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A COMPARATIVE STUDY ON THE SYNTHESIS OF THE CdS NANOPARTICLE BY THERMAL DECOMPOSITION USING AUTOCLAVE AND γ -RADIATION

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The nanocrystalline CdS was prepared by the two methods, thermal decomposition using autoclave about 300°C and by γ -radiation at room temperature. Cd(oleate)₂ solution was used as the cadmium ion sources. Na₂S·9H₂O was used as the S sources. X-ray Powder Diffraction (XRD) and Transmission Electron Microscopy (TEM) were used to characterize the structure and to observe their morphology and size of produced CdS nanoparticle, respectively. The photoluminescence (PL) spectrum was obtained to characterize the optical properties of CdS nanoparticle.

Keywords: cadmium sulfide nanoparticles; γ -radiation

INTRODUCTION

The synthesis with defined nanometer scale features is of great interest in material chemistry. Considerable progress has been made in of group II-VI semiconductor crystallines and its nanocrystallines. In the past, the methods involved solid reactions, aqueous solutions, gas reactions and the precursor ones. But such II-VI materials have been prepared at high temperature (above 500°C) [4–8]. It has been reported that the nanoscale products have different properties from each method. The new nanoscale materials have potential applications in both mesoscopic research and the development of nano-devices. Little research has been done on the one-dimensional nanomaterials such as nanowires, nanorod or nanotube at ambient conditions [1]. Li and co-workers reported on the preparation of

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the ordered CdS nanowires in an anodic alumina oxide (AAO) template [9]. In the present study, radiation of radioactive such as γ -ray was used to initiate the growth of cadmium sulfide particles by the reaction of solvated electron with the thiol (3-mercapto-1, 2-propandiol RSH) to release HS^- ions, and the resultant colloidal solutions including nanoparticle were studied. Recently γ -ray radiation has been used to produce nanocrystalline metals, alloys, metal oxide, and composites (ZnS, PbS and CuS etc.) [1–3,5]. Yi Xie and co-workers reported a novel simultaneous in situ formation (SISF) technique for the synthesis ZnS nanowires via γ -irradiation [1]. Due to the work at atmospheric pressure and in the room temperature, this method can be used as a new method for the synthesis of inorganic nanoparticles via more simple process than one in the past. In this paper, we synthesized CdS nanoparticle by both thermal decomposition using autoclave and γ -ray radiation. The produced CdS nanoparticles were studied on the crystal structure and on the optical properties.

EXPERIMENT

Cadmium chloride hemipentahydrate ($\text{CdCl}_2 \cdot 5/2\text{H}_2\text{O}$, 99%), sodium oleate ($\text{C}_{17}\text{H}_{33}\text{COONa}$, 98%) and sodium sulfide nonahydrate ($\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$, 98⁺%) were obtained from Aldrich. All reagent-grade chemicals were used as received, and house-distilled water was passed through a four-cartridge Barnstead Nanopure II purification train consisting of Macropure pre-treatment, organic free, and a 0.2 μm hollow-fiber final filter for removing particles. Its resistivity was determined as 18.4 $\text{M}\Omega$ and used throughout. Sodium oleate (1 mmol) was dissolved in 80 ml of water to obtain aqueous solution. $\text{CdCl}_2 \cdot 5/2\text{H}_2\text{O}$ (0.5 mmol) was dissolved in 1 ml of water and mixed with the prepared sodium oleate solution. $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ (1 mmol) powders were added into mixed solution of sodium oleate and CdCl_2 and then continuously stirred for 2 h. Then, the reactant solution were filled only 80 vol% on a cavity of the autoclave. Before the temperature of the reaction autoclave to be maintained at 300°C for 2 h, the temperature was slowly increased up to 300°C (60°C per 30 minutes). Finally, the autoclave was cooled down to the room temperature. On the other hands, the same reactants were irradiated with γ -ray of a dose of 1.8×10^4 Gy in the field of a ^{60}Co γ -ray source. The product was washed with acetone, filtered and then dried at room temperature. The color of both products are yellow powders. The samples were characterized by X-ray powder diffraction (XRD) patterns to investigate the structure. X-ray powder diffraction (XRD) spectra were collected using a Philips, X'Pert-MPD system. Transmission Electron Microscopy (TEM) was investigated to study on the morphology and particle sizes. Transmission electron microscopy (TEM)

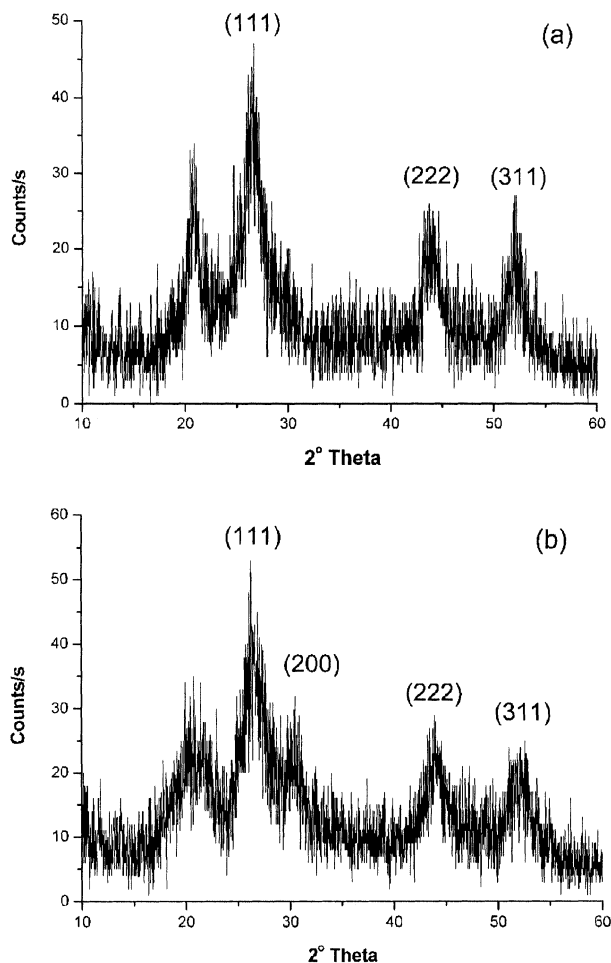


FIGURE 1 XRD patterns of the synthesized CdS nanoparticles: (a) thermal decomposition using autoclave at 300°C (b) γ -ray radiation in room temperature.

images were obtained using a Jeol, JEM-2010. The photoluminescence (PL) spectra was obtained to study on the optical properties of the synthesized CdS nanoparticles. Photoluminescence spectra were recorded on a Perkin-Elmer (LS50B).

RESULTS AND DISCUSSION

Figure 1 shows the XRD patterns of the CdS nanoparticles synthesized by thermal decomposition using autoclave and γ -ray radiation. The discernible

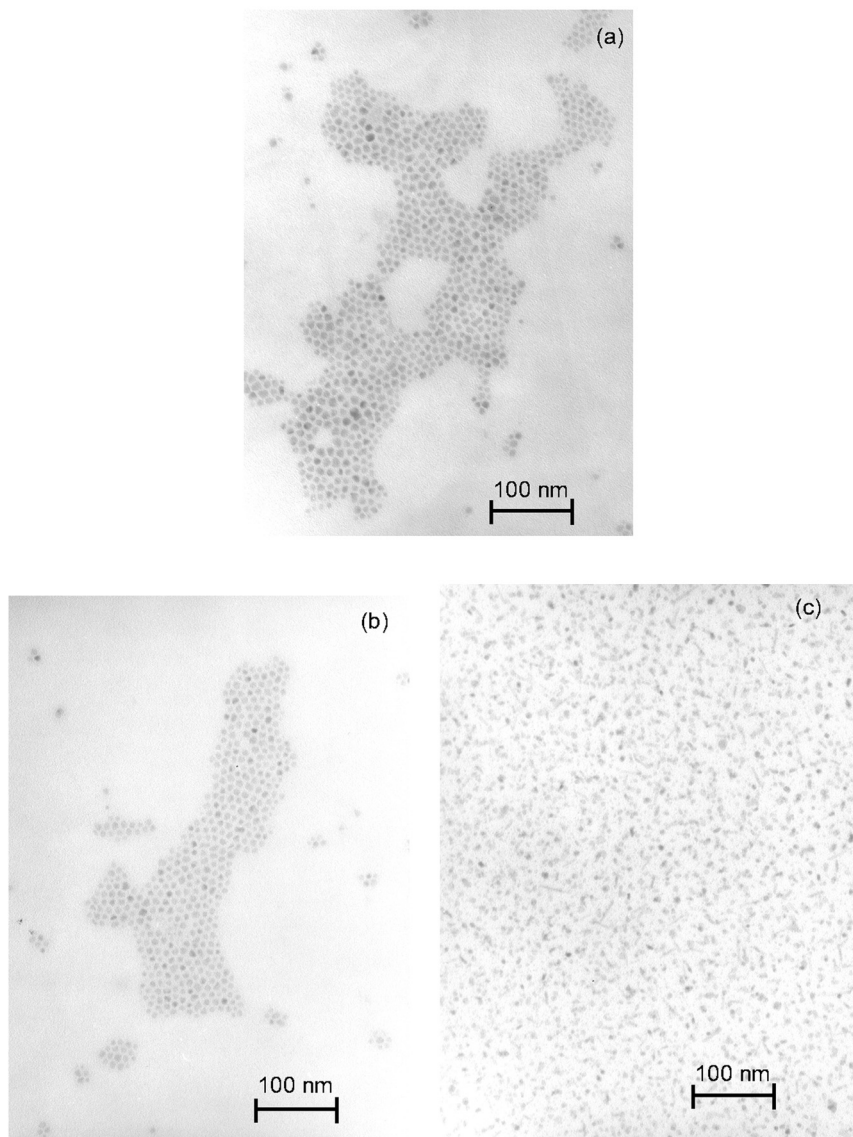


FIGURE 2 TEM images of the synthesized CdS nanoparticles: (a) thermal decomposition using autoclave at 300°C, (b and c) γ -ray radiation in room temperature.

peaks can be indexed to (111), (220), and (311) planes of the cubic structure of CdS with cell constant $a = 5.811 \text{ \AA}$ as Figure 1(a). In Figure 1(b), the peaks are also assigned to (111), (200), (220), and (311) planes of the cubic structure of CdS nanoparticle with cell constant $a = 5.811 \text{ \AA}$. These are corresponding to the structure of CdS (JCPDS, card no. 80-0019). Both products showed the same structure. A drop of

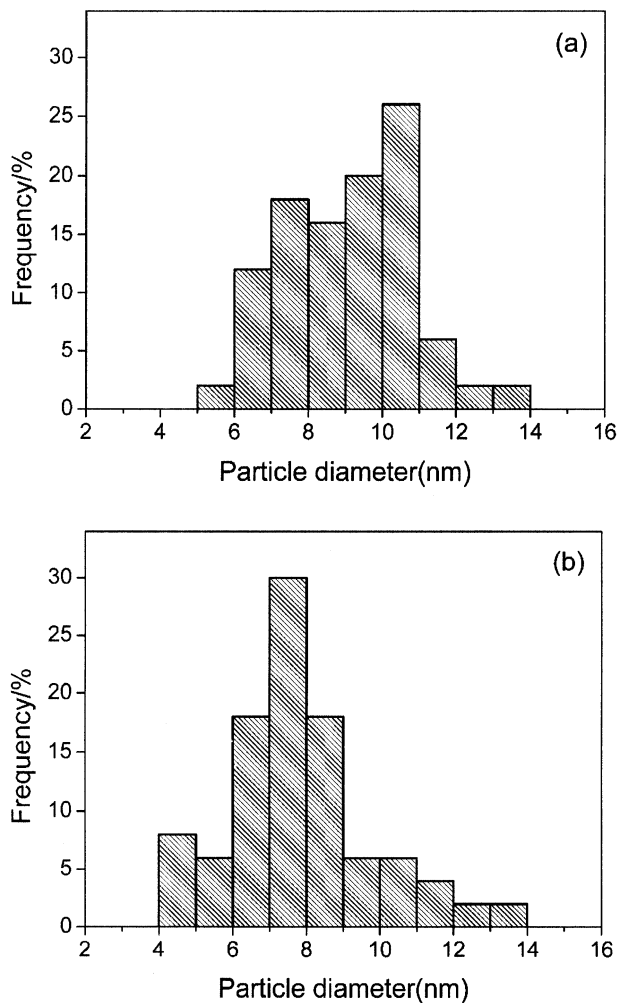


FIGURE 3 Histograms on the size distribution of the synthesized CdS nanoparticles: (a) thermal decomposition using autoclave at 300°C (b) γ -ray radiation in room temperature.

CdS nanoparticle in iso-octane was placed on the grid and dried in air. TEM image in Figure 2(a) shows that CdS nanoparticle synthesized by thermal decomposition using autoclave has a uniform irregular ball-shaped. The size of CdS nanocrystallines has been determined as 7–10 nm size from the TEM image as Figure 3(a). On the other hand, the synthesized CdS nanoparticle by γ -ray radiation shows to have a mixture of two types as Figure 2(b), 2(c) on the same sample. These are obtained in the different

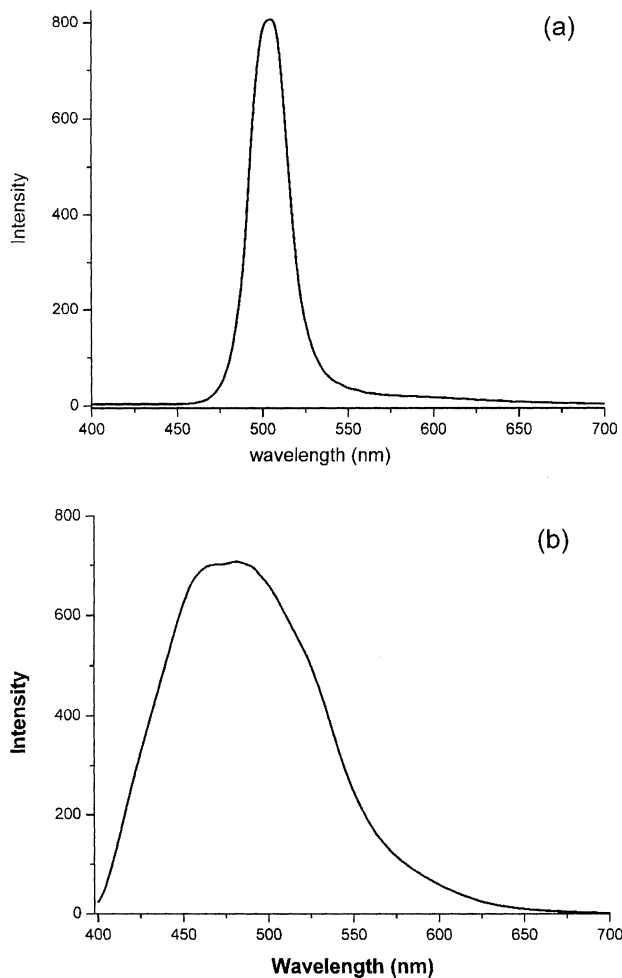


FIGURE 4 Photoluminescence spectra for the synthesized CdS nanoparticles dispersed in iso-octane, $\lambda_{\text{exc}} = 365$ nm: (a) thermal decomposition using autoclave at 300°C (b) γ -ray radiation in room temperature.

parts of the same sample. Figure 2(b) shows CdS nanoparticles with the diameters of 4–14 nm, but the most of the nanoparticles are in the size range of 8–9 nm as shown in histogram of Figure 3(b). Figure 2(c) is the TEM image of CdS nanoparticle synthesized by γ -ray radiation and obtained in the different part of the same sample. The size distribution of CdS nanoparticle was determined as 4 ± 1.2 nm from the TEM image. This is a contrasting one to the size distribution of Figure 2(b). The particle size was not uniform compared to Figure 2(b) and also mixed with irregular spherical shape and rod shape. This result indicates that the synthetic procedure of CdS nanoparticle by γ -ray radiation should be carried out by controlling the condition such as molar ratio among reactants, a dose of γ -ray and radiation time more carefully. The furthermore control of such conditions will be reported on the following report.

The optical properties of as-prepared CdS powders were studied with photoluminescence spectra as Figure 4. The photoluminescence spectrum of CdS nanoparticle synthesized by thermal decomposition using autoclave is shown in Figure 4(a). This shows the narrow emission band of 480–550 nm. While the photoluminescence spectrum of CdS nanoparticle synthesized by γ -ray radiation is shown in Figure 4(b). The spectrum shows the wide band at the wavelength of 400–600 nm. It is considered as the mixture of the two types of the CdS nanoparticle by γ -radiation presumably consists of them as Figure 2(b) and (c). Fluorescence photographs of CdS nanoparticles synthesized by thermal decomposition as Figure 2(a) and γ -ray radiation as Figure 2(b) and (c) shows the different colors. The CdS nanoparticle synthesized by thermal decomposition showed a green color and CdS nanoparticle synthesized by γ -ray radiation shows yellow-green color. The latter is caused by the mixture of the differently sized ones as Figure 2(b) and (c).

CONCLUSIONS

CdS nanoparticle was synthesized by two different methods of thermal decomposition using autoclave and γ -ray radiation. The CdS nanoparticle synthesized by different methods showed the different size distributions, morphology and optical properties. CdS nanoparticle was synthesized by two different methods of thermal decomposition using autoclave has a rather narrow and homogeneous size distribution than the one by γ -ray radiation. The CdS nanoparticles of γ -ray radiation were not similar with each other and they showed the distinct two segregated groups of size distributions as size range of 4–14 nm and 4 ± 1.2 nm and morphologies. Photoluminescence spectra also showed a distinct comparison between them by two different synthetic methods. The CdS nanoparticles prepared

by using autoclave showed the narrow emission band because of homogeneous size distribution. This method was done at high temperature and high pressure. On the other hand, the CdS nanoparticles prepared by γ -ray radiation was done at the room temperature and the atmospheric pressure, but showed the wide band due to two kinds of particles size distributions.

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