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Synthesis and Characterisation of CDSE Nanoparticles by Chemical Precipitation Method

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ABSTRACT

CdSe nanoparticles were synthesized using a simple chemical precipitation method at room temperature in different Cd:Se ratios. The nanoparticles were characterized using x-ray diffraction(XRD),high resolution scanning electron microscopy (HRSEM),energy dispersive X-ray analysis (EDAX). XRD pattern reveals that the nanoparticles are in well crystalline Hexagonal phase.The broadening of diffraction peaks indicated the formation of particles in the nanometer size regime.HRSEM images of Cdse nanoparticles of CdSe in different Cd:Se ratios shows different morphologies. The elemental analysis from EDAX shows that the prepared samples are exactly stoichiometric.

Keywords*; CdSe nanoparticles, x-ray diffraction, nanocrystals, sem image*

1. INTRODUCTION

Research on nanomaterials increased remarkably in the past years due to their unique characteristics such as quantum confinement effect was introduced to explain a wide range of physical and chemical properties of nanostructured materials in response to changes in dimensions or shapes within nanoscales[1-5]. The reasons for this behaviour can be reduced to two fundamental phenomena .First is the high dispersity of nanocrystalline systems; ie. The number of atoms at the surface is comparable to the number of those which are located in the crystalline lattice [6]. The second phenomenon arises from quantum mechanics where it is well known that electrons and holes confined by potential barriers to a space comparable are smaller than the De Broglie wavelength of the particles ,have directly allowed energy states rather than a continuum [7-8].The size dependent emission properties particularly for CdSe nano crystals, renders it significance in modern technologies such as large screen liquid crystal display[9]light emitting diodes[10]thin film transistors [11]fluorescent probes in biologicalimaging[12] photovoltaicdevices[13] gamma ray detectors[14], lasers[15], biomedical tags[16] photoconductors[17-18]etc. There are various methods [19-20] of the preparation of CdSe nanoparticles. Some of the above mentioned methods have some drawbacks. Most of the methods need long reaction times and special instruments to synthesize CdSe nano particles because of the presence of the complexant in the reaction media. Used precursors are unstable, are an environmental hazard and require very high temperatures. [21-22]. These methods are not cost effective either. Hence a simple chemical precipitation method has been preferred.

In this paper we report the synthesis and structural characterization of CdSe nanoparticles of two different Cd:Se ion concentrations by chemical precipitation methods.

2. EXPERIMENTAL

2.1 Synthesis of CdSe nanoparticles

High quality pure CdSe nanoparticles of three different Cd:Se ion concentrations were prepared by chemical precipitation method using cadmium chloride $CdCl_2$ and sodium selenide(Na_2SeO_3) as precursors. Double distilled water was used as solvent

.The reaction was performed at room temperature (32°c) and standard pressure. In the reaction process initially 100 ml solutions of 0.07mol/l $CdCl_2$was prepared by using double distilled water. The stirring was continued for half an hour at a particular speed in the solution 0.07mol/l Na_2SeO_3(100ml) solution was ordered drop by drop with constant stirring under progressive reaction. The precipitate thus obtained as the final product of the reaction was recovered by centrifugation , washed several times by acetone to remove the impurities, unreacted reactants and dried in hot air oven at 60°c for about 5h.In order to prepare different samples the amounts of $CdCl_2$, Na_2SeO_3 were taken in the ratios of 2:1 and 4:1were named as sample1and sample2 respectively.

2.2 Characterization

The pure CdSe nanoparticle samples were characterized with structural and morphological properties. The X-ray diffraction patterns for the samples were recorded using Schimadzu Labx XRD 6000 X-ray powder diffractometer with CuKα – radiations for the 2θ values ranging from 0° to 90° with scanning rate 10 ° per minute. FE1 quanta FEG 200 high resolution scanning electron microscope was used to study the morphology and compositional analysis of different CdSe samples.

3. RESULTS AND DISCUSSION

3.1 X-ray Diffraction studies

Fig.1 shows the diffraction patterns of CdSe nanoparticles were prepared with different Cd : Se ratios. It is observed that the peaks observed at 2θ =23.6,27.24,35.2,42.12,48.81 and 50.71indexed as (100),(101),(102),(110),(200) and (112)planes corresponding to hexagonal phase (JCPDS No.77-2307).The lattice parameters and cell volumes of cdse nanoparticles having different molar ratios are compared with standard values and presented in table1.

It is observed that the diffraction patterns of nanoparticles are broadened as the concentration of cadmium is increased. The broad peaks were due to the small size effect. No other crystalline impurities were detected within the detection limit indicating that as synthesized product was of high purity. The average crystallite size of the sample have been calculated using the Debye scherrer formula, where d is the mean crystallite size in nanometer.λ is the wave length of the X-ray used(1.5406Å),β is the FWHM of the diffraction peak in radians and θ is the diffraction angle. It is observed that crystallite size is 2.17nm and 2.34nm for sample 1 and sample 2 respectively.

Fig.1 X ray diffraction patterns of pure CdSe nanoparticles(a) sample 1and (b) sample 2

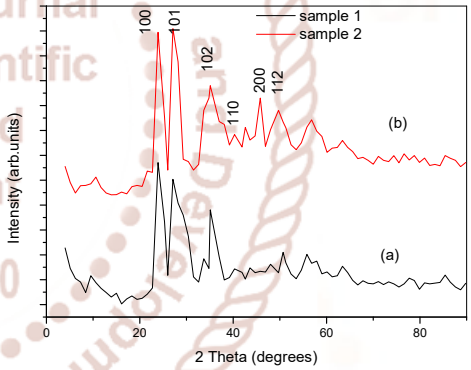


Table 1: Comparison of lattice parameters and cell volume for CdSe nanoparticles of different Cd:Se ion ratio with standard CdSe

Standard Parameters	Sample 1	Sample 2	CdSe (Standard) (Hexagonal)
Lattice parameter (a) Å	4.3480	4.4241	4.299
Lattice parameter (c) Å	6.5731	6.8829	7.01
Lattice volume (V) Å	107.6165	116.670	112.20

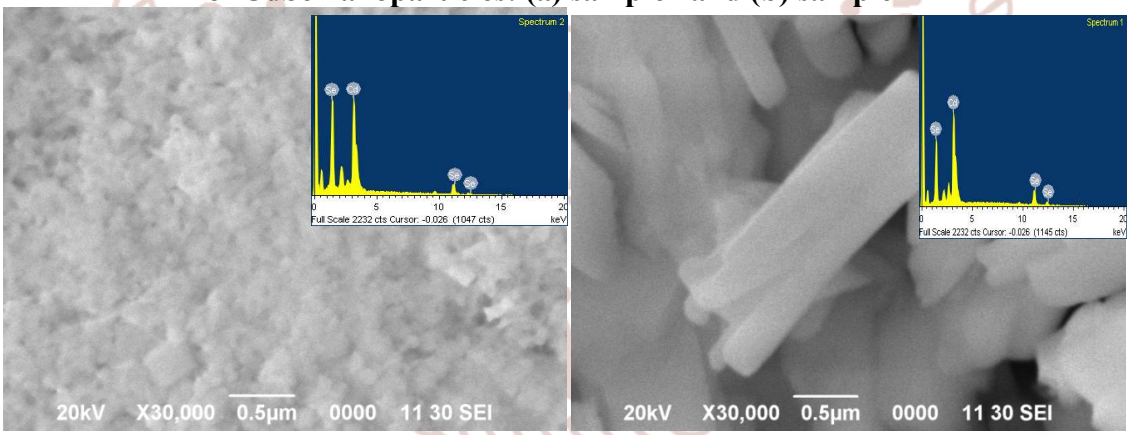
Table 2. . Comparison of 2θ values for CdSe nanoparticles of different Cd:Se ion ratio with standard CdSe

CdSe Standard (Hexagonal)				Sample 1	Sample 2
h	k	ℓ	2θ	2θ	2θ
1	0	0	23.882	23.98	24.234
1	0	1	27.097	27.133	27.133
1	0	2	35.136	34.864	35.096
1	1	0	41.999	40.178	40.178
1	0	3	48.888	43.811	45.744
1	1	2	49.718	50.807	49.358

3.2 Morphological studies

The surface morphology of CdSe nanocrystals with three different concentrations were studied by SEM and is shown in fig.3(a andb). The morphology shows the structure in (a)spherical structure and in(b) rod like structure . It can be seen that the monodispersity and homogeneity of samples. An overview of images shows the product that consists of mono disperse structure of rod shape.

Fig.3. HR-SEM image and corresponding EDAX pattern of CdSe nanoparticles: (a) sample 1and (b) sample 2



The Energy Dispersive Xray Analysis(EDAX) spectrum confirms that the sample only contains Cd and Se with an average atomic percentage ratio of exactly 50:50.The compositional value of the elements in weight percentage of CdSe of different concentrations are presented in Table 3.

Table 3. Compositional values of elements present in CdSe nanoparticles of different Cd:Se ion ratio (a) sample1and (b) sample 2

	Element	weight%	atomic%
(a)			
	Se L	34.96	43.34
	Cd L	65.04	56.66
(b)			
	Se L	27.04	35.44
	Cd L	72.96	64.56

CONCLUSION

The reported work presents a chemical precipitation method for the synthesis of pure CdSe nanoparticles at different Cd:Se ion ratios at room temperature. X-ray diffraction analysis reveals that CdSe nanoparticles at different Cd:Se ion ratios, exhibit hexagonal structure without any impurity phase and the average grain size is found to be in the range of nm. HRSEM images also reveals the spherical and rod shaped CdSe nanoparticles with relatively with uniform surface and the EDAX spectrum confirms the presence of Cd,Se ions.

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