Preparation and characterization of Hibiscus rosa-Sinensis leave extract stabilized Cadmium Sulfide Nanoparticles

R.Venugopal[1,2 *], Ch.Srinivas[2]
*** Author for Correspondance: venumrc.iisc@gmail.com**

Abstract

The Cadmium Sulfide Nanoparticles (CdS NPs) are drawn up using the green synthesis method. In the existing interpretation, Hibiscus rosa sinesis leaves extract is utilized as a natural stabilizer. Cadmium is obtained from sources being cadmium chloride, cadmium nitrate, cadmium sulphate, and sodium sulphide applied as a determinant of sulphur. In pursuance of determining the nature of a material, band gap, functional compounds, elemental analysis, as well as surface morphology of the synthesized material, the rehearsed CdSNPs prevail, being characterized by carrying out X-Ray Diffraction (XRD), UV-Visible Absorption spectroscopy, Fourier Transform Infrared (FTIR) spectroscopy, furthermore Scanning Electron Microscopy (SEM) experimental techniques. These tests have shown that the rehearsed materials were found as cubic crystalline cadmium sulphide with an average particle size of less than 10 nm. FTIR has demonstrated that a thin molecular layer has developed on the surface of the cadmium sulphide nanoparticles.

Keywords: Natural Surfactants, CdS NPs, Green Synthesis, Semiconducting Nanoparticles, Hibiscus rosa sinesis.

1. Introduction

The abridged magnitude of NPs provides favourable tools for electronic and optoelectronic devices that favour curtailing the bigness of electronic and optoelectronic devices. The particles preserving enormity less than 100 nm are ascribed as NPs. Nanomaterials' optical and electrical terrains, melting points, and crystal structure endure substantially contrasting correlates to their bulk depiction and confide in crystallite size for the quantum confinement effect. The disparate approaches outlawed by the researchers to synthesize CdSNPs include the Chemical precipitation method [1], Solvo thermal method [2], Laser ablation method [3], Hydrothermal method [4], Photochemical method [5], Microwave heating method [6], Photo etching [7] and Ultrasonic irradiation method [8].

Even though various chemical methods are available that employ synthetic materials to produce NPs, The Green Synthesis approach attracted more attention from researchers because the natural extracts such as plants and plants products remarkably affect the characteristics of nanoparticles such as morphology, size, and

distribution of NPs [9]. The foremost advantages of these natural extracts are that they are available, cost-effective, need only ambient room temperature, and their byproducts are also not toxic. Semiconducting NPs comparatively CdS, ZnS and CdSe are extensively studied by reason of its solitary magnitude-abased optical along with electronic terrains from the antipodal two decades [10, 11]. A great engrossment has been delineated in Cadmium Sulfide (CdS) because of the possibility of tunable energy gap, discrete energy levels, size vulnerable optical attributes, agreeable chemical stability furthermore apparent preparation approaches. The CdS is a semiconducting material belongs to II – VI group confining direct band gap of 2.42 eV in bulk state at room temperature.

The CdS NPs have abeyant applications in Pigments, Photo resistors which endure sensitive to visible and near infrared light, CdS can be combined with other layers for use in solar cells, Photo catalytic activities, Optical sensing applications. Owing to the eminences of Green Synthesis process, in the present study the CdSNPs are rehearsed by adopting the hibiscusrosa sinesis leaves extract as stabilizing or capping agent, Cadmium Chloride, Cadmium Nitrate, Cadmium Sulfate subsist utilized as a informant of cadmium along with Sodium Sulfide used as a provenience of sulfide.

2. Experimental

2.1. Rehearsal of natural stabilizer

The Young and fresh leaves of Hibiscus rosasinesis were collected from Jangaon region of Telangana, India. The Collected leaves cleaned assiduously primarily amidst normal usage water hounded by distilled water and dipped these leaves in distilled water for around 12 hours and then rinsed once again to remove the dust particles. 100 grams of hibiscus rosasinesis leaves taken in to a beaker with 400 ml millipore water, heated at around 50^0C on hotplate of magntic stirrer for 6 hours. Then filtered the extract thrice with whatman filter paper and kept it in refrigerator over a night at around 4^0C to get clear solution. This extract solution used in the synthesis process.

2.2. Synthesis of Cadmium sulfide nanoparticles

All the raw materials of analytical reagent grade were employed to prepare CdS NPs. Furthermore, all the solutions were contrived with Millipore water. Firstly

a $Na_2S.H_2O$ solution of 0.1M is primed by adding 0.7804 g of sodium sulfide into 100 ml Millipore water. Then a disembodied disposed green extract (Hibiscus rosa sinesis leaf extract) is gradually supplemented to this solution held down strenuous stirring. Cadmium salts ($CdCl_2$ - 1.8331 g, $Cd(NO_3)_2$ - 2.36 g, and $CdSO_4.8H_2O$ -2.0847 g distinctly supplemented into 100 ml milli-Q water) solution of 0.1M is primed distinctly.Further, it mixed with the above resultant solution. Now this final solution turns up yellow or orange over the pH value at this juncture is 6; this solution was stirred continuously for 4 hours at 60^0C temperature to obtain a precipitate. The obtained residue is cleansed several times with methanol after that, centrifuged and dried to get a yellow-colored powder. The primed powder is characterized by applying various experimental techniques such as XRD, UV Visible spectroscopy, FTIR, SEM, EDS, TEM, and SAXS to study and confirm its crystal structure, band gap, functional groups, morphology, composition moreover, particle size respectively.

The XRD spectrums abide inscribed employing the PHILIPS PW1830 Generator Diffractometer at 40 kV and 25 mA with Cuk_α radiation wavelength of 1.540560 A obsessed among graphite monochromator. The UV Visible spectrum is inscribed by utilizing the UV-Visible spectris 2092 spectrophotometer. The FTIR spectrum is marked by applying the SHIMADZU EUROPE FTIR-8400S spectrometer over a wave number range of 7800 cm^{-1} – 350 cm^{-1}. The material's morphology is examined using ZEISS Special Edition18 Scanning Electron Microscope.

3. Results

3.1. Powder Characterization

The X-Ray Diffraction technique is exploited to know the crystal texture of the as-prepared sample.

The standard crystallite size of the CdS NPs is obtained using the Scherrer formula [12].

$$D = \frac{0.89\lambda}{\beta \cos\theta} \rightarrow (1)$$

Where D concerns an average crystallite size, λ represents the wavelength of the X-ray radiation, β prevails the full width at half maximum (FWHM) of the X-ray diffractogram in radians, and θ represents the Bragg's angle.

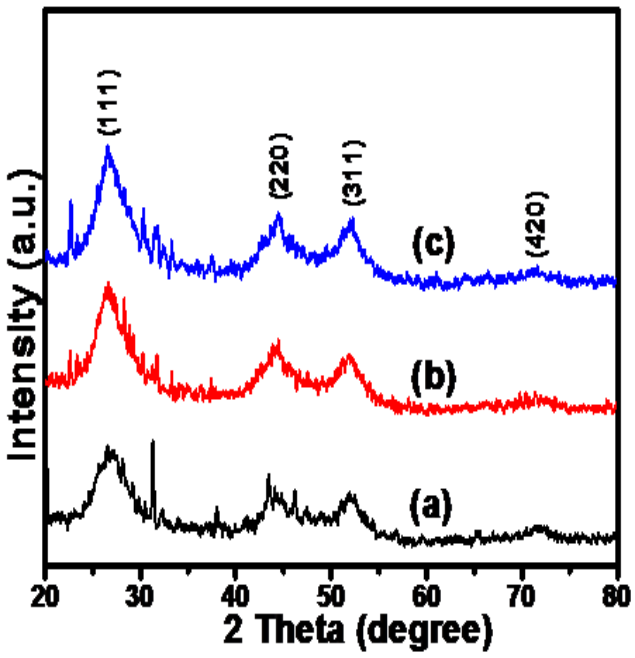

Fig. 1 XRD spectra of hibiscus rosa-Sinensis leaf extract - preserved CdSNPs possession out of (a) cadmium chloride, (b) cadmium nitrate, as well as (c) cadmium sulfate precursors.

The XRD spectra of Figure 1 show hibiscus rosasinesis leaf elicitation-stabilized CdS NPs in which cadmium chloride, cadmium nitrate, and cadmium sulfate salts are applied as precursors. XRD peak at 2θ value 26.573^0 corresponding to (111) was observed to be the most prominent peak. Other peaks at 2θ values of around 44.549^0, $52.224^{0,}$ and 71.528^0 correspond to (220), (311), and (420) planes, respectively, endure also observed. These peaks agreed well with the cubic phase of CdS (JCPDS:89-0440). Along with these peaks, there are other small intense peaks at two theta values around 23.370^0 which could be indexed to scattering from the (111) plane of cubic sodium sulfide (JCPDS:72-2149). This peak may be due to an incomplete dissolution of sodium sulfide, which was operational during the preparation of CdNPs. Two peaks at around two theta values of 33.266^0 and 37.445^0 may be attributed to reflect from (111) and (200) planes of cubic cadmium oxide (JCPDS:78-0653). The peak at 46.183^0 is found to be reflected from the (009) plane of rhombohedral cadmium chloride (JCPDS:89-1568). The broadness of peaks by means of the XRD spectrum represents nanosize particle elaboration of CdS.

The standard crystallite sizes of the CdSNPs stabilized by hibiscus rosasinesis leaf extract abled out of possession of cadmium chloride, cadmium nitrate, as well as cadmium sulfate as forerunner being observed to be 2 nm, 2 nm, and 3 nm correspondingly, the lattice parameter calculated out of the utmost intense peak (111) of cadmium sulfide is 5.8056 Å which is agreed good with that of reported value 5.8304 Å with a difference of 0.0248 Å.

3.2. Surface Morphology Studies and Elemental Analysis

The SEM micrographs of synthesized CdS NPs associated with hibiscus rosasinesis leaf extract are shown in Figure 2 (a-c). It is pleasant from the SEM images that a small agglomeration of CdS NPs has taken place to some extent. The concerned morphology of CdS NPs is a combination of both spherical and irregular shapes. The uniform size of as-prepared CdS NPs contributed to the association of green stabilizer with CdS NPs and an average size of CdS NPs being calculated to be around 3.02 nm, 2.95 nm, and 2.80 nm for powder samples in Figure 2 (a) to 2 (c)) respectively which is deliberated by Image J software.

Figure 2 (d-f) shows the EDS images of hibiscus rosasinesis leaf extract capped CdSNPs. These images give clear evidence for the presence of Cd and S elements mostly apart from sodium, chloride, and oxygen, and little presence of these other elements as a consequence of sodium sulfide and cadmium chloride employed during the course of the reaction.

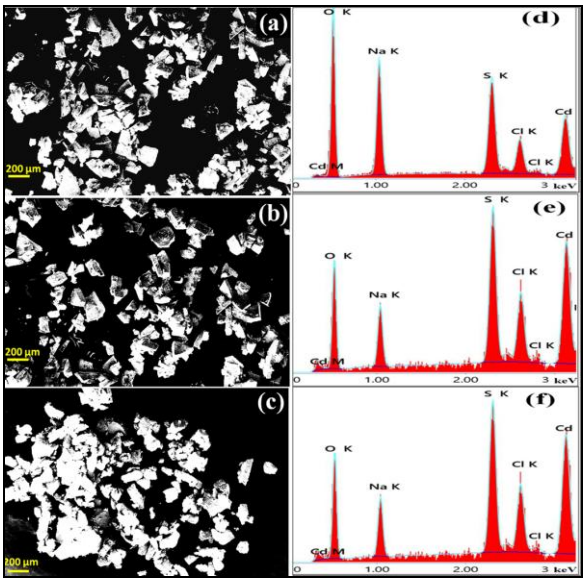

Fig. 2 SEM micrographs (a-c) as well as EDS spectra (d-f) of hibiscus rosa-sinesis encapsulated CdSNPs primed in distinction to (a, d) cadmium chloride (b, e) cadmium nitrate as well as (c, f) cadmium sulfate

3.3. Functional Groups Studies

FTIR spectrum of hibiscus rosasinesis leaf surfactant stabilized CdSNPs synthesized out of (a) cadmium chloride (b) cadmium nitrate, along with (c) cadmium sulfate depicted in Figure 3 (a-c). Figure 3 (d) depicts the FTIR spectrum of the hibiscus rosasinesis stabilizer.

The broad peak observed in CdS FTIR spectra at around 3473.62 cm^{-1} indicates – OH stretching and N-H stretching [13,14].

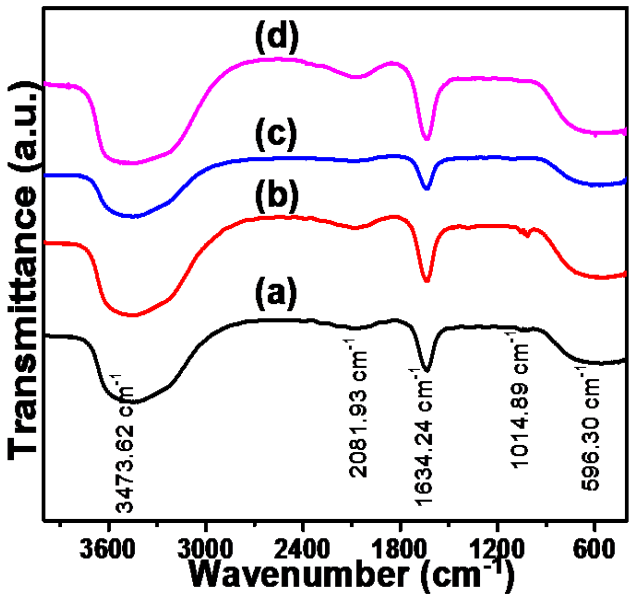

Fig. 3 FTIR spectrum of hibiscus rosa-sinesis leaf extract encapsulated CdSNPs synthesized out of possession of (a) cadmium chloride, (b) cadmium nitrate, (c) cadmium sulfate and (d) FTIR Spectrum of hibiscus rosa-sinesis stabilizer

The small broad peak in the vicinity of 2081.93 cm^{-1} is assigned to intermolecular hydrogen bonds or stretching of C-H groups. A small intensity sharp peak at 1634.24 cm^{-1} correlates to the bending vibration of water molecules. The small spike at about 1014.89 cm^{-1} contributed to the C-O stretching of the vibration of methanol. The peak around the lower wave number around 596.30 cm^{-1} is possibly because of Cd-S stretching.

3.4. Optical Studies

The UV-Visible Absorption spectra of hibiscus rosasinesis leaf extract stabilized

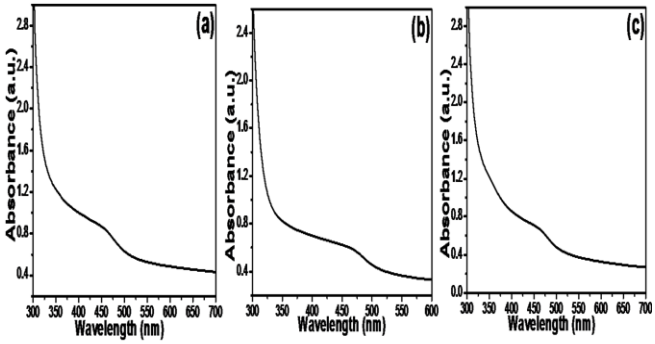

Fig. 4 UV- Visible Absorption graphs of hibiscus rosa-sinesis leaf extract prepared CdSNPs attained out of possession of (a) cadmium chloride (b) cadmium nitrate as well as (c) cadmium sulfate

CdS NPs are shown in Figure 4 (a-c) and contain absorbance on the vertical axis and wavelength on the horizontal axis. The onset absorption of the absorbance curve can be used to determine the energy band gap of CdSNPs. The absorbance curve of CdSNPs shows that the highest absorption occurred at wavelengths of 457 nm, 469 nm, and 462 nm, which are prepared from precursors of cadmium chloride, cadmium nitrate, as well as cadmium sulfate, respectively, all of which are in the visible range. According to Figures 4 (a), 4 (b), and 4 (c), the band gap values for these CdS NPs endure 2.71 eV, 2.64 eV, and 2.68 eV, correspondingly. Due to the quantum confinement effect, the energy gap of the

CdSNPs is greater than the 2.42 eV of bulk CdS.

4. Discussions

The CdS NPs prepared through green synthesis using cadmium precursors, including cadmium chloride, cadmium nitrate, and cadmium sulfate; for the sulfide source, sodium sulfide was used and hibiscus rosasinesis employed as a natural surfactant in the synthesis process. After recurrence with diverse sources of cadmium as well as sulfur in various molecular proportions, different reaction allotments, stirring rapidity, as well as multiple ratios of natural surfactant, it has been finalized that the time required for a reaction involving 0.1 M cadmium precursor as well as 0.1 M sodium sulfide at a stirring motion of 900 rpm is 4 hours gave the best outcome of the stabilizing agent of hibiscus rosasinesis leaf extract.

The XRD spectrums have verified the emergence of Cadmium Sulfide using hibiscus rosasinesis surfactant; the multiple broad XRD peaks clearly give the sign of NPs, and apart from this, there are some sharp peaks present which means the synthesized CdS powder contains both nano and microstructures. This result confirms that the presence of the natural surfactant significantly reduced the CdS NPs' crystallite size.

The band gap measurements are made from the UV-visible absorption spectra and obtained from rehearsed CdS powder band gap in the nano range. The FTIR spectroscopy observations of as-prepared CdSNPs confirmed the periphery changes of functional groups. The peak observed in the lower wavenumber range of less than 600 cm^{-1} of FTIR spectra proved the confirmation of Cd-S stretching, which is the fingerprint evidence for CdS. The CdSNPs exhibited a distinguished morphology along with some agglomeration. The poly dispersion of the material is possibly because of the deficit presence or lower proportionate of natural surfactant. The rich cadmium and sulfur in the EDS spectra of all samples signature the CdS NPs formation. Further, the agglomeration inculcated in the CdSNPs can be correlated to their large surface energy that evinces much that the surface interaction is caused by van der Waals forces. [15].

5. Conclusions

In overview, the CdS NPs have been prepared by the green synthesis method, which makes use of hibiscus rosasinesis leaf

extract as a natural stabilizer or capping agent reporting along with innumerable cadmium precursors being cadmium chloride, cadmium nitrate, and cadmium sulfate and sodium sulfide for the sulfide source are maneuvered. The reaction conditions are optimized to maximize the effectiveness of the natural stabilizer. There is also the formation of a thin layer of natural surfactant surrounding the surface of the CdSNPs. Quantum confinement effects and a blue shift in the optical absorption edge have reportedly been seen in CdSNPs fabricated from all three cadmium precursors. The CdS NPs appear to have been made from high elemental purity and have an average particle size of less than 10 nm.

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Conflict of Interest

The author declares no conflict of interest.

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