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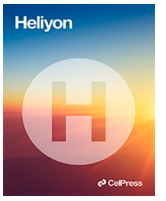

Research article

Thermal annealing gradient tailoring on ammonium nitrate-capped CdS nanospheres synthesized via green precipitation

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A B S T R A C T

This study explores the impact of thermal annealing gradients on the physical properties and structural evolution of cadmium sulphide (CdS) nanospheres capped with ammonium nitrate as a modifier, which were fabricated through precipitation and subsequent annealing within 160–480 °C temperature range. The properties were characterized using X-ray diffraction (XRD), ultraviolet–visible (UV–Vis), Fourier transform infrared (FTIR), scanning electron microscopy (SEM), thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) techniques The XRD results show that the present CdS exhibits superior crystallinity compared to pure CdS without capping, transitions from a cubic to a hexagonal phase structure, and increases in crystallite size and crystallinity with increasing temperature. The FTIR spectra postulate that a vibrational band presence evidences ammonium nitrate capping on CdS, with another distinct band that represents CdS in the lower wavenumber region, both intensifying at elevated temperatures. The UV–Vis analysis reveals that CdS exhibits strong ultraviolet (UV) absorption suitable for effective photoreaction under UV light and has a broader band gap compared to bulk CdS. SEM images show an extensive distribution of homogeneous nanospheres over the surface, with increased growth in size when capped with ammonium nitrate and at higher temperatures. As validated by TGA and DSC results, CdS with a smaller crystallite size improves thermal stability and energy transfer, as evidenced by reduced weight loss and a lower endothermic temperature, respectively. Varying the annealing temperature with ammonium nitrate capping can improve the structural and physical properties of CdS, which are beneficial for varied applications such as optoelectronics, energy storage, and photocatalysts.

1. Introduction

Nowadays, nanomaterial fabrication and characterization have received substantial interest because of their typical features and extensive prospects for numerous potential applications in technology. Examples include photovoltaic devices, optoelectronics, light-emitting diodes, and transistors. Meanwhile, semiconductor-based nanomaterials exhibit extraordinary electrical, optical, piezoelectric, and thermal properties, which substantiate a vital contribution to numerous applications due to their exceptional properties. Researchers are increasingly interested in synthesizing semiconductor structures in the nanometer range, which are less than or about 100 nm in size, due to the specific properties of these nano-dimensioned semiconductors in comparison to the bulk ones [1–8].

Cadmium sulphide (CdS) is an inorganic semiconductor substance with a band gap of 2.42 eV that belongs to the II-VI compound family. CdS has undergone extensive study attributed to its tunable band gap, intense optical absorption, high reaction efficacy, reliability, and affordability. Thus, CdS has emerged as a crucial member of the metal chalcogenide family. Besides, researchers have thoroughly studied the impact of various reaction parameters, solvents, and surfactant types on the nanomorphology of CdS. Hence, various methods have been developed to synthesize CdS in nanoscale dimensions, resulting in multiple shapes and structures like nanospheres, nanorods, nanocrystals, nanotubes, nanowhiskers, nanowells, nanowires, etc. Synthetic methods such as chemical

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precipitation, one-pot synthesis, photochemical, hydrothermal, and solvothermal produce various physical and chemical properties. Among them, dropwise precipitation is considered one of the most simple, efficient, practical, and cost-effective methods for fabricating CdS [1,5,9–15]. Ammonium nitrate is well recognized as an essential sustainable green inorganic substance since it comprises environmentally friendly nitrogen, hydrogen, and oxygen components and is widely utilized as a fertilizer, antibiotic, and yeast nutrition. From the literature, Pingali et al. [9] have found that adding ammonium nitrate to spraying solutions during spray pyrolysis can evidently decrease the size of Ag, Ru, and Ni nanoparticles from 200 nm–300 nm to 10 nm–20 nm, indicating that ammonium nitrate would act as an effective capping agent for size reduction and nanoparticle formation.

Until now, the current reported fabrication of CdS nanostructure from precursors still necessitates high temperatures and stringent experimental conditions. Therefore, further development and advancement in this subject are still needed, especially for the preparation of CdS with low-cost efficiency and unique properties. Through this investigation, the CdS nanoparticles were synthesized by dropwise precipitation and subsequent heating of the material at varying temperatures between 160 °C and 480 °C. The chemical synthesis involved a reaction between thiourea and cadmium chloride, with thiourea as a S source and cadmium chloride as a Cd source, and potassium hydroxide (KOH) used as an alkali medium for ion production in the solution. Due to the low solubility of CdS, ammonium nitrate (NH_4NO_3) was added to the solution as a capping agent in the reaction, which functions as a buffer and pH controller. The crystalline structure, surface morphology, optical and thermal properties of CdS were characterized in detail in this study.

2. Materials and method

2.1. Reagents

All the necessary raw ingredients used for material synthesis were provided by Sigma Aldrich and consumed directly without any special treatment. Cadmium chloride ($CdCl_2$), thiourea $CS(NH_2)_2$, potassium hydroxide (KOH) and ammonium nitrate (NH_4NO_3) were utilized as received from Sigma Aldrich without additional purification.

2.2. Synthesis procedure

Ammonium nitrate serves as a surfactant and capping agent during reactions in order to facilitate product solubility, avoid agglomeration of particles, and maintain the pH solution at a desirable level. Initially, the solutions listed above were mixed with the appropriate concentrations, and then thiourea (0.10 M) was slowly added until the solution turned yellow. Next, the solution was heated at a constant temperature of 80 °C for 2 h, with the stirring remaining at 300 rpm, until a yellow precipitate was visible at the bottom of the solution. After that, the precipitate was obtained through a filtration process, washed several times with distilled water and acetone, and then dehydrated at ambient temperature for one day. Finally, CdS was annealed in a furnace for 3 h at various temperature gradients, namely 160 °C, 320 °C, and 480 °C, then left to cool under ambient conditions before subsequent measurements were conducted. The schematic diagram in Fig. 1 depicts the synthetic procedure for CdS nanospheres.

2.3. Properties characterizations

The final freestanding powder product was characterized using several instruments, as stated below. XRD (Rigaku) was utilized to elucidate the sample crystallinity, crystalline structure, and content purity of CdS nanoparticles, as well as for evaluating their crystallite size. FTIR (Bruker Invenio) was used to assess the functional groups and structural bonding, while SEM (Vega Tescan) was performed to gain insight into the surface morphology of the CdS nanoparticles. The optical absorption spectra of the samples were

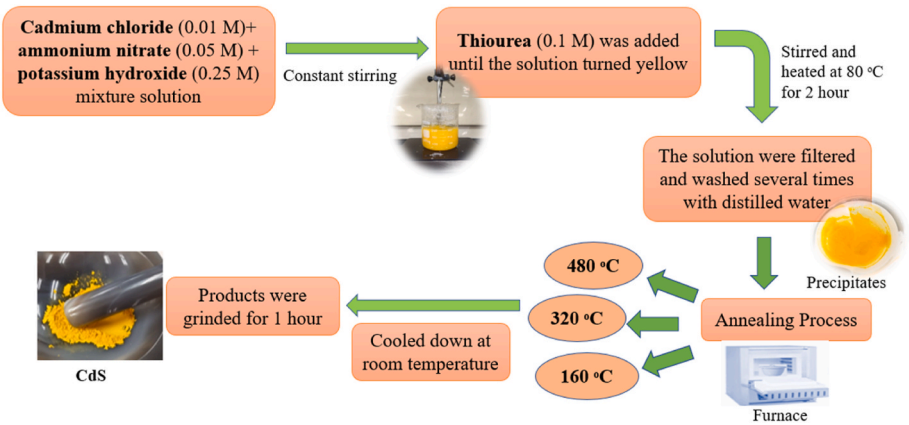

Fig. 1. Schematic synthetic procedure of CdS nanospheres.

accessed using a UV–vis spectrophotometer (UV-1800, Japan). The thermal stability and transition were characterized using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC).

3. Results and discussion

3.1. Compositional analysis

The FTIR analysis was conducted to better understand the functional groups and intermolecular bonding of the CdS nanospheres capped with ammonium nitrate as the surfactant. Fig. 2 exhibits the FTIR spectra of CdS created at various annealing temperatures within the wavenumber range of 4000 to 400 cm^{-1}. By referring to the figure, the absorption band that emerged within a region of 3500 cm^{-1} to 3000 cm^{-1} might represent the O–H stretching vibrations due to the effect of water molecules trapped on the sample surface [16–26]. Meanwhile, it is obvious that increasing the annealing temperature reduces the intensity of the aforementioned peak. This is because the water molecules released from the molecular surface vanish at higher annealing temperatures. Besides, the sample annealed at 160 °C facilitates easier water molecule reattachment to the surface after being removed from the furnace and allowed to cool naturally. There is a relatively small peak observed at 2350 cm^{-1}, attributable to the vibrational mode of the S–H bond [5,19], whereas a peak detected at position 2000 cm^{-1} can be associated with the C–H bending mode [2,18,27]. The vibrational peak located at 1630 cm^{-1} correlates with surrounding CO_2 gas molecules adsorption on the particle surface. The CO_2 tends to detach from the surface, as evidenced by the peaks that diminish at higher annealing temperatures. Indeed, water and carbon dioxide molecules more conveniently adhere to powder samples exposed to the surrounding environment, particularly nanosized particles that possess an enhanced surface area. Aside from that, the peak protruding at 1100 cm^{-1} can be ascribed to NH bending modes (amine) due to residual ammonium nitrate capped on the CdS molecular surface [2,4,21]. Interestingly, the related peak does not appear on the spectrum of pure CdS prepared at ambient temperature without being capped with ammonium nitrate. This means that the acquired results are consistent, implying that ammonium nitrate was effectively capped on the surface of the CdS sample at various annealing temperatures. Besides, as the temperature rises, the capping effect becomes more efficient due to the stronger peaks found, which is beneficial for infrared optical transmission. Furthermore, all the samples exhibit a broadband centred around 615 cm^{-1} within the lower wavenumber region of 400–700 cm^{-1}, which mainly represents the Cd–S stretching vibrational mode [5,6,23,28]. This peak becomes stronger and more dominant at elevated temperatures because the CdS molecules are more regularly arranged, as evidenced by XRD analysis, which is responsible for the light transmission in order to penetrate more easily throughout the structure and hence intensifies the peak intensity, which becomes stronger and sharper. Meanwhile, the finding also reveals that when the annealing temperature increases, the CdS tends to convert into its purest form without any unidentified compounds traced, simply because many other organic molecular bonds have already disappeared or weakened, for instance, the O–H bonds between water molecules and the absorbed CO_2 molecules.

3.2. Structural analysis

Fig. 3 demonstrates the XRD patterns of ammonium nitrate-capped CdS nanospheres annealed at different temperatures. From the figure, three dominant peaks are clearly distinguished at (111), (220), and (311), respectively, for the samples annealed at a temperature of 160 °C. This peak group corresponds instantaneously to the JCPDS card file (75–1546) regarding the cubic phase structure. The widening of the peaks indicates that tiny nanospheres are generated at 160 °C, as compared to pure CdS without capping. Besides,

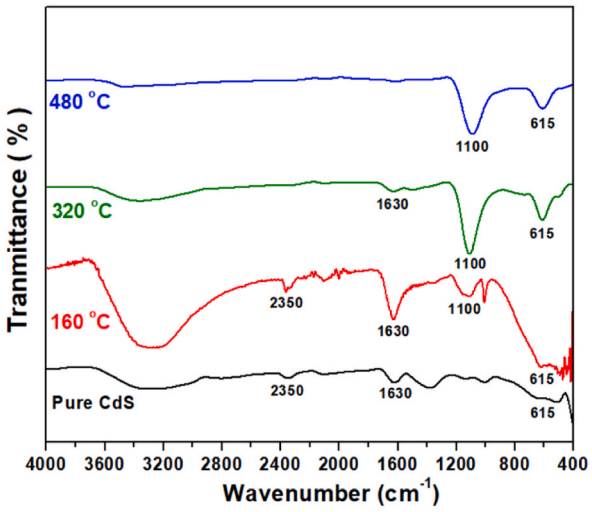

Fig. 2. FTIR spectra of ammonium nitrate-capped CdS nanospheres annealed at different annealing temperatures.

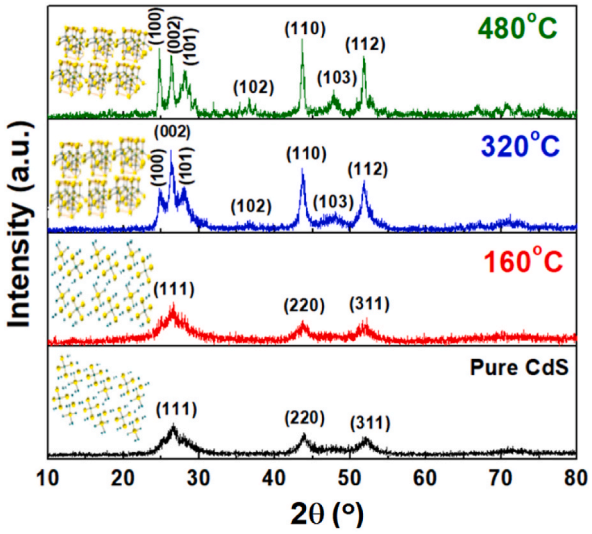

Fig. 3. XRD pattern of ammonium nitrate-capped CdS nanospheres synthesized at varying annealing temperatures.

as for CdS annealed at temperatures of 320 °C and 480 °C, the occurrence of seven diffraction peaks is clearly observed at $2\theta = 24.84^{o}$, 26.44^{o}, 28.06^{o}, 43.53^{o}, 47.82^{o}, 51.62^{o}, and 70.53^{o}, which belong to (100), (002), (101), (110), (103), (112), and (211) miller indices, respectively. These peaks represent the formation of hexagonal-type CdS structures, as designated by the JCPDS card number (80-6), which indicates the evolution of CdS from a cubic phase to a hexagonal phase [18,20,21]. Interestingly, normal small crystallites exhibit a cubic lattice at lower temperatures, which is considered a noteworthy observation. Nevertheless, the structure exhibits a preferred tendency for hexagonal phase growth, with more aggregated nanoparticles formed at higher temperatures [15,17,22,29]. Therefore, such findings demonstrate that the proportion of hexagonal structure increases with temperature, while CdS annealed up to 480 °C has completely transformed into the pure hexagonal phase [11,23,30]. The broadening of XRD bandwidth implies that the samples consist of ultrafine particles, which are crystalline and nanosized in nature, while the numerous diffracted crests in the diffractogram denote the polycrystalline nature of CdS. In contrast, pure CdS without ammonium nitrate capping shows a cubic phase with lower crystallinity and a narrower bandwidth, as compared to other CdS samples. Afterwards, the crystallite size was determined utilizing the Scherrer-Debye equation (1), as given underneath [8,9,26]:

$$D = 0.9\,\lambda / \beta \cos\theta \tag{1}$$

where D is the crystallite size, λ represents the wavelength of CuKα X rays, β indicates the full width at half maximum (FWHM) and θ is the diffracted Bragg's angle [3,16]. The crystalline phase and average crystallite size of the present CdS nanospheres are tabulated in Table 1. This result indicates that the size of crystallite increases slightly with an increase in annealing temperature from 160 °C to 480 °C, accompanied by the transformation of the cubic into a hexagonal structure. The previous work reported by Sivasubramanian et al. [19] supports this finding, which revealed that particle size increases and a crystalline phase transition occurs with annealing temperature. Meanwhile, it is worth noting the tunability of peak widths and intensities, as, for instance, when the sample is annealed at 480 °C, the peaks of the (100) and (101) planes become sharper and more pronounced. Moreover, the peaks located at (102) and (103) become more obvious and stronger as compared to the one annealed at 320 °C, which is relatively broad and insignificant.

3.3. Optical properties

UV–visible spectroscopy has become a powerful tool for capturing optical absorption spectra. Fig. 4 displays the UV–visible absorption spectra of the present ammonium nitrate-capped CdS nanospheres at varying annealing temperatures. The studied UV–visible region exhibits well-resolved smooth and continuous spectral curves, indicating good optical absorption of CdS nanospheres with homogeneous and extensive size distributions, as also confirmed by the SEM images. The absorbance is most prominent in the ultraviolet region less than 400 nm, then the intensity gradually diminishes at higher wavelengths and finally flattens towards the visible

Table 1
Structural parameters determined for the present CdS nanospheres.

Annealing Temperature (°C)	Average crystallite size (nm)	Crystalline phase	Peak Intensity
Pure CdS	3.21	Cubic	Moderate
160	2.31	Cubic	High
320	7.89	Hexagonal	Strong
480	14.64	Hexagonal	Very Strong

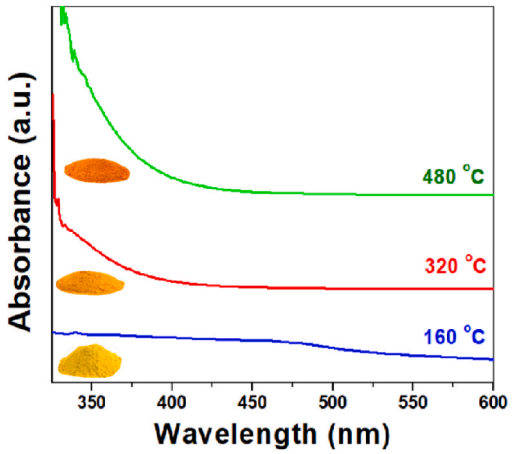

Fig. 4. UV–visible absorption spectra of present ammonium nitrate-capped CdS nanospheres.

Afterwards, the band gap energy (E_g) can be calculated by Tauc's model, which is given by equation (2).

region. Also, when CdS is annealed at 160 °C, it shows a maximum absorption at 244 nm. Subsequently, as annealing temperatures elevate up to 320 °C and 480 °C, a small and sharp absorption peak is visible in the UV region at a wavelength position of 330 nm. The prominent UV absorption suggests that UV light excitation can further stimulate the interactions of CdS with other photosensitive

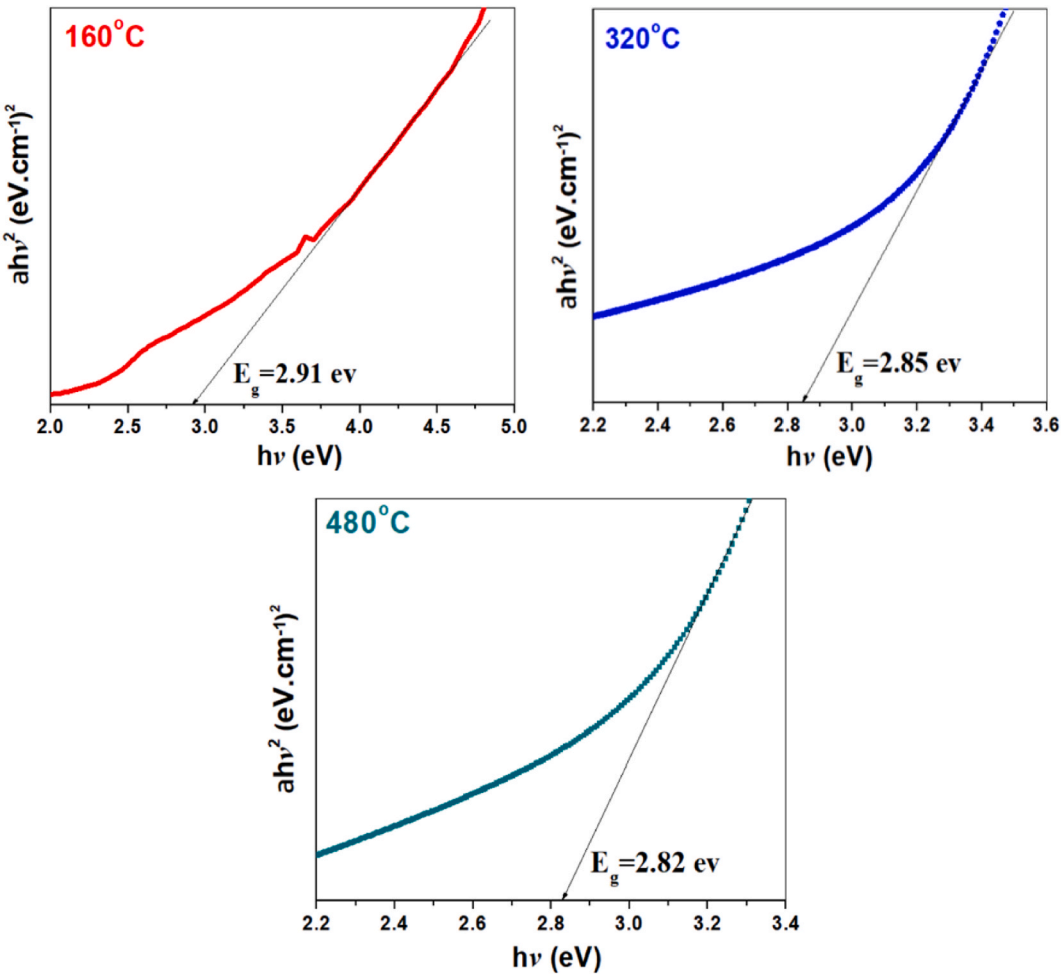

Fig. 5. Band gap determination of present ammonium nitrate-capped CdS nanospheres.

compounds, which may assist in promoting efficient photochemical or photocatalytic reactions. Besides, it is worth noting that the absorbance intensity of CdS in the UV–visible region strengthens as the annealing temperature rises. This phenomenon is primarily driven by an increase in molecule absorption centers due to the formation of larger crystallite sizes with reduced surface imperfections. Beneficially, increased absorbance can enhance photon capture ability during photocatalytic reactions and, in turn, result in an increase in the charge carrier transition efficiency [23,31,32].

$$(\alpha h\upsilon) = A\ (h\upsilon - Eg)^{n} \quad (2)$$

where n = ½ is designated for direct band gap semiconductor material, A represents a proportionality constant, α is an optical absorbance, and (hυ) denotes the photon energy [2,27,28]. When $(\alpha h\upsilon)^2$ is plotted as a function of (αhυ), as shown in Fig. 5, resulting in E_g at the point where the proportional portion of the linear plot is extended to $(\alpha h\upsilon)^2 = 0$. Besides, the band gap, absorption peak and crystallite size of the CdS nanoparticles prepared at various annealing temperatures are recorded in Table 2. The band gap of the CdS nanosphere capped with ammonium nitrate produced in KOH solution, ranging from 2.82 eV to 2.91 eV, is found to be significantly higher than that of the literature value of bulk CdS (2.40 eV), and is also a little larger than the values of (2.16–2.60) eV as reported in the work by Sheng et al. [31], in which the CdS was produced in NaOH without any capping agent. The finding demonstrates that the band gap of CdS becomes smaller at higher annealing temperatures due to an increase in crystallite sizes resulting from aggregation, as affirmed by XRD analysis. A narrower band gap is beneficial for photocatalytic activity since less photon energy is required to photoexcite the charge carrier transition for instantaneous degradation activity on organic pollutant molecules.

3.4. Surface morphology

The scanning electron microscope (SEM) was used to investigate the surface morphology of the ammonium nitrate-capped CdS nanospheres. SEM micrographs of CdS grown at different annealing temperatures are depicted in Fig. 6. From the figure, it can be clearly seen that the surface of the CdS nanoparticles is smooth and homogeneous, accompanied by an extensive outgrowth of CdS nanospheres in a conventional manner. The SEM images also reveal that the nanostructure has uniform spherical sizes lying between 67 nm and 130 nm. The average nanoparticle size is determined to be about 67 nm, 85 nm, and 98 nm for the samples annealed at various temperatures of 160 °C, 320 °C, and 480 °C. The trend shows that the size rises with annealing temperature, owing to the phase transition of the cubic into a hexagonal structure, which is compatible with XRD analysis. In addition, the average nanoparticle size is smaller than the CdS nanoparticles without capping, as reported by Sheng et al. [31], which is obtained within 70–150 nm. This result indicates that the ammonium nitrate capping effectively reduces the CdS particle size, which is useful for an effective catalytic reaction. On the other hand, these nanoparticles might be built up by the aggregation of numerous small nanocrystallites, as previously scrutinized by Debyde-Scherer's method. Worthily, high-temperature annealing causes nearby nanocrystallites with high surface energy to converge due to weak forces, which reduces total surface energy and results in agglomeration with larger particles or grain formation [23]. However, pure CdS without capped ammonium nitrate has a finer size, with an average diameter of approximately 58 nm, as compared to the sample with annealing treatment. Advantageously, ammonium nitrate has prevented the CdS nanospheres from clumping during heating and has retained their initial shape, albeit with a larger size.

3.5. Thermal stability

The thermal analysis, including TGA and DSC was carried out on ammonium nitrate-capped CdS nanospheres contained in a dynamic atmosphere of vaporous oxygen at a flow rate of 30 ml min^{-1} while heating from 30 °C to 700 °C at a rate of 10 °C min^{-1}. The TGA curves of the CdS synthesized at various annealing temperatures are depicted in Fig. 7(a). According to the figure, the sample annealed at 160 °C demonstrates the highest thermal stability of the prepared nanoparticles, with weight reduction being exceptionally low and progressively stable up to 450 °C. This result is due to the existence of cubic structures and the smaller crystallite sizes for CdS annealed at 160 °C, as proven by XRD analysis. Besides, the samples annealed at higher annealing temperatures show a gradual weight reduction from 200 °C to 500 °C, which is caused by the destruction of covalently bound organic bonds [7,10,15]. As previously discussed in the XRD analysis, the cubic structure has been gently transformed into a hexagonal structure with larger crystallite sizes. From the TGA analysis, present CdS demonstrates exceptional thermal stability up to 450 °C and, as such, can be incorporated as a colour pigment in paints and plastic. Table 3 presents the total weight loss and endothermic temperature determined for CdS. Differential scanning calorimetry (DSC) is an approach for measuring the quantity of heat released or absorbed by a sample as its temperature fluctuates or rises at a regulated and uniform rate. Fig. 7(b) displays the DSC curves of CdS at various annealing temperatures. In the present investigation, all the DSC curves indicate an endothermic peak around 550 °C and the corresponding decay owing to the decomposition of CdS.

Table 2
Band gap, absorption peak and crystallite size of ammonium nitrate capped CdS at varying annealing temperatures.

Annealing temperature (°C)	Band gap (eV)	Absorption peak (nm)	Average crystallite size (nm)
160	2.91	244	2.31
320	2.85	330	7.89
480	2.82	338	14.64

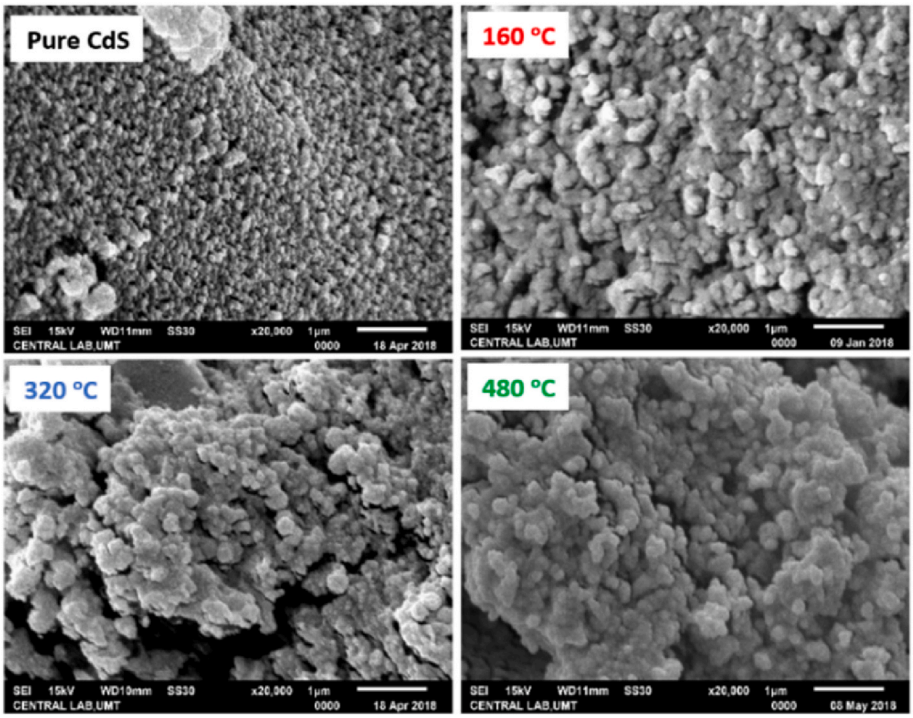

Fig. 6. SEM images of ammonium nitrate-capped CdS at various annealing temperatures.

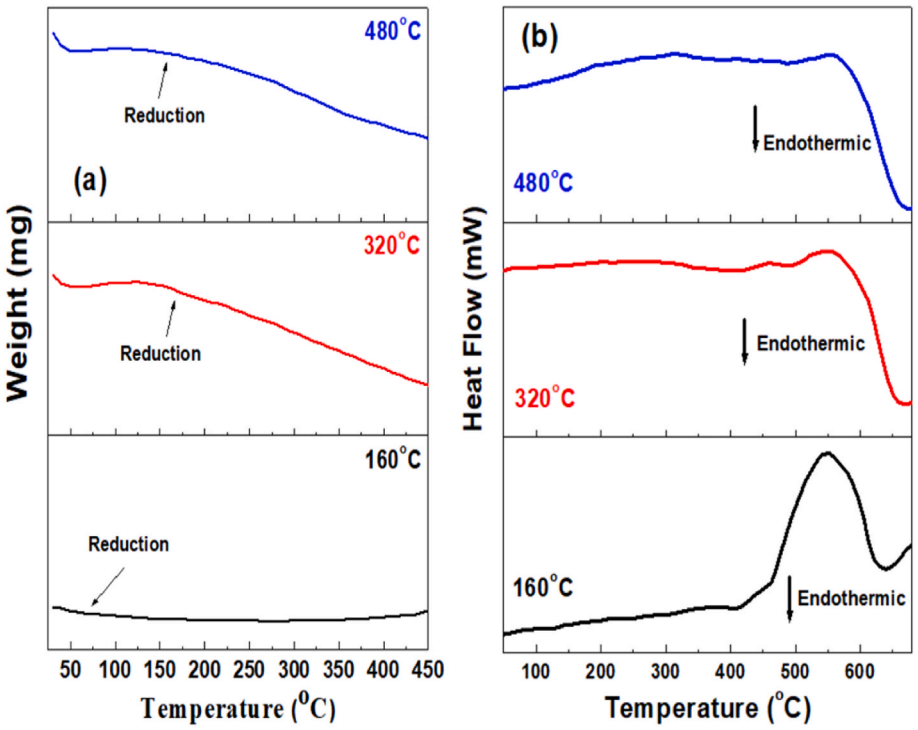

Fig. 7. (a) TGA and (b) DSC curves of ammonium nitrate-capped CdS nanoparticles.

Table 3
Weight loss and endothermic peak position for the ammonium nitrate-capped CdS prepared at different annealing temperatures.

Annealing temperatures (°C)	Initial weight (mg)	Final weight (mg)	Weight Loss Δm (%)	Endothermic peak position (°C)
160	18.26	18.16	0.55 %	548
320	5.19	4.69	9.63 %	552
480	5.58	4.76	14.70 %	557

4. Conclusion

This study has successfully investigated the effect of annealing temperature on the structural phase and morphological transformation of CdS nanospheres capped with ammonium nitrate as a surfactant. The samples were analyzed using various techniques, including XRD, FTIR, SEM, UV–Vis, TGA, and DSC. The as-synthesized CdS showed better crystallinity than pure CdS with no ammonium nitrate capping, but its crystalline structure transformed from an initial cubic to a hexagonal phase accompanied with increased crystallinity and size. The CdS nanospheres were found to be nearly spherical in shape with nano-size dimensions, while the shape remains the same but the size increases slightly when capped with ammonium nitrate and also annealed at higher temperatures. The UV–Vis analysis reveals that CdS exhibits stronger absorption and a smaller band gap as annealing temperature rises. The CdS nanoparticles with a smaller crystallite size exhibit improved thermal stability and heat diffusion, according to TGA and DSC data. The study suggests that thermally driven annealing temperatures with ammonium nitrate capping on CdS can significantly improve the optical absorption, thermal stability, morphology, crystallinity, and crystallite size of CdS nanoparticles for desired future applications in optoelectronics and photocatalysts. This work resolves a relevant issue in CdS nanospheres tunability by highlighting the limitations and possible obstacles of CdS and suggesting a solution based on the production of CdS with ammonium nitrate as a capping agent. The creation of CdS spheres capped with ammonium nitrate using precipitation methods is novel and contributes to improving the properties of CdS as compared to traditional approaches.

CRediT authorship contribution statement

Chan Kok Sheng: Writing – review & editing, Supervision, Resources, Project administration, Methodology, Funding acquisition, Formal analysis, Conceptualization. **Yousef Mohammad Alrababah:** Writing – original draft, Visualization, Software, Methodology, Investigation, Formal analysis, Data curation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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