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RESEARCH ARTICLE

WET CHEMICAL SYNTHESIS AND SIZE-DEPENDENT OPTICAL PROPERTIES OF ZINC OXIDE (ZnO) QUANTUM DOTS

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ABSTRACT

Quantum dots are semiconductors whose exciton (electron-hole pairs) experience quantum confinement in all three spatial dimensions. As a result, they have properties that are between those of bulk semiconductors and those of discrete molecules. This confinement dramatically alters the optical properties of these dots as compared to the bulk material having applications in transistors, light-emitting diodes (LEDs), laser diodes, sensor devices, telecommunication, biophysics and photovoltaic devices. In this work, Zinc oxide quantum dots were synthesized via the wet chemical colloidal synthesis method at 65 °C using zinc acetate, sodium hydroxide and isopropanol. The optical absorption properties of the synthesized Zinc oxide quantum dots were studied and it was found that the quantum dots absorbed light in the wavelength range of 200-800 nm with maximum absorbance value of 250- 300 nm. The energy band gap of the synthesized ZnO quantum dots varied from 3.81-4.07 eV depending on the size of the sample. The size of the prepared quantum dots were found to be 3.2 nm, 3.6 nm and 4 nm for ageing time of zero second, 300 seconds and 600 seconds with a calculated band gap energy of 4.07 eV, 3.90 eV and 3.81 eV respectively. The optical spectra revealed that there was a narrow size distribution and a blue shift in the energy band gap which indicated that the ZnO quantum dots exhibited a strong confinement effect.

KEYWORDS

Zinc oxide, quantum dots, semiconductor, quantum confinement, band gap, and spectroscopic studies.

1. INTRODUCTION

Zinc Oxide (ZnO) is one of the groups II-VI semiconductor compounds possessing unique and novel physical properties with some exceptional and comparable properties to their II-VI counterparts. ZnO has drew attention due to its exceptional properties owing to its wide bandgap of 3.4 eV at room temperature with large exciton energy of 60 meV making it a promising material for optical devices that are based on excitonic effects, having applications in optoelectronics in the blue/Ultra-Violet (UV) region, including light emitting diodes, laser diodes, photo-detectors among others (Srivastava and Katiyar, 2022; Sauvik and Ahmaruzzaman, 2022).

Also, its unique position among semiconducting oxide due to the availability in a large area single crystal and its piezoelectric and transparent conductive properties has promoted its relevance. Zinc Oxide is a versatile, multifunctional inorganic compound that has gained significant and sustained attention as a result of its unique electrical, optical and chemical properties (Sharma, et al., 2022). Even though Zinc Oxide is a robust, stable and non-toxic semiconductor, it can be reduced to the nanoscale and at the nanoscale, it behaves in an extraordinary new way.

Nanotechnology has made it possible to process zinc oxide into diverse forms of nanostructures, including nanoparticles, nanotubes and nanoflowers. Among these, zinc oxide quantum dots (ZnO QDs) are exciting class of nanomaterials. Zinc oxide quantum dots can be defined as nanocrystals with dimension below 10 nm, a scale at which they exhibit quantum confinement effects and this dominate their properties. The

quantum confinement effect is due to the quantum dot size that is smaller than the Bohr radius of bulk ZnO, which is approximately 2.34 nm. This effect leads to a tunable optical and electronic properties as well as a discrete electronic structure that are not observed in their bulk counterpart (Xiao, 2021). By tuning the size of zinc oxide quantum dots, their optical bandgap are modulated and their colours can be precisely engineered both in absorption and emission. This made zinc oxide quantum dots a promising and valuable material for future technologies like sensitive UV sensors, ultra-efficient displays, and high-performance solar cells. A clear manifestation of this is a shift to left (blue shift) in the ultraviolet-visible (UV-VIS) absorption spectrum of ZnO quantum dots compared to its bulk counterpart. This is attributed to the quantum confinement effect (Xiao, 2021).

Zinc oxide quantum dots are highly effective in catalytic and sensing application due to the large surface area to volume ratio; demonstrate photocatalytic activity and antibacterial properties. (Tejaswi, et al., 2023). Zinc oxide quantum dots have different synthesis routes; gas-phase and liquid-phase routes. The liquid-phase route is particular popular because of their low cost, scalability, superior size control and surface functionalization (Xiao, 2021). In this paper, ZnO quantum dots were synthesized and characterized using the Ultraviolet-Visible (UV-VIS) spectroscopy.

The relationship between the particle size and the band gap can be expresses through the effective mass approximation model (Brus, 1986) as:

$$E = E_g + \frac{\hbar^2 \pi^2}{2m_0 r^2} \left(\frac{1}{m_e^*} + \frac{1}{m_h^*} \right) - \frac{1.8e^2}{4\pi\epsilon_0\epsilon_r r} \quad (1)$$

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Where E in eV is the effective band gap, E_g is the bulk band gap, m_0 is the free electron mass, m_e^* and m_h^* are the effective electron and hole mass respectively, ϵ_0 and ϵ_r are the permittivity of free space and relative permittivity respectively, e is the charge of an electron.

2. MATERIALS AND METHODS

The wet chemical colloidal synthesis method was used in this work. The method is based on three component systems that include an organic surfactant, a solvent and a precursor. In some cases one chemical can act as both the surfactant and solvent. This method was used due to its advantage of simplicity, popularity, easy size and shape control, and efficiency in producing large quantities of high-quality nanoparticles (quantum dots) (Houtepen, et al., 2025; Yin and Alivisatos, 2005). Zinc oxide (ZnO) quantum dots were synthesized using zinc acetate ($Zn(CH_3CO_2)_2 \cdot 2H_2O$) as the surfactant, sodium hydroxide as the precursor and isopropanol as the solvent.

Before the synthesis, the cuvettes, beakers, conical flasks, pipettes, syringes and Petri-dishes used for the synthesis of the quantum dots were soaked in hydrochloric acid for 24 hours, after which, they were washed with detergent solution and rinsed many times with distilled water. They were allowed to dry and later rinsed twice with isopropanol. Then, 0.1 g of zinc acetate was weighed out using an electronic balance into a 25 ml of isopropanol in a beaker. This was stirred vigorously while heating with the magnetic stirrer hotplate until the zinc acetate dissolved completely. The above action took place in about fifteen (15) minutes and the zinc acetate was completely dissolved.

The zinc acetate solution appeared cloudy and few drops of acetic acid were added to make it clear and also to stop premature growth of the dots. Then 125 ml of chilled isopropanol at 0 °C was measured and added to the zinc acetate solution, then transferred to the cold water bath for chilling at 0 °C for two hours. 0.2 g of sodium hydroxide pellets were weighed into a 100 ml of isopropanol in a 250 ml beaker and were stirred vigorously until it dissolved completely and was chilled at 0 °C for two hours.

After chilling, both solutions were brought out. The chilled zinc acetate solution was placed on the magnetic stirrer hotplate and vigorous stirring was applied. Meanwhile, with the aid of a syringe the chilled sodium hydroxide solution was added by running it slowly into the chilled zinc acetate solution under vigorous stirring as shown in plate 1.



Plate 1: Addition of sodium hydroxide solution to that of zinc acetate

The mixed solution was then transferred into a hot water bath kept at a constant temperature of 65 °C (338 K) and samples were collected immediately at an interval of five minutes into a cuvette.

A total of three samples were taken (at an interval of five minutes) and

scanned for absorbance using the spectrophotometer. Plate 2 shows some samples of prepared ZnO quantum dots in the cuvettes.

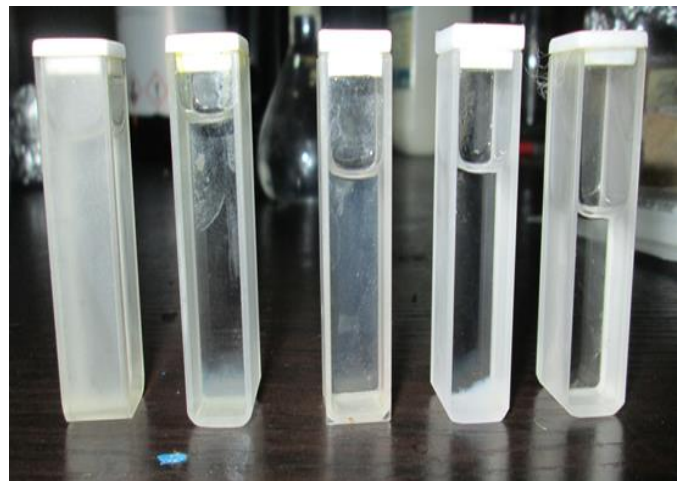


Plate 2: Some samples of prepared ZnO quantum dots in the cuvettes

3. RESULTS AND DISCUSSION

The prepared ZnO quantum dots were transparent and changed from transparent to cloudy while aging. The optical absorption of the ZnO quantum dots were studied using the 752 UV-VIS Spectrophotometer. Absorption measurements were taken using the UV-VIS spectrophotometer (UV-752) at a wavelength range of 200 nm to 800 nm.

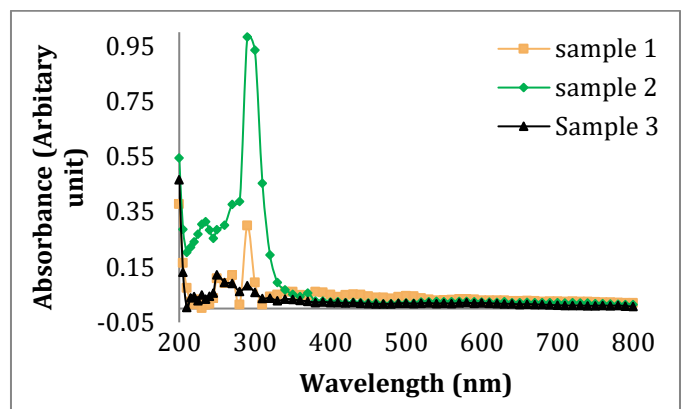


Figure 1: The UV-VIS spectra of the ZnO quantum dots.

Figure 1 shows that all the samples exhibit the same variation of absorbance with wavelength in same pattern with an absorption peak that is very sharp indicating a narrow size distribution of the quantum dots which is in agreement with the result obtained (Siriboon et al., 2023). The peak wavelengths in the absorbance spectra in this work were found to be at 290 nm, 300 nm, and 250 nm which are in the ultra-violet region for sample one (collected at 0 minutes), two (collected at 5 minutes), and three (collected at 10 minutes) respectively. Absorption occurs at higher energies, so a shift towards shorter wavelengths observed in the spectra was obvious, meaning that from the qualitative perspective, it is reasonable for a shift to the left to occur, validating the observed spectra in this investigation and confirming quantum confinement effect in the synthesized quantum dots. The energy band gap was determined using optical method. In semiconductors such as ZnO, the absorption of light only occurs when the frequency (and hence energy) is greater than or equal to the energy required to excite an electron from the valence band to the conduction band of the material (Osiele 2000).

The condition is;

$$h\nu \geq E_g \quad (2)$$

At the absorption edge, equation (2) becomes,

$$h\nu_o = E_g$$

therefore,

$$E_g = \frac{hc}{\lambda_c} \quad (3)$$

where h is the Planck's constant, c is the speed of light in vacuum and λ_c is the cut-off wavelength. The extrapolated cut-off wavelengths were used to calculate the energy band gap and these are shown in Table 1. The cut-off wavelength is derived from the intersection of the tangent of the peak absorbance with the wavelength axis. This method reveals more details about the band structure of the material and can be used to distinguish between direct and indirect band semiconductors (Chopra and Kaur, 1983).

Table 1: Extrapolated cut-off wavelength and energy band gap

QUANTUM DOTS	Peak Wavelength (nm)	Cut-off Wavelength (nm)	Energy Band Gap (eV)
Sample 1	290	304.54	4.07
Sample 2	300	318.55	3.90
Sample 3	250	325.96	3.81

Energy band gap is an important factor in terms of the uses of ZnO semiconductors. It is an indispensable property. The extrapolated energy band gaps of the quantum dots in this research were found to be 4.07 eV, 3.90 eV, and 3.81 eV for samples one, two, and three quantum dots respectively, whereas in bulk ZnO, the energy band gap value is 3.4 eV. A higher energy band gap observed for sample one, two and three is in good fate and is consistent with the blue shift of the absorbance peaks on the UV-VIS spectra which confirms successful quantum confinement. The sharp absorption peak indicate mono-disperse size distribution, which can be attributed to the size focusing effect during the early growth stage of the prepared quantum dots.

The linear absorption coefficient, α per unit length is defined as (Osiele, 2000)

$$k = \frac{\text{absorbance}}{4\pi} \quad (4)$$

and

$$\alpha = \frac{4\pi k}{\lambda}$$

where k is the extinction constant/coefficient and λ is the wavelength.

Substituting equation (4) into (5) we have

$$\alpha = \frac{\text{absorbance}}{\text{wavelength}} \quad (5)$$

The linear absorption coefficients for the zinc oxide quantum dots were calculated and figure 2 shows the variation of absorption coefficient with wavelength. The linear absorption coefficient depends on the wavelength of light that was actually absorbed.

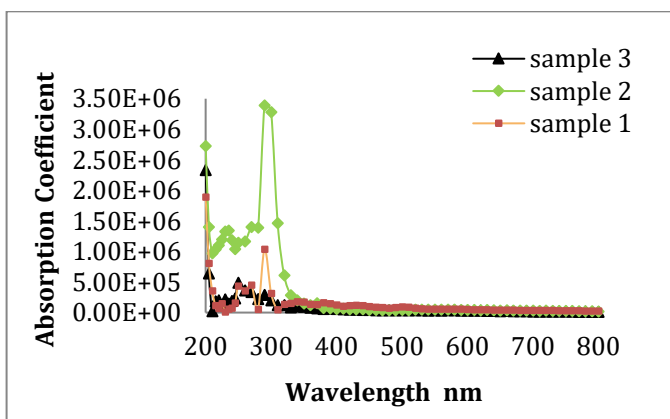


Figure 2: Variation of absorption coefficient with wavelength for ZnO quantum dots.

The quantum dots sizes were determined using the effective mass model derived by Brus, (1992).

$$E_g^* = E_g^b + \frac{\hbar^2 \pi^2}{2r^2} \left[\frac{1}{m_e^*} + \frac{1}{m_h^*} \right] - \frac{1.8e^2}{4\pi\epsilon\epsilon_0 r} - \frac{0.124e^4}{\hbar^2(4\pi\epsilon\epsilon_0)^2} \left[\frac{1}{m_e^*} + \frac{1}{m_h^*} \right]^{-1} \quad (6)$$

where, E_g^* is the energy band gap of the quantum dots determined from the UV-VIS absorbance spectrum. E_g^b is the energy band gap of bulk ZnO at room temperature, which have a value of 3.4 eV. \hbar is the reduced

Planck's constant; $\hbar = h/2\pi$, where h is Planck's constant (6.625×10^{-34} J.s). r is the radius of the quantum dots in nanometer (nm). m_e^* is 0.24, the effective mass of a conduction band electron in ZnO. m_h^* is 0.59, the effective mass of a valence band hole in ZnO. e is 1.6×10^{-19} C, is the elementary charge. ϵ_0 is 8.854×10^{-12} C² N⁻¹ m⁻² (Permittivity of free space). ϵ is 8.66 (Relative permittivity of ZnO).

Equation (6) can be rearranged to give a quadratic equation as a function of the radius, r ;

$$E_g^* r^2 = E_g^b r^2 + \frac{\hbar^2 \pi^2}{2} \left[\frac{1}{m_e^*} + \frac{1}{m_h^*} \right] r^2 - \frac{1.8e^2 r}{4\pi\epsilon\epsilon_0} - \frac{0.124e^4}{\hbar^2(4\pi\epsilon\epsilon_0)^2} \left[\frac{1}{m_e^*} + \frac{1}{m_h^*} \right]^{-1} r^2$$

$$\left[E_g^* - E_g^b + \frac{0.124e^4}{\hbar^2(4\pi\epsilon\epsilon_0)^2} \left[\frac{1}{m_e^*} + \frac{1}{m_h^*} \right]^{-1} \right] r^2 + \frac{1.8e^2 r}{4\pi\epsilon\epsilon_0} - \frac{\hbar^2 \pi^2}{2} \left[\frac{1}{m_e^*} + \frac{1}{m_h^*} \right] = 0$$

Comparing with

$$ar^2 + br + c = 0$$

Then the radius of the quantum dots is

$$r = \frac{-b \pm \sqrt{b^2 - 4ac}}{2a} \quad (7)$$

Therefore;

$$a = \left[E_g^* - E_g^b + \frac{0.124e^4}{\hbar^2(4\pi\epsilon\epsilon_0)^2} \left[\frac{1}{m_e^*} + \frac{1}{m_h^*} \right]^{-1} \right] \quad (8)$$

$$b = \frac{1.8e^2}{4\pi\epsilon\epsilon_0} \quad (9)$$

$$c = -\frac{\hbar^2 \pi^2}{2} \left[\frac{1}{m_e^*} + \frac{1}{m_h^*} \right] \quad (10)$$

Table 2 shows the calculated quantum dots sizes with their ageing time and corresponding energy band gap. The quantum dots sizes were obtained from the energy band gap inferred from the optical absorption spectra.

Table 2: Summary of synthesized quantum dots properties

Quantum Dots	Ageing Time (s)	Energy Band Gap (eV)	Radius (nm)	Quantum Dots Size (nm)
Sample one	0	4.07	1.60	3.2
Sample two	300	3.90	1.80	3.8
Sample three	600	3.81	2.00	4.0

The quantum dots prepared showed an increase in size as the ageing time increases. The sizes of quantum dots obtained from this method are a little larger compare to that obtained by other techniques such as X-ray diffraction (XRD) or Transmission electron microscope (TEM) by about 2 nm, reason being that the cut-off wavelength is related more to the size distribution of the particles and hence the estimated size appears a little larger compare to the average particle size (Penny et al., 2005).

A higher energy band gap observed at a lower particle size for sample one, two and three are consistent with the shift of the peaks on the UV-VIS spectra to lower wavelength. This can be ascribed to the fact that fewer molecular orbital states are being summed and added to the total possible states and so a greater band gap in energy exists between the highest occupied molecular orbit and lowest unoccupied molecular orbit. Thus, the higher energy band gap observed in the smaller quantum dot particles size is expected by theory. A group researcher also reported a very small particle size of 1nm for CdS which gave a band gap of 3.9 eV against the bulk CdS with 2.45 eV energy band gap (Banerjee, et al., 2000). Large energy band gap and highly blue shifted absorption edge confirmed that the prepared ZnO quantum dots showed strong quantum confinement effects which is in agreement with (Soosen, et al., 2009).

From the results presented in Table 2, a graph of the variation of energy band gap with quantum dots radii for sample one to three was plotted (figure 4). The graph of the variation of quantum dots energy band gap with radius (Figure 4), show an inverse relationship between the particle size and the energy band gap which is consistent with quantum confinement theory (Viswanatha, et al., 2004). Smaller quantum dot particle of size 3.2 nm exhibited a larger energy band gap of 4.07 eV while larger quantum dot particle of size 4.0 nm showed a smaller energy band gap of 3.81 eV.

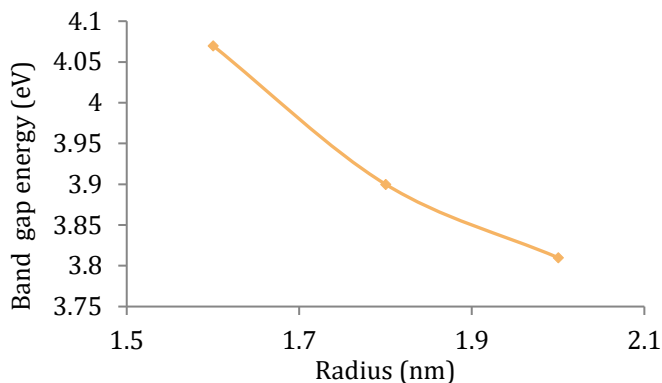


Figure 4: Variation of quantum dots energy band gap with the radius.

4. CONCLUSION

Zinc oxide quantum dots with sizes ranging from 3.2 to 4.0 nm were prepared successfully using zinc acetate, sodium hydroxide and isopropanol via the wet chemical colloidal synthesis method. The synthesized quantum dots absorbed light whose wavelength lies in the range of 200-800 nm and were found to have high absorbance in the ultra violet region and depreciate as the wavelength increased. Each prepared sample of the ZnO quantum dots showed maximum peak at different wavelength indicating that they are of different sizes and the effective mass model was used to calculate the sizes of the synthesized ZnO quantum dots. A higher energy band gap was observed at a lower quantum dot size for sample one, two and three.

The quantum dots exhibited size dependent properties with energy band gaps of 4.07-3.81 eV, showing significant blue shifts compared to the bulk ZnO (3.4 eV). The sharp absorption peaks confirmed narrow size distribution and the relationship between the energy band gap and particle size follows the effective mass approximation, validating the Brus model for ZnO quantum dots. With all these features of the synthesized zinc oxide quantum dots of samples one, two and three, it is very possible to use them in light-emitting diodes (LEDs), diode lasers, transistors, sensor devices, telecommunication, biophysics, photovoltaic devices and other technological applications where smaller quantum dots and large energy band gaps are required.

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