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Synthesis and Characterization of Cadmium Sulfide Nanoparticles for the Applications of Dye Sensitized Solar Cell

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ABSTRACT: Cadmium sulfide nanoparticles were prepared by chemical precipitation method in aqueous medium using cadmium acetate and sodium sulfide. The synthesized nanoparticles were characterized by using UV-Vis studies, X-ray diffraction studies (XRD), field emission scanning electron microscopy (FESEM), energy dispersive x-ray analysis (EDAX) and BET analysis. The X-ray diffraction pattern revealed that the synthesized cadmium sulfide nanoparticles were polycrystalline nature and grain size 7 nm was calculated by using Scherer method. Specific surface area, pore volume and pore size were estimated by nitrogen adsorption-desorption analysis. The specific surface area of the synthesized material is 74.26 m^2/g. The temperature and frequency dependence of dielectric constant, dielectric loss and AC conductivities were studied over a range of frequency (50 Hz to 5 MHz) and temperature (40 - 200°C). These results demonstrate that the CdS nanoparticles has a potential applications in DSSC's.

KEYWORDS: Cadmium sulfide, Solar cell, dielectric constant, precipitation method, DSSCs.

I. INTRODUCTION

Semiconductor nanoparticles such as PbS and CdS have attracted considerable attention due to the their unique properties from that of their bulk materials[1].Generally, the nanoparticles exhibits variation in their properties to the bulk material due to two factors namely the increase in surface to volume ratio and change in electronic structure of the material by the quantum confinement effect[2-3].In past decade, semiconductor nanopaticles have been synthesized through various methods including hrdrothermal[4], micro-emulsion[5], sol-gel[6], chemical co-precipitation[7], sonochemical[8], microwave irradiation[9-10] and solvothermal methods[11] and so on. However these methods normally consist of two or more steps and vigorous conditions, such as high pressure or high temperature are required to attain nanoparticles[12-14]. Futhermore co-precipitation method has some advantages like inexpensive, less time, need less instruments, convenient operation and produce high yield of product formation with very low temperature condition. Generally in coprecipitation method CdS nanoparticles obtained with agglomerated particles without using capping agents. So the presence of the surfactant are also used as stabilizer to prevent agglomeration [5-6]. Study of the effect of the residual capping agents on particle surface has unveiled various adverse and favourable behaviours in catalytic applications.

In this paper, the CdS nanoparticles were synthesized by chemical precipitation method with tetrabutylammonium bromide as a capping agent and their effect on surface morphology of nanoparticles, structural, optical and dielectric properties were reported. The present process provides a effective route for preparation of CdS nanoaparticles which can be used in the fabrication of opto-electronic devices.

II. EXPERIMENTAL

2.1 Synthesis of CdS nanoparticles

All the chemicals used in the experiment were purchased from Loba chemie Pvt ltd and used without further purification. Millipore double distilled water was used throughout the experiment. The CdS nanoparticles were synthesized by chemical precipitation method using Cadmium acetate and sodium sulfide as the starting materials. 0.1 M solution of $Cd(CH_3COO)_2$ and Na_2S reactants was prepared using millipore water. 50 ml of 0.1M cadmium acetate solution was taken in 250ml Round bottom flask and stirred for 30 mins using magnetic stirrer. After 20 mins 0.01M tetrabutylammonium bromide (TBAB) dissolved in water was added to it and continued stirring for 30 mins to get homogeneous solution and then 0.1M solution of sodium sulfide was added dropwise to the solution of cadmium acetate under continuous stirring until the formation of yellow precipitate and stirring was continued for 2h to complete the reaction. The pH was maintained at 9-10 by adding ammonia solution in basic medium. The chemical reaction is,

$$Cd(CH_3COO)_2 + Na_2S \longrightarrow CdS + 2CH_3COONa$$

The solution was allowed to settle down for 2h, and the particles were precipitated into ethanol. The solid yellow product was washed by suspending them in ethanol. The reaction product was repeatedly washed with ethanol to clear the surfactant impurities from the CdS nanoparticles and centrifuged for 20 mins for three times with ethanol at high rpm level. And then the product was dried at 150ºC for 3h under reduced pressure at ambient temperature. The dried samples were ground using mortar and pestle to make fine powder for better characterization. The flowchart synthesis of pure and capped CdS nanoparticles is given in Fig.1.

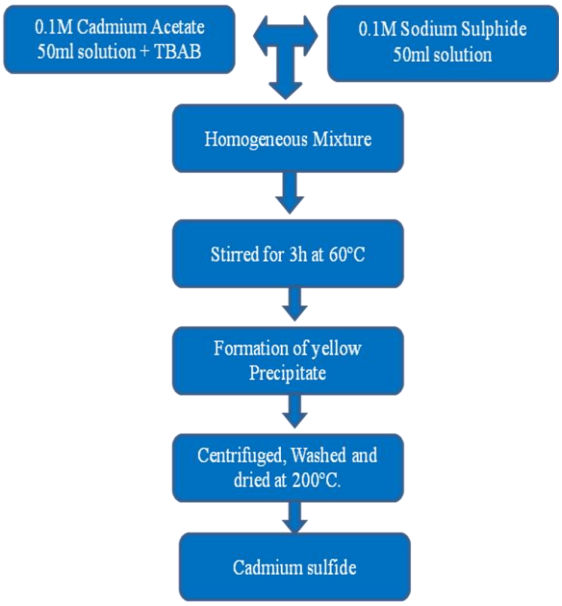

Fig. 1 Flow chart of synthesis of CdS nanoparticles by precipitation method

2.2 Characterization:

The optical absorption was recorded by using Shimadzu-Pharmonper 1700 UV-vis spectrophotometer in the range 200 to 1200 nm. For optical characterization the absorbance and transmittance spectrum were recorded. The crystallinity and crystal phases were determined by X-Ray diffraction pattern using Bruker D5005 diffractometer (Cu Kα radiation (λ=1.5404Å) in 2θ range from 10° to 70°. The microstructures of the samples were characterized by FEI Quanta 200 Environmental SEM with EDAX analysis. The BET nitrogen absorption desorption analysis were carried out by

Quantachrome NOVAWIN instruments. Dielectric studies were conducted by using HIOKI 3532-50 LCR Hi TESTER model LCR meter as the function of frequency 50Hz – 5MHz at the temperature ranges from 40°-200°C.

III. RESULTS AND DISCUSSIONS

3.1 Structural studies:

Figure.2 shows the X-ray diffraction pattern of the CdS nanoparticles prepared at room temperature in the diffraction angle of range 10° -70°. The obtained peaks were matching with the reported value of (JCPDS file No.41-4019) which confirms the hexagonal phase of these samples. The broadening peaks observed in capped CdS nanoparticles which clearly indicates the particle size is decreased when increasing the concentration of TBAB. The average crystallite size was calculated using the scherrer's formula.

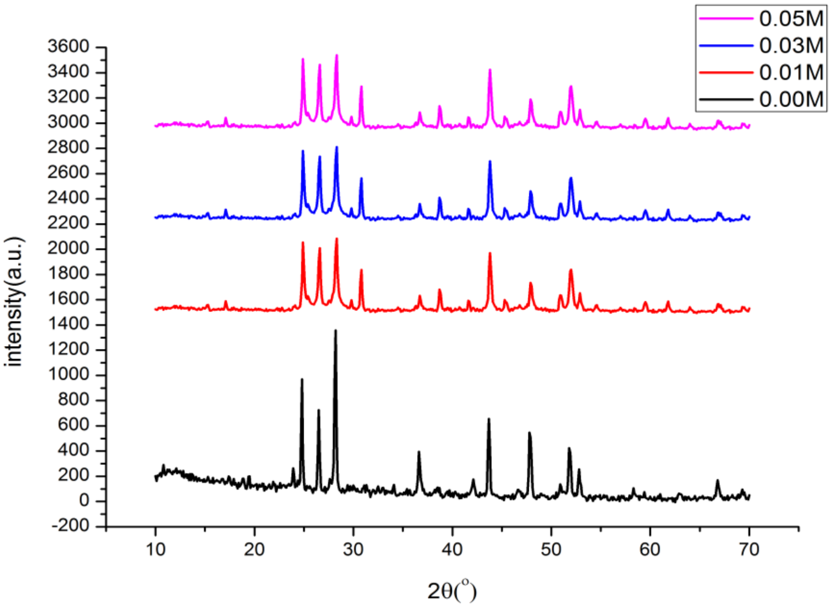

Fig. 2. XRD pattern of the CdS nanoparticles synthesized by various concentrations of apping agent TBAB

$$T = \frac{0.89\lambda}{\beta \cos \theta} \qquad \_\_\_\_\_(1)$$

Where T= crystallite size, λ is the wavelength of X-rays(1.5404 Å),β is the fullwidth half maximum in radian and Θ is the bragg's diffraction angle obtained from 2Θ value corresponds to the maximum intensity peak in XRD pattern.
The strain and crystallite size of the sample were calculated from Williamson Hall equation[14],

$$\beta \cos \Theta = K\lambda/t + 2\varepsilon \sin \Theta \qquad \_\_\_\_\_\_ \qquad (2)$$

where K- debye sherrer's constant (0.89), t – average crystallite size, ε – lattice strain. In this method while plotting the graph 2 sinΘ against β cosΘ gives a straight linear plot, the intercept gives crystallite size and slope gives the strain(ε). The calculated average crystallite size and stain was found to be 7nm and 4.78 x 10^{-3}.

3.2. **Morphological studies:**

The surface morphology, grain size and shape of CdS was studied by SEM analysis. The Fig.3 shows the SEM image of as synthesized CdS nanoparticles were polycrystalline nature and the synthesized particles exhibited spherical granules like structures[13]. Also the figure indicating that the TBAB capped CdS nanoparticles have monodispersed spherical crystallites. The EDAX spectrum and elemental mapping in Fig.4 shows that the nanoparticles posses stochiometric composition with 59.35 at.% cadmium and 40.65 at.% of sulphur.

The nitrogen absorption desorption BET analysis showed that the synthesized materials has the specific surface area values of 74.26 m2/g with the pore size 13.17 Å. The Nitrogen gas was used as a adsorbent for the analysis time 120 mins. The pore volume of the synthesized materials are 1.87x10^{-2}cc/g in the 273K.

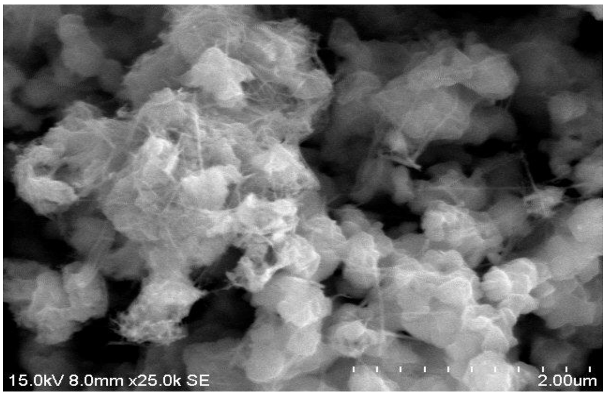

Fig. 3 FESEM image of as synthesized CdS nanoparticles

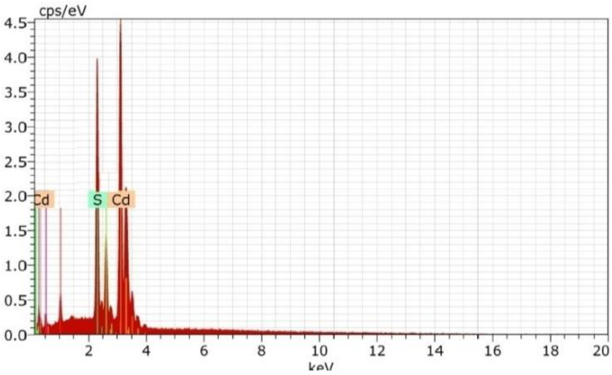

Fig.4.EDAX spectrum of as synthesized CdS nanoparticles

3.3**. Optical studies:**

Fig.5 shows the absorbance and transmittance UV-vis spectrum of CdS nanoparticles. The absorption edges were found to be at 415 and 428nm.The optical transition between the levels in the conduction and valence bands depends on the size of nanoparticles. The bandgap values of corresponding edges are 3.21eV and 3.81eV. The optical size was calculated using the Brus equation [7] which relates the energy gap and optical size of nanoparticles. The calculated optical sizes of CdS nanoparticles are 5.8nm and 5.2nm respectively.

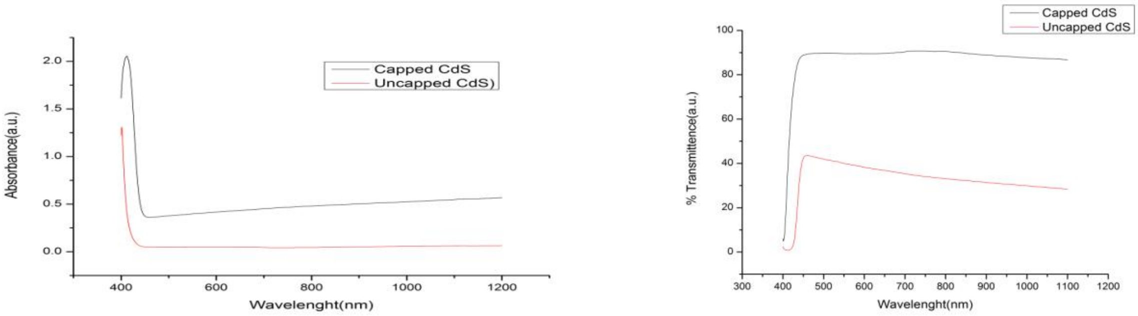

Fig. 5a and Fig.5b UV-vis absorption and transmittance spectrum of as synthesized CdS nanoparticles

3.4. **Dielectric Studies:**

Dielectric studies were conducted by using HIOKI 3532-50 LCR Hi TESTER model LCR meter as the function of frequency 50Hz – 5MHz at the temperature ranges from 40°-200°C. The samples were made into the pellet form and kept in between the parallel plate capacitors. The dielectric constant is calculated by the formula,

$$\varepsilon_r = cd / A\varepsilon_o \qquad \_\_\_\_\_\_\_\_(3)$$

where d- thickness of the sample, A is the surface area of the sample, ε_0 is the permittivity of free space (8.85×10^{-12} $C^2N^{-1}m^{-2}$) and C is the capacitance. From the Fig.6a and Fig.6b it is observed that the dielectric constant and dielectric loss were high values in low frequencies but when the frequencies increases there is decrease in the dielectric constant values and at constant values for some interval. As the temperature increases, which increases the charge carriers exponentially due to space charge polarization. In dielectric materials, dielectric losses occurs due to the absorption of current.

Fig.6a & 6b Dielectric loss and dielectric constant of CdS nanoparticles as the function of frequency at different temperatures.

IV. CONCLUSION

Cadmium sulfide nanoparticles have been synthesized by low cost, easiest, high yield chemical co-precipitation method. The CdS nanoparticles shows cubic hexagonal phase with grain size of 7nm were obtained successfully. The capping agent plays an important role of controlling the size of nanoparticles by adjusting their molar concentrations. UV results shows optical absorption edge is located at 415nm. The optical band gap of the CdS nanoparticles were obtained from UV-Vis absorption spectrum which was calculated to be 3.81eV.The SEM images showed spherical like structure of CdS nanoparticles. The composition was determined by EDS and it exhibits mainly Cd and S peaks, further confirming that the sample is pure CdS. The specific surface area of the synthesized material is 74.26 m^2/g. Depends upon the above properties of the CdS nanoparticles were used in solar cell applications.

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