Facile synthesis of mesoporous CdS nanospheres and their application in photocatalytic degradation and adsorption of organic dyes

Yuming Guo, Jinfeng Wang, Zhikai Tao, Fangfang Dong, Kui Wang, Xiaoming Ma, Penghao Yang, Panpan Hu, Yiting Xu and Lin Yang*

College of Chemistry and Environmental Science, Henan Normal University, Xinxiang 453007, PR China

Key Laboratory of Green Chemical Media and Reactions, Ministry of Education, Henan Normal University, Xinxiang 453007, PR China

E-mail: yanglin1819@163.com

1 Experimental Details

1.1. Synthesis of mesoporous CdS nanospheres

Cadmium nitrate ($Cd(NO_3)_2 \cdot 4H_2O$), thioacetamide (TAA), ammonia solution ($NH_3 \cdot H_2O$, 25%~28% by weight), polyvinylpyrrolidone (PVP, Mw=40000), ethanol (EtOH) were purchased from China National Pharmaceutical Group Corp. All the chemicals were of analytical grade and were used as received without further purification.

In a typical experiment, 0.58 g of $Cd(NO_3)_2 \cdot 4H_2O$ and 0.20 g of PVP were dissolved into 25 ml of $EtOH/H_2O$ mixed solvent (2:3, v/v) and stirred for 30 min under ambient conditions. Subsequently, 25 mL of TAA solution (100 mM) in ethanol/water mixed solvent was added quickly under moderate stirring, followed by dropping 0.4 mL of $NH_3 \cdot H_2O$ (1:1, v/v). Then, the reaction system was incubated at room temperature for 1 h. Afterward, the yellow products were centrifuged,

washed with double distilled water (DD water) and absolute ethanol for several times, and dried

under vacuum at 40   for 8 h.

In order to investigate the influence of the PVP on the formation of the CdS NSs, two control

experiments were performed with 0.01g or withoug PVP under identical conditions as the typical

experiment.

1.2 Characterization

The crystal structure of the sample was determined by X-ray powder diffraction (XRD, Bruker

D8Advance) with graphite monochromatized Cu Kα radiation. The high resolution transmission

electron microscopy (HRTEM) investigations were conducted on JEOL JEM-2010 accompanied by

selected-area electron diffraction (SAED). Field-emission scanning electron microscopy (FESEM)

images were recorded by JEOL JSM-6700 FESEM with acceleration voltage of 15 kV. The UV-vis

absorption spectrum of the as-prepared CdS NSs was recorded on a Perkin-Elmer Lambda 17

UV-visible spectrophotometer. A Shimadzu RF-540 PC instrument was used to record the

photoluminescence (PL) spectrum of the sample.

1.3 Photocatalytic activity evaluation

For photocatalytic activity evalution, 12 mg of CdS NSs were suspended in 30 mL of freshly

prepared RhB or MB aqueous solution (20 mg/L). Then the suspension was moderate stirred in the

dark for 1 h to establish an adsorption/desorption equilibrium. Afterwards, the suspension was

exposed to the irradiation of 500 W xenon lamp (380~780 nm) for photocatalysis under moderate

stirring. At specified time intervals, 5 mL of suspension was taken from the reactor and centrifuged

to separate the catalyst. The content of the organic dyes in the supernatant was determined by

UV-vis absorption spectroscopy at 554 nm for RhB and 664 nm for MB. The photodegradation rate

of RhB was calculated with equation 1.

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

where A_0 was the initial RhB absorbance and A was the final RhB absorbance after treated with photocatalysts for certain times.

For comparison, the photocatalytic activity of commercial photocatalyst P25 was also determined under same conditions.

1.4 Adsorption equilibrium and kinetic experiments

Adsorption equilibrium experiment was performed as follows. 4 mg of the CdS NSs were suspended in 10 mL of Congo red (CR) aqueous solutions with different pre-set concentrations (10 mg/L ~ 80 mg/L) at pH 7. Above suspension was constantly stirred for 6 h in the dark at room temperature and centrifuged to separate the adsorbent. Then the final concentration of CR remained in aqueous phase was determined by UV-vis absorption spectroscopy at 498 nm. The amount of CR at equilibrium q_e (mg/g) on the adsorbent sample was calculated from equation 2.

$$q_e = \frac{(C_0 - C_e) \, V}{W} \quad \text{(Equation 2)}$$

Where C_0 and C_e (mg/L) are the initial and equilibrium concentrations of CR, respectively, V (L) is the volume of the solution and W is the mass of the adsorbent used (g).

The procedure of the adsorption kinetic experiments was similar to that of isotherm experiment, except for fixing the concentration of CR at 40 mg/L. The aqueous samples were taken from tubes at given time intervals, and the concentration of CR was measured similarly. The amount of adsorption q_t (mg/g) at time t was calculated by equation 3:

$$q_t = \frac{(C_0 - C_t) \, V}{W} \quad \text{(Equation 3)}$$
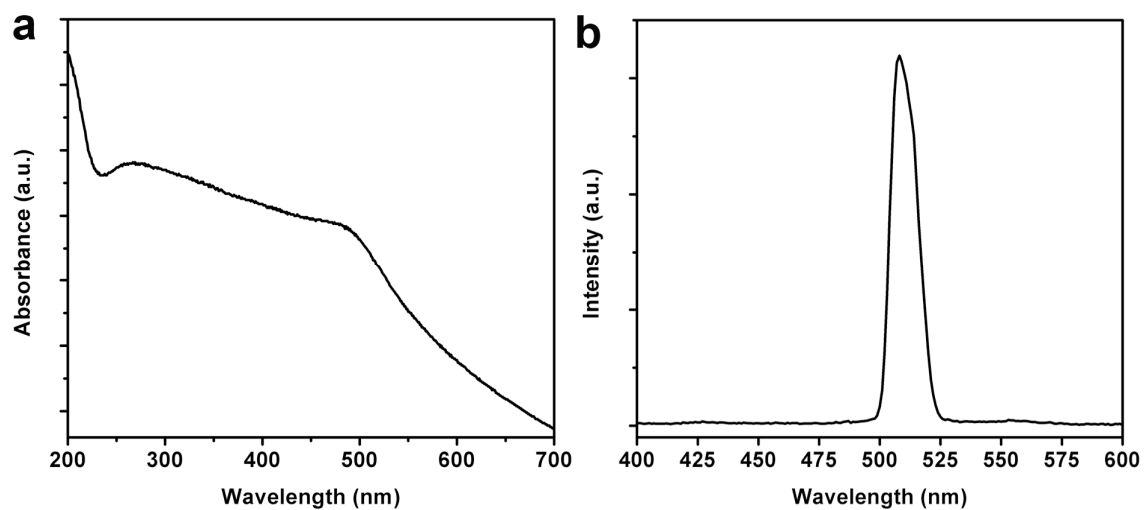
Where C_0 and C_t (mg/L) are the initial and immediate concentrations of CR respectively. V is the

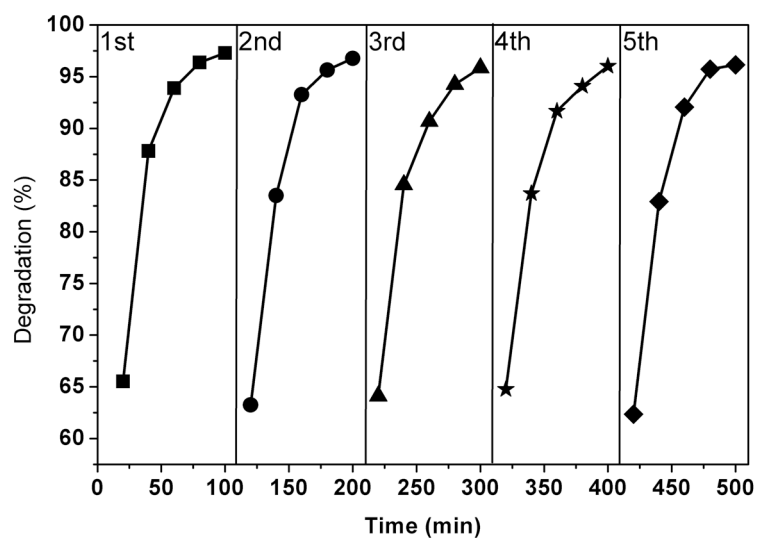volume (L) and W is the mass of the adsorbent used (g).
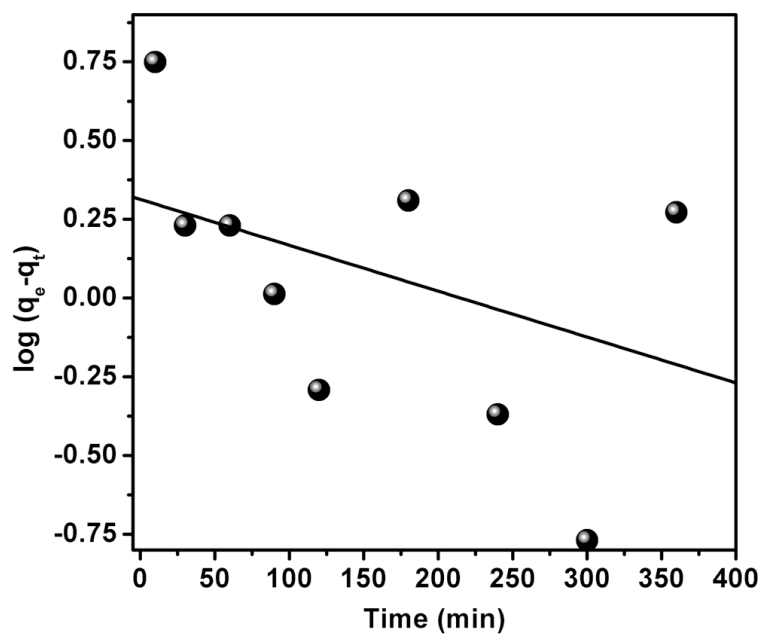
2 Results and Discussion
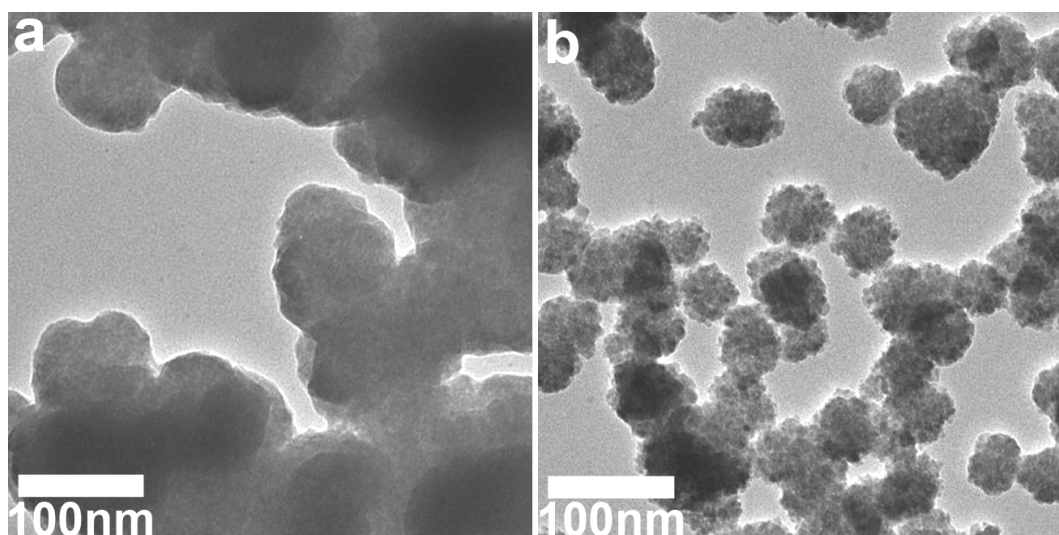
Fig. S1 TEM images of the CdS structures obtained in the (a) absence and (b) presence of PVP.
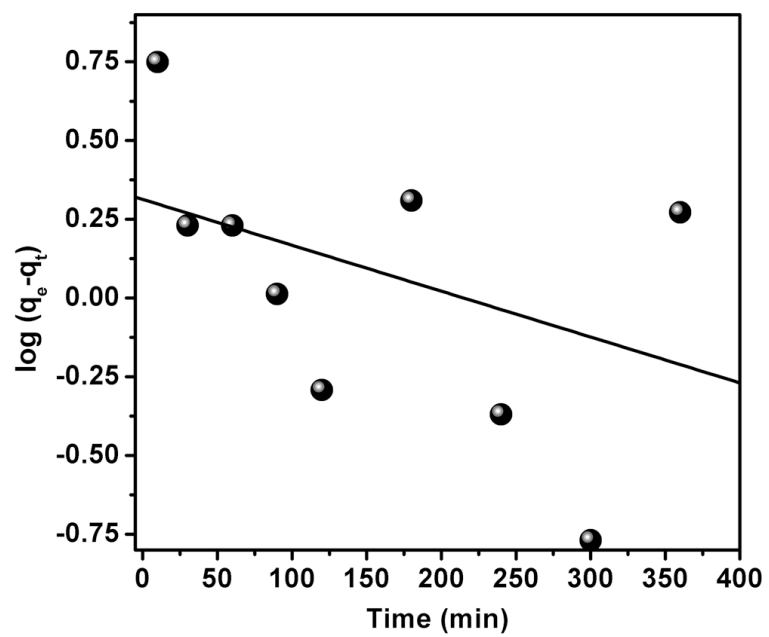

Fig. S2 (a) UV-vis absorption spectrum and (b) PL spectrum of the CdS NSs under 255nm

excitation.

Fig. S3 The recycling of the CdS NSs on the photocatalytic degradation of MB.

Fig. S4 Plot of pseudo-first-order kinetics.

Fig. S5 Adsorption isotherm for the adsorption of CR on the CdS NSs adsorbent.