



The role of potassium tellurite as tellurium source in mercaptoacetic acid-capped CdTe nanoparticles

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ABSTRACT

Water-soluble CdTe nanoparticles were synthesized in aqueous solution with the assistance of mercaptoacetic acid (MAA) molecules by wet chemical route and microwave-assisted method. A series of cadmium telluride (CdTe) nanoparticles capped with a bifunctional molecule, which contains both thiols and carboxylic acid groups were prepared using different pH values and using potassium tellurite as tellurium source. Thiol-capped nanocrystals of CdTe can be isolated as powders using 2-propanol. The synthesized thiol-capped CdTe were characterized with EDAX, TEM, Raman, FT-IR, UV-Visible absorption, fluorescence spectroscopy and X-ray diffraction (XRD) for the particle size determination and to understand their optical properties. The particles crystallize predominantly in cubic phase with narrow photoluminescence emission. Potassium tellurite as source of tellurium improves the photoluminescence efficiency and also avoids the cumbersome processes associated with H_2Te or $NaHTe$ sources.

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1. Introduction

Semiconductor nanoparticles are currently being studied in the context of their size-dependent photophysical and photochemical properties. The size of the nanoparticles in all three dimensions is less than the de Broglie wavelength of the electronic subsystem excitations. Therefore, many properties of these materials can be systematically described and understood in terms of quantum confinement effects [1–5]. From the viewpoint of basic science, a nanoparticle is an intermediate state of matter between molecule-like clusters and bulk crystals, and therefore offers a possibility for an evolution of electronic and optical properties of the matter from small atomic clusters to bulk solids. In the context of applications, nanoparticles can be efficiently used in novel light-emitting devices, optical transformers, photonic switches, nanoelectronic circuitry, and other applications [6–11].

Bulk CdTe is a direct band gap semiconductor with an absorption onset at 826 nm ($E_g = 1.56$ eV at 300 K) and an exciton Bohr radius of 7.3 nm [12]. Compared with other II–VI semiconductors such as CdS and CdSe, CdTe has a smaller band gap and a larger Bohr exciton radius and thus is expected to show a stronger quantum confinement effect for smaller size particles. Due to the strong confinement of charge carriers, CdTe nanoparticles should enhance

nonlinear optical properties, which can be useful in electro-optics. CdTe was used in IR detectors, solar cells, and light-emitting diodes. Because of high quantum efficiency and multicolor availability, CdTe nanoparticles can find applications in solid-state lighting, displays, optical communication, sensors, as well as in biological imaging and detection [13]. Currently, two synthesis strategies, namely; nonaqueous synthesis and aqueous synthesis, are used to prepare CdTe nanoparticles. As compared to the nonaqueous synthesis, aqueous synthesis is more reproducible, cheaper, less toxic, and the “as-prepared” samples are more water-soluble and bio-compatible [14]. On the other hand, microwave dielectric heating is fast emerging as a widely accepted new processing technology. With comparison to conventional thermal techniques, microwave dielectric heating has three dominating merits: (a) temperature can be rapidly raised due to high utilization factor of microwave energy, and the kinetics of the reaction rate are increased by 1–2 orders of magnitude, (b) thermal gradient effects can be effectively reduced due to the volumetric heating of microwaves, which is favorable for realizing homogeneous heating and producing a more uniform product formation, and (c) reaction selectivity is enhanced since different kinds of substances have a varied dipole constant [15]. Therefore, microwave methodology has been widely used in various fields including plasma and analytical chemistry, chemical catalysis, and organic reactions [16]. Besides these, it is a rapidly developing area of research, which has attracted much attention as a new method for preparing

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nanoscale inorganic compounds [17]. It was also demonstrated that high-quality quantum dots could be successfully prepared by microwave dielectric heating [18]. In addition, CdTe NCs, whose Photo-Luminescence Quantum Yield (PLQY) was increased to 60% under optimum conditions, were obtained by microwave irradiation in the aqueous phase [19].

Colloidal nanoparticles have been investigated for many years, and it has been reported that thiolates are effective stabilizer agents for preparation of nanometer-sized particles [20,21]. Mercaptoacetic acid (MAA) is a kind of thiolate that has been used in preparation and assembly of CdS [22] and CdTe [23,24] nanoparticles as the stabilizer agent. MAA is a bifunctional molecule, which contains both thiols and carboxylic acid groups. Both carboxylic acids and thiols are known to be absorbed on metal surface, forming well-packed monolayers [25–27]. CdTe nanoparticles were prepared in aqueous solution with H_2Te or $NaHTe$ as a source for tellurium [14,24] in the literature. Al_2Te_3 lumps were used as source for the generation of H_2Te and are transported to the reaction chamber with a slow nitrogen flow to synthesis the precursors. These precursors are then converted to CdTe nanocrystals by refluxing the reaction mixture at 100 °C. The refluxing time controlled the size of the particles. When sodium hydrogen tellurite ($NaHTe$) was used as source, it was synthesized with suitable reaction of sodium borohydrate with tellurium in water. The reaction was carried out in an ice cooled reacting system and proper care should be taken to avoid the excess pressure from resulting hydrogen. In order to overcome, these cumbersome processes to get the tellurium source, effort was made to use potassium tellurite as tellurium source in the present work.

Potassium tellurite was used as tellurium source to prepare different sizes of CdTe nanoparticles by wet chemical route and microwave-assisted method with different pH values as 7.4, 10.5, and 11.2 and stabilized with MAA. The structure of phonons, absorption and emission of nanoparticles are studied through X-ray diffraction (XRD), EDAX, TEM, Raman, FT-IR, and fluorescence spectroscopy and UV-Visible optical absorption analysis. The results are discussed in detail.

2. Experimental section

All chemicals used were of the highest purity available. De-ionized water obtained from a Millipore Milli-Q Plus purification system was used in all experiments. In the present work, CdTe nanoparticles were prepared by two different methods (wet chemical and microwave-assisted processes).

In the wet chemical method, the precursor, $CdCl_2 \cdot 5H_2O$ (0.046 g) was dissolved in 250 ml of de-ionized water and an appropriate amount (0.23 g) of the MAA was added under stirring; this was followed by adjusting the pH by dropwise addition of a 1 M solution of KOH. Occasionally, the solution can remain slightly turbid at this stage because of the incomplete solubility of Cd thiolate complexes, but this does not influence the further synthesis. The solution was placed in a three-necked flask fitted with a septum and valves and was deaerated by N_2 bubbling for 30 min.

0.063 g of potassium tellurite is dissolved in 250 ml of de-ionized water in the presence of appropriate amount (0.23 g) of the MAA under stirring. Potassium tellurite solution was then added to the $CdCl_2 \cdot 5H_2O$ solution under continuous (vigorous) stirring. After addition of potassium tellurite, the solution was stirred for two hours then CdTe precursors are formed at this stage. In the microwave-assisted method, CdTe precursor solution was injected into the exclusive vitreous vessel with a volume of 50 ml, and reacted in microwave furnace (GMS 19 A Microwave Oven) for 5 min. The reactions were operated at 700 W power in a cyclic mode (on: 15 s; off 6 s) to avoid intense boiling of the solution. The size and optical properties of the CdTe nanoparticles were controlled by controlling the microwave irradiation time and the concentration of Cd^{2+} , Te^{2-} , and MAA. The reaction was stopped by removing the vessel from the microwave furnace and cooling naturally to room temperature. The molar ratio of $Cd^{2+}/MAA/Te^{2-}$ was set as 1:10:1. The particles were extracted by precipitation with the addition of 2-propanol to the solution. The resulting powder was separated by centrifuging and then dried at room temperature. Six samples were prepared having different sizes (Table 1), hereafter called (S_1 , S_2 , S_3 , S_4 , S_5 , and S_6).

X-ray powder diffraction analysis was carried out with a Philips X'Pert PRO diffractometer with $CuK\alpha$ ($\lambda = 0.15406$ nm) radiation and EDAX were performed on EDS Noran System Six (Thermo Electron Corporation). Transmission electron microscopy (TEM) was performed by a JEM-1011 (accelerating voltage of 100 kV) in order to analyze the size and structure of the resulted samples. IR spectra were recorded with a Perkin Elmer FT-IR spectrometer within the range of 500–4000 cm^{-1} . Raman spectroscopy was performed for powders of CdTe nanoparticles with Raman Systems-R 3000. Room temperature UV-Vis absorption spectra were measured using a Cary 5E UV-Vis-NIR spectrophotometer. Photoluminescence measurements were performed at room temperature using JY Fluorolog-3-11 spectrofluorometer (at the excitation wavelength $\lambda_{exc} = 350$ nm). All optical measurements were performed at room temperature.

3. Results and discussion

3.1. Structural characterization

3.1.1. XRD, EDAX analysis and TEM image

Thiol-stabilized CdTe nanoparticles belonging to the cubic (zincblende) crystalline phase were formed in aqueous solution by reaction of Cd^{2+} and Te^{2-} ions in the presence of thiols as stabilizers. Structural characterization of the CdTe nanoparticles was carried out with powder X-ray diffraction (XRD) analysis. XRD patterns observed for CdTe nanoparticles (pH = 7.4, 10.5 and 11.2 for two different preparation methods) are shown in Fig. 1. XRD spectra shows direct evidence for the crystalline nature of the nanocrystals and CdTe samples exhibit four peaks that can be assigned to (200), (220), (222) and (400) diffractions corresponding to cubic zinc blende structure. These peaks are Scherrer broadened due to finite crystalline size. The crystallite size, d_{XRD} , was further

Table 1
Sample name, preparation method, pH values and Scherrer sizes of CdTe nanoparticles capped with mercaptoacetic acid.

Samples	Preparation method	pH	Scherrer size (nm)	Absorption edge (nm)	Emission peak (nm)
S_1	Wet chemical route	7.4	8	495	493
S_2	Wet chemical route	10.5	10	509	506
S_3	Wet chemical route	11.2	12	530	511
S_4	Microwave method	7.4	15	540	517
S_5	Microwave method	10.5	17	562	526
S_6	Microwave method	11.2	20	574	537

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