

Fabrication and Characterization of CdS Nanoparticles Annealed by using Different Radiations

Aneeqa Sabah, Saadat Anwar Siddiqi, Salamat Ali

Abstract—The systematic manipulations of shapes and sizes of inorganic compounds greatly benefit the various application fields including optics, magnetic, electronics, catalysis and medicine. However shape control has been much more difficult to achieve. Hence exploration of novel method for the preparation of differently shaped nanoparticles is challenging research area. II-VI group of semiconductor cadmium sulphide (CdS) nanostructure with different morphologies (such as, acicular like, mesoporous, spherical shapes) and of crystallite sizes vary from 11 to 16 nm were successfully synthesized by chemical aqueous precipitation of Cd^{2+} ions with homogeneously released S^{2-} ions from decomposition of cadmium sulphate (CdSO_4) and thioacetamide (CH_3CSNH_2) by annealing at different radiations (microwave, ultrasonic and sunlight) with matter and systematic research has been done for various factors affecting the controlled growth rate of CdS nanoparticles. The obtained nanomaterials have been characterized by X-ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Thermogravimetric (DSC-TGA) analysis and Scanning Electron Microscopy (SEM). The result indicates that on increasing the reaction time particle size increases but on increasing the molar ratios grain size decreases.

Keywords—CdS nanoparticles, Morphology, Oxidation, Radiations

I. INTRODUCTION

RECENT developments in nanotechnology and demonstration of various quantum size effects in nanoscale particles, implies that many novel devices of the future will be based on properties of nanomaterials [1].

There is no strict dividing line between nanoparticles and non-nanoparticles. The size at which materials display different properties to the bulk material are material dependant and can certainly be claimed for many materials much larger in size than 100 nm.

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Two principle factors, *Increased relative surface area* and *Quantum effect* causes the properties of nanomaterials to differ significantly from other materials.

These factors can change and enhance the properties such as reactivity, strength, electrical, optical and magnetic characteristics. Size-dependent properties are observed such as quantum confinement in semiconductor particles, surface Plasmon resonance in some metal particles and super Para magnetism in magnetic materials.

Although a broad definition, have materials that have one dimension in nanoscale (and are extending in other two dimensions), the structure known as quantum well. Materials that are nanoscale in two dimensions (and extended in one dimension), said to be quantum wire, The extreme case of size reduction of semiconductors, in which all three dimensions reach to the low nanometer range, is called quantum dots. This has led to allow tighter control of factors like particle growth and size, solubility and emission properties. Recently, quantum dots have found applications in composites, solar cells and fluorescent biological labels (for example to trace a biological molecule). Recently monolayer-protected, high-quality, monodispersed, crystalline quantum dots as small as 2 nm in diameter has been prepared which can be conveniently treated as a typical chemical reagent.

Quantum dots of II-VI semiconductors have attracted particular attention, because they are relatively easy to synthesize in size range required for quantum confinement. CdS is a wide band gap semiconductor with $E_g = 2.5\text{eV}$ at 300K. It is slightly soluble in water; highly soluble in acid.

Bulk CdS has a hexagonal wurtzite type (W) crystal structure with $a=0.4160\text{ nm}$ and $c=0.6756\text{ nm}$. Two other structures are observed only in nanocrystalline CdS, a) cubic zinc belende (Z) phase under ambient conditions, and b) a high pressure rock salt phase.

The wurtzite to rock salt transformation involves not only change in symmetry (hexagonal to cubic) but also a change in the nearest neighbor atomic co-ordination from four to six, but wurtzite to zinc belende transformation involves only a change in symmetry.

In recent years, nanocrystalline CdS attracted much attention because of considerable change at nanoscale, when crystallite radius becomes comparable to or less than the Bohr exciton radius, there is a considerable enhancement of the

exciton

binding energy. Nanoparticles of CdS are by far the most studied system among the entire semi conducting nanoparticles due to their extensive use such as field effect transistors, light emitting diodes, photo catalysis and biological sensors.

The conductivity of CdS increases when irradiated with light (leading to uses as photo resistors).

CdS frequently exhibits the phenomena of cathodoluminescence and phosphorescence. Solid CdS can act as a solid state laser. Synthetic cadmium pigments are valued for their good thermal stability, light and weather fastness, chemical resistance and high opacity. Biologically Cadmium sulfide is produced by sulfate reducing bacteria is being investigated as a means of producing nano- crystalline CdS. Cadmium sulphide semiconductor used in solar cells, photodetectors, laser, light emitting diodes etc. CdS nanoparticles are attractive candidates for optoelectronic applications. However CdS is unstable material and can easily change into other materials on heating in air.

The behavior of metal sulphides ZnS, CdS, GaS, Ti_2S , Sb_2S_3 and Sb_2S_5 during thermal treatment in an oxidizing agent were studied by means of DTA, TG and DTG curves. Obtained results show that during oxidation, the metal sulphides follow either the sulphate or oxide theory depending on method of production and crystal lattice theory [2].

CdS films are impressive for terrestrial photovoltaic applications. Using photo acoustic technique, the thermal properties of CdS thin films grown by spray pyrolysis were measured [3]. Hybrid photovoltaic (PV) cells based on cadmium sulphide (CdS) single crystal and phthalocyanine (Pc) films have been developed and their PV performance was measured [4].

In 20th century, few reports have been available in the literature on cubic to hexagonal phase transformation in CdS [5]-[7]. CdS generally shows dimorphism of (i) zinc blende type (cubic form) and wurtzite type (hexagonal form) at relatively low temperatures, and (ii) only wurtzite type at relatively high temperatures. The complete transformation has been observed from cubic to hexagonal phase around 600 °C.

Cadmium uptake by iron (oxy) hydroxides is critical to its environmental fate and transport. The sorption of Cd^{2+} in the presence of the anions phosphate (PO_4^{3-}), sulphate (SO_4^{2-}), Oxalate ($\text{OOC}=\text{COO}^{2-}$), citrate ($\text{HOC}(\text{CH}_2\text{COO})_2\text{COO}^{3-}$), and humate were examined in 1999. Electrostatic enhancement of the sorption of Cd^{2+} by iron oxides is important in soils, particularly those which are treated with inorganic fertilizers. However, this enhancement is likely to be reversible and ionic strength-dependent [8].

Microwaves, applications in synthesis of nanoparticles are a large and dynamically growing research field. Basically a search for methods that permit production of nanoparticles with well controlled and tailored properties like degree of crystallinity and/or defect free crystalline lattice, doped or alloyed with optically or magnetically active ions, crystals size and crystal size distribution, shape, capping with functional

layers, etc.

The combination of Microwave-Hydrothermal techniques has been used in the last few years for synthesis of nanocrystalline powders. A Novel Microwave-Solvothermal technique for synthesizing nanocrystalline CdS particles to see the effect of different parameters such as time, temperature, solvents and molar ratio of Cd^{2+} to thiourea on the phase(s) formation have been investigated by using microwaves of 2.45 GHz frequency [9]. In 2003 PbS microcrystal with the flower shaped structure have been successfully synthesized in a simple aqueous system by using microwave irradiations, employing $(\text{CH}_3\text{COO})_2\text{Pb}$ and $\text{Na}_2\text{S}_2\text{O}_3$ as the starting materials. Morphology and sizes of crystals has been controlled by changing molar ratio of $\text{Pb}^{2+}/\text{S}_2\text{O}_3^{2-}$ and microwave irradiation power [10].

Again in 2006, microwave assisted synthesis of CdS nanoparticles in a water-in-oil micro emulsion has proven itself as a very promising method for the production of a material displaying properties different from those of the same material produced by conventional thermostatic conditions [11].

The interrelation between particle size, crystal structure and optical properties in semiconductor quantum dots has attracted particular attention in the year of 2000. Hence an attempt at relating the size induced transformation from hexagonal to cubic structure in CdS nanoparticles to a change in band gap have reported by R. Banerjee, R. Jayakrishnan and P. Ayyub [12].

Oxidation of metal sulphides is complicated exothermal heterogeneous process, which is affected by number of complicated processes involving reaction kinetics, crystal chemistry, adsorption, mass exchange, heat exchange and others.

Hence systematic investigations have been carried out on the oxidation of CdS in 2002 by using DTA, and TGA, as well as chemical, morphological, Phase X-ray analysis and SEM. The stability of phase changes have been discussed at increase temperature ranges. The result of roasted sulphide concentrated in the oxidation of CdS, a very porous and mechanically weak CdO crust [13].

Interaction of radiation with matter is always found to be an attractive method for synthesis of nanoparticles. An efficient method was introduced for synthesis of technologically important mesoporous CdS semiconductor nanoparticles by gamma ray irradiation in oil-in-water micro emulsion [14].

The interrelation between particle size, crystal structure and optical properties in semiconductor nanocrystalline thin films has wide spread interest. An attempt to relating the size and structure of cadmium sulphide (CdS) nanocrystalline thin film with variation of pH of the solution have been carried out. CdS particle sizes between 3.5 to 5.3 nm were obtained by varying the pH from 1.6 to 2.2 and using polyvinyl alcohol as a capping agent in a chemical route. [15].

In brief, nanotechnology is one of the most active research areas with both novel science and useful applications that has

gradually established it in the last decades. Expenditure on nanotechnology research is significant; however, the research is continuously moving forward motivated by immediate profitable return generated by high value commercial products. The Architecture, Engineering, and Construction (A/E/C) industry might accommodate broad applications of nanotechnology and nanomaterials.

Nanomaterials often require very different production approaches, classified as 'top-down' and 'bottom-up'. Using the bottom-up method, (molecular self assembly by chemical and physical triggering) involves to building of larger and more complex systems molecule-by-molecule.

Different methods are used in order to optimize specific properties of the nanomaterials. These properties include, size distribution, symmetry, surface properties, surface coating, purity, ease of manipulation, yield and suitability for scaling up.

It is still a challenge for chemists and material scientists to find some convenient, economical, less energy consuming and environmentally friendly synthesized routes to these semiconductor nanocrystals.

The wet chemistry techniques currently offer the best quality nanoparticles from a number of points of view. They produce nanoparticles that are already in the form of dispersion, hence high inter-particle forces can be designed in to prevent agglomeration. The formation of aggregates can be reduced or eliminated. (By capping through organic element)

The chemical composition and morphology can be closely controlled.

In wet chemistry precipitation processes the solutions of the different ions are mixed under controlled conditions of temperature and pressure to form insoluble precipitates. This approach enables the relatively straightforward production of significant quantities of nanoparticles material at modest capital cost. This method is simple, inexpensive and provides high quality materials to ensure meaningful results.

Sonochemistry and Microwave techniques are very useful synthetic method that has been in use for some time now for synthesis of well controlled shapes and size of nanoparticles could be critical for their applications.

In Sonochemistry methods, acoustic cavitations are used to control the process (Gedanken, 2004). The high local temperatures and pressures, combined with extraordinarily rapid cooling, provide a unique means for driving chemical reactions under extreme conditions.

Microwave heating is particularly useful for reactions carried out under elevated pressures, (Dielectric heating) because of contact less delivery of energy to the reading fluids, high energy density possible and short heating times leading to nanoparticles weakly agglomerated, with high crystallinity and narrow grain size distribution.

II. EXPERIMENT

In the present investigation, CdS nanoparticles were prepared by chemical aqueous phase precipitation technique involving the reactions of cadmium sulphate $3\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$ with thioacetamide CH_3CSNH_2 . For controlled synthesis of nanocrystals with narrow size distribution and uniform shape

or with different morphologies, two elements are very important, *Molar Ratio and Annealing at different Radiations*.

This is one of the facile methods to prepare shape controllable CdS particles; this technique is simple, fast, low cost and not much time consumption to provide material with best outcomes.

Basic principle of used technique is "interaction of radiation with matter".

A. Precipitation Route and Stoichiometric Weighing

The starting materials used for the fabrication of CdS are powder of $3\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$ and Thioacetamide CH_3CSNH_2 of purity better than 99.9 % and water as solvent. Digital balance (Adventurer OHAUS) was used for accurate weighing the starting materials, under desired molar ratio ($\text{Cd}^{2+}/\text{S}^{2-} = 1:3$) and ($\text{Cd}^{2+}/\text{S}^{2-} = 1:4$).

B. Solution Preparation

In typical experiment, all compounds were analytically pure and used without further purification. A series of aqueous solutions of $3\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$, and CH_3CSNH_2 were prepared in separate flask using 100 ml distilled water.

C. Stirring

The formation of transparent liquids indicating complete dissolution of all materials in distilled water. Cadmium sulphate solution was added drop wise to thioacetamide solution. The homogeneous mixing of both solutions was done under constant magnetic stirring. The initial colorless thioacetamide solution started turning to light yellow after some hours indicating formation of CdS nanoparticles to some extent. Subsequently, the color of the solution changed to light yellow and thereafter it remain unchanged.

D. Annealing at Different Radiations

For completion of synthetic reaction and to explore the shape and size evolution of CdS particles with irradiation time, both solutions were divided into three equal parts and placed under various energy sources, *Microwaves, Ultrasonic and Sunlight* for different time and power.

When solution were heated by these above mentioned energy sources, CH_3CSNH_2 was finally hydrolyzed to S^{2-} ions and CdSO_4 gives Cd^{2+} ions after that reaction of these S^{2-} ions with Cd^{2+} ions to produce CdS nanoparticles takes place. Hence the colorless solution was appeared bright yellow. Under various environment, materials decomposes into ions, at different rates and it is found that fastest chemical reaction is possible under microwave (Dielectric heating), then sonication (Acousting cavitations) and very slow reaction takes place under sunlight (photochemical reduction).

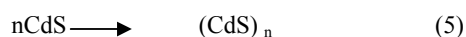
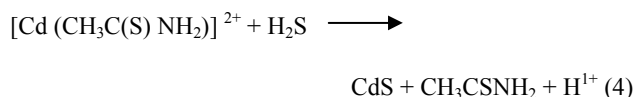
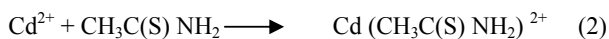
E. Washing and Heating

Then the Solutions were washed with distilled water many times by using centrifuge machine to remove remnants or side products. After application of various energy sources, and washing bright Yellow precipitates of CdS nanoparticles were collected, dried in oven at 70°C to collect pale yellow powders. This colloid was stable with time and did not show

any observable precipitates over a long period.

III. REACTION MECHANISM

Reaction mechanism involved in formation process of CdS nanoparticles can be formulated as follows:



IV. RESULTS AND DISCUSSION

The bright yellow precipitates of CdS were collected after irradiation, then washed and dried at 60 °C, finally characterized by XRD (X-Ray Diffraction), FTIR (Fourier transform infrared spectroscopy), DSC-TGA (Differential Scanning Calorimetry-Thermogravimetric) and SEM (Scanning Electron microscopy) techniques.

Phase Analysis, Cell Dimension, Grain Size Measurement has been deduced from XRD patterns about each sample.

The Average particle size was found to be in the range of 11-16 nm by using Scherer's formula. The result show that size of nanoparticles varies under different environment. Three broad and symmetric peaks were observed at $2\theta = 26^\circ$, 44° and 52° belonging to (111), (220), (311) corresponding to the cubic phase of CdS [16] shown in Fig 1.

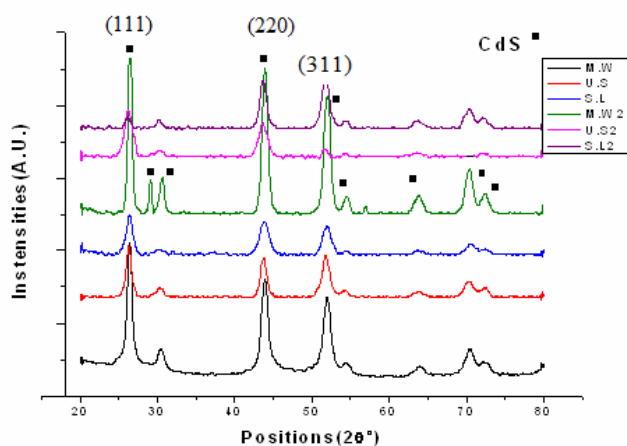


Fig. (1) The comparative patterns of all prepared samples, which shows that the morphological changes increase as ratios increases.

The particle size increases as the reaction time increases under different energy sources. As samples that have been treated under microwave are of smaller sizes than those, which are prepared in sunlight and Ultrasonic environment because microwave heating requires short reaction time in comparison with sunlight and ultrasonic environment. Application of various radiations is an appropriate approach for precise control of the size of nanocrystalline materials.

Increase in molar ratio results in the reduction of crystallite size.

XRD results indicate that all samples are almost in single cubic zinc belende phase with small traces of hexagonal phase, and X-ray data has been indexed according to FCC structure with space group Fm43 for all products. *Lattice Parameters* were calculated for each sample. The Particle size calculated through scherrer's formula was found to be in the range of 11-16 nm. *The results show that the particle size increases as the reaction time increases. As long a molarities increases, crystallite size of prepared CdS nanocrystals decreases.*

SEM images represent changes in the structure of CdS prepared in distilled water solution (100ml). The Grain size, shape, texture and surface properties were observed by SEM techniques, with molarities ($\text{Cd}^{2+}/\text{S}^{2-} = 1:3$), ($\text{Cd}^{2+}/\text{S}^{2-} = 1:4$).

The SEM images of synthesized CdS nanocrystals with different morphologies were shown in Fig.2 (a), (b) and (c).

Fig. 2 (a) represents fast growth acicular structure of CdS sample fabricated by microwave irradiations for 30 min (180 watt). The size of particles is in range from 50 nm to 100 nm.

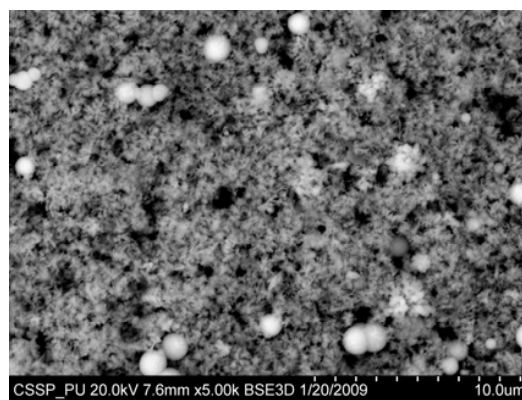


Fig. 2 (a) Acicular (Needle like components) structure of CdS Nanoparticles

Fig. 2 (b) shows that the Mesoporous CdS nanoparticles fabricated by the Ultrasonic irradiations for 480 min (240 watt). The size of particles is not uniform, but most of them range from 300 nm to 500 nm. Mesoporous structure has potential impact in the areas such as heterogeneous catalysis, molecular separation and the composite nanomaterials. The mesoporous CdS nanoparticles could be formed by a growth and assembling process of CdS nuclei.

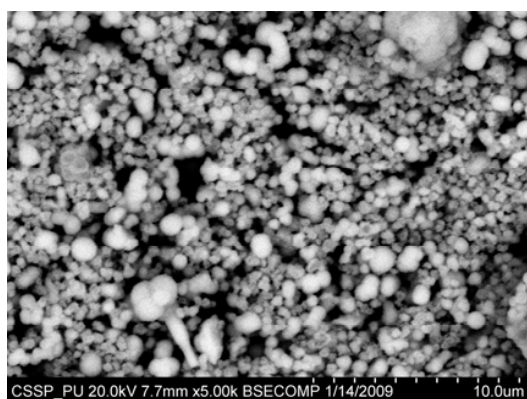


Fig. 2 (b) Mesoporous Spherical CdS Nanoparticles

In Fig. 2(c) the morphology of Spherical CdS nanoparticles prepared by sunlight energy for 240 min (at 40 °C) is clearly visible. The particle sizes vary from 200 nm to 400 nm under sunlight.

So the result indicates that to nucleate and grow inorganic ordered spherical shaped CdS nanostructure, controlled environment is required. *Furthermore growth and size of nanoparticles can be controlled by varying the time, molarity and different energy sources.*

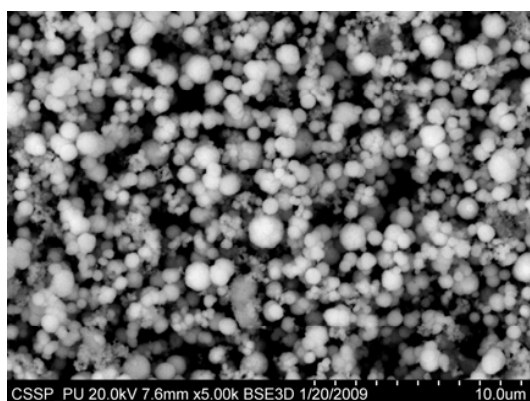


Fig. 2 (c) Spherical CdS nanoparticles

FTIR measurements have been made in the wave number range 500 cm^{-1} to 4000 cm^{-1} . The samples have been admixed with KBr, thoroughly mixed and palletized by pressing under a pressure of 10 tons for few minutes under vacuum, before FTIR analysis. The FTIR spectra and corresponding data of these samples have been provided in Table I.

Analyzing the FTIR spectra of CdS nanoparticles, prepared for different molarities and environment were almost similar there is negligible shifting in absorption peaks of all spectra. Hence only one FTIR spectra is shown in Fig 3. One can observe many absorption bands, which are give in Table I.

The very weak absorption bands at 3699.7 cm^{-1} is assigned to O-H stretching vibration of water molecules, due to

presence of moisture in the sample [17].

CdS particles showed two stretching bands, asymmetric and symmetric, around 2920.2 cm^{-1} and 2852.6 cm^{-1} are associated with C-H stretching (Tang et al 2005). Very weak bending vibrations of water molecules appeared at 1587.6 cm^{-1} , C-C stretching [25], Medium strong band positions in the range of 1376 cm^{-1} -1460 cm^{-1} , are possibly due to stretching vibrations of sulphate group. Traces of SO_4^{2-} ion as impurity is seen as there are small absorptions appears in the range of 1089 cm^{-1} - 1151 cm^{-1} . Absorption at 1018.1 cm^{-1} and 1106.4 cm^{-1} were assigned to SO_4^{2-} (Williams and Fleming 2004). There are medium to strong absorption bands at 613 cm^{-1} and 723 cm^{-1} (closely related to band positions 669.0 cm^{-1} and 718.9 cm^{-1} have been assigned to Cd-S stretching by Periasamy et al 1997), possibly due to Cd-S stretching.

Hence the existences of above mentioned bands identify the presence of CdS and also the impurities that the samples consisted of water molecules or hydroxide ions.

TABLE I
VIBRATIONAL ASSIGNMENTS OF CdS NANOMATERIALS WITH POSITIONS AND INTENSITIES OF ABSORPTION SHOWN BY IR SPECTRA

Sr No.	Positions (Cm-1)	Intensities	Assignments
01	610.9-723.4	Medium	Cd-S stretching
02	1089-1150	weak	SO_4^{2-} as traces
03	1375.9-1460.1	Medium strong	Sulphate group S=O
04	1587.5-1604.0	weak	C-C stretching, bending vibrations of H_2O
05	2853.4-2922.6	Doublet	C-H stretching
06	3699.7	Weak Triplet	O-H stretching

The DSC-TGA (Differential Scanning Calorimetric-Thermal Gravimetric Analysis) analysis of each specimen was carried out by the ramp method. The exothermal peaks are considered in up direction.

Analysis of DCS curves of all samples shows that the oxidation process for all CdS nanoparticles, prepared for different molarities and environment were almost similar, hence one graph of typical molarity and environment has been presented here.

Thermo gravimetric analysis was carried out under following experimental conditions:

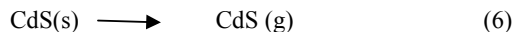
Heating rate: 5°C/min,

Sample mass: 21mg,

Static air atmosphere, DTA: 20 miliwatt. The analysis was made in platinum crucible using Al_2O_3 as the reference material.

Results from thermo gravimetric analysis (TGA) are shown in Fig. 4 (a), (b) and (c). First in Fig. 4 (a), there is decrease in

sample mass below 400 °C. This region exhibits endothermic peak in DSC curve. This initial weight loss in temp range below 400 °C mainly corresponds to evaporation of residual solvent or maybe some part of CdS appear as an endothermic peak in the DSC curve. Hence possible chemical reaction as follows:



The TG curve shows some gain in mass in the temperature range 400 °C to 800 °C. The process was

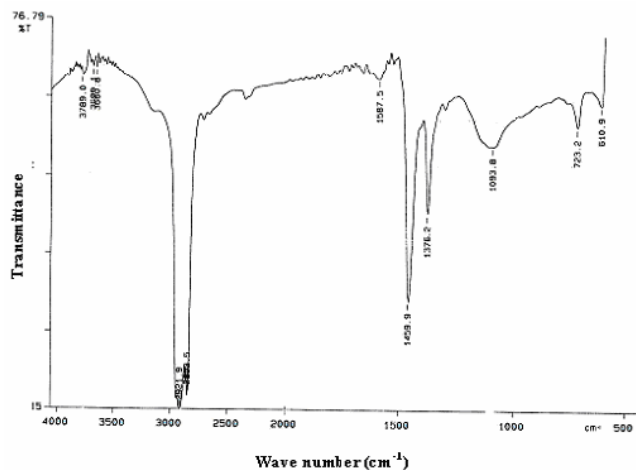
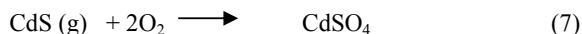
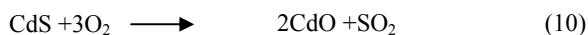


Fig. 3 FTIR Pattern of CdS

exothermic within $\Delta m = 12\%$. This gain is most probably indicating the cadmium sulphate formation. Theoretical increase in sample mass for the complete oxidation of CdS to CdSO_4 is 44.3 %, this might mean that CdS is not completely oxidized to CdSO_4 [18], this is the beginning of oxidation. Possible reaction is as follows:



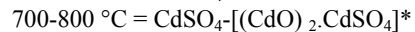
Before the complete formation of CdSO_4 , its decomposition start, began to react with unreacted sulphide, indicating the formation of intermediate product $2\text{CdO} \cdot \text{CdSO}_4$, as a result various endothermic peaks were observed in temperature range 820 °C to 1000 °C and at the same time the mass of sample continued to decrease within $\Delta m = 26\%$ at ≈ 980 °C. This is basically due to the reaction between the sulphate, sulphide and evaporation of CdS. Possible chemical reaction is:



Theoretically, if the whole quantity of CdS turns into CdO

Δm would be $\approx 11\%$. The probable reason for this is the sublimation of CdS as well as oxidation [19].

The stability of phase changes with increase in temperature can be shown as follows:



[*]-phase stability under certain temperature.

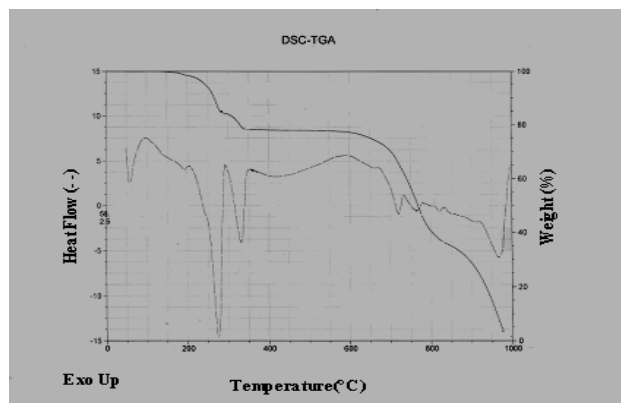


Fig. 4 (b) shows the CdO XRD pattern.

Fig. 4(c) represents thermo gravimetric analysis of CdS samples within Ar (Argon) Flow at heating rate of 5°C/min. Sample mass: 21 mg.

TGA shows two weight loss regions as shown in both Fig. 4 (a) and (c). These regions are exhibited in DSC curves as endothermic peaks. The initial weight loss in temperature range below 400°C mainly corresponds to evaporation of CdS or evaporation of residual solvent. There was a considerable decrease in mass at 850 °C i.e. indicating 99 % weight loss. This is because of sublimation of CdS. The sublimation and dissociation of CdS which resulted in the emission of sulphur vapor has been confirmed by others [19].

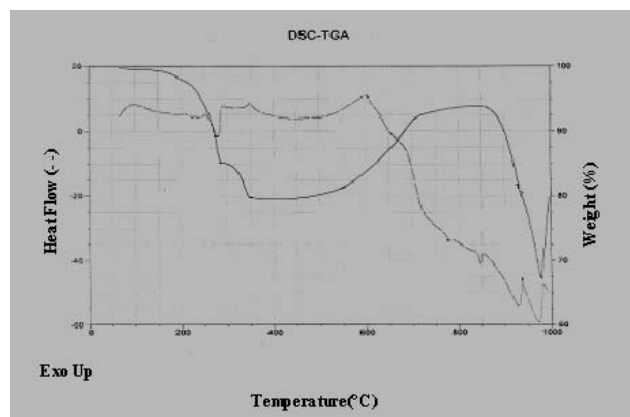
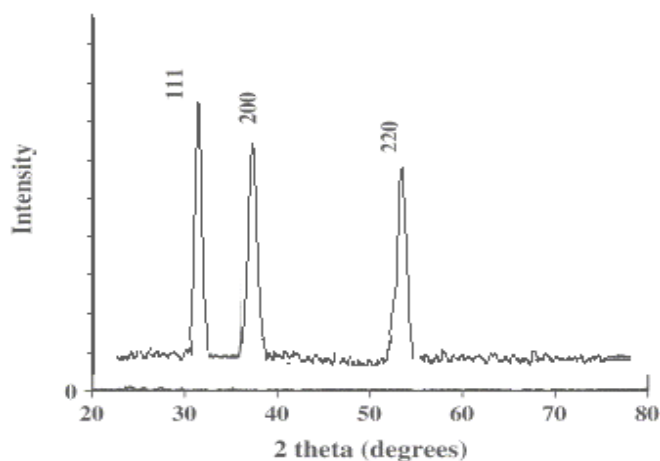


Fig. 4 (a) DSC Pattern of CdS



(Oxidation Process)
Fig. 4 (c) DSC Pattern of CdS
(Sublimation Process)

Fig 4(b) XRD Pattern of CdO appears at $2\theta = 33.08^\circ, 38.54^\circ$ and 55.12°

Hence in oxidation of synthetic powdered CdS, prepared by precipitation under the certain conditions used above, i.e. in absence of air and in Ar flow, intermediate sulphate products are obtained due to deficiency of oxygen, under these conditions, compositions of gas phase changes. Oxidation of CdS is complicated heterogeneous exothermic process, which is affected by number of complicated process involving reaction kinetics, crystal chemistry, mass and heat exchange, and many others.

There are some differences in the temperature range 800 to 900 °C in that the transitions from CdS-CdSO₄-CdO occur comparatively at low temperature under normal heating than by thermodynamic analysis. The dissociation of CdSO₄ to CdO is successfully completed at $T > 800^\circ\text{C}$, while according to TGA, CdO phase is quite stable at $\approx 900^\circ\text{C}$. These differences are due to the complicated nature of the heterogeneous processes affected by the chemical, adsorption, mass-heat exchange processes etc.

The sublimation of CdS is also a big cause of these differences [19] shown in fig 4 (c).

IV. CONCLUSION

Five different samples of CdS nanoparticles are prepared from stoichiometric weights of Cadmium sulphate 3CdSO₄·8H₂O and Thioacetamide NH₃CSNH₂ by using aqueous precipitation techniques with different molar ratios. Samples have been treated at different radiations like microwave, ultrasonic and sunlight to control the size, shape and growth of CdS nanoparticles.

XRD results indicate that all samples are almost in single cubic zinc belende phase with small traces of hexagonal

phase; The Particle size calculated through scherrer's formula was found to be in the range of 11-16 nm. The results show that the particle size increases as the reaction time increases. As long a molarities increases, crystallite size of prepared CdS nanocrystals decreases. The weight changes under evaporation and Oxidation (Transition from CdS-CdSO₄-CdO) of CdS were observed in DSC-TGA analysis. There was a considerable decrease in mass at 850 °C i.e. indicating 99% weight loss. This weight loss is because of sublimation of CdS. The presence of organic content and presence of chemical functional groups in the samples of CdS nanoparticles was also identified in the FTIR analysis. There are medium to strong absorption bands at 613 cm⁻¹ and 723 cm⁻¹ possibly due to Cd-S stretching. Hence confirms the presence of CdS nanoparticles. Morphological characteristics of CdS nanoparticles have been studied by SEM images at different magnifications. The formation of CdS nanocrystals with different shape sizes and texture (such as Acicular (needle like), mesoporous and spherical shape) are confirmed through SEM analysis.

Hence it has been concluded that shape and size controlled synthesis of CdS nanoparticles has been successfully possible by interaction of radiation with matter at different time and also stoichiometric ratios. It is suggested that controlled growth of CdS nanoparticles can also be possible by using Microwave radiations at different time and power. Agglomeration of nanoparticles can be prevented by capping through organic solvents.

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REFERENCES

- [1] Efros AL, Rosen M Annu, Rev. Mater. Sci. vol. 30, 2003, pp. 475.
- [2] R. I. Dimitrov and B. S. Boyanov, Journal of Thermal Analysis and Calorimetry. Vol 61, 2000, pp. 181-189.
- [3] P. Raji, C. Sanjeeviraja and K. Ramachandran, Cryst. Res. Technol, vol 28, 2005, pp. 233-238.
- [4] Pushpa Ann Kurian, C. Vijayan, K. Sathiyamoorthy, C. S. Suchand Sandeep, Reji Philip Res, Lett. 2, 2007, pp. 561-568.
- [5] J. Jakimavicius, A. Alisauskas and A. Sirvaitis, Materials Chemistry and Physics, Vol. 11, 1971, pp. 493.
- [6] J. Jakimavicius, A. Alisauskas and A. Sirvaitis, Chem, vol. 76, 1972, pp. 38400.
- [7] K. Matsumoto, K. Takagi and S. Kaneko, J. Electrochem. Soc, vol. 130, 1983, pp. 423.

- [8] Clare R. Collins, K. Vala Ragnarsdottir, David M. Sherman, *Geochemical et Cosmochimica Acta*, vol. 63, 1999, pp. 2989-3002.
- [9] Murugan, Vadivel, Kale, B.B.Swaminathan, V.Sonawane, vol. 4594, 2001, pp. 440-446.
- [10] Yonghong Ni, Fei Wang, Hongjiang liu, Gui Yin, Jianming Hong, Xiang Ma, and Zheng Xu, *J. Cryst. Growth*, Vol. 262, 2004, pp. 399-402.
- [11] Eugenio Caponetti, Delia Chillura Martino, Maurizio Leone, Lucia Pedone, Maria Luisa Saladino and Valeria Vetri, *Journal of Colloid and Interface Science*, vol. 304, 2006, pp. 413A-418.
- [12] R. Banerjee, R. Jayakrishnan and P. Ayyub, *J. Phys. Condens. Matter*. Vol. 12, 2000, pp. 10647-10654.
- [13] R.I.Dimitrov, N. Moldovanska and I. K. Bonev, vol. 385, 2002, pp. 41-49.
- [14] Jun Chen, Xinbo Wang and Zhicheng Zhang, *Materials letters*, vol. 62, 2008, pp. 787-790.
- [15] J. Barman, J. P. Borah, K. C. Sarma, *Chalcogenide Letters*, vol. 5, 2008, 265 – 271.
- [16] R. Devi, P. Purkayastha, P. K. Kalita and B. Sarma, *Sci. vol.* 30, 2007, 123-128.
- [17] C. Yohannan Panicker, Hema Tresa Varghese and Daizy Philip, *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, vol. 65, 2006, pp. 802-804.
- [18] R. I. Dimitrov and B. S. Boyanov, *Journal of Thermal Analysis and Calorimetry*, vol. 61, 2000, pp. 181-189.
- [19] R. I. Dimitrov, N. Moldovanska and I. K. Bonev, *Cadmium sulphide oxidation*, vol. 385, 2002, pp. 41-49.