

STRUCTURAL, MORPHOLOGICAL AND OPTICAL INVESTIGATION OF CdS NANOPARTICLES SYNTHESIZED BY MICROWAVE ASSISTED METHOD

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CdS is one of the II-VI compounds and is a promising material for its biolabeling properties and optoelectronic applications. In this work, CdS nanoparticles are grown by microwave assisted method using Cadmium-Acetate and Thioacetamide as a precursor solution. CdS nanoparticles were characterized for structural, optical and morphological study by using X-ray diffraction (XRD), UV-visible spectroscopy, scanning electron microscopy (SEM) and Transmission electron microscopy (TEM) respectively. The optical band-gap of CdS nanoparticles is found to be 3.84 eV from the absorbance spectra. The XRD peaks confirm the cubic structure of CdS and the particle size of synthesized CdS nanoparticles was found to be 5-7 nm.

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1. Introduction

Recent development in the field of material science has led to the immense research for developing alternate materials to show the fluorescent properties of nanoparticles. Semiconductor nanomaterials are attracting researchers from different domains of physics, chemistry and biology for both fundamental research as well as industrial development. The numerous applications depend upon the transition of electrons from conduction band to valence band and vice-versa in the energy band of the nanoparticles. There may be a presence of different energy level apart from valence band and conduction band. The recombination of electron hole pair generates the fluorescent properties [1]. The application of II-VI semiconductors includes the wide areas of optoelectronics, optical devices, nanosensors, solar cells or photoresistive film, biomedical applications, photosensing nanodevice and biolabeling [2-4, 8, 19-21]. In II-VI compounds, the optical properties depends upon the size of nanocrystals for their applications as biosensor, solar cells or photoresistive films [5, 19]. II-VI compound semiconductors such as ZnS, CdS, CdSe, etc., are promising materials for technological application, mostly due to their large band gap [6].

CdS is a II-VI compound semiconductor which has a direct band transition and has the application in optoelectronic devices because of its relatively low electron affinity, high chemical inertness, and sputter resistance [2]. This new advancement has led to the development of semiconductor nanocrystals as a building material for the numerous devices for various applications. CdS semiconductor quantum dots have been developed for application as nanosensor having good biolabeling properties for bioimaging. These QDs have many desirable properties which includes high quantum yields, high molar extinction coefficients, long fluorescence lifetimes, narrow emission spectra and chemical degradation [7, 13].

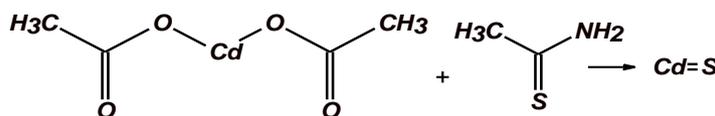
II-VI semiconductor nanoparticles have been synthesized by a variety of methods which include solvothermal method, biosynthesis method, sonochemical reduction method, co-

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precipitation method, sol-gel process, solid state reaction, chemical bath deposition method and microwave irradiation method [6,9-17]. In this work, CdS nanoparticles are grown by using microwave assisted method using cadmium-acetate and thioacetamide as a precursor solution. The synthesized CdS nanoparticles were characterized for structural, optical and morphological study by using X-ray diffraction (XRD), UV-visible spectroscopy, scanning electron microscopy (SEM) and Transmission electron microscopy (TEM) respectively.

2. Experimental

For the synthesis of CdS nanoparticles all the chemicals used were taken of AR grade. In this experiment freshly prepared aqueous solution of cadmium acetate (1M) and thioacetamide was taken. A solution of cadmium acetate ($\text{Cd}(\text{CH}_3\text{COOH})_2 \cdot 2\text{H}_2\text{O}$) is mixed with thioacetamide (CH_3CSNH_2) stoichiometrically. The volume of the solution was made to 800 ml by adding distilled water and then it was put on magnetic stirrer for an hour. Further, the solution was microwaved with a power of 900W by adding a few drops of NaOH to get the precipitated bright yellow colored CdS nanoparticles. The CdS nanoparticles were dried at 100°C in an oven for the next 6 hours. The resulting bright yellow colored CdS nanoparticles were then characterized for structural, optical and morphological study.



Scheme 1. Synthesis of CdS nanoparticles

3. Results and Discussions

The powder XRD analyses of the nanoparticles were performed in the 2θ range from 10 to 80° using Rigaku X-ray diffractometer with $\text{CuK}\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$). The Debye-Scherrer formula gives the crystalline size considering the full width at half maxima of XRD pattern shown in Fig. 1. The average nanoparticle size is calculated by the formula,

$$D = \frac{0.9\lambda}{\beta \cos \theta}$$

Where, λ is the wavelength of the incident ray, θ is the Bragg's angle and β is the full width at half maxima [18]. The average particle size is found to be 6-8 nm. The diffraction peaks obtained in the XRD pattern shows the cubic structure of CdS (JCPDS 10 – 454). It has been reported that the CdS (zinc blend structure) nanoparticles of size 6-9 nm shows high intensity fluorescent maxima tunable from 480 to 600 nm and has excellent resistance to photobleaching which has the application as biolabeling for tissues and cells [7].

The energy band gap of the CdS nanoparticles was evaluated by plotting a graph between $(\alpha h\nu)^2$ versus $h\nu$ and by extrapolating the linear region of the curve to the energy axis.

$$\alpha h\nu \propto A(h\nu - E_g)^{1/2}$$

Where α is the absorption coefficient, $h\nu$ is the photon energy, E_g is the direct band gap energy, and A is a constant.

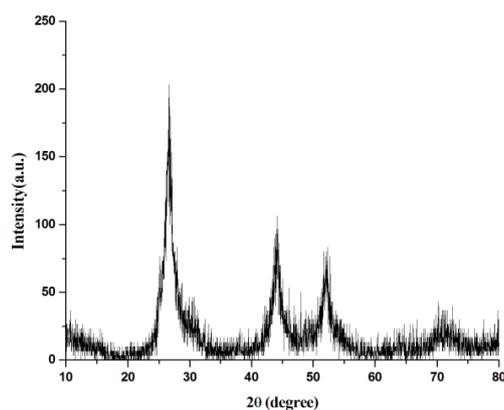


Fig. 1 XRD pattern of the CdS nanoparticles synthesized by microwave irradiation

The fluorescence property of a material is used as a basic tool for the exploration and differentiation of the labeling of the nanocrystals. Tissues and cells conjugated with QDs can be analyzed by the laser scanning confocal microscopy. Specific type of labeling may be accomplished when a targeting molecule interacts with the quantum dots.

SEM characterization of CdS nanoparticles was made by JEOL Model JSM - 6390LV. Fig 2a and Fig. 2b show the surface morphology which has been taken by SEM.

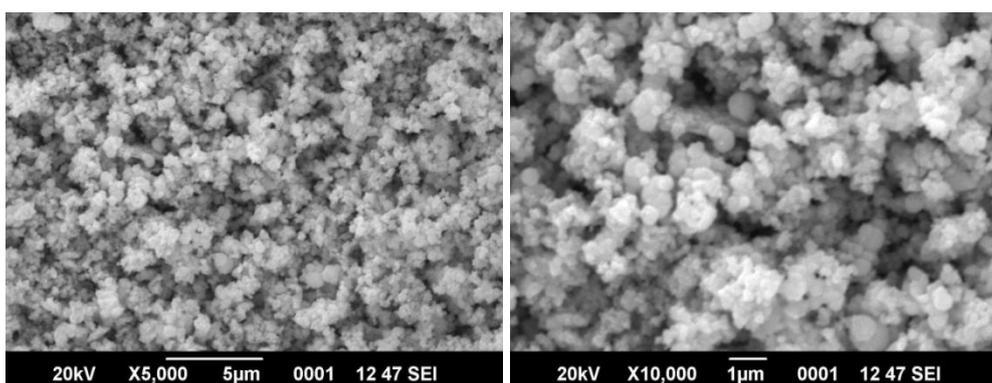


Fig. 2 SEM of the CdS nanoparticles synthesized by microwave assisted irradiation.

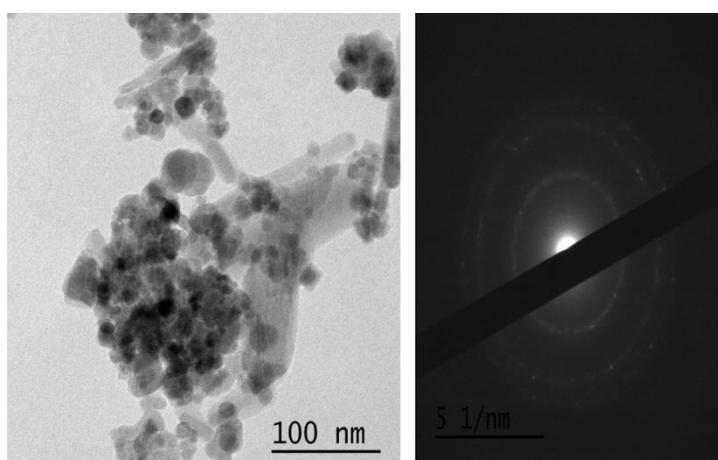


Fig.3. CdS nanoparticles: a) TEM image of CdS nanoparticles; b) selected area diffraction pattern (SAD)

The morphology of the as synthesized nanoparticles was analysed by HRTEM. TEM was taken to visualize the shape and size of synthesized CdS nanoparticles. It also confirms the nanocrystallinity of the sample. Figure 3a. shows the typical image of microwave synthesized CdS nanoparticles. It was also observed from the image that the particles were in abundance and were clearly well separated.

The average size of the nanoparticles was around 8-10 nm which is in good agreement with the size of nanoparticles calculated from XRD. Fig 3b showed the selected area electron diffraction (SAED) pattern of the nanoparticles. It was clear from the image measurements that the particles were crystallized and also the diffraction rings matched the XRD peaks quite well.

In this study, we were able to synthesize the nanoparticle of CdS of size 6-8 nm, which may be very useful in biolabeling of tissues and cells. Fig. 2 shows the SEM micrograph of the CdS nanoparticles.

The absorption spectrum of CdS nanoparticles is shown in Fig. 4 using UV- visible spectrophotometer (Rayleigh UV-2601). The intercept of the graph on X- axis gives the value of band gap as shown in Fig. 5. The optical bandgap is found out to be 3.84 eV.

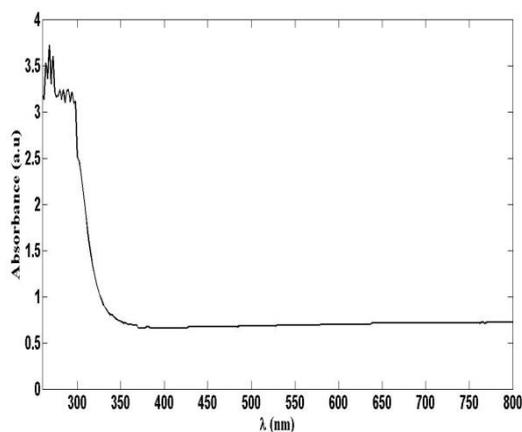


Fig. 4 Absorbition Spectrum of CdS nanoparticles synthesized by microwave irradiation.

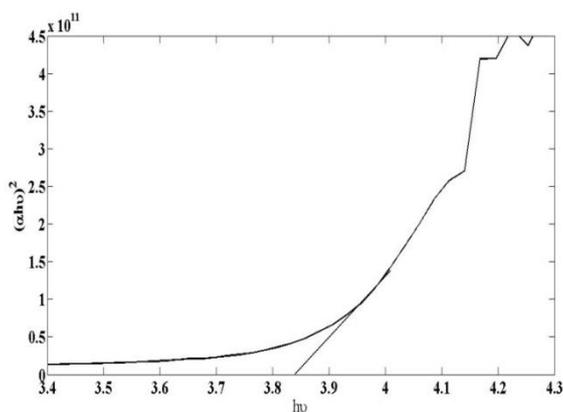


Fig. 5 Optical bandgap of CdS nanoparticles synthesized by microwave irradiation.

4. Conclusion

CdS nanoparticles of cubic phase with an average particle size of around 6-8 nm were obtained using a novel method of microwave irradiation. The synthesis was made using the aqueous solution of cadmium acetate and thioacetamide. The particle size was calculated using Debye-Scherer formula. CdS nanoparticles may have an application as biolabeling of tissues and cells. The fluorescence property of a material is used as a basic tool for the exploration and

differentiation of the labeling of the nanoparticles. Specific type of labeling may be accomplished when a targeting molecule interacts with the QDs. The optical band gap of synthesized CdS nanoparticles were obtained by UV- visible spectroscopy and it was found to be 3.84 eV.

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