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Influence of Temperature on the Stability and Optical Characteristics of TGA Capped ZnS Quantum Dots for Utilization in Blue Light Emission

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Abstract

A convenient and efficient one-pot synthesis method was employed to produce Thioglycolic Acid (TGA) capped Zinc Sulfide (ZnS) Quantum Dots (QDs). Compared to other QDs like CdSe, CdS, CdTe, and ZnSe, confining ZnS QDs was observed to be remarkably sensitive and challenging. Transmission electron microscopy characterization reveals a uniform dispersion of ZnS Quantum Dots (QDs) with an average diameter of 1.2 nm. Complementary calculations using the Bruce equation also estimate a size of 1.5 nm, affirming the consistent size of the QDs. The optical properties analysis indicates an initial optical band gap of 4.01 eV at room temperature. Notably, an intriguing observation is the reduction in band gap as temperature rises. Moreover, the size of the QDs was observed to increase with temperature, while the Urbach energy, revealing of localized states in the bandgap, also exhibited an increase with rising temperature. These temperature-dependent optical properties highlight the tunability of ZnS QDs, which could be advantageous for various applications. The Photo Luminescence (PL) response of the ZnS QDs exhibited a broad blue Photoluminescence band when subjected to a temperature of 90°C. This blue emission is a favourable characteristic for blue light emitters, making ZnS QDs potential candidates for applications in optoelectronic devices and displays. The Fourier-transform Infrared Spectroscopy (FTIR) spectra provided evidence for the successful encapsulation of ZnS QDs with TGA as the capping agent, further supporting the stability and passivation of the QDs' surfaces.

Keywords: *Opto Electronic Device ZnS Quantum Dots, Quantum Confinement, Thioglycolic Acid, Urbach Energy*

1.0 Introduction

The increasing global demand for sustainable and renewable energy sources has incentivized researchers to explore diverse options in alternative energy and novel devices. One promising avenue lies in the innovation of energy efficient and cost effective advanced optoelectronic devices using zero dimensional nanostructured composites, such as Quantum Dots (QDs). These exceptional semiconductor materials offer unique physical, chemical, and size dependent optical properties that can be controlled through precise synthesis techniques. QDs have proven to be valuable optical markers due to their broad absorption and emission spectra, excellent luminescence, and photochemical stability[1]. As a result, QD based optical emitters have found applications in various fields, including biology, bioanalysis, biosensors, and electronics[2].

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To achieve specific spectrum selectivity, researchers employ various synthetic techniques to tune the size of QDs. However, this field faces challenges due to the need for extensive efforts and synthesis protocols. While QDs like CdSe, CdS, CdTe, PbS, PbSe, and extensive research has been conducted on ZnO synthesized using various methods, research on ZnS QDs remains relatively sparse[3-5]. Despite their potential, the successful application of ZnS QDs depends on understanding their stability and optical properties under different temperature conditions[6]. This knowledge is vital for designing energy efficient and cost effective optoelectronic devices.

To synthesize ZnS QDs, researchers have explored methods like one pot non injection, colloidal synthesis, cationic inverse injection, and chemical precipitation, utilizing different encapsulating agents like TGA, MPA, PVP, and GSH, with different concentrations and refluxing temperatures[3,7,8]. Among these capping agents, Thioglycolic Acid (TGA) has been preferred due to its superior stability and surface passivation effects. Numerous investigations have effectively produced environmental friendly TGA-capped ZnS QDs using ligand exchange methods.

However, attempts to precipitate ZnS colloidal particles in aqueous solutions resulted in large-sized particles that quickly settled, hindering their application[9,10]. Furthermore, achieving stable colloidal ZnS QDs at pH 8 with capping molecules yielded unsatisfactory luminescence in as-synthesized samples[11]. Scientists have additionally investigated other capping agents like 2-mercaptoethanol and thiols to enhance photoluminescence[12,13].

In this article, we investigate the influence of temperature on the stability and optical characteristics of TGA stabilised ZnS quantum dots. Through a facile one pot synthesis method, we synthesize ultrafine TGA capped ZnS QDs and analyse their morphological and optical characteristics under varying temperature conditions. The results of this study shed light on the temperature dependent behaviour of ZnS QDs, providing valuable insights for optimizing their performance in blue light emission applications and advancing the field of optoelectronics towards more sustainable and energy efficient solutions.

2.0 Materials and Methods

2.1 Materials

In this study, Zinc Sulfide (ZnS) Quantum Dots (QDs) were synthesized using a one-pot unify method. The precursors employed for the creation of included Zinc Acetate Dihydrate ($Zn(Ace)_2 \cdot 2H_2O$), Sodium Sulfide (Na_2S), Sodium Hydroxide (NaOH), and Thioglycolic Acid ($C_2H_4O_2S$) from Merck, with a purity greater than 98%. Thioglycolic Acid (TGA) was utilized as the capping ligand during the synthesis process.

2.2 Experimental Details

The proposed method for synthesizing Thioglycolic Acid (TGA) capped Zinc Sulfide (ZnS) Quantum Dots (QDs) yields nanoparticles of the highest purity. The synthesis procedure can be broken down into three steps:

I. **Synthesis of TGA stabilized Zn precursor:** To initiate the process, a 0.02 M aqueous solution of Zinc Acetate Dihydrate $(CH_3COO)_2Zn \cdot 2H_2O$ is prepared and agitated for a duration of 10 minutes (Equation-1). Subsequently, under constant stirring, 0.04 M of Thio Glycolic Acid ($C_2H_4O_2S$) is injected as the sulfur source. The acidity level of the reaction mixture is adjusted to 9 using gradual addition of 1 M Sodium Hydroxide (NaOH). Initially, zinc acetate reacts with NaOH, succeeding in the formation of zinc hydroxide, causing a white precipitate (Equation-2). Further addition of NaOH dissolves the white precipitate; bring about in a clear solution (Equation-3).

II. **Preparation of Sodium sulfide precursor:** The Sodium Sulfide (Na_2S) precursor is prepared using 0.1 M of sodium sulfide. Subsequently, the sulphide ions was introduced into the reaction mixture while employing vigorous stirring. (Equation-4). The mixture is agitated for 30 minutes to ensure thorough mixing and reaction.

III. **The growth of TGA-capped ZnS QDs:** As a consequence of the reactions described above, TGA capped ZnS quantum dots are formed in the solution, appearing transparent and colorless (Equation-5) under ambient conditions. Aliquots of the reaction solution are collected for

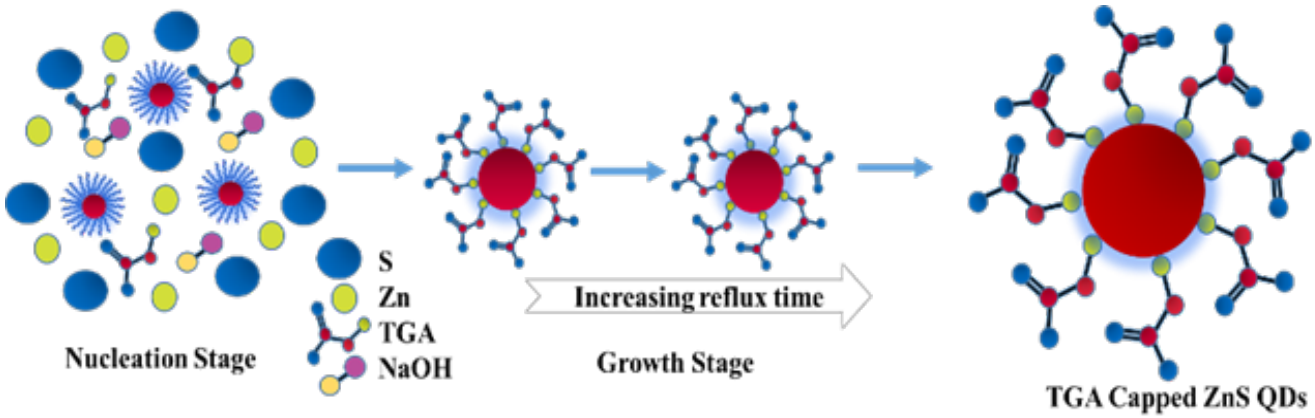
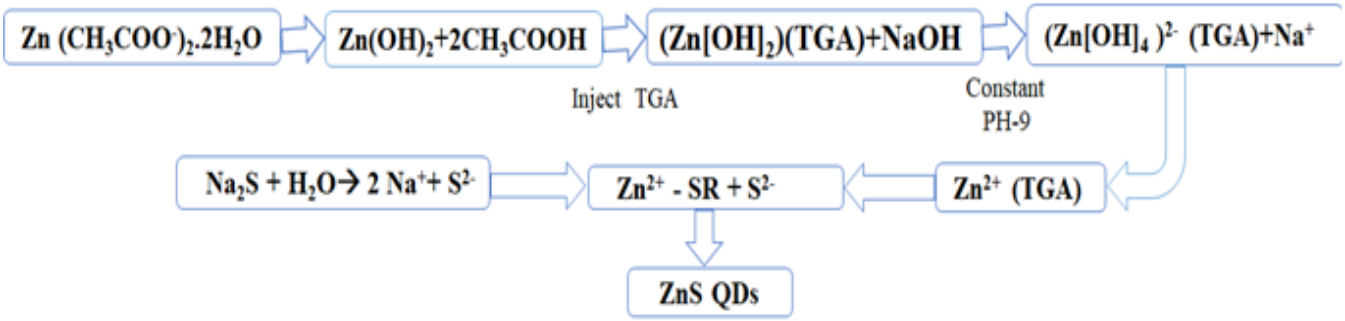


Figure 1. Diagram and descriptions illustrates the Experimental details of synthesis of TGA capped ZnS QDs.



Flow Chart 1. Synthesis process of TGA capped ZnS QDs.

various characterization techniques at ambient atmosphere. Additionally, the heat is slowly increased to check the growth of the quantum dots, and samples are collected at different temperatures.

The entire synthesis process is illustrated in Figure 1 and the reaction mechanisms are self-explanatory[14-16].

$$Zn(CH_3COO^-)_2\,2H_2O \rightarrow Zn(OH)_2 + 2CH_3COOH \quad (1)$$

$$Zn(OH_2)(TGA) + NaOH \rightarrow (Zn[OH]_4)^{2-}(TGA) + Na^+ \quad (2)$$

$$(Zn[OH]_4)^{2-} \rightarrow Zn^{2+}(TGA) + (\text{Other bi products}) \quad (3)$$

$$Na_2S + H_2O \rightarrow 2\,Na^+ + S^{2-} \quad (4)$$

$$Zn^{2+} - SR + S^{2-} \rightarrow ZnS \quad (5)$$

2.3 Instrumentation

The synthesized TGA capped ZnS quantum dots (TGA:ZnS QDs) underwent a comprehensive characterization to assess their optical and morphological properties The optical characteristics were examined through the utilization of an Ocean Optics USB 4000 UV-Vis-NIR spectrometer and a Photoluminescence (PL) measurement was performed using an RF-6000 spectrometer. The morphological properties of the TGA:ZnS QDs were examined using a Transmission Electron Microscope (TEM) Technai T20, USA. The study of chemical bonding and capping ligand through Alpha-II FTIR spectroscopy.

3.0 Results and Discussions

3.1 FTIR Study

The Fourier Transform Infrared Analysis (FTIR) obtained for the ZnS Quantum Dots (QDs) (Figure 2) provide valuable insights into the occurrence of specific chemical bonds and functional groups within the QDs, along with the role of the capping ligand, Thioglycolic, in

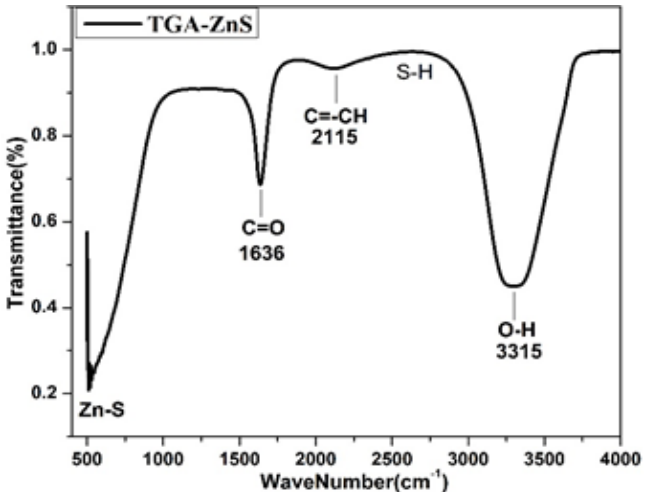


Figure 2. FTIR Spectrum of TGA capped ZnS QDs.

their stabilization. The initial peak observed in the FTIR spectra, ranging from 520 cm^{-1} to 560 cm^{-1}, correlate to the ZnS vibrations, indicating the existence of zinc-sulfur bonds within the QDs. This peak confirms the successful synthesis of ZnS quantum dots and their characteristic vibrational modes. A prominent and strong peak at 1636 cm^{-1} is attributed to the carbon-carbon double bond (C=O) stretching wave number, indicating the presence of carbonyl groups within the capping ligand, Thioglycolic acid. Additionally, a slight bending peak at 2115 cm^{-1} specifies the existence of a terminal carbon-hydrogen bond (C=-CH)[17]. These observations confirm the involvement of Thioglycolic acid in capping and stabilizing the ZnS quantum dots. Interestingly, the FTIR analysis also show the disappearance of the thiol group, observed between

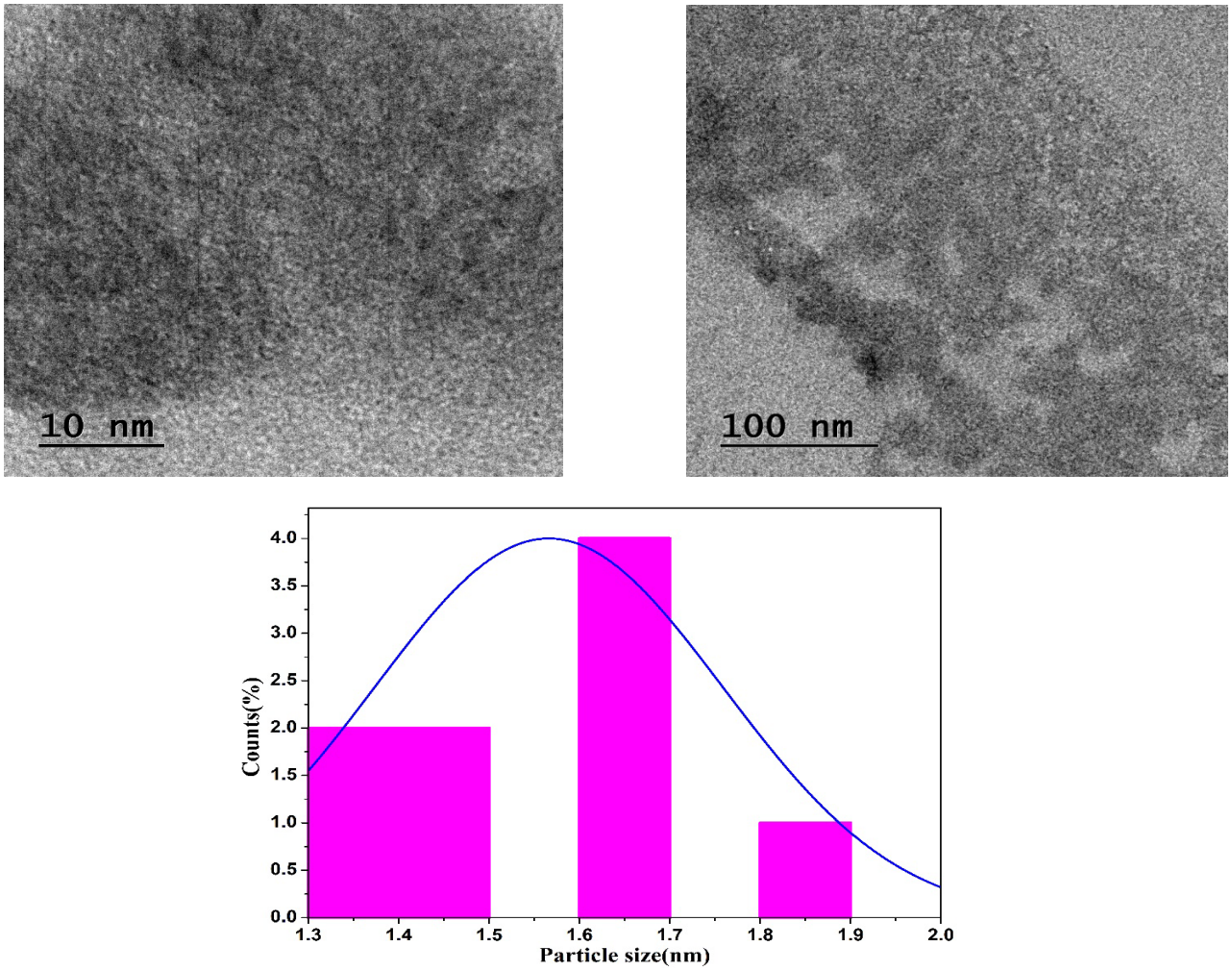


Figure 3. TEM images of TGA:ZnS QDs and corresponding particle size distribution.

2560 cm^{-1} and 2590 cm^{-1}. This disappearance is ascribed to the replacement of thiol ligands on quantum dots, achieved by breaking the S-H bonds during the synthesis process. This suggests a transformation in the surface chemistry of the quantum dots, where the thiol groups from the initial reaction are substituted by the capping ligand, TGA. The existence of a broad and strong band at 3315 cm^{-1} is attributed to -OH groups from the TGA capping ligand[18]. These hydroxyl groups play an important role in the binding of metal ions, and their presence contributes to the resilient ability of TGA to form a stable capping layer on quantum dots. This strong interaction between the sulfur electron pair of TGA and the zinc atoms on quantum dots surface facilitates the creation of the TGA capping, ensuring the stability and integrity of ZnS QDs. Comparable interpretations have been reported in the literature for other nano particles derived from glucose, such as gluconic acid nano particles, further supporting the validity of the findings in this study[19].

3.2 TEM Analysis

Figure 3 presents the TEM images of the TGA capped ZnS Quantum Dots (QDs) and its corresponding size distribution histogram. The TEM images offer visual proof of the uniform dispersion of the quantum dots, and the lattice stripes observed on the QD surface indicate good crystallinity. This statement endorses that all ZnS quantum dots were effectively capped with a sufficient amount of TGA ligands, ensuring their stability and preventing agglomeration[20]. The corresponding size distribution histogram illustrates the quantum dot sizes, and it shows a Gaussian peak with a mean quantum dot size of 1.52 nm. This finding is in close agreement with the QDs sizes obtained from UV Visible spectroscopy, which also provided insights into the optical and size characteristics of the TGA capped ZnS quantum dots.

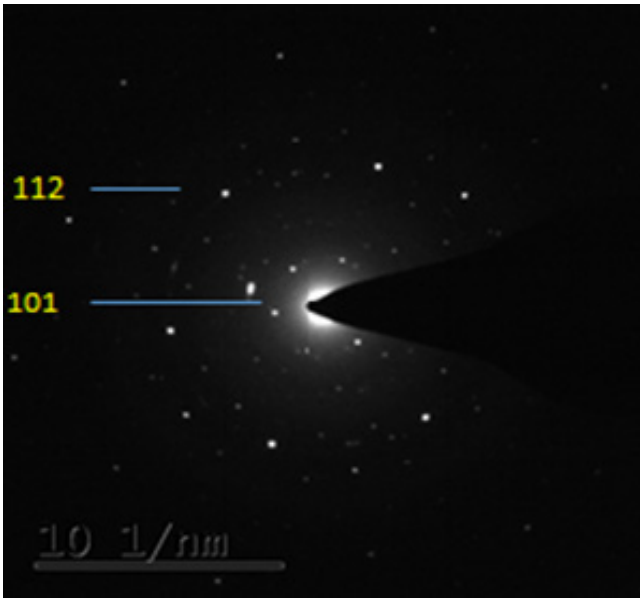


Figure 4. Pattern of Selected Area Electron Diffraction (SAED) of TGA: ZnS QDs.

Figure 4 displays the pattern of Selected Area Electron Diffraction for TGA capped ZnS Quantum Dots (QDs). The image form bright field resulting from the SAED pattern offers valuable insights into the crystalline properties of the TGA:ZnS QDs. The observed diffraction pattern confirms the existence of polycrystalline characteristics within quantum dots. The diffraction pattern reveals distinct diffraction spots corresponding to lattice fringes of cubic blend ZnS QDs. Notably, the occurrence of diffraction spots corresponding to the planes (101) and (112) indicates the crystallographic orientation of the quantum dots. The d spacing, determined from digital micrograph analysis, was 2.6 Å, which aligns with the diffraction plane (101) of ZnS. The preferential growth along the (101) diffraction plane further supports the notion that the TGA ligands effectively controlled the formation and growth of the ZnS quantum dots, ensuring their uniformity and stability[21].

3.3 UV Visible Spectra Analysis

The optical studies conducted on the TGA capped ZnS Quantum Dots (QDs) revealed intriguing characteristics in their UV Visible absorption and transmittance spectra (Figure 5 and Figure 6, respectively). The absorption band of the TGA: ZnS QDs is primarily limited to the UV region, with a weak absorption band observed in the visible light region. This sharp transmittance peak in the spectra is indicative of a clear excitonic state, suggesting the presence of well-defined quantum confinement effects in the quantum dots. The absorption edge observed at around 300 nm in the UV Visible spectra corresponds to the first electronic transition of ZnS bands, leading to blue emission properties. Remarkably, the absorption bands remained consistent without shifting or broadening, even with an increase in stirring time or prolonged synthesis at ambient temperature. This persistent absorption peak at a

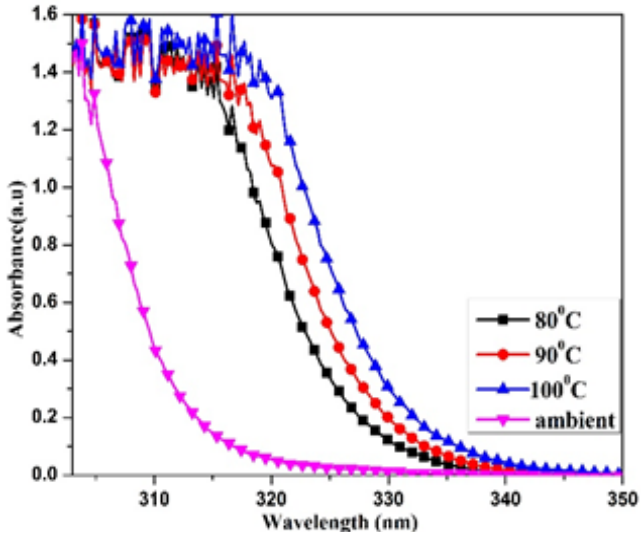


Figure 5. UV Visible Absorption spectra.

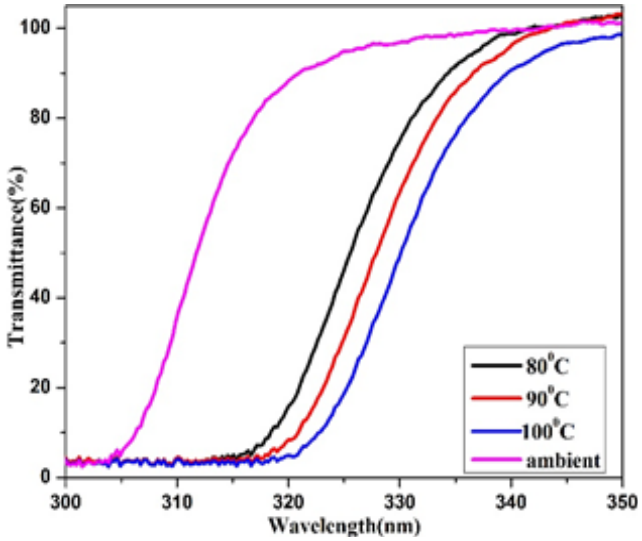


Figure 6. UV Visible Transmittance spectra.

fixed wavelength suggests the development of magic-sized quantum dots, which exhibit unique and stable optical properties[22]. Nevertheless, with the rise in temperature, the absorption peak shifts towards longer wavelengths, also endorse through transmittance spectrum.

Comparatively, the absorption shoulder at 350 nm,[23,24] characteristic of bulk ZnS particles, confirms that the synthesized TGA:ZnS QDs exhibit a blue shift in their absorption properties. This blue shift is consistent with the reduction in the size of the quantum dots, indicating quantum confinement effects due to their nanometre-scale dimensions[5,25].

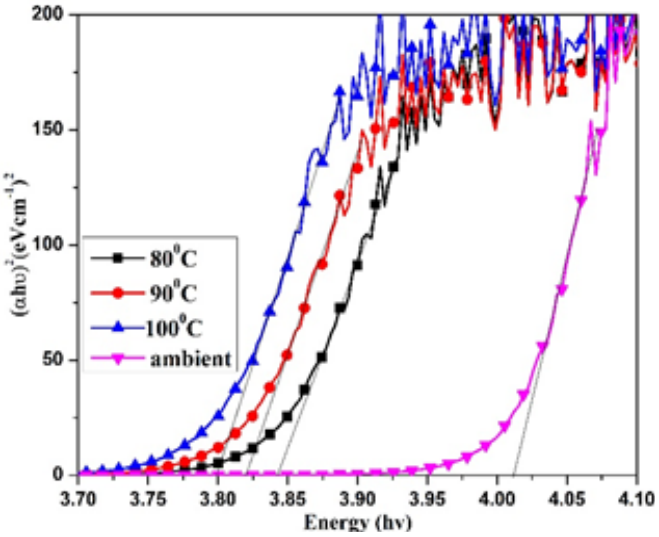


Figure 7. Tauc plot of ZnS QDs.

The precise energy band gap measurements for the TGA:ZnS QDs were calculated using Tauc's formulation,[26] and the outcomes are illustrated in Figure 7.

$$\alpha hv = A(hv\text{-}Eg)^n; \quad (9)$$

Where hv is photon energy, A is a constant and E_g is optical band gap of QDs respectively.

At ambient temperature, the TGA:ZnS QDs exhibited an energy band gap of considerable magnitude and this gap diminished as the temperature rose. However, compared to the band gap energy of bulk ZnS (3.68 eV), all temperature variation samples displayed higher band gap energy values. This trend implies a reduction in the dimensions of the TGA:ZnS QDs with increasing temperature.[27]

To estimate the dimensions of the quantum dots, Brus' equation (equation 10) was utilized, and the size was expressed in terms of the band gap energy.

$$r = \frac{-\left[\frac{1.786e^2}{4\pi\epsilon_0\epsilon_r}\right] + \sqrt{\left[\frac{1.786e^2}{4\pi\epsilon_0\epsilon_r}\right]^2 + [E_g^{QD} - E_g^{Bulk}]\frac{h^2}{2}\left[\frac{1}{m_{e*}} + \frac{1}{m_{h*}}\right]}}{2[E_g^{QD} - E_g^{Bulk}]} \quad (10)$$

Where: r represents the radius QDs, E_g^{QD} and E_g^{Bulk} refer to the energy band gaps of the quantum dots and bulk particles, ϵ_0 and ϵ_r represent the absolute and relative permittivity, m_{e^*} and m_{h^*} signify the effective masses of electron and hole respectively. The chosen for approximating the size are as follows: energy band

Table 1. Comparison of quantum dot size

Temperature (°C)	Band gap (eV)	QD size (nm)	TEM (Average)
80	3.84	3	1.52 nm
90	3.81	3.8	
100	3.79	4.4	
Ambient	4.01	1.5	

gap = 3.68 eV, εr =5.2, m_{e^*} =1.71 mo, m_{h^*} =3.04 mo, where mo signifies absolute mass of the electron.

Effective mass approximation according to Brus provided an estimation of the QDs' radius. The magnitude of the TGA:ZnS QDs synthesized at ambient temperature was determined to be approximately 1.5 nm, whereas for temperature variation samples, the average dimensions was determined to be 3.7 nm tabulated in (Table 1)[28]. This indicates that the quantum dots synthesized at ambient temperature are under strong quantum confinement due to their Bohr exciton radius. It is worth noting that the discrepancy between the QD size estimated from UV Visible absorption spectroscopy and TEM is recognized to the assumption of spherical geometry in the effective mass approximation.

Urbach energy, also known as the Urbach tail, is a crucial parameter in the study of Quantum Dots (QDs). It provides valuable information about the structural as well as electronic characteristics of the QDs. The Urbach energy can be determined using UV Visible spectroscopy, and it is derived from the photon energy and absorption coefficient data acquired from the spectroscopic measurements. In the case of TGA:ZnS quantum dots, the Urbach energy was measured to be 0.05 eV under ambient environmental conditions. Additionally, when the quantum dots were subjected to elevated temperatures (100°C), the Urbach energy increased to 0.06 eV. This observation implies that the quantum dots displayed higher crystallinity, less disorder, and improved stability in dimension under ambient environmental conditions as opposed to the conditions of high-temperature[29].

3.4 Photoluminescence Study

Figure 9 presents the luminescence spectra of TGA capped ZnS Quantum Dots (QDs) synthesized with a concentration ratio of TGA:Zn:S = 6:4:2 under two different conditions: ambient temperature and 90°C for two hours, while maintaining a constant pH of 9 during the synthesis process. For the analysis of the emission band characteristics, the Photoluminescence (PL) spectra

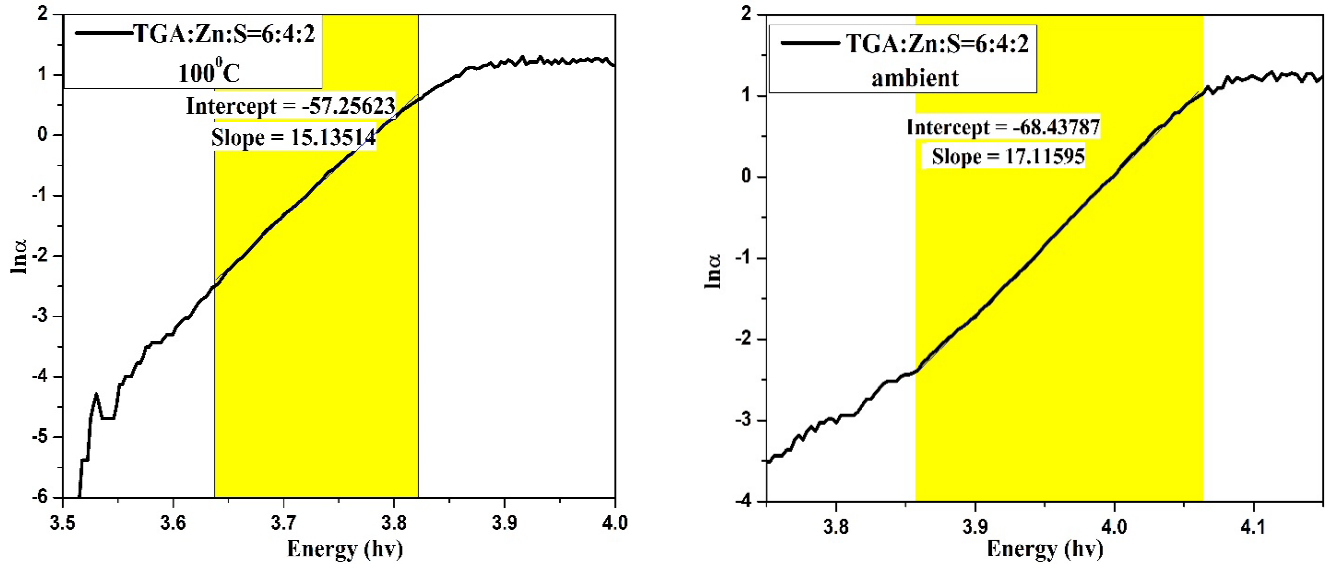


Figure 8. Urbach energy of ZnS QDs.

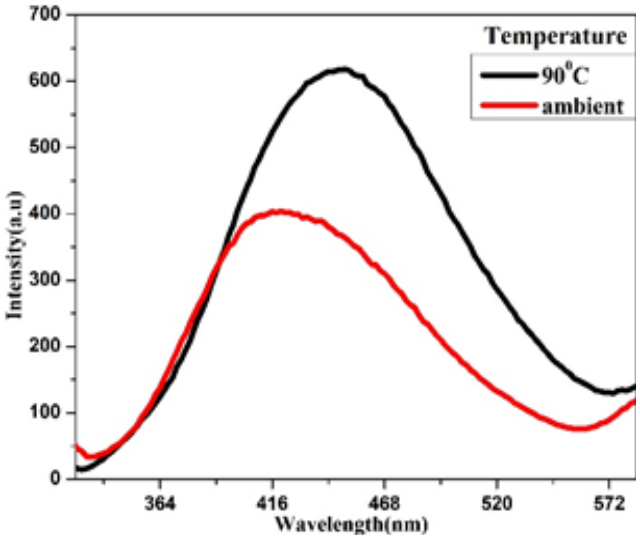


Figure 9. Photoluminescence spectra of TGA capped ZnS QDs.

were acquired with a constant excitation wavelength of 295 nm for both temperature settings. The resultant spectra reveal wide bands featuring dominant peaks at approximately 420 nm and 450 nm consistent across both ambient and 90°C conditions. Remarkably, with escalating temperature, there is an observable shift of the emission band towards longer wavelengths, indicating a greater emission efficiency at higher temperatures. The QDs synthesized under 90°C condition exhibit a distinctive blue emission, which can be attributed to the presence of trap state emission. This phenomenon involves the recombination of electrons at the sulphur vacancy donor level with holes trapped at the zinc vacancy acceptor level. The trap states are accountable for emitting light at longer wavelengths in comparison to the band edge emission of ZnS QDs[2,30]. This study's outcomes propose that with a rise in temperature, the QDs size enhances and by achieving an optimal concentration ratio TGA:Zn:S, the band gap of the QDs becomes adjustable, thereby allowing control over the emitted wavelength. This controllable emission wavelength holds significant potential in various applications, particularly within the realm of blue Light-Emitting Diodes (LEDs).

4.0 Conclusion

This study investigated the influence of temperature on the stability and optical properties of TGA capped ZnS Quantum Dots (QDs) with a focus on their potential utility in generating blue light emission. The synthesis process using TGA as a capping agent proved to be an effective and straightforward method to produce ZnS QDs. The FTIR analysis provided evidence of strong binding between the –C–O and –OH groups of TGA and the ZnS QDs, indicating the successful capping of the QDs with TGA. UV-Visible absorption peaks demonstrated that increasing the temperature resulted in the growth of larger QDs with a corresponding decrease in band gap, confirming the temperature-dependent size enhancement of the QDs. TEM images unveiled distinct and well-defined polycrystalline diffraction patterns within the Quantum Dots, signifying the establishment of stable and organized quantum structures. This finding aligned harmoniously with the Urbach energy assessments, underscoring diminished energy band disorder within the QDs and thereby reinforcing their structural stability. In terms of Photoluminescence (PL), the spectrum shows cased emissions spanning both the blue and green spectral regions. The ability to govern QDs' dimensions and band gaps through temperature adjustments amplifies their prospects for applications in blue Light-Emitting Diodes (LEDs) and a spectrum of other optoelectronic devices.

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