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# Preparation of CdTe nanocrystals and CdTe/SiO<sub>2</sub> nanocomposites in glycol

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### Abstract

CdTe nanocrystals were prepared in glycol using the reaction between  $Cd^{2+}$  and NaHTe in the presence of thioglycolic acid (TGA) as the stabilizing agents. And CdTe/SiO<sub>2</sub> nanocomposites were fabricated in situ. The structure of CdTe/SiO<sub>2</sub> nanocomposites was discrete CdTe nanocrystals embedded in a cross-linked silica matrix with the size of ca. 28 nm. This kind of nanocomposites was water-soluble and exhibited stronger stability than the pure CdTe nanocrystals, which could be applied in biological labeling and other fields. © 2004 Elsevier B.V. All rights reserved.

Keywords: CdTe; CdTe/SiO2 nanocomposites; Glycol

# 1. Introduction

Semiconductor nanocrystals show unique size-dependent optical properties and are currently of great interest for fundamental research and industrial development in recent years [1–3]. CdTe nanocrystals are an important II–VI semiconductor material, which has been used in numerous applications, such as in light-emitting devices [4], photonic [5] and biological labeling [6]. The stability of nanocrystals is essential for their applications. However, a simple thiol capped CdTe nanocrystals surface was not sufficient to accomplish a permanent stabilization [7,8].

Silica coating is one possible way to improve the stability and avoid the coagulation of the colloid, which has the following advantages: (1) optical transparency; (2) chemical inertness; (3) photochemical stability even under laser photolysis; (4) cheapness; (5) readily introduced functional groups for further linkage; (6) easily transferred into a wide range of solvent.

In this paper, CdTe nanocrystals and CdTe/SiO<sub>2</sub> composites were prepared in glycol. The choice of glycol is based on its high boiling point, which always means faster growth rate, better crystallinity and narrower size distributions of the nanocrystals. Moreover, it could prevent the hydrolysis of thiols and reduce the amount of water in the system, so that we can easily control the formation and the growth of silica. The CdTe/SiO<sub>2</sub> nanocomposites could be easily transferred into water, which is important for their potential application in biological labeling.

# 2. Experimental section

# 2.1. Chemicals

Tellurium (reagent powder), thioglycolic acid (TGA), tetramethylammonium hydroxide (TMAH, 25% in methanol) were purchased from Acros. Methyltriethoxysilane (MTES, 99%) was purchased from Aldrich. 3-Mercaptopropyltrimethoxysilane (MPS) was purchased from

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Tokyo Kasei Kofyo Co., Ltd. NaBH<sub>4</sub> (A.R.), dihydrous cadmium acetate (A.R.) and tetraethoxysilane (TEOS) were acquired from standard source and TEOS was redistilled before used. Water was purified with Milli-Q ( $18.3 \text{ M}\Omega$ ) water system.

#### 2.2. Preparation of CdTe nanocrystals

CdTe nanocrystals were prepared by adding N<sub>2</sub>-saturated cadmium acetate hydrate in glycol to NaHTe solution in the presence of TGA as stabilizer. The molar ratio of  $Cd^{2+}/TGA/HTe^{-}$  was 2:4.8:1, and the concentration of HTe<sup>-</sup> was fixed at 2 mM. The mixture then heated at 120 °C for several hours. For the purposes of comparison, the CdTe nanocrystals were prepared in the similar conditions except water instead of glycol, and refluxed for several hours.

#### 2.3. Preparation of CdTe/SiO<sub>2</sub> nanocomposites

 $5 \,\mu$ l of MPS was added to  $50 \,\text{ml}$  CdTe nanocrystals prepared in glycol under vigorous stirring. After the mixtures were stirred for 12 h, 15  $\mu$ l MTES and 15  $\mu$ l TEOS were added in batches, and then reacted for about 24 h at 40 °C. TMAH was used to maintain the pH range of 8–9.5. The resulting CdTe/SiO<sub>2</sub> nanocomposites were transferred from glycol media to water by centrifugation at 8000 rpm and redispersion.

### 2.4. Instruments

Transmission electron micrograph (TEM) images were recorded by a JEOL-2010 electron microscope equipped with CCD systems operating at 200 kV. The diluted CdTe/SiO<sub>2</sub>

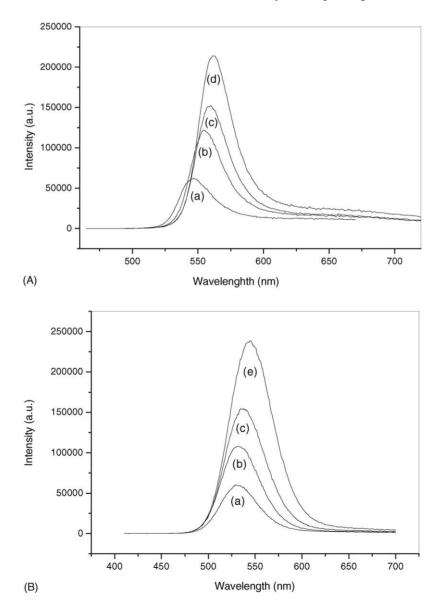


Fig. 1. Luminescence spectra of CdTe prepared in glycol (A) and water (B). Reaction time: (a) 15 min; (b) 30 min; (c) 1 h; (d) 1.5 h; (e) 2 h. The excitation wavelength is 400 nm.

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