Ultra-low-temperature growth of CdS quantum dots on g-C₃N₄

nanosheets and their photocatalytic performance

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1. Material Preparation

First, cadmium chloride (15 mg) was dissolved in deionized water (10 ml) in a 250-ml Teflon beaker, and ethanol (90 ml) was added to the solution to form a homogeneous solution under magnetic stirring. Subsequently, 100 mg of g-C₃N₄ (XFNANO) was dispersed into the cadmium chloride solution and kept in ultrasonic bath for 90 minutes. Meanwhile, sodium sulfide (6 mg) was dissolved in deionized water (10 ml) in a 250 ml-Teflon beaker, and ethanol (90 ml) was added to the solution. The abovementioned two solutions were transferred into a temperature-controlled chamber. the temperature inside chamber was set to -60°C, the solutions were cooled down to -60°C with magnetic stirring for another 60 minutes. Afterwards, the sodium sulfide solution was slowly added into the cadmium chloride solutions, and the mixture was magneticly stirred for another 90 minutes. Subsequently, the as-obtained CdS/g-C₃N₄ was collected by suction filtration at -60°C and washed three times with deionized water. Finally, the CdS/g-C₃N₄ was dried in oven at 60°C overnight. As control, room temperature (RT) CdS/g-C₃N₄ was also prepared at the same condition except the -60°C reaction temperature was changed to 25°C.

2. Characterization

The phase of the samples were measured by X-ray diffraction (XRD). The structure of crystals were collected using X-ray diffractometer (D/max 2500V) with Cu target. Further structural characterizations and morphological were investigated by transmission electron microscopic (TEM) (JEOL-2010) and high-resolution transmission electron microscopy (HRTEM) (TecnaiTF20) observations. X-ray photoelectron spectroscopy (XPS) measurements were performed using X-ray photoelectron spectrometer (Escalab 250Xi) equipped with an AI Kα X-ray radiation. UV-vis absorption spectra was characterized by UV-visible spectrophotometer (UV-2600, Shimazu) with BaSO₄ as a reluctance standard. Transient photocurrent responses were characterized by CHI 660 electrochemical workstation with a standard three-electrode system.

Photocatalytic H₂ production activity

The photocatalytic H₂ evolution experiment took place in a 250ml two-necked Pyre flask. 25 mg of CdS/g-C₃N₄ samples were scattered in 50 ml of 0.1M L-ascorbic acid solution. The pH value was measured to be 4.0 by KOH solution. 0.5wt% Pt as co-catalyst was loaded by adding 300 ul of H2PtCl6 (0.08 wt%) into the above mentioned solution . The reaction was triggered by a 300-W xenon lamp (Microsolar 300, perfectlight, Beijing, China) with a UV cut-off filter ($\lambda >$ 420 nm, 75mW/cm²). Before being transferred into the two-necked Pyre flask, the solution was purgedwith nitrogen to expel residual oxygen. The composition of produced gas was collected every 60 minutes and analyzed through an Agilent 7890A gas chromatograph (CG) with TCD detector.

Photocatalytic organic dye Rhodamine B degradation activity

Rhodamine B (RhB) was used for evaluating the photocatalytic activity of CdS/g-C₃N₄. The RhB solution (5.0×10^{-5}

mol L⁻¹) with CdS/g-C₃N₄ powders (1 mg ml⁻¹) was dispersed with ultrasound for 60 minutes and magnetic stirring for 60 minutes in dark. A simulated solar light (Microsolar 300, perfectlight), with a 420 nm edge filter, offered visible light irradiation (75mW/cm²). The concentration of RhB was determined using a UV-visible spectrophotometer (UV-2600, Shimazu) at a time interval of 15 min.

3. Supplementary Figures



Fig. S1. Size distribution of the grains of the $CdS/g-C_3N_4$



Fig. S2. Size distribution of the grains of the RT CdS/g-C $_3N_4$



Fig. S3. UV-vis absorption spectra of CdS/g-C $_3N_4$, RT CdS/g-C $_3N_4$, g-C $_3N_4$ and CdS.



Fig. S4. XPS spectra of (a) Cd 3d and (b) S 2p.



Fig.S5. PL spectra of CdS/g-C₃N₄ RT CdS/g-C₃N₄ and g-C₃N₄ at excitation wavelength of 325 nm.



Fig. S6. The photocatalystic H₂-evoluiton rate of CdS/g-C₃N₄, RT CdS/g-C₃N₄, g-C₃N₄ and CdS.



Fig. S7. The photocatalystic RhB-degradation rate of CdS/g-C₃N₄, RT CdS/g-C₃N₄, g-C₃N₄ and CdS.



Fig. S8 The XPS of CdS/g-C₃N₄ after H_2 evolution stability test. (a) Cd 3d and (b) S 2p



Fig.S9 The XRD of CdS/g-C₃N₄ after H₂ evolution stability test. The dark cyan line is the standard pattern of the



Fig. S10 The XPS of CdS/g-C₃N₄ after after RhB degradation stability test. (a) Cd 3d and (b) S 2p



Fig. S11 The XRD of CdS/g-C₃N₄ after RhB degradation stability test. The dark cyan line is the standard pattern of the CdS (JCPDS No. 75-0581).



Fig. S12. Schematic of the photocatalytic mechanisms for the $CdS/g-C_3N_4$. (a) H_2 evolution. (b) Degradation of RhB.