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SYNTHESIS AND STRUCTURAL ANALYSIS OF DOPED AND UN DOPED CADMIUM SULPHIDE NANO PARTICLES

Maria Bernadette Leena,* K. Raji*

Department of Physics, Holy Cross College,,Tiruchirappalli 2 (TN)
Associate Professor ,Department of Physics, Holy Cross College,,Tiruchirappalli 2 (TN)

ABSTRACT:-*The Cobalt doped and un doped Cadmium Sulphide nano particles were prepared by co precipitation technique at room temperature. The effect of nano stochiometry on structural , micro structural properties of the powders were studied. The structural properties of the nano powders were investigated by X ray diffraction. The Scanning Electron Microscopic studies were carried out using (Quanta FEG 250) and Elemental Analysis was carried out with an EDAX using Energy dispersive X ray spectrophotometer. The particle size was also calculated and compared. The present investigation is done on the recommendation of usage of microbial and photo catalytic activity of some materials.*

Key words*:Doping, nano particles, co precipitation, diffraction and micro structure*

Introduction

Cadmium Sulphide is an important group of II-VI compound semiconductors with the excellent physical properties and wide band gap energy of 2.42 eV. Cadmium sulphide (CdS) has two abilities for photo catalytic reaction. Its band gap responses to visible light and its conduction band is more negative than the reduction potential of H^*/H_2 [1]. It is obviously known that the CdS has many properties such as crystalline phase, size, morphology, specific surface area which can affect its photo catalytic activity. It has been offered that that morphology of photo catalyst could be effective for the constraint of recombination between photo generated electron and holes and for the separation of H_2 evolution sites from oxidation reaction sites [2].

Metal doped semiconductors have been appealed scientific attention due to their probable applications. Different techniques such as chemical precipitation, hydrothermal method, chemical vapour deposition, spray pyrolysis and other chemical routes have been used to synthesize metal doped CdS[2]. Metal (Ni,Sb, Co,etc…) doped CdS has drawn remarkable attention as it presents to a great opportunity to combine electrical and optical properties into a single material [2],[3],[4],[5].

These catalyst can trap light induced electrons and act as the active sites for H_2 production and degradation of dyes from water [6],[7]. With the assistance of Co catalysts, researchers have successfully developed various CdS based photo catalysts possessing visible light catalytic activity. These modified photo catalysts are valuable, since they can efficiently promote water splitting under visible light irradiation and provide high hydrogen production rates.[8],[9],[10].

Experimental

Samples of CdS and Cobalt doped CdS has been prepared by chemical Precipitation method

Synthesis

The undoped CdS materials have been prepared by precipitation method using Cadmium Acetate and Sodium sulphide. In this method aqueous solutions of 0.1M of Cadmium Acetate($CH_3COO)_2$ Cd.$2H_2O$) and 0.1M aqueous solution of Sodium Sulphide (Na_2S) were prepared separately. The nano particles initially purified by double distilled water and they are centrifuged 1000 rpm for 5 minutes. The orange coloured CdS nano particles were obtained as precipitate after being evaporated at room temperature.

Cobalt- doped CdS nano particles

0.1M of Cadmium Acetate dehydrate ($Cd(AC)_2$ $2H_2O$)) and 0.005M of Cobalt Acetate tetrahydrate (Co ($CH_3COO)_2$,$4H_2O$) were prepared with double distilled water. Simultaneously 0.1M of Sodium Sulphide solution was also prepared. Aqueous solution of Na_2S was added dropwise into the Mixed Solution, which resulted in of orange coloured precipitation of Co:CdS nano particles.The nano particles initially purified by double distilled water and they

are centrifuged 1000 rpm for 5 minutes.The orange coloured cobalt doped CdS nano particles were obtained as precipitate after being evaporated at room temperature.

Thus undoped CdS and Transition metal ion doped CdS were obtained.

Characterization

The undoped CdS and Co doped CdS NPs were characterized by X-ray diffractometer (Model: X'PERT PRO PANalytical). The diffraction patterns were recorded in the range of 20° - 80 ° for the CdS samples where the monochromatic wavelength of 1.5405 A° was used.

Results and Discussion

The X ray diffraction patterns of the synthesized doped and un doped CdS nano particles are shown in the Fig (1),and Fig (2). According to literature review, CdS nanoparticles shows either cubic or hexagonal structure. The figures to confirm the crystal structure of X−ray diffractogram of Co doped CdS nanoparticles and that of pure CdS. The broadening in the peaks is observed in all samples due to nanocrystalline nature. The XRD patterns of all samples are in accordance with the cubic structure of CdS and the peaks observed in the XRD patterns and d−values match well with the standard JCPDS data (80−0019) of cubic CdS with no secondary phase found.

The three most intense peaks (111), (220), (311) at prominent Bragg's angle for all samples measured. The host CdS may have accommodated Co^{2+} ions into its lattice because Co^{2+} possesses smaller ionic radii (0.74 Å) than Cd^{2+} (0.96 Å) [11] but no significant change in 2θ is observed. Further, the electro negativities of both cations differ with sulfur (1.88 for Co, 1.69 for Cd, Pauling Scale) hence there is all possibility that Co can be incorporated into the CdS lattice at vacancy sites. Doping of Co in CdS does not lead to any structural phase transformation. The average crystallite size of the samples is determined by Debye Scherrer's equation [12]. The average Crystallite size for undoped cadmium sulphide is 11 nm and that Cobalt doped cadmium sulphide is 7 nm. For both the samples the strain is 0.04 .

The SEM images of undoped and Co doped CdS nano particles are shown in micro graphs,3a,3b and 4a,4b,and 4c. The SEM images shows the particle size in few microns and in nano meter range.

Morphological and Compositional Analysis

Surface Morphology of the sample has been studied using FEI Quanta FEG 200 High Resolution Scanning Electron Microscope (HRSEM) with energy dispersive X-ray spectrometry (EDAX). SEM images show the agglomerated nano particles with cubical shape which coincides with XRD result of the prepared samples.
The chemical composition analysis of the Co doped CdS nano particles was done using EDAX technique, and it confirms the purity of the samples and also confirms presence of Cobalt content in the doped samples. It is evident from the fig. (5) and also Table.1 provides the elemental composition in terms of atomic and weight percentage. The data gives the good control on each element.

Conclusions

1. Energy dispersive analysis of X−rays (EDAX) shows some foreign impurity content in the samples and the amount of Co present in CdS increased depending on the increase of Co concentration in the solution.
2. XRD pattern shows strong diffraction peaks which is an indication of good crystallinity of the samples and are matching with the JCPDS data (JCPDS file no. 80−0019). Small shifts in the peaks position in Co doped CdS nanoparticles ascertain to the incorporation of Co^{2}+ into the CdS lattice and also exhibits zero alteration in the phase property of CdS.
3. The XRD results also indicate that the Co^{2+} ions incorporation in the CdS lattice without changing the cubic structure, there is no indication of any secondary phase or clusters, confirming that the samples are only one single phase but considered peak shifting and slightly variation in lattice constant with Co concentration. The estimated crystallite size was in the range between 7-11 nm.
4. The SEM images of pure and Co doped CdS nanoparticles appeared cubical in shape No alteration in the size and shape is observed due to the incorporation of dopant. These crystals exhibit poly crystalline nature with Cubic phase.

This study demonstrated an easy method for preparation of efficient visible light photocatalysts, namely metal doped CdS nanoparticles, by chemical precipitation method. Crystallite size of metal doped CdS catalysts was decreased by adding metals such as Cobalt. The EDAX study confirms the presence of the Cobalt and other elements. The results are promising for further study.

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leenavijay.jj@gmail.com

K. Raji*

Associate Professor in Physics

Department of Physics
Holy Cross College
Tiruchirappalli 2 (TN)

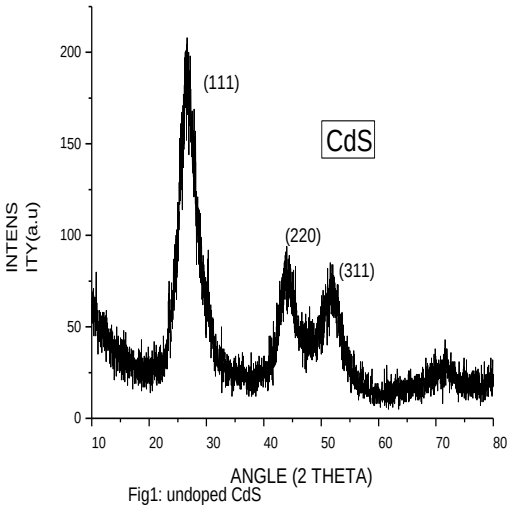

Fig1: undoped CdS

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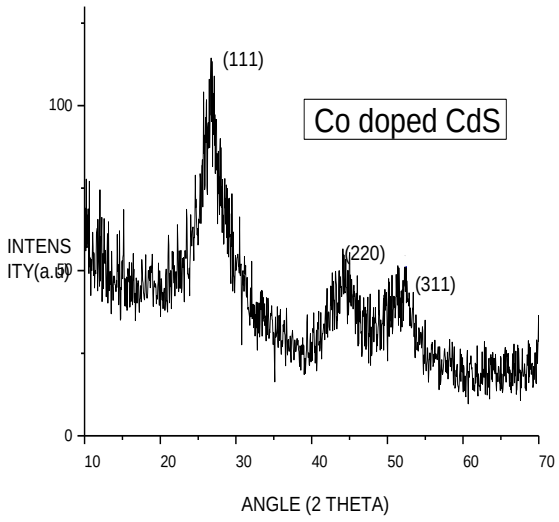

Fig 2: Cobalt doped CdS

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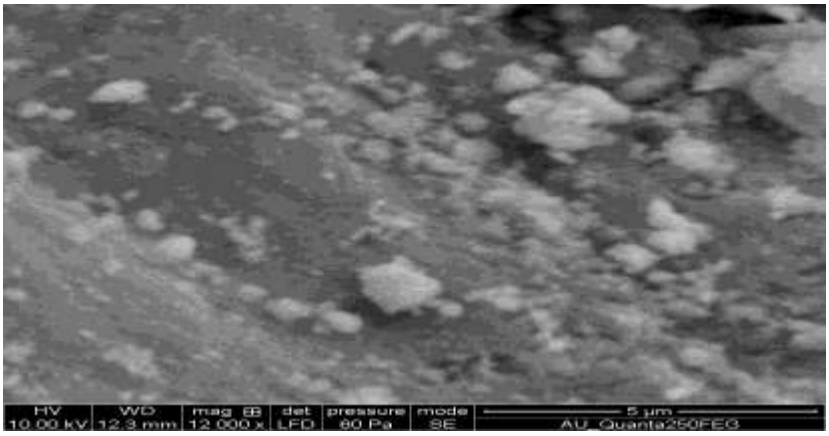
Structural and Elemental Analysis



3(a) micro structure of cobalt doped cds nano particles 5µm

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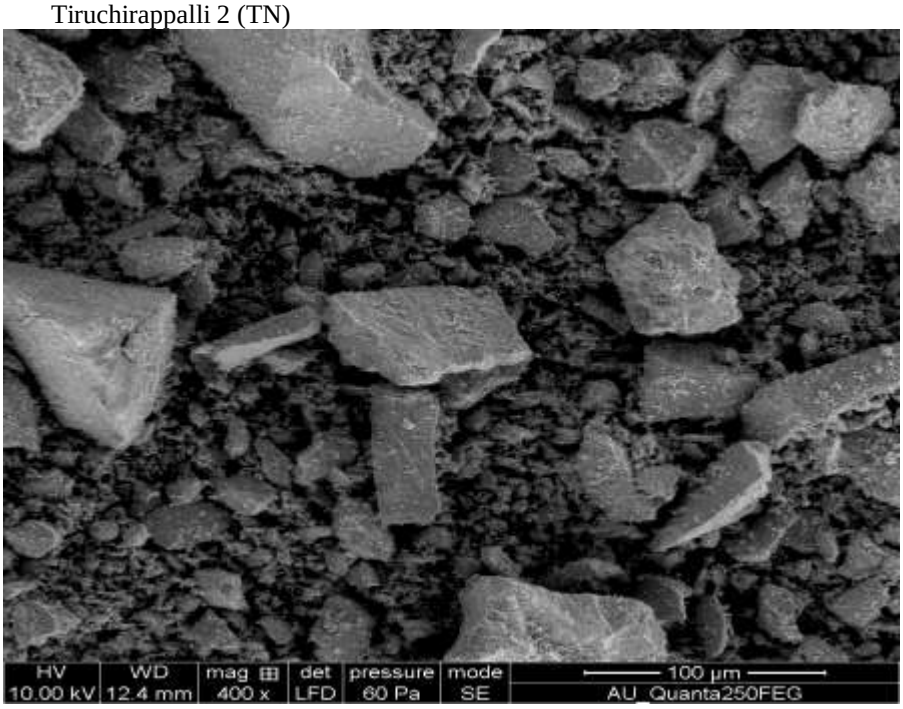

3(b) **Micro Structure of Cobalt doped CdS nano particles at 100µm**

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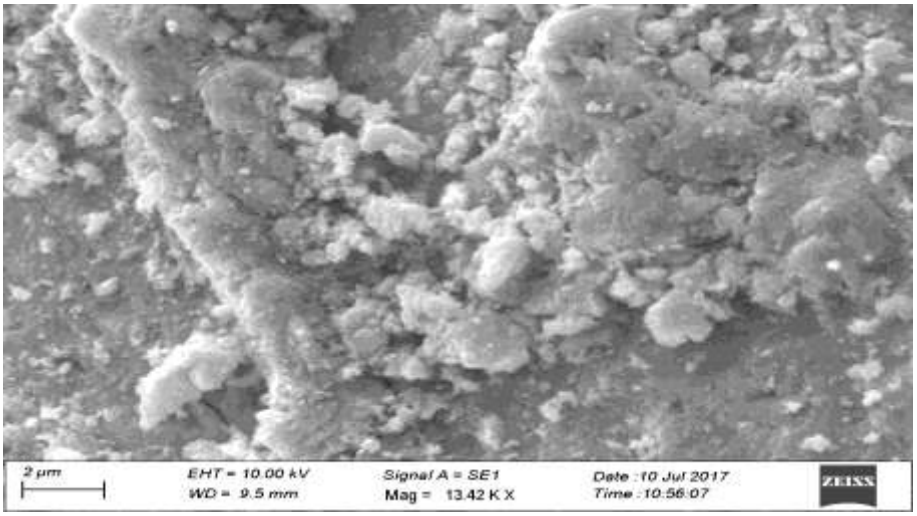

4(a) **micro structure of undoped Cds at 2µ m**

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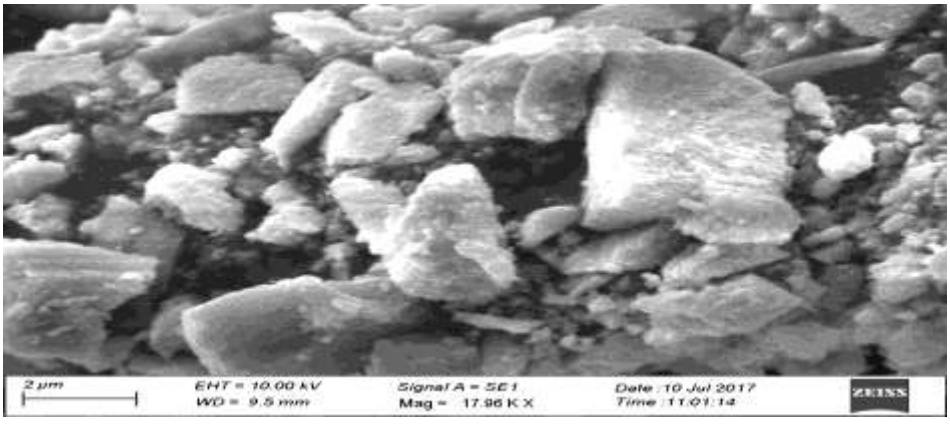

4(b) **Near cubical in shape and having agglomeration of CdS at 2µm**

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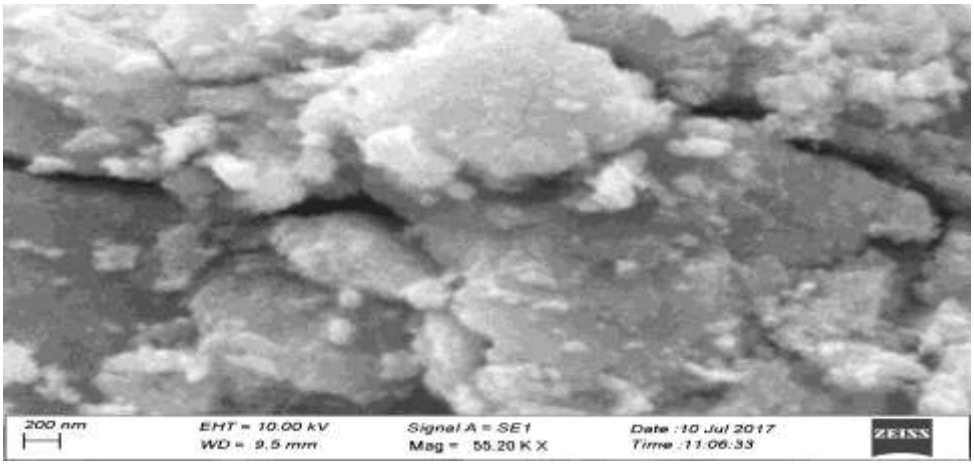

4(C) micro structure of undoped CdS at 200nm

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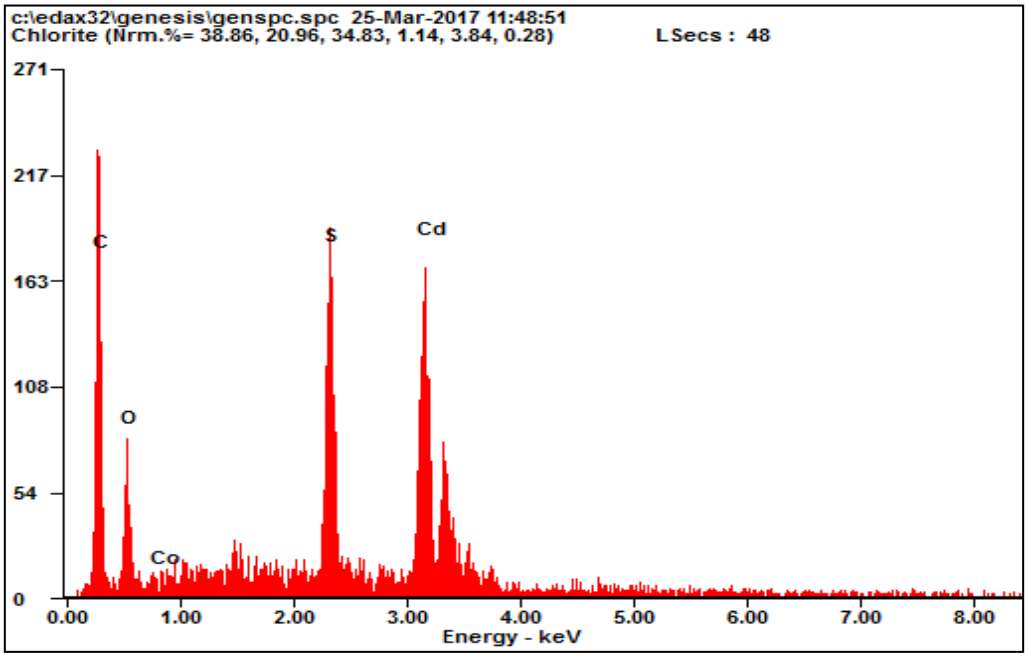

Fig.5. EDAX of Cobalt doped CdS

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Table1 : Composition of Elements in EDAX (in %)

Element	Wt%	At%
C K	19.5	54.86
O K	6.56	13.69
CoL	0.59	0.034
CdK	12.68	13.19
S K	60.49	17.93

