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Facile additive-free solvothermal synthesis of cadmium sulfide flower-like three dimensional assemblies with unique optical properties and photocatalytic activity

Yuming Guo,[a] Jinfeng Wang,[a] Lin Yang,*[a] Jie Zhang,[a] Kai Jiang,[a] Wujv Li,[b] Lingling Wang[a] and Lili Jiang[a]

[a] *College of Chemistry and Environmental Science, Key Laboratory of Green Chemical Media and Reactions, Ministry of Education, Henan Normal University, Xinxiang 453007, P. R. China. Fax: +86-373-3328507; Tel: +86-373-3325999; E-mail:yanglin1819@163.com*

[b] *Luohe Vocational Technology College, Luohe 462000, P.R. China.*

Experimental details

Chemicals and Materials

$CdCl_2 \cdot 2.5H_2O$, thiourea, and ethanol were all of analytical grade, purchased from Sinopharm Chemical Reagent Beijing Co., Ltd and used without further purification.

Experimental Procedures

In this work, the mixed solvent of water and ethanol was used to prepare the CdS 3 D assemblies. However, no template or additive was involved in our method.

In the typical experiment, 1 mmol of $CdCl_2 \cdot 2.5H_2O$ was dissolved into 12.5 mL of ethanol/water mixed solvent (2:3, v/v) at 30℃ under magnetic stirring for 30 min to prepare $CdCl_2$ working solution. 2.5 mmol of thiourea was dissolved into 12.5 mL of ethanol/water mixed solvent (2:3, v/v) to prepare thiourea working solution. Then 12.5 mL of thoiurea working solution was slowly added to the $CdCl_2$ working solution under vigorous magnetic stirring for 30 min. The thiourea/$CdCl_2$ molar ratio was 5:2 and the pH was adjusted to 5.4. 18

mL of the reaction mixture was transferred into a 25 mL Teflon-lined stainless steel autoclave and maintained at 160℃ for 9 hr. After cooling to room temperature, the yellow precipitation was filtered, washed with double distilled water and absolute ethanol for several times, respectively. Then the samples were dried at 40℃ in vacuum for 4 hr.

Characterization

The size and morphology of the as-prepared products were characterized by scanning electron microscopy (SEM, JSM-6390LV, JEOL). High-resolution transmission electron microscopy (HRTEM) investigations, accompanied by selected-area electron diffraction (SAED), were conducted on a JEOL JEL-2010 transmission electron microscope. To prepare the TEM samples, a 5 μL droplet of dilute suspension was dripped onto a carbon coated copper grid, then dried at room temperature. The phases of the as-prepared products were determined by powder X-ray diffraction (XRD) using a DX-2000 X-ray diffractometer (Dandong Fangyuan Instrument, Co., Ltd) with graphite monochromatized Cu Kα radiation (λ = 0.15406 nm). A scanning rate of 0.05 deg/s was applied to record the pattern in the 2θ range of 20-70°.

Photocatalytic activity evaluation

For photocatalytic activity evaluation, 45 mg of the as-prepared CdS 3D assemblies was suspended in 24 mL of DD water by sonicating for 30 min. Then 6 mL of 50 ppm organic dyes aqueous solution was added into the CdS suspension. Before photocatalytic experiment, the suspension was moderate stirred in dark for 30 min to establish an adsorption/desorption equilibrium. Afterwards, the suspension was exposed to the irradiation of 500 W xenon lamp for photocatalytic degradation of organic dyes under moderate stirring. At specified time

intervals, 4 ml of the suspension was taken from the reactor and centrifuged to separate out the catalyst. The content of the organic dyes in the supernatant was determined by UV–visible absorption spectroscopy at 463 nm for methyl orange (MO) and at 554 nm for rhodamine B (RhB) and the photodegradation rate was calculated with equation 1.

$$\text{Photodegradation rate } \% = (A_0\text{-}A) \times 100\% / A_0 \qquad \text{(Equation 1)}$$

where A_0 was the initial absorbance intensity of organic dyes and A was the final absorbance intensity after treated with photocatalysts for certain times.

Results and Discussion

CdS prepared using sodium sulfide as the sulfide source in double distilled water at room temperature

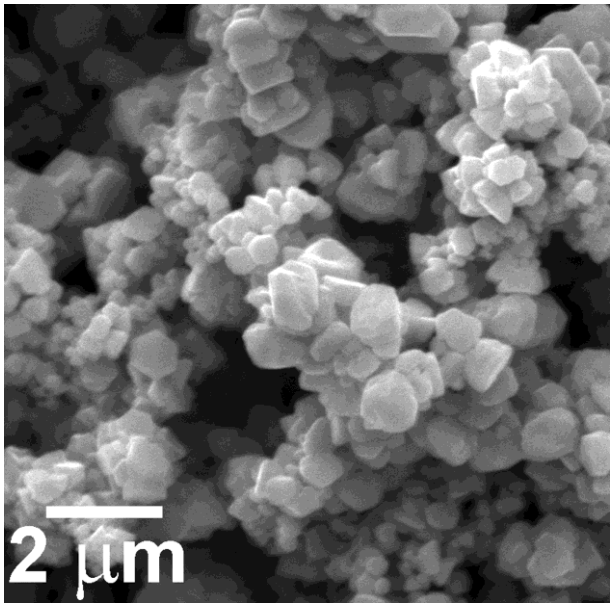

Figure S1 The CdS prepared using sodium sulfide as the sulfide source in double distilled water at room temperature.

Degradation spectra of MO after irradiated with visible light in the presence of the as-prepared CdS

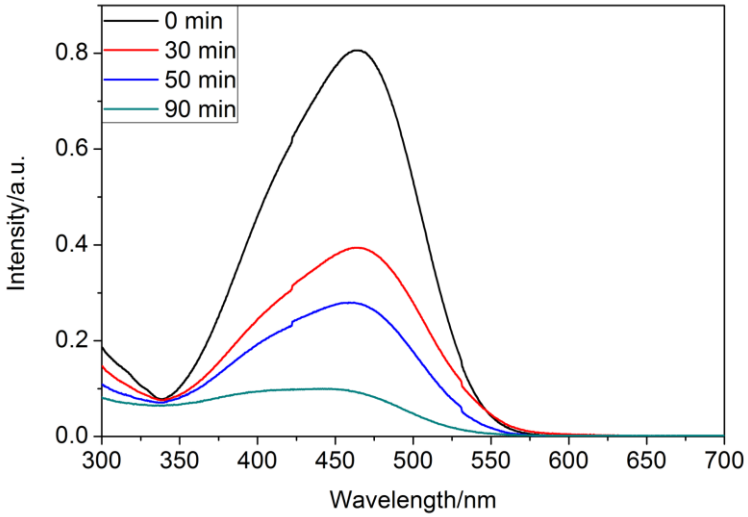

Figure S2 Degradation spectra of MO after irradiated with visible light for different periods of time in the presence of the as-prepared CdS.

Degradation spectra of RhB after irradiated with visible light in the presence of the as-prepared CdS

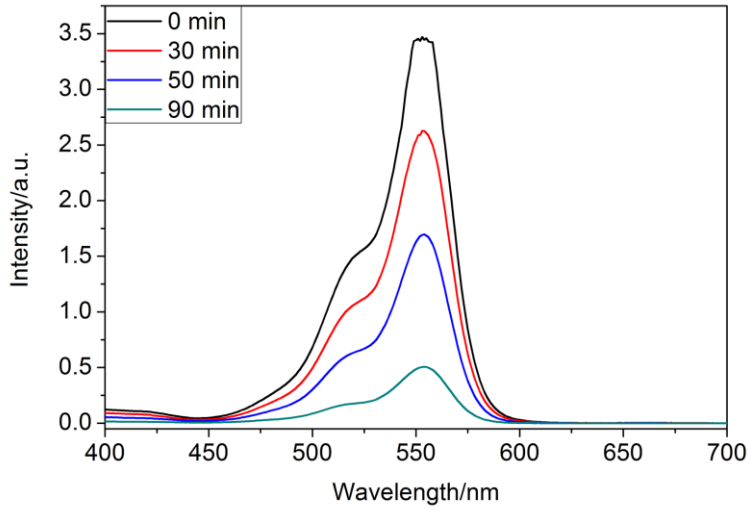

Figure S3 Degradation spectra of RhB after irradiated with visible light for different periods of time in the presence of the as-prepared CdS.