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Synthesis and Characterization of CdSe Nanoparticles

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Abstract. CdSe capped with TSC nanoparticles was prepared by chemical method at room temperature. Spin coating method has been used to deposited thin film on the glass substrates at room temperature. The morphological, structural and optical properties of the thin film are characterized by transmission electron microscope , atomic force microscope and optical absorption and Photoluminescence measurements. The average roughness of CdSe thin film is 0.935 nm and the average diameter is 44 nm. The direct optical band gap (Eg) has been determined from the absorption coefficient which is equal to 2.2 eV. The increase in the band gap value of our thin films as compared to bulk is due to the quantum confinement effect. These nanoparticles were found to display strong photoluminescence with emission peaks at the green region. Increases of excitation wavelength lead to an increase in emission intensity.

INTRODUCTION

There are two main operators cause the properties of the material at Nanoscale being unique: the surface and quantum effects [1]. Their large surface area of nanomaterials can modify or improve properties such as mechanical, optical and electrical properties. The unique and tunable chemical, physical, and optoelectronic properties of nanoscale semiconductors ,such as CdS, CdSe, CdTe, etc.which influenced by the shape , size as well as composition give More attention to use these material in deferent applications[2,3].

The direct band gap of Nanocrystalline CdSe can be varying to show tremendous electrical and optical properties. There are three main well-known structures of CdSe; a haxagonal wurtziete structure, a cubic zinc blende (sphalirite) structure, and the rock salt. Only the wurtzite and the sphalirite exist at atmospheric pressures Usually the cubic exists in thin layers, while the bulk CdSe has the haxagonal structure [4]. it is a promesing semiconductor for many photovoltaics[5], ultrasensitive detection[6] , diode laser [7], biosensors[8] , light emitting diodes[9] .

In our work, we have prepared CdSe NPs by chemical method. Characterization of prepared samples have been done by using TEM, AFM, XRD, EDX measurements. The optical band gap determined, from absorption measurements.

EXPERIMENTAL

Sodium sulfite (Na2SO3), cadmium acetate [Cd (CH3COO) 2], and Selenium powder (Se) were used as precursors for Selenium and cadmium. Trisodium citrate (TSC) used as a complex agent while ammonia solution used as pH adjuster.

Sodiumselenosulfate (Na2SeSO3) solution was prepared by refluxing 3.15 g of sodium sulfite (Na2SO3) in 50 ml deionized water with 0.98 g of Se powder at 70C0 for 7 hr... And left overnight and filtered. The filtrate solution used as Se source. Cd precursor was prepared by refluxing 3.45 gm of cadmium acetate [Cd (CH3COO) 2] in 50 ml deionized water. 0.1 M TSC was prepared to use as complex agent. 10 ml from 0.1 M TSC solution added to 10 ml from cadmium acetate the PH of this solution attuned to 11 by add drops of ammonia , 7.5 ml from sodium

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selenosulfate (Na2SeSO3) solution added slowly and the final solution kept on stirrer at room temperature for 7 hrs. Spin coating method has been used to deposited thin film on the glass substrates at room temperature.

The absorption spectra of the thin film has been measured by a UV/VIS/NIR by using computer controlled spectro-photometer (Shimazdu 1800) from 400 to 800 nm. Photo-luminescence (PL) spectrum of our thin film is record in visible region on a luminescence spectrophotometer LS-03 (Perkin Elmer Instruments).The surface morphology of the samples is recorded by an atomic force microscopy (AFM) by using CSPM model AA3000 AFM supply by Angstrom Company.

RESULTS AND DISCUSSION

The size and morphology of CdSe nanoparticles have been examined by transmission electron microscopy (TEM) and atomic force microscopy(AFM) . The image of the CdSe NPs. From TEM test showing in Fig. 1, display spherical monodispersed particles of 20-90 nm with an average diameter about 44 nm.

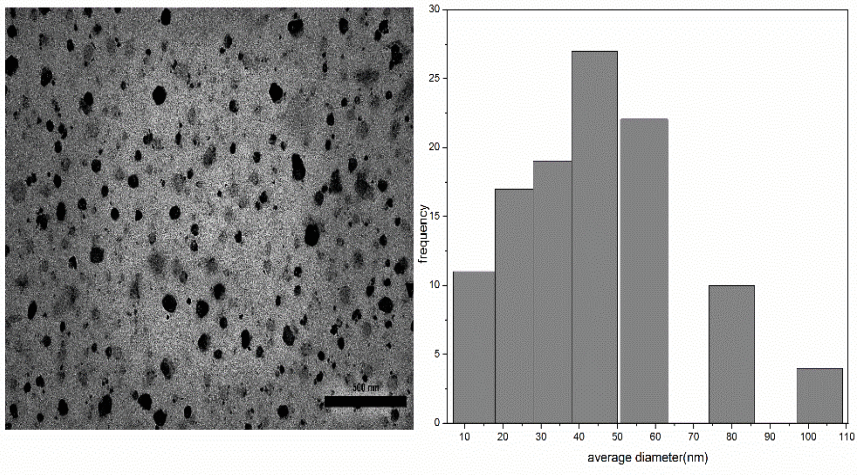
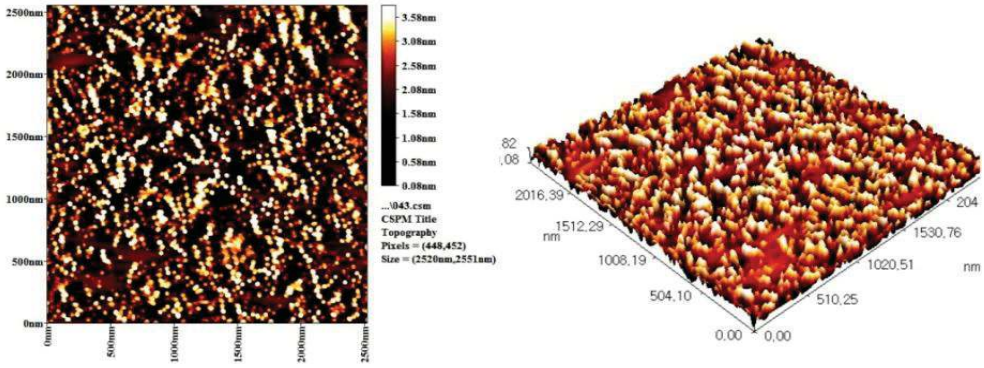
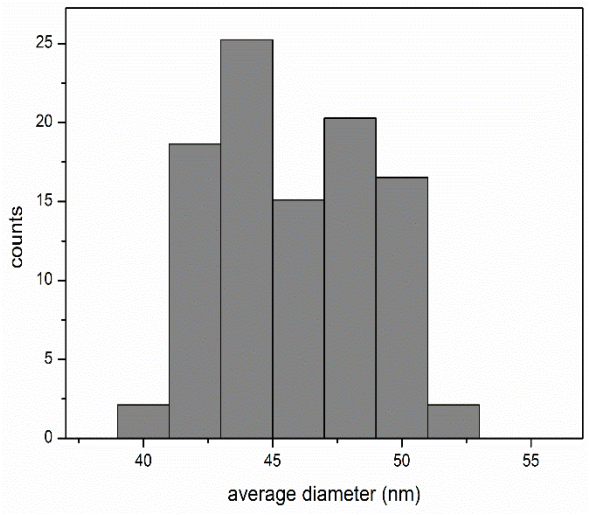


FIGURE. 1. The (a) TEM image and (b) size distribution of CdSe NPs.

the surface of CdSe NPs has been examined by AFM analysis. Fig.2 shows two and three-dimensional representation of 2500X2500 nm area of the CdSe NPs which revealed the surface roughness of thin film. The RMS value is 1.08 nm and the thin film average roughness is 0.935 nm. As can be seen from the flowchart in fig. 2, the average diameter of the sample which has been estimated from the AFM image is equal to 44 nm.



(a) (b)



(c)

FIGURE. 2. AFM (a) 2D, (b) 3D Images and (c) size distribution of CdSe thin films

XRD is a great non-destructive technique that can be applied for identification of phase, orientation determination, lattice parameter measurement, crystal quality assessment, and crystal structure. X-ray diffraction (XRD) patterns were recorded from 10o to 80o to examine the prepared NPs.

The peaks obtained in the fig.3 are matched with the standard pattern of the cubic crystal of CdSe (JCPDS 19-0191); The XRD patterns with their broadbands indicate that the CdSe films involve nano-size particles[10]. No peaks resultant from impurities were detected, indicating the high purity of the mater.

to calculate the average size of a crystal Debye-Scherrer equation has been used

$$D = \frac{0.9\lambda}{\beta\cos\theta} \quad \text{.....(1)}$$

"Where λ is the wavelength of X-ray, β is called FWHM (Full Width at Half Maxima), θ is the angle of Diffraction".

Interplanar distance spacing d of lattice plane, h,k,l was calculated by using the formula[11].

$$d = \frac{\lambda}{2\sin\theta} \quad \text{.....(2)}$$

Where, λ = 1.5406 nm and θ is Bragg diffraction angle.

The average microstrain which define as "disarrangement of the lattice was developed in the prepared NPs" and calculated by using the relation as given below[12].

$$\varepsilon = \frac{(\beta\cos\theta)}{4} \quad \text{.....(3)}$$

The particle sizes, interplanar distance spacing d and the average microstrain have been estimated for all peaks are in Table 1

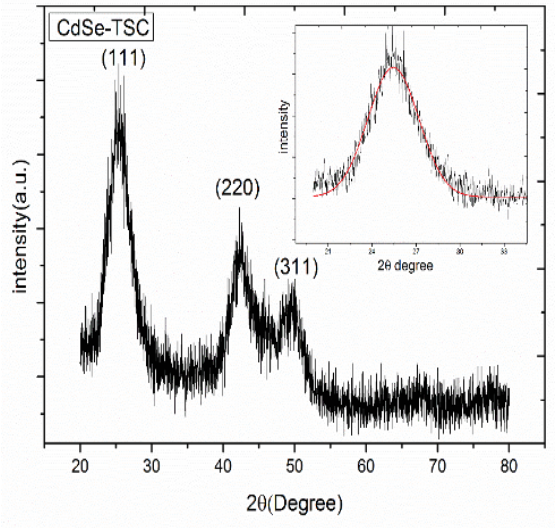
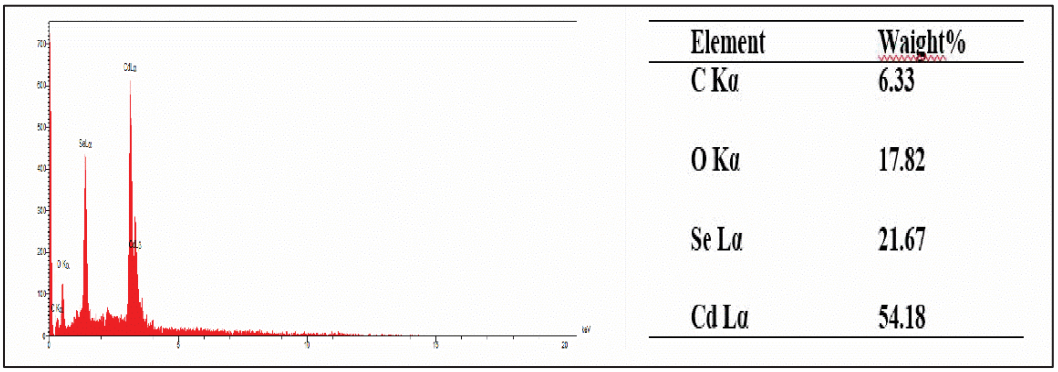


FIGURE. 3. XRD pattern of the CdSe NPs.

TABLE 1. interplanar spacing'd', (h k l), the average microstrain and grain size values for CdSe NPs for different XRD peaks

2θ(degree)	Plane(hkl)	d spacing(A^o)	FWHM(rad.)	ε lin-2m-4 $*10^{-3}$	G.size(nm)
25.46129	**111**	**3.495293**	**0.08482841**	**20**	**1.65**
42.61668	**220**	**2.119642**	**0.09510683**	**22**	**1.56**
49.27541	**311**	**1.847658**	**0.07215993**	**16**	**2.11**

The chemical compositional of the CdSe NPs has been carried out using EDX analysis. The appeared peaks in the fig. 4 are relate to Se and Cd elements. The elemental constitution of the thin film was found to have weight percentage at 21.67% of Se and 54.18% of Cd .



Element	Waight%
C Kα	6.33
O Kα	17.82
Se Lα	21.67
Cd Lα	54.18

FIGURE 4. EDX pattern of (a) CdSe Nanoparticles (b) elemental composition table.

UV-VIS absorption spectra for CdSe nanoparticles shown in Fig. 5, The range of the absorption edge lies between 650 nm to 420 nm, which is a pronounced blue shift from 712 nm of the bulk CdSe[13], indicating that particles in nanoscale[14].

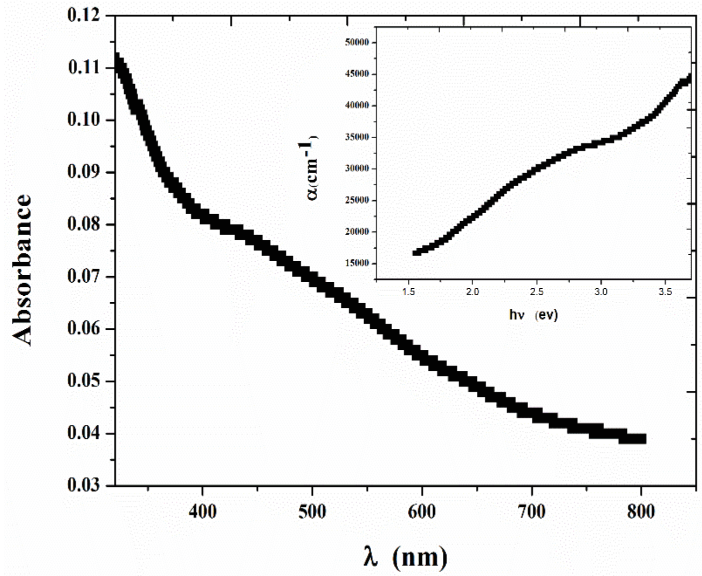


FIGURE.5. absorption spectra of CdSe nanoparticles the relationship between absorption coefficients Versus Photon energy of cdse nanoparticles

The relation used to find the absorption coefficient (α) from the absorptions (A) and thin film thickness(d) is[2]:-

$$\alpha = \frac{2.303}{d} A \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (4)$$

The relationship between the absorption coefficient and photon energy of cdse nanoparticles shown in Fig. 5 . The significant of (α) value is to determine of transition type. The (α) value shown in fig. 4 was greater than (104 cm-1) which refer to the direct transition.

The relation between α and the photon energy hv gives by the theory of optical absorption, as,

$$\alpha = \frac{A(h\nu - Eg)^m}{h\nu} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (5)$$

A is a constant and m is (1/2, 3/2) for allowed direct transitions, direct forbidden and (2, 3) for transitions indirect allowed and indirect forbidden. Eg is the optical band gap[15].

According to equation (5) and fig. 5 which indicated on the type of transition, the optical band gap determined, from plotting of (αhν)2 vs. photon energy is shown in fig. 6, it was found equal to 2.2 ev. The diameter of crystallites has been calculated by using the effective mass approximation Formula [2].

$$Eg = Eg^{bulk} + \frac{h^2}{\mu 8R^2}) \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (6)$$

Where R is the diameter of crystallites and μ is the effective mass of electron–hole pair. R was found to 2.8 nm where match with grain size that calculated from diffraction peaks of XRD .

The difference between the diameter was calculated from eq. 6 and that calculated from AFM and TEM up to aggregation and accumulation of particles together..

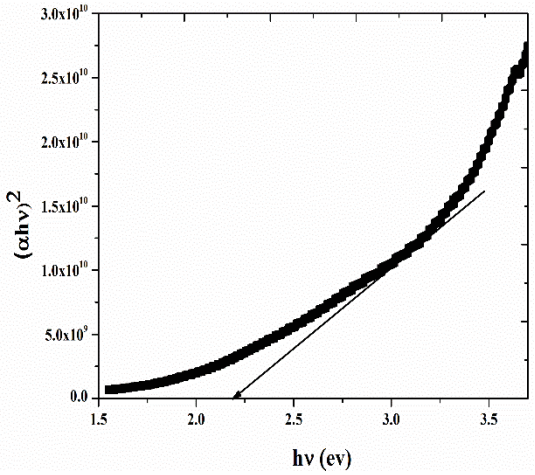


FIGURE 6. The relationship between $(\alpha h \nu)^2$ versus Photon Energy for CdSe nanoparticles

Photoluminescence (PL) is a very public emission spectroscopy characterizetion method forstudying the properties of nanomaterials since it is simple and direct[16] .Fig.7 show the Photoluminescence emission spectra of CdSe nanoparticles at the different excitation wavelength. It is clear from the figure that the emission peaks of all three excitation wavelength nearby the green, at 548 nm. From PL peak, The band gap of CdSe nanoparticle are founded equal to 2.13 eV. There is a blue shift compared with the bulk CdSe (720 nm) because of the quantum confinement effect[17].

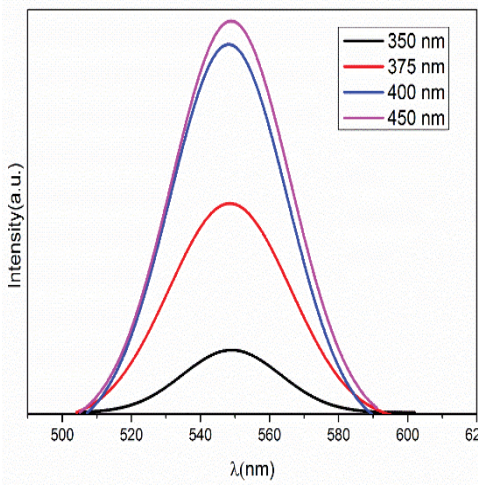


FIGURE. 7. Photoluminescence emission spectra of CdSe Nanoparticles at different excitation wavelength

Fig.8 shows the relationship between excitation wavelength and PL intensity .the PL intensity has been increased with increasing of excitation wavelength.

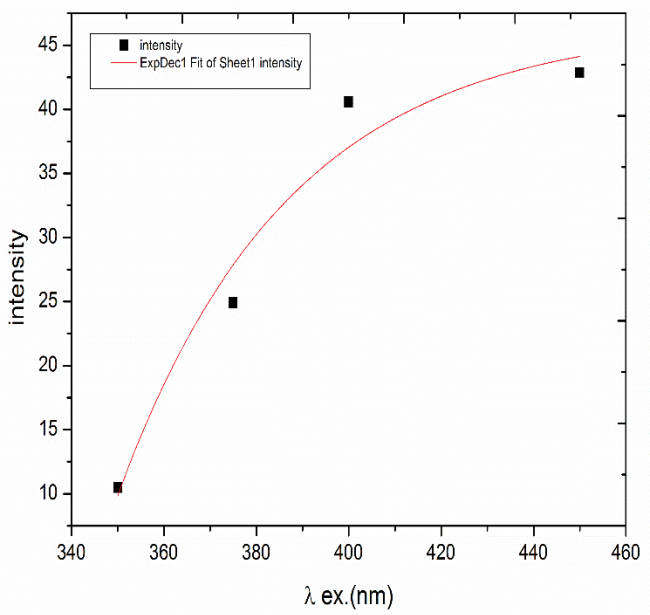


FIGURE.8. the relationship between the excitation wavelength and intensity of emission wavelength

CONCLUSIONS.

CdSe nanoparticles were synthesized by chemical method, CdSe nanoparticles examined by TEM ,AFM,XRD ,PL and UV-VIS . the NPs. have cubic structure with average diameter 44 nm was obtained from flowchart of AFM and TEM also shown an obvious blue shift both in the UV–visible absorbance and photoluminescence (PL) emission spectra. They emit strong, relatively narrow photoluminescence (at the green, 548 nm) and therefore are favorable candidates for optoelectronic applications.

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