS

CdS was prepared by typical precipitation method. Briefly, 140 mL of Na₂S solution (0.1 mol L⁻¹) was added dropwise into same volume and same concentration $Cd(NO₃)₂$ solution at room temperature. After vigorously stirred for 2 h, the resultant suspension was transferred into an autoclave and treated at 180 °C for 12 h. The obtained orange-yellow powder was dried at 60 °C for 12 h and denoted as CdS.

2.2. Synthesis of CdS(M) samples

The $Na₂MoO₄$ injected CdS samples (CdS(M)) were prepared during H2 production of CdS. Typically, the above obtained CdS (50 mg) was dispersed in 80 mL of lactic acid solution (10 vol%). Subsequently, a calculated volume of sodium molybdate (Na_2MO_4) solution $(0.01 \text{ mol L}^{-1})$ was injected into the CdS system and stirred for 1 h. To remove O_2 , N_2 was bubbled for 15 min. Then, the suspension was irradiated by Xenon lamp ($\lambda \ge 420$ nm) for 5 h. At last, the resulting sample was dried at 60 °C for 12 h. To discuss the effect of the Na_2MoO_4 amount on the H_2 -evolution activity of CdS, CdS(M-4), CdS(M-12), CdS (M-22), CdS(M-44) and CdS(M-220) were corresponded to the concentration of Na₂MoO₄ solution at 4, 12, 22, 44 and 220 µmol L⁻¹, respectively. For comparison, CdS was treated via a similar method without $Na₂MoO₄$ solution and denoted as CdS(AF).

2.3. Characterization

The powder XRD patterns were detected on a X-ray Diffractometer (Ultima III, Rigaku, Japan) with Cu- K_{α} radiation. FESEM images were obtained by a field emission scanning electron microscope (JSM-7500, JEOL, Japan). Ultraviolet–visible absorption spectra were performed by a spectrophotometer (UV-2450, Shimadzu, Japan) with BaSO₄ as

internal standard. XPS analysis was done using X-ray photoelectron spectrophotometer (XSAM800, KRATOA, UK) with monochromatic Al K_{α} radiation source and the adventitious C 1s peak (284.6 eV).

2.4. Photocatalytic H_2 -evolution rate measurement

The photocatalytic H_2 -evolution rate was measured in a closed system. Typically, 50 mg of CdS was dispersed in 80 mL of lactic acid solution (10 vol%). First, the suspension was outgassed with N_2 gas for 15 min. Then, Xe lamp (350 W) with a 420 nm cutoff filter was positioned ca. 20 cm away from the above reactor and irradiated. Finally, the H_2 amount was recorded by a gas chromatograph (thermal conductivity detector) (GC-2014C, Shimadzu, Japan) with a 5 Å molecular sieve column.

2.5. Photoelectrochemical measurements

All the photoelectrochemical measurements were conducted on an electrochemical workstation (CHI660E, Shanghai Chenhua, China) using a standard three-electrode system in different electrolytes, where the prepared CdS sample on FTO glass acts as working electrode in addition to Pt-counter electrode and Ag/AgCl-reference electrode. The working electrode was prepared on fluorine doped tin oxide (FTO) conductor glass according to our previous works [\[9,41\].](#page--1-12) The curves of linear sweep voltammetry were tested at the potential range from 0.1 to -1.2 V (scan rate: 10 mV s⁻¹). The *i-t* curves were conducted at the bias potential of −0.3 V during repeated ON/OFF irradiation cycles. A 3-W LED (420 nm, 90 mW cm⁻²) was applied as light source.

3. Results and discussion

3.1. The photoinduced-stability phenomena of CdS photocatalysts and the corresponding photocatalytic performance

For CdS photocatalyst as one of the photocatalytic H_2 -evolution materials, both efficiency and stability are the main challenge due to the recombination of photogenerated charge pairs and serious photocorrosion. At first, the photostability phenomena of CdS before and after the injection of $Na₂MoO₄$ solution can be observed by the schematic diagram in [Fig. 1](#page-1-0). The CdS sample is dispersed into lactic acid to form an orange-yellow suspension (the left picture of [Fig. 1](#page-1-0)). After irradiated for 5 h, the black-yellow CdS(AF) suspension [\(Fig. 1](#page-1-0)-(1)) is obtained. The color change can be attributed to the serious photocorrosion of CdS, namely, S^{2-} in the CdS crystal lattice is easily oxidized to form S^0 . Once Na₂MoO₄ solution is injected into the CdS system, the phenomena become obviously different from the above. When the concentration of Na₂MoO₄ solution is 12 µmol L⁻¹, the color of CdS(M-12) ([Fig. 1](#page-1-0)-(2)) changes from orange-yellow to gray-yellow, suggesting that the photocorrosion of CdS is slightly inhibited. With the

Fig. 1. The photostability phenomena of CdS after the injection of Na₂MoO₄ solution.

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