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Advances in Applied Science Research, 2014, 5(5):99-105

ISSN: 0976-8610
CODEN (USA): AASRFC

Structural properties of CdS nano particles prepared in the presence of organic solvent

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ABSTRACT

CdS being a wide band gap II-VI semiconducting material with better lattice matching properties is made suitable option for solar cell applications. In the present work, CdS nanoparticles were prepared by chemical precipitation technique in the presence of organic solvent. Cadmium sulphate [$3CdSO_4 \cdot 8H_2O$] and thiourea [$(NH_2)_2CS$] were used as precursor materials and N, N- Dimethylformamide (DMF) was used as an organic solvent. NaOH solution is used for maintaining pH 8 and 12. The obtained CdS nano particles were characterized by X-Ray Diffraction (XRD), Particle Size Analyser (PSA), Scanning Electron Microscope (SEM), Energy Dispersive X-ray spectrometry (EDX), Transmission Electron Microscope (TEM) and Fourier Transform Infrared spectroscopy (FTIR) for average crystallite size, average particle size, morphology, elemental composition, shape and bond analysis respectively. After analysing the results, the obtained nanoparticles were in the nano range and it has been used in various applications.

Keywords: CdS nanoparticles, Chemical Precipitation Technique, XRD, TEM, FTIR

INTRODUCTION

Cadmium sulphide is an important II–VI semiconductor material with Energy band gap of 2.42 eV (515 nm) at room temperature [1]. Semiconductor nanoparticles have interesting physical and chemical properties such as wide band gap, non-linear optical effects and improved photoluminescence effect [2]. These factors can change and develop the properties such as reactivity, strength, electrical, optical and magnetic characteristics. There are three reasons for the change of electronic states in nano sized particles: one reason is the quantum confinement in semiconductor particles, second is surface plasmon resonance in some metal particles and the thirdis super para magnetism in magnetic materials [3]. Based on these properties CdS nanoparticles were used in various applications such as solar cells, photochemical catalysis, gas sensor, detectors for laser, infrared photo detectors, light - emitting diodes and biological labels [4-5]. Preparation of CdS nanoparticles can be done by soft chemical reaction, solid-state reaction, sol-gel process, sonochemical preparation, microwave heating, photo etching and reverse micelle [6]. Most of the researchers prepared CdS nanoparticlesin the presence of different surfactant materials and capping agents [7]. But in the present work, CdS nanoparticles were prepared in the presence of organic solvent such as N, N – DMF at different pH values 8 and 12.

MATERIALS AND METHODS

Experimental Details-

CdS nanoparticles were synthesized by a chemical precipitation technique with cadmium sulphate and thiourea as starting materials in the presence of organic solvent N, N – DMF. CdS nanoparticles were obtained by maintaining two pH values such as 8 and 12. Cadmium sulphate [$3CdSO_4 \cdot 8H_2O$] andThiourea [$(NH_2)_2CS$] were taken as reactant materials and 5 ml of N, N-DMF organic solvent is added to the above solution. At pH 12 the solution has changed its colour to dark yellow which was transparent light yellow at pH 8. The above precipitate was washed several times with ethanol, water and further dried at 80ºC for 60 minutes which lead to the formation of CdS nano particles[8].

Characterization Techniques-
The obtained CdS nanoparticles were characterized by XRD for average crystallite size, Lattice Parameters and Strain using Bruker D8 advanced X-ray diffractometer having Cu$K\alpha$ radiation. HORIBA SZ-100 Particle Size Analyser for average particle size. HITACHI S 3400N SEM used for Morphology, Particle size and Elemental compositions. From TEM analysis Size and Shape was measured by JEM-100CXII. PERKIN FTIR was used for Bond analysis of the chemical reaction.

RESULTS AND DISCUSSION

X-Ray Diffraction-
Figure 1 shows the XRD pattern of the CdS nanoparticles at pH 8 and 12. The structure of the CdS nanoparticles is in hexagonal phase.

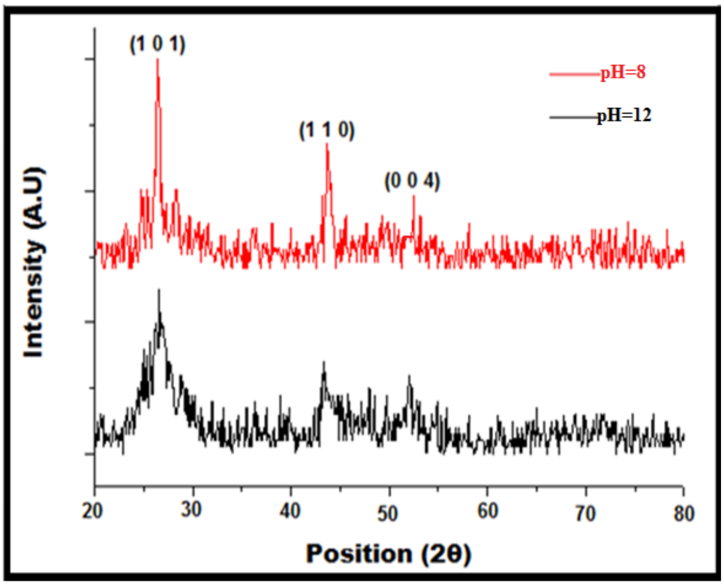

Fig. 1.XRD Pattern of CdS nanoparticles at pH 8 and 12

The extended peaks are representing the dimensions of the nano range particles. Peaks were observed at 28°, 43° and 54° with the corresponding (h k l) values (1 0 1), (1 1 0) and (0 0 4). The lattice parameters were in good agreement with JCPDS card number 41 – 1049with a =b = 4.140 A°, c=6.719A°.

The average crystallite size was calculated by Debye – Scherrer formula,

$$D = \frac{K\lambda}{\beta . Cos\theta} \quad (1)$$

Where D – is the average crystallite size, λ – is the wavelength of the radiation, β – is the full width half maximum (FWHM) of the peak, K – is the Debye – Scherrer's constant and θ – is the Bragg's angle.

The measured average crystallite size of CdS nanoparticles were 32 nm and 18nm for pH 12 and 8 respectively.

The micro strain and crystallite size of the particles were measured from the Williamson – Hall equation.

$$\beta \cos\theta = \frac{K\lambda}{t} + 2\varepsilon \sin\theta \quad (2)$$

Where β – is the full width half maximum (FWHM) of the XRD corresponding peaks, *K* – is Debye-Scherer's constant, *t* – is the crystallite size, λ – is the wave length of the X-ray radiation, ε – is the lattice strain and θ – is the

Bragg's angle. In this process 2sinθis plotted against βcosθ, using a linear extrapolation to this plot where the intercept gives the crystallite size and slope gives the strain (ε).

The crystallite sizes were measured as 25 nm and 17 nm; the obtained strainswere 5.86 x 10^{-3} and 7.025 x 10^{-3} for pH 12 and 8 respectively.

The lattice parameters of the hexagonal phase were measured by the following equation.

$$\frac{1}{d^2} = \frac{4(h^2 + hk + k^2)}{3a^2} + \frac{l}{c^2} \qquad (3)$$

The measured lattice parameters (*a = b= 0.40819 nm and c = 0.67196 nm)*which are similar to the valuesof XRD patterns [9, 10].

Particle Size Analyser-
Asprepared CdS nanoparticles were ultra-sonicated and suspended in the ethanol solution. The sizes of the agglomerated colloids in the suspensions were estimated using particle size analyser and the histograms are shown in figure. 2 for both pH values.

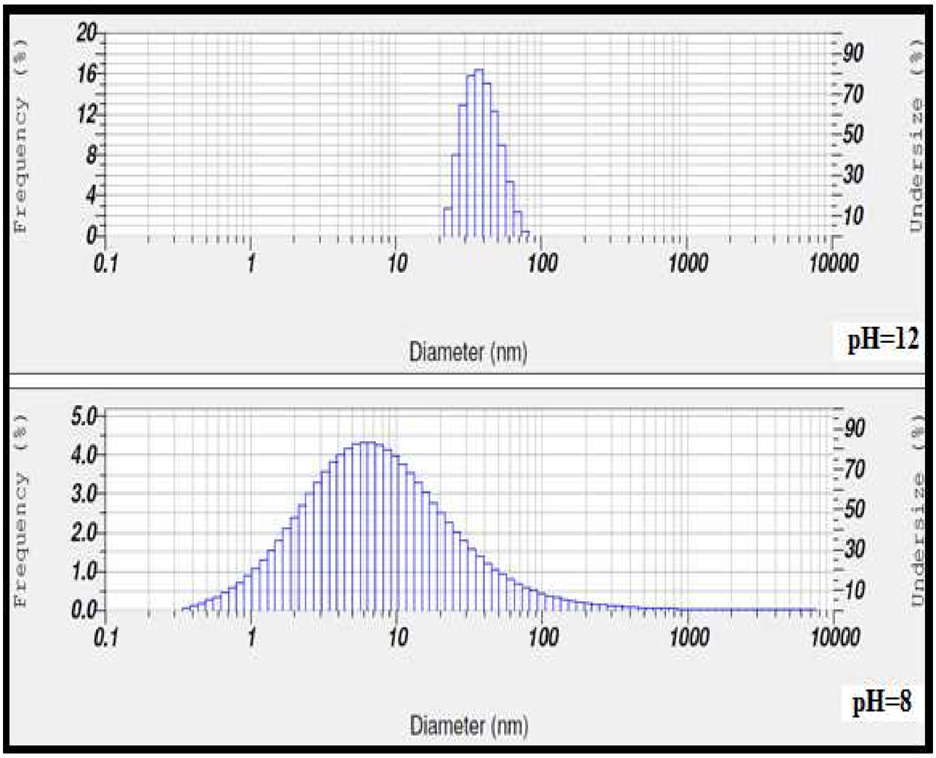

Fig. 2.Particle distribution for CdS Nanoparticles at pH 8 and 12

The CdS nanoparticles which were prepared at pH 8 shows uniform distribution in the dynamic light scattering compared with pH 12. The average particle sizes were 21 nm and 39 nm for pH 8 and 12 respectively. It is correlating with the average crystallite size of XRD pattern (i.e. 18 nm and 32 nm).

Scanning Electron Microscope-
The grain size, shape and surface morphology were observed by Scanning Electron Microscope. SEM images of CdS nanoparticles with different magnifications at both pH values as shown in figure 3a, 3b, 4a & 4b.

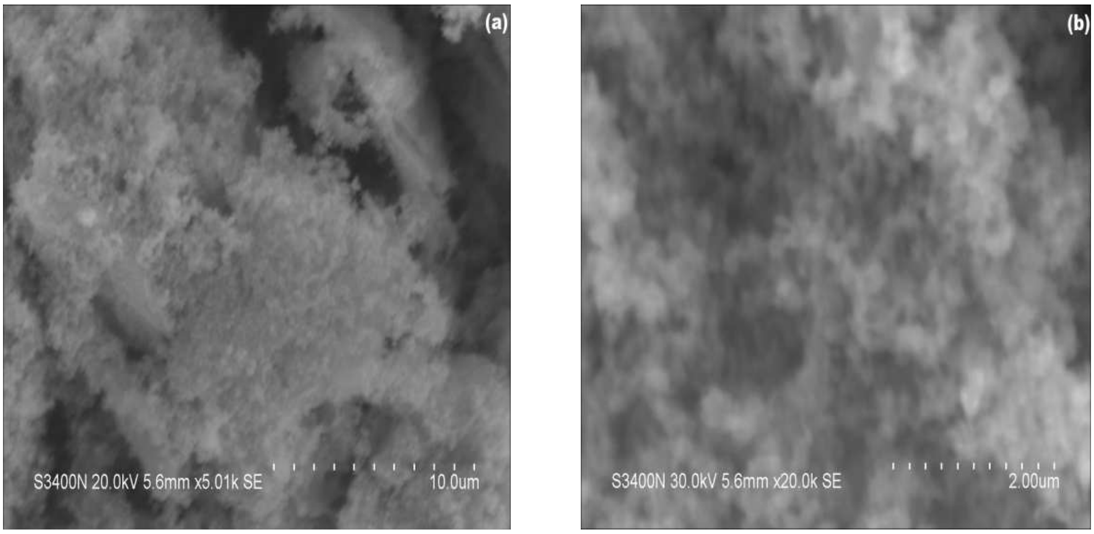

Fig. 3a.& 3b.SEM Images of CdS nanoparticles at pH 12

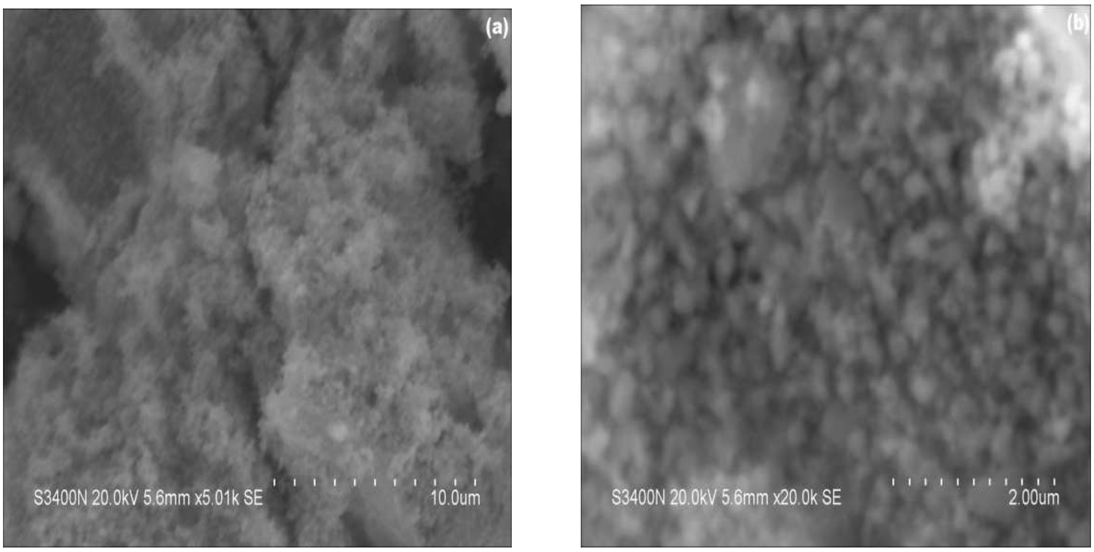

Fig. 4a.&4b.SEM Images of CdS nanoparticles at pH 8

They show that the particles exhibit spherical granular structure and the size of the particles are in the range of 100 nm to 150 nm and 80 nm to 100 nm for pH 12 and 8 respectively [11].

Energy Dispersive X-ray Spectroscope-
The elemental percentages are obtained from EDX pattern. Figure 5 shows the EDX pattern of CdS nano particles at pH 12 and 8 showing varying weight percentages of elements.

Elements	Weight percentages at pH=12	Weight percentages at pH=8
Cadmium	71.24 %	61.82 %
Sulphur	28.76 %	38.18 %
Total	100.00 %	100.00 %

Table 1.Elemental compositions of CdS nanoparticles

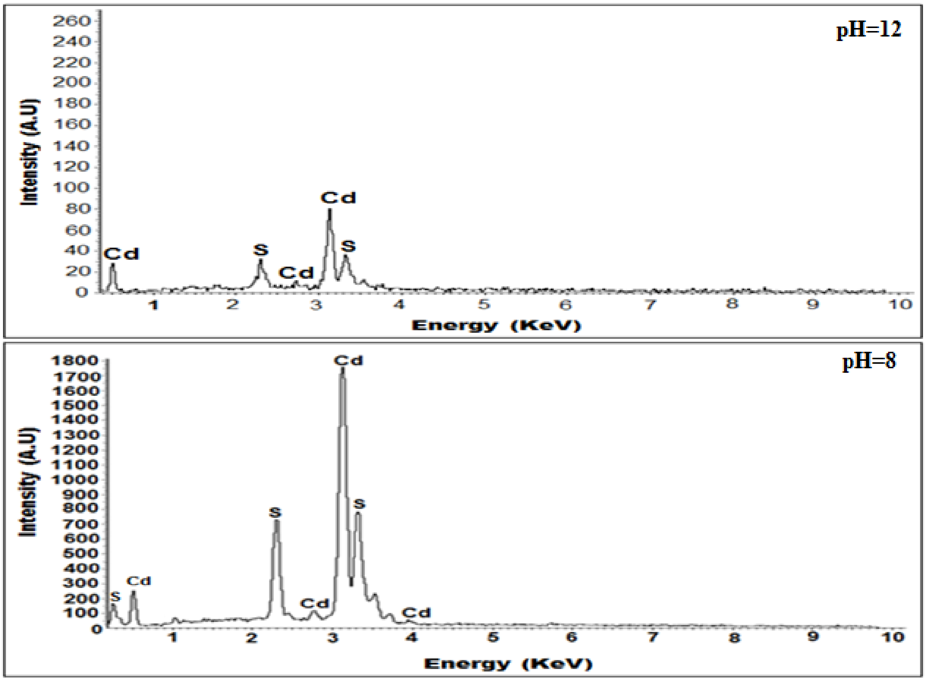

Fig. 5.EDX Pattern of CdS nanoparticles at pH 8 and 12

This table infers that the weight percentages of CdS nanoparticles prepared by maintaining pH 8 contains appropriate cadmium and sulphur weight percentages in the reaction compared with weight percentages of CdS nanoparticles prepared by maintaining pH 12 because the solution has become less acidic and more alkaline [12].

Transmission Electron Microscope-
The morphology and structure arrangement were observed by TEM. TEM images of CdS nanoparticles with different magnifications at both pH values are shown in figure 6a, 6b, 7a & 7b.

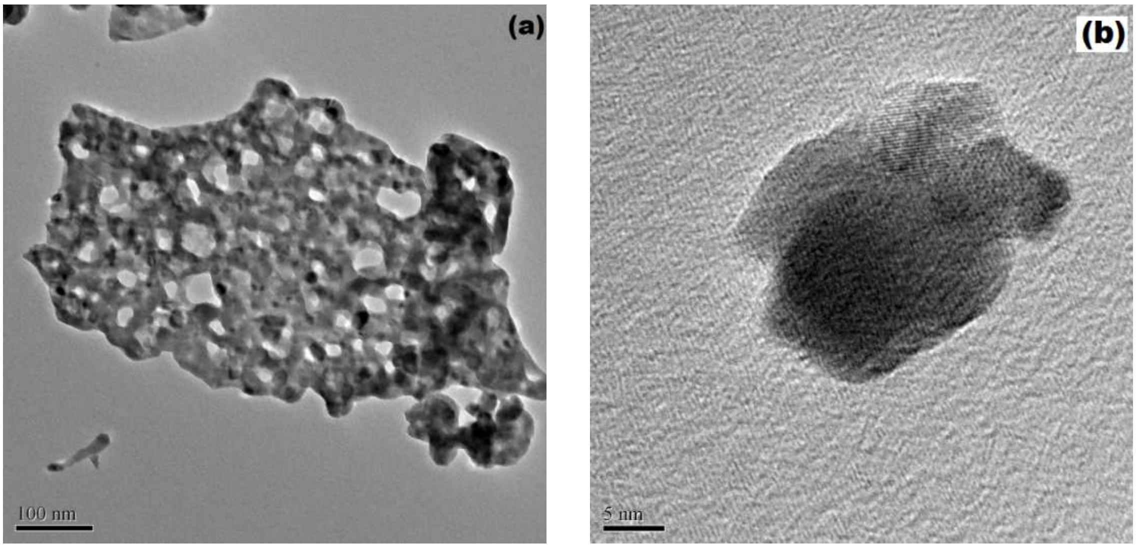

Fig. 6a & 6b.TEM Images of the CdS nanoparticles at pH 12

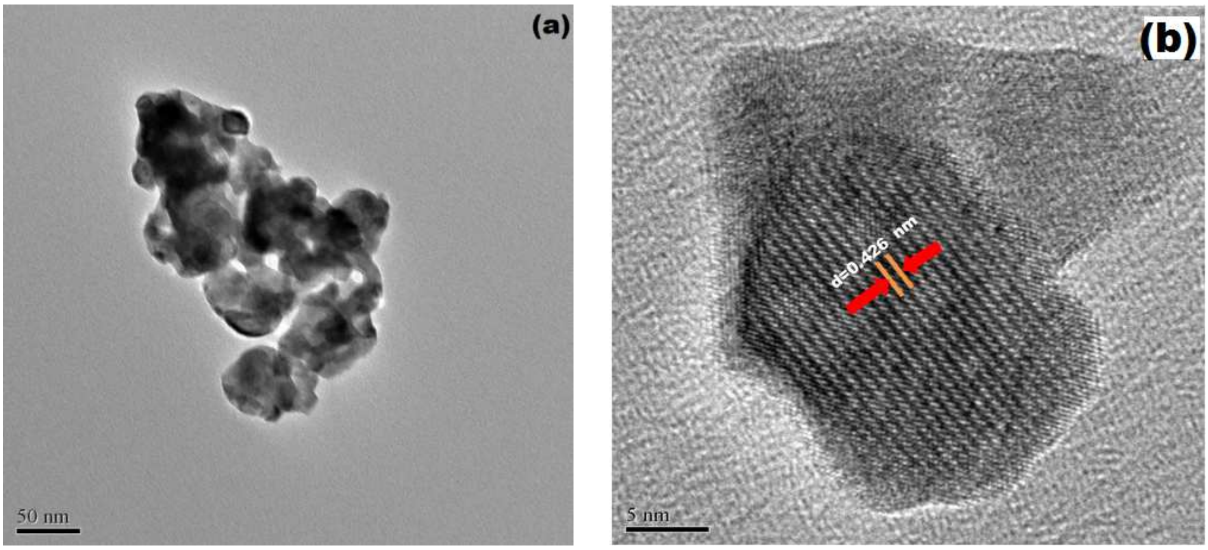

Fig. 7a & 7b.TEM Images of the CdS nanoparticles at pH 8

The above images show that the fine CdS nanoparticleswerefound to have plane crystalline nature. The shape of the CdS nanoparticles was spherical shape.The SAED patterns of CdS nanoparticles explainthat the diffraction rings of CdS nanoparticles match with the hexagonal phase. The calculated d-spacing value was d=0.426 nm, the valueobtained is nearly equal to XRD d-spacing value [13].

Fourier Transform Infrared Spectroscopy-

FTIR analysis was made from 450 cm^{-1} to 4000 cm^{-1}, were in the samples have been prepared with KBr medium. FTIRspectrum of CdS nanoparticles are as shown in figure 8.

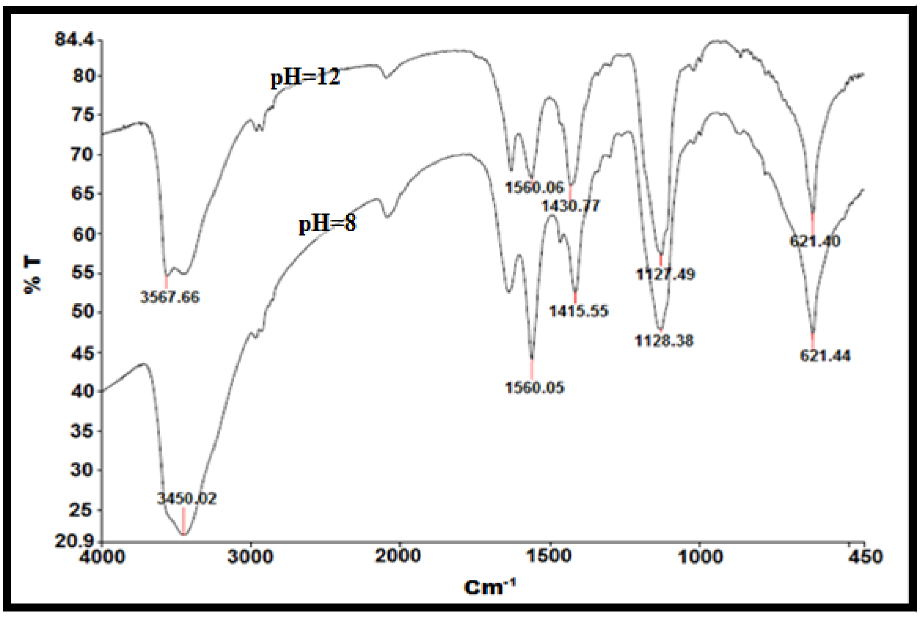

Fig. 8.FTIR Spectrum of CdS nanoparticles

From FTIR, very weak absorption bond is observed at 3567.66 cm^{-1} is due to O-H stretching vibration of water molecules, due to presence of moisture in the sample. Very weak bending vibrations of water molecules are appeared at 1560.06 cm^{-1}. C-C Stretching, medium strong band positions are appeared in the range of 1430 cm^{-1} to 1560 cm^{-1}and are possibly due to stretching vibrations of Sulphate group, traces of SO_4^{-2} ion. The narrow absorption peak centred at around 1120 cm^{-1} occurred in the above figure is ascribed to the C=O bonding. There are medium to strong absorption bands at 620 cm^{-1}, possibly due to Cd-S stretching. Hence the existences of above mentioned bands identify the presence [14].

CONCLUSION

The CdS nanoparticles had been successfully synthesized by the Chemical Precipitation Technique at pH 8 and 12 in the presence of organic solvent N, N-DMF. The XRD results indicate that the CdS nanoparticle shows hexagonal structure. The average crystallite sizes were measured from Debye-Scherrer's formula and lattice strain were measured by Williamson-Hall equation. By decreasing the pH value, the crystallite size was decreased and the micro strain was increased because the solution has become less acidic and more alkaline.The average particle sizes were decreased with decreasing the pH value. SEM images of the particles range from 100 – 150 nm and 80 – 100 nm for pH 12 and 8 respectively. EDX pattern gave the Cd and S elements presence in the final sample. The d-spacing value from TEM images was nearly equal to XRD values and it shows hexagonal structure. The FTIR analysis confirms the Cd – S stretching at 620 cm^{-1} which was involved in the chemical reaction. These CdS nano particles can be used in vast applications: optoelectronics, sensors and drug delivery systems.

Acknowledgements

My special thanks to Head of the Department, Centre for Nano Science and Technology, Institute of Science and Technology, JNTU Hyderabad, Kukatpally, Telangana for providing DST – Nano Mission funding.

REFERENCES

[1] Dumbrava A, Badea C, Prodan G, Ciupina V,*ChalcogenideLett*, **2010**, 7, 111.
[2] Puja Chawla, Geeta Sharma, Lochab S P, Nafa Singh, *Bull Mater Sci*,**2010**, 33, 535.
[3] Elilarassi R,Maheshwari S, Chandrasekaran G, *Optoelectronics and Adv Mater–Rapid Communications*,**2010**, 4, 309.
[4] Maleki M, SasaniGhamsari M, MirdamadiSh, Ghasemzadeh R, *Semiconductor Phy Quantum Electronics & Optoelectronics*,**2007**, 10, 30.
[5] Martinez-Castanon G A, Sanchez-Loredo M G, Martinez-Mendoza J R, Facundo Ruiz, *Adv in Tech of Mater and Mater Processing*, **2005**, 7, 171.
[6] ManjunathaPattabi, JayasheelaUchil, *Solar Energy Mater & Solar Cells*,**2000**, 63, 309.
[7] Pawar M J, Caure S S, *ChalcogenideLett*,**2009**, 6, 689.
[8] Vineet Singh, Pratima Chauhan, *ChalcogenideLett*,**2009**, 6, 421.
[9] Sedaghat Z, Taghaviniaand N, Marandi M, *Nanotechnol*,**2006**, 17, 3812.
[10] Nandakumar P, Vijayan C, Dhanalakshmi K, Sundararajan G, Kesavan Nair P, Murti Y V G S, *Mater Sci and Eng B*, **2001**, 83, 61.
[11] Aneeqa Sabah, Saadat Anwar Siddiqi, Salamat Ali, *World Academy of SciEng and Technol*,**2010**, 69, 82.
[12] Reyes P, Velumani S,*Mater Sci and Eng B*,**2012**, 177, 1452.
[13] Srinivasa Rao B, Rajesh Kumar B, Rajagopal Reddy V, Subba Rao T, *ChalcogenideLett*, **2011**, 8, 177.
[14] Saravanan L, Diwakar S, Mohankumar R, Pandurangan A, Jayavel R, *NanomaterNanotechnol*,**2011**, 1, 42.