SYNTHESIS, CHARACTERIZATION AND PHOTOLUMINESCENCE PROPERTIES OF CdTe NANOCRYSTALS

T. SURIWONG*, A. PHURUANGRATb, S. THONGTEMc, T. THONGTEmd,e
aSchool of Renewable Energy Technology, Naresuan University, Phitsanulok 65000, Thailand
bDepartment of Materials Science and Technology, Faculty of Science, Prince of Songkla University, Hat Yai, Songkhla 90112, Thailand
cDepartment of Physics and Materials Science, Faculty of Science, Chiang Mai University, Chiang Mai 50200, Thailand
dDepartment of Chemistry, Faculty of Science, Chiang Mai University, Chiang Mai 50200, Thailand
eMaterials Science Research Center, Faculty of Science, Chiang Mai University, Chiang Mai 50200, Thailand

Cadmium telluride (CdTe) nanocrystals were solvothermally synthesized using Cd(NO₃)₂·4H₂O and Te powder in ethylenediamine (C₅H₄(NH₂)₂) at 200 °C for 12–72 h. Phase, morphology and photoluminescence (PL) property of the products were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and photoluminescence spectroscopy. The product synthesized at 200 °C for 72 h was specified as cubic CdTe nanocrystals (41.39 nm) evaluated by the Scherrer equation, with 550 nm emission peak excited by 300 nm wavelength.

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1. Introduction

Semiconducting cadmium telluride nanocrystals are a great attractive material for fundamental research and applications in optoelectronic and photovoltaic devices, including in the fields of thermoelectric, electronic, optical, catalyst, biological and magnetic [1, 2]. Cadmium telluride (CdTe) with 1.56 eV energy gap at 300 K is the most important material of the II-VI semiconducting compound with high PL quantum efficiencies [2–4]. It illuminates within visible spectral range from 500 to 730 nm influenced by the particle size and quantum confinement effect [2, 5]. The PL spectroscopic analysis indicates that CdTe nanoparticles (NPs) show an emission peak at 572 nm reported by Wang et al [6]. But for Shen et al [7], they reported the green- to red-emitting CdTe quantum dots (QDs) with a maximum photoluminescence quantum yield of 56.68 %. The as-synthesized CdTe nanodisks show a photoluminescence peak influenced by thickness of the disks [8]. In this paper, CdTe nanocrystals were synthesized by a solvothermal process at 200 °C for 12–72 h. The phase, morphology and photoluminescence property of the products were then characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and photoluminescence (PL) spectroscopy.

*Corresponding authors: phuruangrat@hotmail.com
2. Experiment

All chemicals were analytical grade and were used without further chemical treatment during the whole synthesis process. In a typical procedure, each of 5 mmol Cd(NO3)2·4H2O and Te powder was dissolved in each 50 ml ethylenediamine (C2H4(NH2)2) with the subsequent addition of 2 ml hydrazine (N2H4) to the Te solution. The obtained solutions were stirred for 30 min, mixed and further stirred for another 30 min. The mixture was transferred to a 200 ml Teflon-lined stainless steel autoclave. Then the autoclave was tightly closed. A few more of the same systems were prepared. They were maintained at 200 °C in an electric oven for 12, 24, 48 and 72 h. At the conclusion of the process, the autoclaves were naturally cooled to room temperature. The obtained precipitates were washed with distilled water and ethanol, dried at 80 °C for 24 h, and collected for further characterization.

The phase and chemical composition of the as-synthesized products were characterized by X-ray diffraction with Cu Kα radiation (1.54056 Å) of a Philips X'Pert MPD X-ray diffractometer (XRD) operating at 30 kV with a scanning rate 0.02 °s⁻¹. The morphology was characterized by scanning electron microscopy (SEM, JEOL JSM-6335F) using LaB6 as an electron gun with an accelerating voltage 15 kV. Photoluminescence (PL) measurement was carried out on a Perkin Elmer LS 50B Fluorescence spectrometer with 300 nm excitation wavelength over the 300–800 nm wavelength range at a 0.5 nm·s⁻¹ scanning rate.

3. Results and discussion

The X-ray diffraction patterns in the 2θ of 10°–60° range of the as-synthesized CdTe products are shown in Fig. 1. The as-synthesized CdTe nanostructure exhibited strong diffraction peaks at 2θ = 23.64°, 39.34°, 46.52° and 57.00° corresponding to the (111), (220), (311) and (400) planes of cubic CdTe phase of the JCPDS database no. 15-0770 [9]. The formation of other cadmium and tellurium compounds such as CdTe, CdO, TeO, and CdTe2O were not detected in this analysis, specified that the as-obtained products were highly purified. They should be noted that the intensity ratio between the (111) and (220) planes of 100 : 94 in the present case is higher than that of 100 : 60 belonging to the JCPDS database no. 15-0770, revealing the growth of CdTe crystal along the [110] direction. Lattice constant (a) for cubic structure was calculated using the following equation

\[
\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2}
\]

where d is the space between adjacent planes in the set (h k l) being the Miller indices [10]. The calculated lattice parameter of cubic CdTe phase equals 6.4776 Å and is in good accordance with the value of the JCPDS database no. 15-0770 with a = 6.4810 Å for zinc blende CdTe structure. The Scherrer equation connected with particle size is given below

\[
d = \frac{0.94 \lambda}{\beta \cos \theta}
\]

where d is the crystallite size, λ is the wavelength of the Cu Kα radiation (λ = 1.54056 Å), β is full width at half maximum (FWHM) of the XRD peak, and θ is the diffraction angle of the corresponding peak [10, 11]. Average size of the crystals was calculated using the Scherrer equation of which the size is 41.39 nm for the 200 °C and 72 h synthesis.
A possible formation mechanism of CdTe was proposed as follows. When Cd(NO$_3$)$_2$·4H$_2$O was dissolved in ethylenediamine (en, a strongly bidentate ligand) [12–14], Cd$^{2+}$ reacted with en to form Cd(en)$_2^{2+}$ complexes by the equation

$$\text{Cd}^{2+} + 2\text{en} \rightarrow \text{Cd(en)}_2^{2+} \text{complexes}$$  \hspace{1cm} (3)

Subsequently, Te powder was reduced to Te$^{2-}$ by hydrazine [12, 15]

$$2\text{Te} + \text{N}_2\text{H}_4 + 4\text{OH}^- \rightarrow 2\text{Te}^{2-} + \text{N}_2 + 4\text{H}_2\text{O}$$ \hspace{1cm} (4)

Under solvothermal condition, CdTe nanocrystals [12–14] formed by a few steps shown below

$$\text{Cd(en)}_2^{2+} + \text{Te}^{2-} \leftrightarrow \text{CdTe} \cdot 2\text{en} \leftrightarrow \text{CdTe} + 2\text{en}$$ \hspace{1cm} (5)

Fig. 2 shows SEM images of CdTe nanocrystals synthesized by solvothermal reaction at 200 °C for 12 h and 72 h. They can be seen that crystalline CdTe presented observable change in grain size when the reaction time was lengthened from 12 h to 72 h. The size of grains was increased when the processing time was lengthened. The particle size of the crystalline CdTe at 200 °C for 12 h was 0.8–1.0 μm and was increased to 1.5–2.0 μm for the crystalline CdTe synthesized at 200 °C for 72 h. In order to decrease their surface energies, the very tiny nanoparticles were agglomerated to form the larger ones [16]. For smaller particles and larger surface area, more numbers of surface atoms exist on the particles. These surface atoms are unsaturated and have vacant coordination sites or dangling bonds. They have very high affinity to form bonds with adjacent particles, leading to agglomerations. Thus smaller particles are always cluster together in groups by forming agglomerations.
Fig. 2 SEM images of CdTe samples synthesized by the solvothermal reaction at 200 °C for (a) 12 h and (b) 72 h.

Fig. 3 shows PL spectrum of CdTe nanocrystals synthesized via the solvothermal reaction at 200 °C for 72 h excited by 300 nm wavelength.

Fig. 3 PL emission of CdTe synthesized by the 200 °C and 72 h solvothermal reaction

PL spectrum shows a visible emission at 550 nm, blue shift with respect to bulk CdTe (863 nm) [17]. Possibly, it associated with defects or surface-trap emission [18, 19]. Menezes et al [20] reported that CdTe nanoparticles have only one fluorescence band with maximum emission at 556.5 nm wavelength.

4. Conclusions

CdTe nanocrystals were successfully synthesized from Cd(NO₃)₂·4H₂O and Te powder in ethylenediamine by solvothermal reaction at 200 °C for 72 h. A possible formation mechanism of CdTe was discussed in this report, and the cubic CdTe nanocrystals show an emission peak at 550 nm excited by 300 nm wavelength.

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References