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Hydrothermal synthesis and defect-driven optical characterization of CdS nanoparticles for semiconductor and solar applications

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ABSTRACT

Nanoparticles (NPs) play a crucial role in advancing technology, particularly by enhancing the performance of energy storage in semiconductor applications. The synthesis of NPs with reduced particle size and increased surface area, along with a higher number of active sites, facilitates improved ion diffusion, making them highly suitable for such applications. Various methods have been employed to reduce the size of NPs, depending on factors such as purity and controlled composition. The present study focuses on controlling both the size and composition of cadmium sulfide (CdS) NPs, aiming to achieve a high surface-to-volume ratio. These NPs were synthesized using a hydrothermal method in a high-pressure autoclave. The evaluation of the synthesized inorganic CdS-NPs for technological applications requires experimental validation of their characteristics, including particle size, energy band gap, thermal stability, temperature response, as well as optical and electronic properties. The results obtained using the proposed methods reveal a bandgap of 2.28 eV, a hexagonal wurtzite structure with an average crystallite size of 10.26 nm, reduced effective mass, and an intense absorption peak at a higher wavelength. These characteristics indicate that the synthesized CdS nanoparticles are suitable for various applications, including high-power semiconductors, solar energy harvesting, optoelectronic devices, and materials for energy and electrical engineering.

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1. INTRODUCTION

Nanoscience and nanotechnology play a vital role across various scientific and technological domains. The composition of cadmium sulfide-nanoparticles (CdS-NPs) are widely used in medical, agricultural, and materials science applications [1]. Metal sulphide nanostructures are commonly employed as efficient photocatalysts in semiconductor nanostructures due to their ability to absorb a broad range of visible and/or UV light, as well as their favorable electronic structure and charge transport properties and

these NPs have also contributed significantly to advancements in solar energy technologies, energy conservation, and water treatment processes. Among the different types of NPs, metal and metal sulfide NPs are particularly noteworthy due to their broad applicability in addressing environmental challenges and enhancing energy efficiency [2]. Group II–VI semiconductor NPs, such as CdS, are especially preferred because they can be synthesized easily within a controlled size range.

CdS NPs, characterized by a wide bandgap of 2.4 eV, have a broad range of applications across multiple scientific and technological domains. They are extensively employed in the fabrication of photodetectors [3], [4], solar cells [5], photocatalysts [6], nonlinear optical materials [7], and in antimicrobial applications [8], [9]. CdS NPs or quantum dots are widely recognized among reported materials for their exceptional optical and electrical properties, enabling their use in diverse fields such as bio-sensing, bio-imaging, photovoltaic cells, nano-medicine, molecular pathology, and drug delivery [10]. In addition, the unique optical properties of CdS NPs, often referred to as quantum dots, support their use in a wide variety of applications, including bio imaging, bio sensing, nano medicine, photovoltaic devices, and targeted drug delivery systems [11], [12].

Various oxide-based nanomaterials can be synthesized using a range of methods that offer flexibility, cost-effectiveness, ease of handling, and consistent quality [13]-[16]. Among these, green synthesis using plant extracts has attracted considerable attention due to its environmentally friendly nature. For example, the use of *Ixora coccinea* extract in the biosynthesis of titanium dioxide (TiO_2) NPs represents a sustainable approach for producing materials with potential applications in organic electronics, merging ecological sustainability with advanced functional performance [17]. The hydrothermal method is another widely employed technique, offering several advantages such as controlled particle size, enhanced stability, and high purity. This method typically operates at temperatures below 450 °C, minimizing electron conduction losses and thereby improving the performance of semiconductor devices and solar energy systems. Extensive research has been conducted on CdS NPs, focusing on their synthesis techniques and detailed investigations of their structural and optical properties. These studies frequently utilize characterization techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), and photoluminescence (PL) spectroscopy to gain insights into their morphology, crystallinity, and emission behavior [18]-[20].

2. EXPERIMENTAL RESULTS

2.1. Synthesis of CdS-NPs

Initially, 0.75 M of cadmium acetate dihydrate ($Cd(CH_3COO)_2•2H_2O$), used as the cadmium source, was dissolved in 50 mL of distilled water and stirred at 60 °C for approximately 2 hours to promote dissociation into Cd^{2+} ions. In a separate beaker, 2.56 M of sodium sulfide (Na_2S) was dissolved in 50 mL of distilled water and stirred at 60 °C for 1.5 hours to generate sulfide ions (S^{2-}). Subsequently, the two precursor solutions were slowly combined at 60 °C and briefly stirred to ensure homogeneity. The resulting mixture was then transferred into a teflon-lined stainless-steel autoclave and sealed. The hydrothermal reaction was carried out at 170 °C for 24 hours, during which the precursors underwent nucleation and growth processes leading to the formation of CdS-NPs.

The elevated temperature and pressure within the autoclave facilitated the controlled synthesis of CdS-NPs. After the reaction, the product was repeatedly washed with distilled water using an ultrasonic cleaner to eliminate impurities and any unreacted precursors. The purified material was then dried overnight at 50–60 °C to remove residual moisture. The final product, a yellow powder, was identified as the synthesized CdS-NPs.

2.2. Characterization

Advanced techniques have been developed to analyze various properties of NPs. In this study, the CdS NPs characterization was performed using six primary methods: X-ray diffraction (XRD), scanning electron microscopy (SEM), differential thermal analysis (DTA), thermos-gravimetric analysis (TGA) (Model-STA7300-Hitachi, Japan), UV-visible spectroscopy (Model UV-2450 Shimadzu Japan), and photoluminescence (PL) spectroscopy. These techniques confirmed the presence of the NPs and provided valuable insights into their size, shape, and structural properties.

XRD patterns were obtained using a Bruker AXS D8 advance diffractometer in the Bragg–Brentano focusing geometry, employing Cu-Kα radiation (λ = 1.54060 Å) as the X-ray source. The instrument operated at 40 kV and 30 mA, with diffraction patterns recorded at a scanning rate of 5.00°/minute over a 2θ range of 10°–80 °C. The SEM (Model make is Zeiss special addition -18 make) was used for in situ analysis of micro areas. The DTA (Model: STA7300 Make: Hitachi, Japan) was used to monitor and record the temperature of the furnace, and the difference in temperature between the sample and the reference.

Optical absorption was recorded with the help of UV-VIS 8400 S spectrometer. PL properties of the CdS NPs were studied using a Shimadzu RF-5301PC Spectrofluoro photometer.

3. RESULTS AND DISCUSSION

3.1. X-ray diffraction studies

The method is based on the constructive interference of incident X-rays on a sample, satisfying Bragg's law (2d sin θ = nλ). This technique provides valuable information about the phase identification and unit cell dimensions of the material. The study provides information on crystalline phases of NPs with narrow peaks in the diffraction pattern. The synthesized CdS NPs were analyzed using Cu-Kα radiation (λ = 1.54060 Å), and the results matched the JCPDS Card No. 41-1049. Figure 1 represents the XRD pattern of the synthesized compound with specific Miller indices corresponding to maximum intensity and angle of diffraction. The sample is indexed with a hexagonal wurtzite crystal structure with a maximum peak at an angle of 26.6° having Miller indices (002). The Scherrer formula was used to determine the crystallite size, given by (1).

$$D = \frac{0.9\,\lambda}{\beta cos\,\theta} \text{ ———} \quad (1)$$

The symbol β denotes the full width half maximum, λ is the wavelength of the X-ray diffraction source, and θ is the angle of diffraction. The average value of crystalline size is found to be 10.26 nm. The reduced size of NPs with narrow peaks is due to quantum confinement and reduced scattering applicable for electronic properties. The (hkl) values, full width at half maximum (FWHM), d-spacing, and relative intensity of the concerned JCPDS card are given in Table 1.

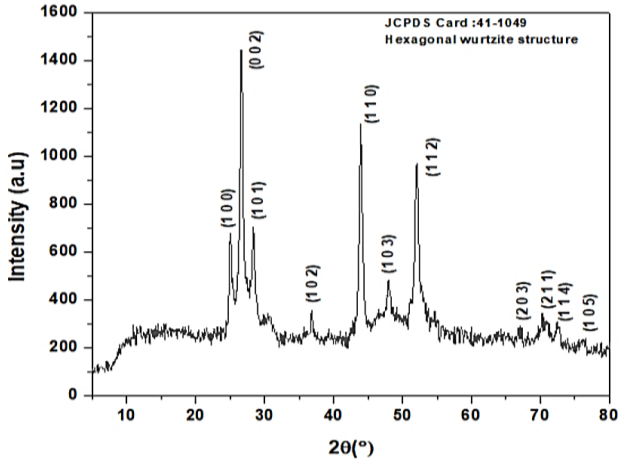


Figure 1. XRD pattern of synthesized CdS NPs

Table 1. XRD data illustrating position, (hkl) values, FWHM, relative intensity, and d-spacing

2θ	hkl	Counts	Peak height	FWHM	Relative intensity	d-spacing
25.05	101	641	335.3	0.48	44.76	3.1632
26.62	002	1432	1000	0.48	100	3.3745
43.94	110	1041.76	746.2	0.64	72.69	2.0674
52.06	112	950.11	653.2	0.64	66.34	1.7629
70.87	211	337.74	87	0.48	23.5	1.3270
72.51	114	311	82	0.8	21,71	1.3072

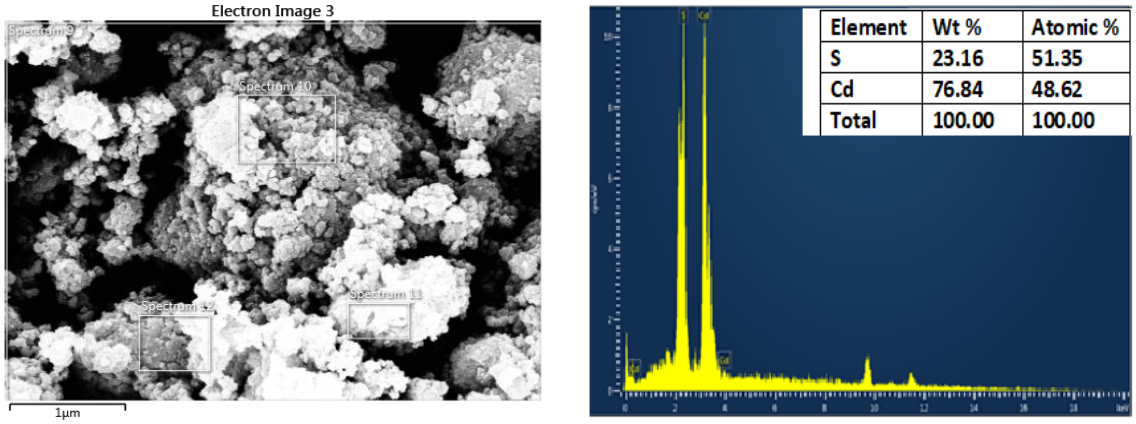
3.2. Surface morphology

SEM offers detailed insights and delivers high-resolution images of the sample at significant magnification levels on the surface, while energy-dispersive spectroscopy (EDS) gives information about chemical composition and weight percentages of elements present in the sample. It relates to the topography on the nanoscale that influences physical and chemical properties and interactions between them. The image

of CdS NPs with a dense morphology captured at one µm size is presented in Figure 2(a), and the elemental composition of the same is illustrated in Figure 2(b), as identified through EDS analysis. The EDS data confirm the presence of chemical elements such as sulfur (S) and cadmium (Cd), which are key components of the synthesized NPs.

3.3. DTA and TGA

Differential thermal analysis is a thermal analysis technique used to characterize materials by monitoring their thermal behavior. The process involves heating or cooling both a sample and a reference material simultaneously at a linear heating rate, while recording the furnace temperature and the temperature difference between the sample and the reference. As shown in Figure 3, desorption is observed as an endothermic shift at 258.8 °C with a heat flow of 23.980 µV, as determined through DTA. Thermo-gravimetric analysis involves measuring the weight of a sample as it either loses or gains mass under specific conditions, with the temperature being increased at a predetermined rate. In the temperature range of 90.1 °C to 503.6 °C (a thermal range of 413.5 °C), the CdS NPs exhibit a noticeable response with a mass loss of 0.997 mg, decreasing from 1.966 mg to 0.969 mg. Between 503.6 °C and 713.4 °C, the sample experiences a small increase in mass of approximately 123 µg, likely due to the buoyancy effect. Beyond this temperature range, no further change in the weight of the CdS NPs was observed, indicating a linear stability in the material.



(a) (b)

Figure 2. Morphological and compositional characterization of CdS nanoparticles: (a) SEM image of CdS nanoparticles and (b) EDS spectrum showing chemical composition and weight percentages

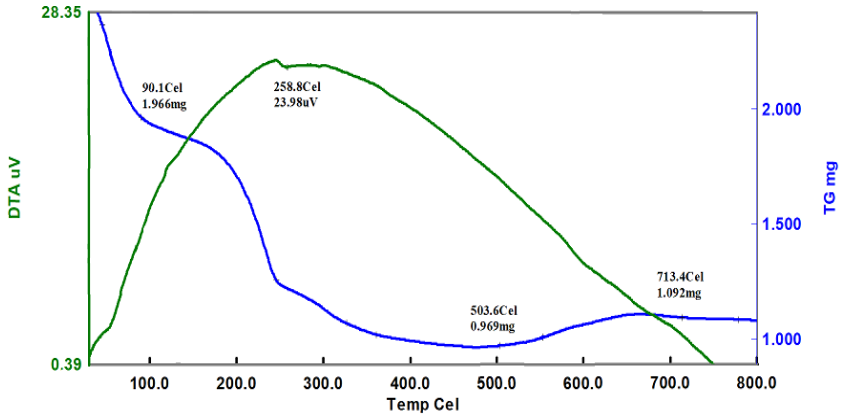


Figure 3. DTA/TGA spectra of CdS NPs

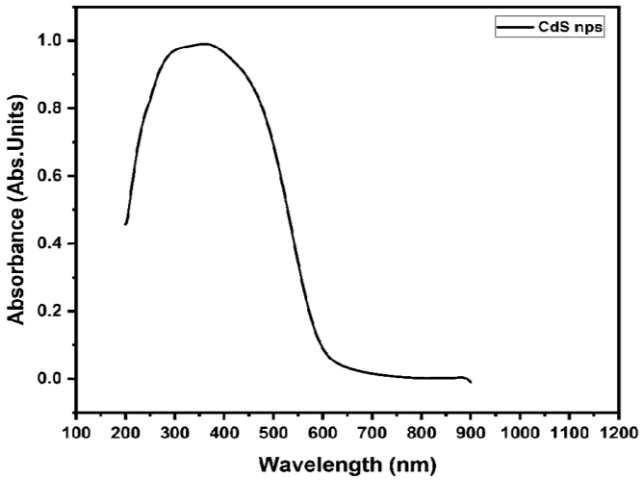
3.4. Optical absorption studies

An effective method determines size and optical properties in the range 200-600 nm at room temperature with stable dispersions after ultrasonication. In this technique, the amount of light absorbed by a sample is directly proportional to the concentration of the absorbing compound. It is widely used to analyze

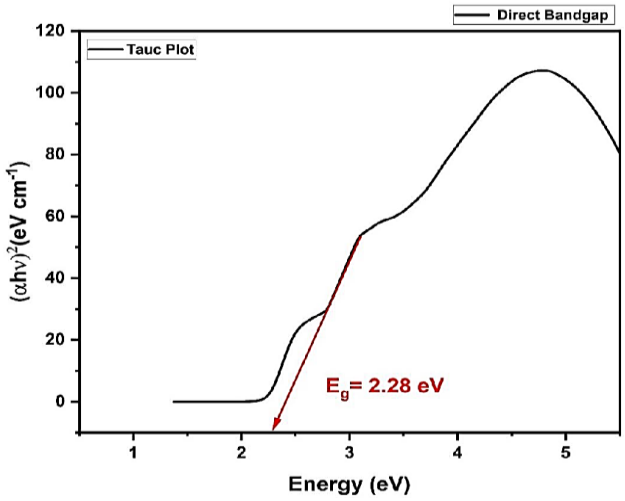
the absorption of light by substances over a range of wavelengths in the UV and visible regions. CdS NPs exhibit a sharp absorption peak between 350 and 450 nm in the UV-visible diffuse reflectance spectrum, measured across a wavelength range from 100 to 1200 nm, as shown in Figure 4(a). This peak indicates an enhanced UV light absorption capacity. Pandian *et al.* [21] investigated the absorbance spectra of CdS NPs and highlighted their optical properties within the wavelength range 371-396 nm, showing a red shift in the absorption wavelength. A strong absorption band in the UV region was observed at a wavelength of approximately 345 nm, which is significantly blue-shifted compared to the absorption edge of bulk cadmium sulfide. As the particle size decreases, the peak shifts towards the lower wavelength side with increased bandgap due to quantum size confinement. The transition associated with electrons across the bandgap is determined by a change in the optical absorption coefficient with wavelength. A graph plotted between photon energy (hν) and $(\alpha h\nu)^2$ is used to determine the energy bandgap of the CdS NPs by (2).

$$\alpha h\nu = A(h\nu - Eg)^n \quad (2)$$

The Tauc plot shown in Figure 4(b) represents the energy gap of CdS NPs, providing a direct bandgap energy of approximately 2.28 eV. This appreciable bandgap enables NPs to be sensitive and applicable for optoelectronic applications in visible and near infrared regions. This electronic property is crucial for the semiconducting behavior of CdS NPs significantly influencing their potential applications in electronic and optoelectronic devices. The bandgap value also indicates the suitability of CdS NPs for photocatalytic applications, particularly under visible light irradiation.



(a)



(b)

Figure 4. Optical characterization of CdS nanoparticles (CdS NPs): (a) absorbance curve of CdS NPs and (b) energy gap of CdS NPs

3.5. Photoluminescence studies

PL spectroscopy is a widely used technique for investigating the optical properties of materials by analyzing the emitted light after excitation with photons. As far as the implications for device integration and stability under illumination are concerned, sulfur vacancies (V_S) defects enhance sub-bandgap absorption but raise recombination losses, lowering voltage and responsivity. Defect emissions broaden spectra and reduce color purity; prolonged illumination accelerates degradation via photo-oxidation. Moderate V_S improves visible-light absorption and charge separation, but excess defects cause trap-assisted recombination and instability. Figure 5 presents the PL spectrum of CdS NPs synthesized via the hydrothermal method. This spectrum provides insights into the excitation and emission wavelengths, as well as the intensity of the emitted radiation. The intensity is plotted as a function of wavelength over the 300-600 nm range. A distinct peak appears at 365 nm in the UV region, while another peak is observed at 505 nm in the visible region. Alipour *et al.* [22] reported PL studies on CdS NPs, identifying emission peaks at 370 nm and 700 nm, along with weaker peaks in the 400–500 nm range, which were attributed to band transitions in CdS NPs. The peaks around 500 and 700 nm are associated with band-edge emission and trap-state emission, respectively [23].

The uniqueness of the study is the existence of an additional peak that addresses defects in NPs that act as trapping centers for charge carriers, not only in the visible region at 505 nm, but also at 365 nm, obtained for synthesized NPs by hydrothermal synthesis. These trapped centers are localized energy states within the material that arise due to the quantum size effect with a controlled bandgap [24]. Synthesis of NPs by the hydrothermal method performed at low temperatures (less than 450 °C) with reduced electron conduction influences the performance of the device, suitable for semiconductor and solar energy applications [25]. The radiative bands at higher and lower wavelengths arise due to sulphur vacancies and recombination due to surface-trapped sites, in addition to residual impurities.

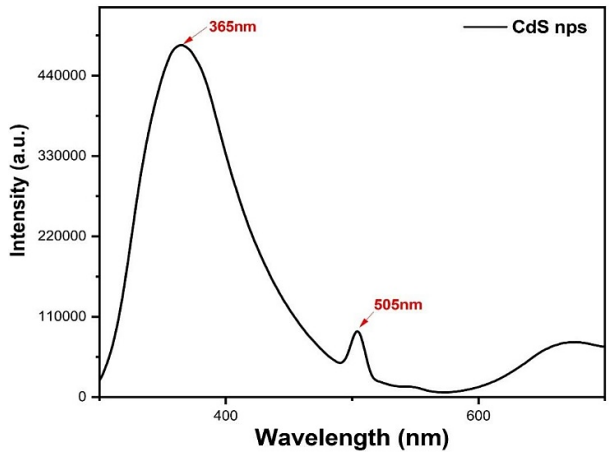


Figure 5. Photoluminescence spectrum of CdS NPs

4. CONCLUSION

CdS-NPs were successfully synthesized via a hydrothermal method without the use of any capping agents, highlighting the simplicity and cost-effectiveness of the process. Surface morphology analysis revealed densely packed structures, while elemental analysis confirmed the successful formation of CdS NPs. X-ray diffraction patterns exhibited sharp and narrow peaks, corresponding to an average crystallite size of approximately 10.26 nm. DTA showed an endothermic peak at 258.8 °C with a heat flow of 23.980 µV, attributed to the desorption process that facilitates the enhancement of photocatalytic reactions. The optical absorption spectrum displayed a distinct peak at 365 nm, with a calculated energy bandgap of 2.28 eV, indicating the semiconducting nature of the material and the presence of surface Plasmon resonance. Photoluminescence spectroscopy revealed emission peaks at 365 nm in the UV region and 503 nm in the visible region, further confirming the optoelectronic activity of the synthesized NPs. Overall, the synthesized CdS-NPs demonstrate promising characteristics suitable for applications in optoelectronic devices, biosensors, solar energy conversion, and photovoltaic technologies. Future work will focus on exploring their thermos-luminescence properties and calculating trap depth to evaluate their suitability for dosimetric applications.

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AUTHOR CONTRIBUTIONS STATEMENT

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Name of Author	C	M	So	Va	Fo	I	R	D	O	E	Vi	Su	P	Fu
Deepti Bhargava	✓							✓	✓		✓		✓	
R.K. N. R. Manepalli		✓				✓				✓		✓		
M. C. Rao	✓		✓	✓		✓				✓	✓		✓	
P. Venkata Ramana Rao	✓						✓	✓	✓					
N. S. Subba Rao	✓						✓	✓	✓					
A. Narendra Babu	✓						✓	✓	✓					
P. Sree Brahmanandam	✓					✓			✓	✓		✓		

C : **C**onceptualization
M : **M**ethodology
So : **So**ftware
Va : **Va**lidation
Fo: **Fo**rmal analysis
I : **I**nvestigation
R : **R**esources
D : **D**ata Curation
O : Writing - **O**riginal Draft
E : Writing - Review & **E**diting
Vi : **Vi**sualization
Su : **Su**pervision
P : **P**roject administration
Fu : **Fu**nding acquisition

CONFLICT OF INTEREST STATEMENT

Authors state no conflict of interest.

DATA AVAILABILITY

The data that support the findings of this study are available on request from the corresponding author. The data, which contains information that could compromise the privacy of research participants, is not publicly available due to certain restrictions.

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