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ORIGINAL CONTRIBUTION

# Preparation and optical properties of blue-emitting colloidal CdS nanocrystallines by the solvothermal process using poly (ethylene oxide) as the stabilizer

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**Abstract** Blue-emitting colloidal CdS nanocrystals have been synthesized through the solvothermal reaction of cadmium acetate and thiourea in *N*,*N*-dimethylformamide using poly(ethylene oxide; PEO) as the stabilizing polymer. The as-prepared CdS colloids were stable at ambient conditions for several weeks. The PEO-stabilized CdS colloids showed a narrow fluorescence band with the maximum at about 420 nm and thus emitting blue fluorescence under the ultraviolet (UV) lamp. A common red shift of fluorescence band is not detected for the prepared CdS colloids in the study, indicating that PEO-stabilized CdS NCs possess few crystalline defects on their surface. In addition, transmission electron microscope micrographs reveal that the sizes of CdS NCs are between 4.4 to 5.4 nm with small standard deviations from 0.5 to 0.7 nm. The particle growth kinetics was studied by monitoring UV-visible absorption onsets versus the reaction time and was found to nearly follow the Lifshitz–Slyozov–Wagner theory for the Ostwald ripening mechanism.

**Keywords** PEO · Colloidal CdS · Solvothermal · Nanocrystals

## Introduction

Semiconductor nanocrystals (NCs) have attracted much attention during the past decades. Among them, CdS is one of the most interesting because it is of great interest for applications in optoelectronics, photovoltaics, catalysis, and biological sensing. For future applications of colloidal NCs in devices of three-dimensional structures and thin films, immobilization of NCs in a condensed phase is required. Various technologies have been developed for anchoring nanoparticles onto inorganic or organic surfaces or trapping them within porous solids [1–5]. To attaint this target, colloidal NCs are usually prepared from organometallic precursors using high-temperature solution chemistry routes [6–10]. As prepared, NCs are surface-capped by strong coordinating organic ligands. Exchange of these ligands allows tailoring the solubility of the NCs in various solvents with preservation of the optical properties. An alternate route is to prepare colloidal NCs in solvents directly by utilizing polymers as steric stabilizers, by which growth of NCs can be controlled [11–20]. This method provides a facile procedure to incorporate NCs into solid substrates or to prepare thin films containing NCs.

Very recently, several groups have explored the preparation of CdS NCs using thiourea and cadmium salts as reactants in a variety of solvents at lower temperatures (<100 °C). Yu et al. [21] called this synthetic route as the *solvothermal process*, by which the morphology and particle sizes of nanocrystalline CdS can be controlled. Carrot et al. [3] used a similar method to synthesize nanosized CdS clusters through the reaction of thiourea with $Cd(OAc)_2$. They used a polyester with thiol end groups as a covalently attached colloid stabilizer. Wada et al. [22] also reported that CdS NCs could be prepared by the microwave-assisted reaction of $Cd(OAc)_2$ with thiourea in *N*,*N*-dimethylformamide (DMF) without using any stabilizer and capping agent. Chen et al. [15] synthesized CdS NCs through the reaction of $Cd(OAc)_2$ and thiourea in DMF using poly(styrene-*co*-maleic anhydride) random copolymer as the stabilizer. He et al. [16] prepared poly(*N*-vinyl-2-

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pyrrolidone; P2VP)-capped ZnS and CdS NCs from Zn $(OAc)_2$ and $Cd(OAc)_2$ by microwave heating. In the above-mentioned studies, the PL spectra of the CdS NCs showed large, red-shifted fluorescence emission in the region of 550–800 nm. The emission band has been confirmed as a result of the radiative recombination of trapped charge carriers in some surface defect states of CdS NCs [23–26].

In the present study, we report that blue-emitting colloidal CdS NCs could be prepared by the simple solvothermal reaction of heating $Cd(OAc)_2$ and thiourea in DMF with PEO as the stabilizer. The resulting CdS colloids exhibit superior stability that no precipitation forms for weeks at room temperature. The peculiar feature of this work is that the CdS colloids, which are prepared by the employed method, show a fluorescence band at ca. 420 nm with a full width at half maximum (FWHM) of only 60 nm. The colloids emit blue fluorescence under the 365-nm ultraviolet (UV) lamp. This narrow fluorescence band can be attributed to excitonic fluorescence, indicating that there are very few defect sites on the surface of the CdS NCs stabilized by PEO. Moreover, the fluorescence emission band of the CdS colloids is independent on the amount of PEO added and the feed ratio of $Cd(OAc)_2$ and thiourea. Although many reports have been dedicated to the synthesis and characterization of CdS NCs from the solvothermal reaction of thiourea and cadmium salts, relatively little attention has been paid on their particle growth kinetics. In the present study, the particle growth kinetics has been investigated by monitoring the evolution of UV-visual (Vis) absorption onsets with reaction time and was found to nearly follow the Lifshitz–Slyozov–Wagner (LSW) theory for the Ostwald ripening mechanism [27, 28].

## Experimental

### Materials

Cadmium acetate dihydrate ($Cd(OAc)_2{\cdot}2H_2O$, 98%, Arcos), thiourea (98%, Showa), PEO (MW=10,000 g/mol, Merck), and *N*,*N*-dimethylformamide (DMF, GC grade 99.5%, Showa) were used as received.

### Synthesis of colloidal CdS NC in DMF

The 0.040-M stock solution of $Cd^{2+}$ was prepared by dissolving 213.2 mg (0.80 mmol) of $Cd(OAc)_2{\cdot}2H_2O$ in 20-ml DMF. The 0.10-M stock solution of thiourea was prepared by dissolving 152.2 mg (2.0 mmol) of thiourea in 20-ml DMF. A typical procedure for the preparation of CdS NCs colloid using PEO concentration of 10 mg/ml in DMF ($EO/Cd^{2+}=450/1$) was as follows: 200.0 mg of PEO was weighted into a 20-ml volumetric flask, then 15-ml DMF was added in the flask to dissolve the polymer. A 500-µl aliquot of $Cd^{2+}$ stock solution and a 500-µl aliquot of thiourea stock solution were added into the 15-ml polymer solution; then added DMF to the 20-ml mark line of the flask. The solution was transferred into a 50-ml flask, which had been wrapped with aluminum foil to prevent the reaction from light. Before the temperature was raised to 90 °C, the reaction mixture was purged with nitrogen stream for 20 min. The reaction mixture was checked to ensure that it was colorless and then reacted at 90 °C for 6 h. The solution turned from colorless to pale yellow and was still transparent and clear, indicating the generation of Cd NCs. For kinetic studies, aliquots were taken form the reaction mixture at different time intervals for recording UV-Vis and photoluminescence (PL) spectra.

### Characterization

UV-Vis absorption spectra were obtained with a Shimadzu UV-160A spectrophotometer. PL spectra were recording on a Hitachi F-2500 Fluorescence spectrophotometer. The images of colloidal CdS NCs under light or UV irradiation were taken with a digital camera. The excited wavelengths of UV irradiation were 365 nm by a UV lamp (UVLS-28, UVP, Upland, CA). A Joel E200EXII operated at 120 kV electron microscope was used to obtain the transmission electron microscope (TEM) images. The samples for TEM

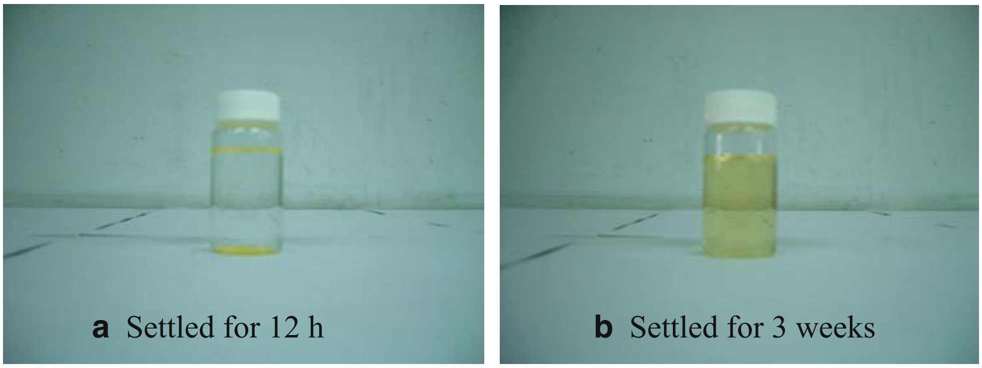

**Fig. 1** Colloidal CdS NCs prepared by the solvothermal reaction of thiourea with $Cd(OAc)_2$ in the absence of stabilizing polymer (**a**) and in the presence of PEO (**b**). The photographs were taken under daylight. $[Cd(OAc)_2]=5\times10^{-4}$ M; [thiourea]=$10^{-3}$ M; PEO=10 mg/ml (200 mg of PEO in 20 ml of DMF)

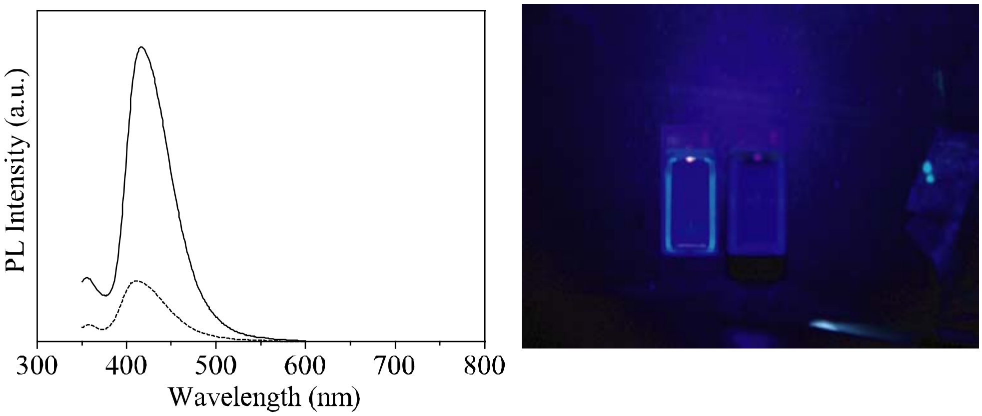

**Fig. 2** The effect of light on the solvothermal reaction of thiourea. **a** Comparison of PL spectra of the sample prepared under daylight (*dash line*) and that prepared in the dark (*solid line*). **b** The luminescence image taken under a UV lamp at 365 nm; the brighter sample was prepared in the flask to prevent exposure from light. $Cd(OAc)_2$ $[Cd(OAc)_2]=5\times10^{-4}$ M; [thiourea]= $10^{-3}$ M; PEO=10 mg/ml (200 mg of PEO in 20 ml of DMF)

were prepared by placing a drop of dilute solution of the colloidal CdS solution on a copper grid coated with a carbon film. The excess solvent was wicked away with a paper tip, and the sample was allowed to dry completely in a vacuum oven.

## Results and discussion

### Solvothermal reaction of Cd(OAc)2 and thiourea with PEO as the stabilizer

DMF is known as a solvent that can well disperse CdS NCs, given a colloidal solution. The dispersity of CdS NCs in DMF is due to the coordination of DMF to cationic cadmium atoms on the surface of CdS NCs [22, 29]. The photographs in Fig. 1 compare the stability of colloidal CdS NCs in DMF with and without adding PEO as the stabilizing polymer in the solvothermal reaction. The colloidal CdS NCs (Fig. 1a), which was prepared by the solvothermal reaction in the absence of stabilizing polymer, was turbid yellow and completely precipitated after 12 h of settling. With adding PEO as the stabilizer in the course of reaction, the resulting CdS colloid was transparent with a pale yellow color. There is no precipitation observed even stored at room temperature up to 3 weeks (Fig. 1b). Furthermore, for the CdS colloids prepared in the absence of PEO, postaddition of PEO to the as-prepared colloids could not prohibit precipitation of CdS NCs. The above results infer that PEO not only serves as the stabilizing polymer but acts as an important role in the solvothermal reaction of $Cd(OAc)_2$ and thiourea.

The solvothermal reaction was affected significantly by light. When the reaction proceeded in a flask in exposure to ambient light, the PL intensity of the colloid CdS solution was only one fifth as high as the sample prepared in the flask wrapped with aluminum foil. Figure 2a shows the differences in PL intensity. Photographs shown in Fig. 2b are the colloidal CdS NCs solutions prepared in the dark (left vial) and under ambient light (right vial) taken under illumination provided by a 365-nm UV lamp. As shown, both samples emitted a blue color under UV irradiation. Obviously, the sample, which was prepared in the condition of preventing light from the reaction mixture, had brighter illumination.

### Kinetics of solvothermal reaction of Cd(OAc)2 and thiourea with PEO as the stabilizer

Growth of the particle size can be monitored by the evolutions of absorption spectra of the colloidal CdS NCs as a function of heating time. Two typical experiment results are shown in Fig. 3. The reaction consisted in mixing PEO (50 or 200 mg) with $Cd(OAc)_2$ ($5\times10^{-4}$ M)

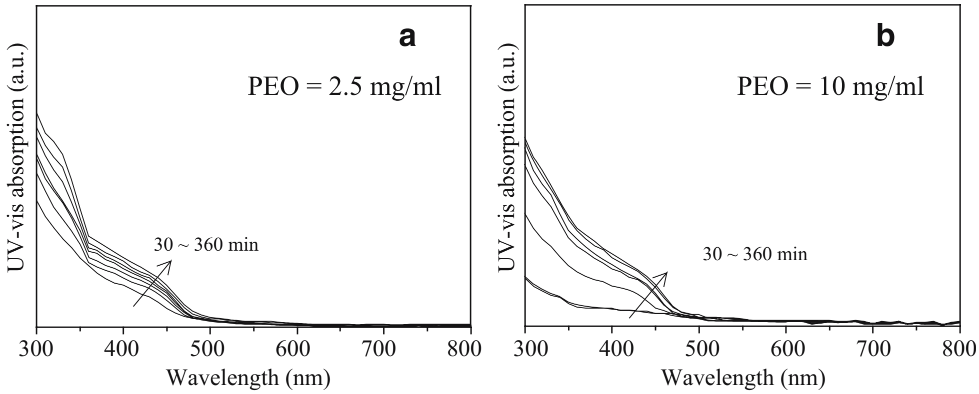

**Fig. 3** The evolutions of UV-Vis absorption spectra of CdS solution as a function of reaction time. **a** PEO concentration= 2.5 mg/ml (50 mg of PEO in 20 ml of DMF) and **b** PEO concentration=10 mg/ml (200 mg of PEO in 20 ml of DMF). $[Cd(OAc)_2]=5\times10^{-4}$ M; [thiourea]=$10^{-3}$ M

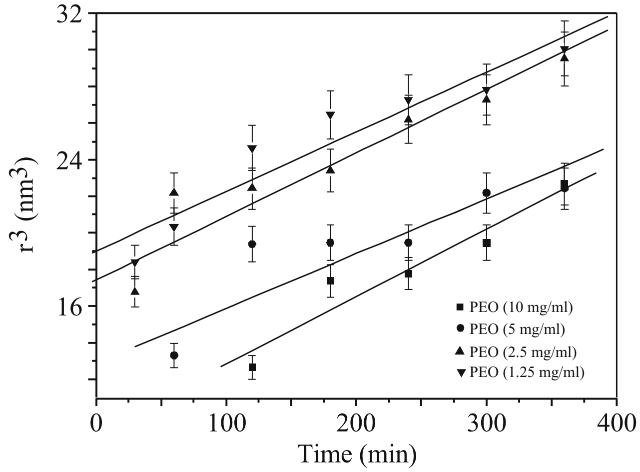

**Fig. 4** Plots of the cubic term of radius of CdS NCs as a function of time by using different amount PEO in the solvothermal reaction. [Cd(OAc)$_2$]=5×10$^{-4}$ M; [thiourea]=10$^{-3}$ M

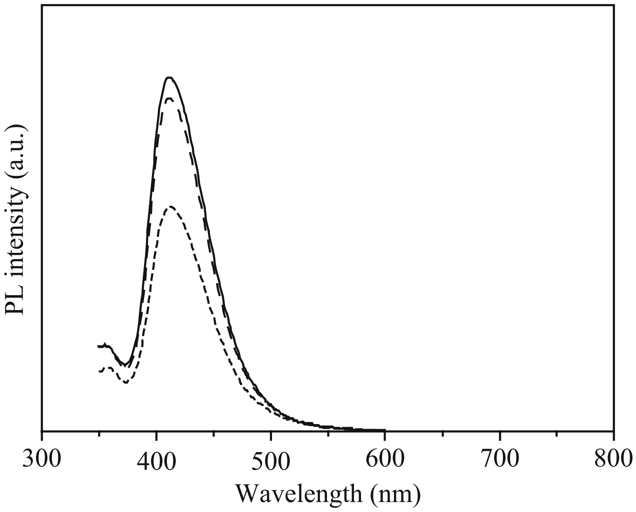

**Fig. 5** PL spectra of colloidal CdS solution at different feeding ratio of Cd(OAc)$_2$ and thiourea. *Solid line*, Cd(OAc)$_2$:thiourea=1:2; *short-dash line*, Cd(OAc)$_2$:thiourea=1:1 and *long-dash line*, Cd(OAc)$_2$:thiourea=1:0.5. [Cd(OAc)$_2$]=5×10$^{-4}$ M; PEO concentration=10 mg/ml (200 mg of PEO in 20 ml of DMF)

followed by the addition of thiourea (10$^{-3}$ M) in 20 ml of DMF. Aliquots were taken from the reaction mixture at different times and analyzed by the UV-Vis spectrometer. Absorbance in the UV-Vis spectrum is proportional to the number of particles formed in the solution. As the PEO concentration is low (2.5 mg/ml, Fig. 3a), the initial rapid increase in absorbance corresponds to the fast nucleation of CdS NCs following the thermal decomposition of thiourea. The absorption onsets are red-shifting with increasing heating times, indicating size growth of CdS NCs in the course of the reaction. The absorption spectrum of the CdS colloid after 360 min of reaction shows a shoulder maximum at ca. 445 nm. While the PEO concentration was raised to 10 mg/ml, no significant absorption above 400 nm is observed until reaction time reaches 120 min. Therefore, during the initial reaction stage, particle nucleation is strongly suppressed at high PEO concentration.

As shown in Fig. 4, the radius of CdS NCs for the different reaction time is estimated from the absorption onset using Brus's effective-mass model [30], and the cubic term of radius is plotted as a function of heating time for syntheses of CdS NCs using different amounts of PEO. The reaction details are listed in Table 1. Two conclusions can be drawn from Fig. 4. First, the increase in the CdS nanocrystal size has approximately linear dependence on the reaction time. Second, the presence of PEO suppresses size growth of CdS NCs; thus, for the same reaction time, the size of NCs is smaller with increasing amounts of PEO present in the reaction mixture.

An equation to describe the growth kinetics of CdS quantum dots prepared from a single precursor [(2,2′-bispyridine)Cd(SC{OPh)$_2$] complex has been reported by Zhang et al. [28]:

$$r_{av}{}^3 = r_{av0}{}^3 + Kt$$

where $r_{\mathrm{av}}$ is the average particle radius, $r_{\mathrm{av},0}$ is the average radius of initially formed particles, and $t$ is the reaction time. In the equation, $K$ (nm$^3$/s) can be regarded as the rate constant for particle growth, and it is a constant proportional to the diffusion coefficient. The deriving of the equation is based on the Gibbs–Thompson equation and LSW model for Ostwald ripening mechanism [27, 31]. In the LSW model, large particles grow at the expense of dissolution of smaller ones [27]. There are two precursors, Cd(OAc)$_2$ and thiourea present in the employed solvothermal reaction, which is different from the reaction of using a single precursor. Nevertheless, the supply of reactants is similar in the two reaction conditions. The precursors in

**Table 1** Summary of reaction results in Fig. 4

| Run no. | PEO (mg/ml) | EO:Cd$^{2+}$ | Particle radius at 180 min (nm) | Particle radius at 360 min (nm) | $K$ (nm$^3$/s) |
|---|---|---|---|---|---|
| CdS-1 | 10 | 450:1 | 2.6 | 2.8 | 0.037 |
| CdS-2 | 5 | 225:1 | 2.7 | 2.8 | 0.026 |
| CdS-3 | 2.5 | 113:1 | 2.9 | 3.1 | 0.032 |
| CdS-4 | 1.25 | 28:1 | 3.0 | 3.1 | 0.033 |

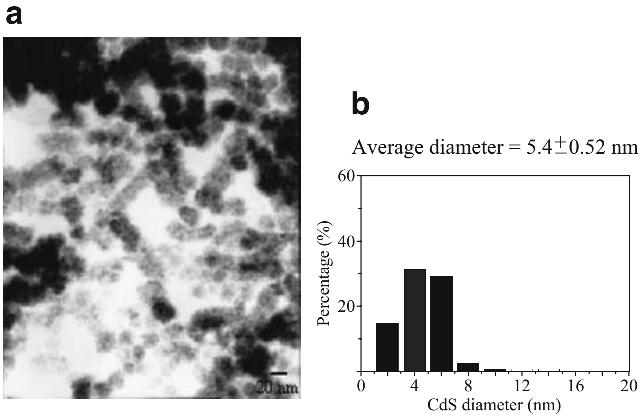

**Fig. 6** **a** TEM of CdS NCs prepared by the solvothermal reaction of thiourea with $Cd(OAc)_2$ in the presence of PEO. The scale bar is 20 nm. $[Cd(OAc)_2]=5\times10^{-4}$ M; $[\text{thiourea}]=10^{-3}$ M; PEO=2.5 mg/ml in DMF. **b** Histogram for the size of CdS nanoparticles obtained from TEM

both reactions are continuously provided throughout the heating period until complete depletion of the precursors.

In Fig. 4, the rising slope ($K$) for the four samples is approximately identical, provided that the least mean square regression is applied to obtain a straight line. Because $K$ is related to the diffusion coefficient, $K$ can be considered as an index of diffusion of reactants through the interfacial boundaries for particle coalescence. The similarity in $K$s (see Table 1) for different PEO concentrations means that the barrier effect of PEO chains in different concentrations is similar. It can also be found in Fig. 4, if we extrapolate the line for the CdS-1 and CdS-2 samples to zero reaction time, that the $y$-intercept will be ca. 16 $nm^3$. This implies the formation of large CdS nanoparticles at initial reaction stage in the case of the PEO concentration below 2.5 mg/ml. No particle forms in the first 25 min of reaction for the PEO concentration is 5 mg/ml. As the PEO concentration is increased to 10 mg/ml, no particles are found until the heating time reaches 100 min. The results indicate that the high concentration of PEO in DMF retards the initial formation of CdS NCs.

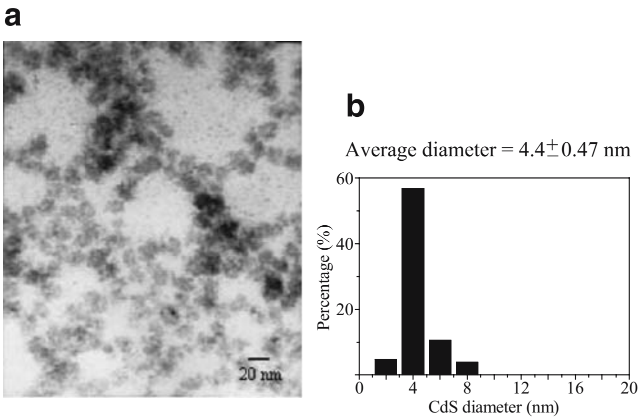

**Fig. 7** **a** TEM of CdS NCs prepared by the solvothermal reaction of thiourea with $Cd(OAc)_2$ in the presence of PEO. The scale bar is 20 nm. $[Cd(OAc)_2]=5\times10^{-4}$ M; $[\text{thiourea}]=10^{-3}$ M; PEO=5 mg/ml in DMF. **b** Histogram for the size of CdS nanoparticles obtained from TEM

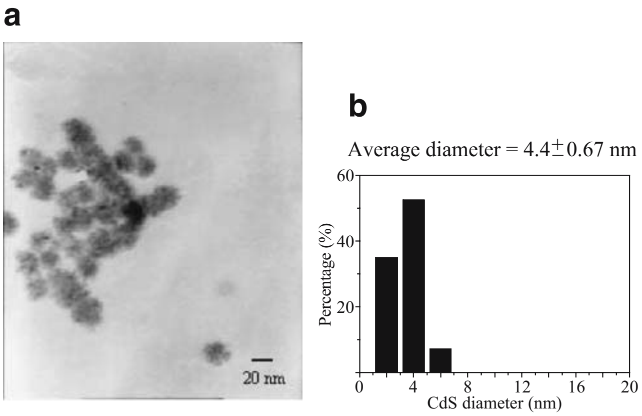

**Fig. 8** **a** TEM of CdS NCs prepared by the solvothermal reaction of thiourea with $Cd(OAc)_2$ in the presence of PEO. The scale bar is 20 nm. $[Cd(OAc)_2]=5\times10^{-4}$ M; $[\text{thiourea}]=10^{-3}$ M; PEO=10 mg/ml in DMF. **b** Histogram for the size of CdS nanoparticles obtained from TEM

Because DMF is a good solvent for PEO, the conformation of PEO molecules in DMF is like random coils. Upon addition of cadmium ions into the solution, complexation of $Cd^{2+}$ with EO units occurs. The interior of the coil can be regarded as a “nanoreactor,” in which $Cd^{2+}$ ions react with $S^{2-}$ to produce CdS NCs. In the nanoreactor, the particle size can be controlled, and CdS NCs are stabilized by EO units. This explains that postaddition of PEO provides no stabilizing effect on the as-prepared CdS NCs in DMF. Table 1 summarizes the effect of the ratio of EO unit to $Cd^{2+}$ ion on the particle size of resulted CdS NCs. At low $EO{:}Cd^{2+}$ molar ratio, there should be considerable free $Cd^{2+}$ ions in the solution. The CdS NCs produced from free $Cd^{2+}$ ions can be only stabilized by DMF and will precipitate slowly. The fact was reflected by the samples CdS-3 and CdS-4. The two samples showed significant precipitation after 1 week.

## Photoluminescence of PEO-stabilized CdS NCs colloids

Figure 5 shows the optical absorption and PL spectra of the various CdS NCs prepared by the solvothermal process. It can be seen that regardless of the feeding ratio of $Cd(OAc)_2$ to thiourea, the emission bands have the same features. Upon excitation with light at wavelength of 340 nm, the spectra are dominated by a strong and narrow band edge emission in the blue region with emission maxima at 420 nm. The FWHM of the emission peaks is pretty narrow (ca. 60 nm) combined with the good agreement of emission band and excitonic transition in the absorption spectra, indicating both a narrow size distribution of NCs and a contribution of all the particle sizes to the observed fluorescence. All CdS NCs colloids show no emission in the 550- to 800-nm region in the PL spectra. For CdS NCs, emission in the 500- to 800-nm region has been confirmed as a result of recombination of trapped electrons/holes in some surface defect states [23–26]. Therefore, it becomes evident that colloidal CdS NCs prepared by using PEO as the stabilizer in DMF bear few defects on their surface.

In the study of Wada et al. [22], they did not use any stabilizer in the microwave-assisted synthesis of CdS NCs. Their results indicated that when the excess $Cd^{2+}$ was added to the reaction mixture, a broad red emission was induced and enhanced with increasing amount of $Cd^{2+}$. This emission is due to the surfer vacancy on the surface of CdS NCs. According to the study of Chen et al. [15], the PL emission peak showed a red shift emerge as increasing heating time (90 °C) of the reaction between thiourea and $Cd(OAc)_2$ using poly(styrene-*co*-maleic anhydride) as the stabilizer. Similar results have also been reported by Carrot et al. [3]; they synthesized CdS NCs by solvothermal reaction using polyester chains with a thiol end group as a covalently attached colloidal stabilizer. Their results showed that as the reaction proceed in DMF, the red shift was more pronounced with increasing reaction time. However, the PL spectra of CdS NCs obtained from THF solutions showed only slighter various with time.

By comparing our results to the mentioned similar systems, it can be concluded that PEO may be the key factor on the PL property in our preparation of CdS NC colloids. It has been known that the PL properties of CdS NCs depend not only on the particle size but the surface composition of particles as well. As described in the discussion of growth kinetics, the PEO coil can be treated as the nanoreactor. It is where the CdS NCs are formed and then stabilized. The irrelevance of PL properties to the feeding ratio of reactants indicates that the particles formed in PEO coils have the identical surface composition.

### TEM micrographs of CdS NCs

Figures 6, 7, and 8 show TEM micrographs for CdS NCs obtained from cast films; the particle size distribution and size standard deviation are derived from the TEM images. Because the syntheses were conducted in DMF, the nanoparticles displayed nonuniformly spatial distributions on solvent evaporation. As shown in the TEM images, CdS NCs aggregate into big clusters. The particle edges are not well defined, which is probably due to the presence of polymer and polymer-caused aggregation of particles. The average diameters of nanoparticles for each preparation are as follows: 5.4±0.52 nm (PEO=2.5 mg/ml); 4.4±0.47 nm (PEO=5 mg/ml); and 4.4±0.67 nm (PEO=10 mg/ml). The size obtained form TEM image is smaller than that estimated from the UV-Vis spectra using the effective mass model derived by Brus. This is because, in calculation, we take the onset wavelength, which is corresponding to the largest particle size. Nevertheless, the TEM results are in agreement with that obtained from the UV-Vis and PL spectra. The more PEO will reduce the particle size, and the PEO-stabilized NCs show a narrow size distribution.

## Conclusions

We have demonstrated that stable CdS NC colloids could be prepared in terms of solvothermal reaction of $Cd(OAc)_2$ and thiourea in DMF, using PEO as the stabilizer. PEO is not only the stabilizer but provides a confined nanospace for the growth of CdS NCs. The colloidal solutions emit bright blue color under irradiation of UV lamp. The kinetic study reveals that a higher $EO/Cd2^{+}$ ratio leads to smaller nanoparticles. It is worth to note that regardless of $EO/Cd2^{+}$ ratio and $Cd^{2+}/S^{2-}$, the PL spectra of the resulting CdS solution are similar, in which only a sharp peak with a narrow FWHM is shown in the blue region.

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