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Promotion effect of nickel loaded on CdS for photocatalytic H_2 production in lactic acid solution

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ABSTRACT

Low-cost Ni modified CdS was prepared via a hydrothermal reduction method. The hydrogen production activity of CdS loaded with 5 wt% Ni under visible light was even higher than that of the one loaded with 0.5 wt% Pt. The highest H_2 evolution rate (3004.8 μmol h^{-1}) occurred when the concentration of sacrificial agent (lactic acid) was 50 vol%. The nickel can quickly transfer excited electrons and enhance the photocatalytic H_2 production activity. It was also found that the hydrogen evolution in this system was generated steadily from both water and lactic acid.

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1. Introduction

The increasingly aggravated environmental problem and energy crisis have drawn attentions to the research of clean and sustainable resources replacing fossil energy. Hydrogen has been considered a promising candidate because it is a new pollution-free and renewable energy. Since Fujishima and Honda found the photocatalytic phenomenon of hydrogen generation with TiO_2 in 1972 [1], TiO_2 has been extensively studied as a photocatalyst for hydrogen production [2–5]. However, TiO_2 is only sensitive to near UV light occupying merely 4% of the sunlight energy [6]. CdS is a good alternative for visible-light responding photocatalyst due to its appropriate band gap and conduction band edge. Nevertheless, bare CdS is unstable and shows low hydrogen evolution activity under irradiation because of photocorrosion and easy recombination of the excited electrons and holes [7–9]. Modifications of CdS with noble metal materials such as Pt, Pd, Au, RuO_2, etc. [10–12] could improve its photocatalytic hydrogen evolution activity and retard photocorrosion via quick transfer of the excited electrons and holes. However, the high cost of noble metals draws back the practical application of photocatalysts, which makes it obliged to develop less expensive co-catalysts as alternatives. In recent years, CdS modified with non-precious metal materials such as MoS_2, WS_2 and NiS [13–15] have been discovered for effectively improving the photocatalytic hydrogen production activity. Other low-cost cocatalysts including CuO_x, CoO_x and various Ni species are also found for enhancing photocatalytic H_2 evolution activity effectively [16–21].

In this work, CdS was modified with Ni through a simple hydrothermal reduction method. The hydrogen production activity of 5 wt% Ni–CdS was even better than that of 0.5 wt% Pt–CdS when lactic acid as sacrificial agent. What is more, we found that the role of nickel in photocatalytic H_2 production in lactic acid solution was different from traditional ones.

2. Experimental

2.1. Preparation of CdS

All of the reagents used were of analytical purity and used without further purification. CdS was synthesized in accordance with a previously reported method [20,21]. 200 mL Na_2S (0.14 M) solution was added to 250 mL $Cd(CH_3COO)_2$ (0.14 M) solution under vigorous stirring and the suspension was then stirred continuously for 24 h prior to be filtrated and washed with deionized water several times. After that, it was transferred into a 200 mL Teflon-lined

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stainless steel autoclave and heated at 200 °C for 24 h and then cooled down to room temperature naturally. The resulting precipitate was then filtrated and washed using deionized water several times, following to be dried at 80 °C overnight.

2.2. *Modification of CdS with Ni via a hydrothermal reduction method*

CdS loaded with Ni was prepared via a hydrothermal method [22]. Certain amount of $NiCl_2·6H_2O$ was dissolved in 60 mL ethylene glycol to form a light green solution. After that, 200 mg CdS was added to the above solution and it was vigorously stirred for 1 h to form a homogeneous suspension. Then, 0.5 g NaOH was added to the system. The solution was then transferred to a 100 mL Teflon-lined stainless steel autoclave and heated at 200 °C for 10 h. The green precipitate was then filtered and washed with acetone, ethanol and deionized water respectively before dried in a vacuum oven at 80 °C overnight. The CdS modified with 0.5 wt% Pt was also prepared in the same way while replacing $NiCl_2·6H_2O$ with H_2PtCl_6.

Samples with different mass ratios of Ni to CdS were prepared. The ratios were 0, 0.08, 0.5, 2, 5, 7, 10 wt% and were labeled as CN0, CN0.08, CN0.5, CN2, CN5, CN7, CN10, respectively. The sample without adding CdS to the solution was also prepared and labeled as N100.

2.3. *Characterization of catalysts*

The crystal structures of samples were confirmed by X-ray diffraction (Rigaku D/max-2200/PC Japan) with Cu kα (40 kV, 20 mA). The UV–vis diffuse reflection spectra (DRS) were determined by a UV–vis spectrophotometer UV-2450 (Shimadzu, Japan) and were converted to absorbance by the Kulbelka–Munk method. The surface areas of the samples were determined by BET measurement (Tristar II, USA). The contents of metal elements were determined by inductively coupled plasma analyzer (ICAP 6000 Radial, Thermo). The transmission electron microscopy (TEM) measurements were conducted using a JEM-2100F (Japan). The surface electronic states of elements were analyzed by X-ray photoelectron spectroscopy (XPS, Shimadzu-Kratos, Axis Ultra DLD, Japan). All the binding energy (BE) values were calibrated by using the standard BE value of contaminant carbon (C1s = 284.6 eV) as a reference.

2.4. *Photocatalytic H_2 production activities test*

The photocatalytic reactions were carried out in a Pyrex reaction cell connected to a closed gas circulation and evacuation system. 0.1 g photocatalyst was dispersed in 50 mL solution containing 10 vol%, 30 vol%, 50 vol%, 70 vol% or 90 vol% lactic acid. The suspension was then thoroughly degassed and irradiated by a Xe lamp (300 W) equipped with an optical cutoff filter ($\lambda > 400$ nm, containing 1 M $NaNO_2$). The rate of H_2 generation was analyzed using an online gas chromatography. The CN5 sample was recycled after the photocatalytic activity test and was labeled as CNR.

3. Results and discussion

3.1. *XRD, UV–vis DRS and BET analyses*

Fig. 1 shows a comparison of the XRD patterns for CN0, CN0.08, CN0.5, CN5, CN10 and N100 samples. The cubic structured Ni [23] was observed for N100 sample, and the hexagonal wurtzite-structured CdS [24] was clearly observed for all the other samples, which indicated that nickels loaded on CdS were in the form of metallic nickel. With the increasing content of nickel, the peak of CdS changed little, indicating that the crystal structure of CdS did not change. No obvious nickel peaks were observed. This might be ascribed to a weak crystallization and high dispersion of Ni on CdS [22,24].

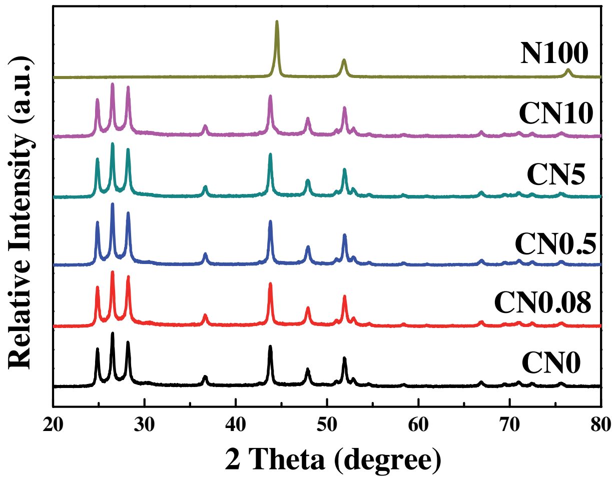

Fig. 1. XRD patterns for CN0, CN0.08, CN0.5, CN5, CN10 and N100 samples.

The UV–vis diffuse reflectance absorption spectra of CN0, CN0.08, CN0.5, CN5, CN10, N100 and CNR are shown in Fig. 2. N100 shows complete absorption in the observed wavelength range, while CdS has an absorption edge at 520 nm, corresponding to the band gap of 2.4 eV [7]. There was no obvious change on the absorption edge with the increase of nickel content. This indicated that nickels were just deposited on the surface instead of being incorporated into the lattice of CdS [20]. The spectra of CdS modified with Ni exhibited obviously enhanced absorption in the region of 540–800 nm, which was ascribed to the absorption of metallic Ni and the change of the color of samples from yellow to dark green [20,24]. As could be observed in the spectrum for CNR, the absorption intensity in this region decreased to the one of the pure CdS, which may because nickels were react with lactic acid during the recycling progress.

The pore structures and BET surface areas of catalysts are investigated. Fig. 3 shows the nitrogen adsorption/desorption isotherms curves of CN0, CN5 and CNR samples have isotherms of type IV from Brunauer–Deming–Deming–Teller (BDDT) classification, indicating the presence of mesopores (2–50 nm) [25,26]. The shapes of hysteresis loops are type H3 at high relative pressure range of 0.8–1.0, indicating the presence of slitlike pores. The isotherms show high absorption at high relative pressure (P/P_0) range,

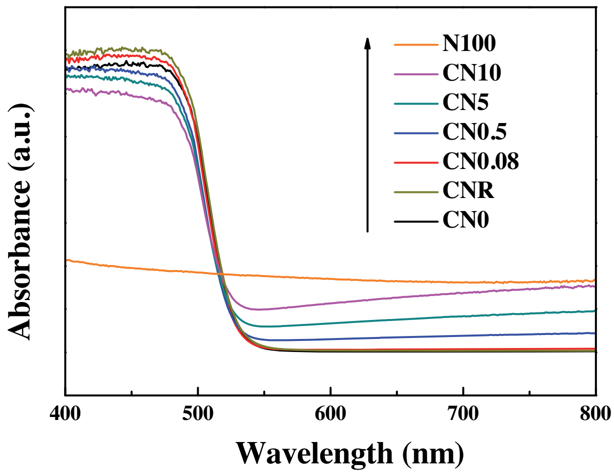

Fig. 2. UV–vis diffuse reflection spectra of CN0, CN0.08, CN0.5, CN5, CN10, N100 and CNR samples.

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