*Vol. 4, No. 4, pp. 205-218, (September 2023)*
DOI: 10.21608/AUJES.2023.200916.1136

**Aswan University Journal of Environmental Studies (AUJES)**
Online ISSN: 2735-4237, Print ISSN: 2735-4229
Journal homepage: https://aujes.journals.ekb.eg/ E-mail: AUJES@aswu.edu.eg

Original research

# Comparative Study on Synthesis ZnO Nanoparticles Using Green and Chemical Methods and Its Effect on Crystallite Size and Optical Properties

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Received: 18/3/2023 Accepted: 2/7/2023



## Abstract:

Zinc oxide (ZnO) nanoparticles attract researchers' attention because of their non-toxicity and multi-potential applications. To synthesize ZnO nanoparticles, various physical, chemical and green methods have been used. In this study, Zinc Oxide nanoparticles were prepared via three different techniques using the zinc nitrate solution as a precursor in a comparative study. The three methods classified Sol-gel and precipitation as chemical methods and environmentally friendly Leidenfrost green synthesis techniques. The resulting samples are characterized by X-ray diffraction analysis (XRD), Fourier Transform Infrared (FTIR) spectroscopy and UV-Visible (UV–Vis) optical absorption. The XRD analysis approves the formation of ZnO nanoparticles for the three methods with the same peaks' orientation (100), (002), (101), (102), (110), (103) and (112) planes. The crystallite size was determined instrumentally to be 17.2, 27 and 65 nm for Sol-gel, Precipitation and Leidenfrost methods, respectively. The results of XRD analysis approve that the chemical methods give the smallest crystallite size whereas the green one shows the highest crystallinity compared with chemical methods. FTIR spectra peaks showed the characteristic transmittance band of the ZnO nanoparticles. The UV-Visible spectrum showed the band gaps of synthesized samples 3.3, 2.03 and 3.2 eV for Sol-gel, Precipitation, and Leidenfrost methods, respectively.

**Keywords**: ZnO nanoparticles, Leidenfrost, sol-gel, precipitation.

## 1- Introduction

Recently, there has been a lot of worldwide attention in the field of nanotechnology. A lot of nanoparticles have distinct crystallite size and morphology, which makes them used in different fields such as pharmaceuticals **(Mahmoud et al., 2017)**, medicine **(Li et al., 2020),** food industry **(Chen et al., 2020)**, water treatment **(Abdelrahman et al., 2021)** and cosmetic **(Khezri et al., 2018)**.

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ZnO is a promising substance due to its eco-friendly nature, low cost, electrochemical activity, wide availability, and relatively broadband gap (3.37 eV) **(Ebnalwaled et al., 2019).** ZnO NPs have many applications as catalysis **(Sun et al., 2016)**, photochemical capability **(Das et al., 2015)**, medical effects **(Bisht & Rayamajhi, 2016)**, fungicidal **(Ali et al., 2022)**, antibacterial **(Qi et al., 2017),** UV filtration **(K.-B. Kim et al., 2017)** and wastewater remediation **(Rambabu et al., 2021).** There are many methods used to prepare nanomaterials including physical and chemical methods such as flame pyrolysis (Mueller et al., 2003), laser ablation **(M. Kim et al., 2017)**, sputter deposition **(Li et al., 2023; Zhu et al., 2023),** and chemical methods as solvothermal **(Dorokhina et al., 2023; Esakki et al., 2023; Ramakrishnan et al., 2018)**, hydrothermal **(Li et al., 2016)**, combustion **(Ghotekar et al., 2023; Laokae et al., 2023)**, chemical vapor deposition (CVD) **(Zhang et al., 2013),** Sol-gel **(Parashar et al., 2020; Pretto et al., 2023)** and precipitation method **(Raoufi, 2013)**. All of these methods could produce nanomaterial's at a higher rate and with better control over size and shape, but these methods require harmful chemicals, 3and produce a hazard waste and discarded a lot of energy **(Yadav et al., 2021)**. Therefore, the development of nanomaterial's production chemically and environmentally requires eco-friendly, cheaper and biocompatible methods, these preparation methods are called "Green synthesis " **)Bhardwaj et al., 2020(.** Green technology is synthesized in a single step, it has a variety of natures, higher stability and appropriate dimensions **(Parveen et al.).** Leidenfrost technique will be taken as an example **(Elbahri et al., 2018)**. In general, the focus will be on Sol-gel and precipitation as chemical methods and Leidenfrost as a green method for ZnO nanoparticles preparation.

Sol-gel is one of the most widely used chemical methods for the synthesis of metal oxide nanoparticles. The texture and surface qualities of the materials can be perfectly controlled with this technique. The sol–gel method can be labeled in definite steps as follow: hydrolysis, polycondensation, aging, drying and calcination **(Parashar et al., 2020).** Firstly, the hydrolysis step, eq. 1, where the required oxygen for metal oxide growth is provided by water or organic solvents. The aqueous sol-gel method refers to the use of water as the reaction medium, while the nonaqueous sol-gel method refers to the use of organic solvent as the reaction medium for the sol-gel process **(Niederberger, 2007; Yoldas, 1979).**

$$\text{M-OR} + H_2O \rightarrow \text{MOH} + \text{ROH} \ldots\ldots\ldots\ldots (1)$$

where M = metal, R = alkyl group ($C_n H_{2n+1}$) **(Parashar et al., 2020).** Secondly, condensation involves the condensation of the closest molecules, where water and alcohol are removed and metal oxide connections are created. As a result, polymeric networks expand to liquid-state colloidal dimensions**.** Condensation makes the solvent more viscous, so the liquid phase is known as a gel **(de Coelho Escobar & dos Santos, 2014)**. Thirdly, the aging step. During the aging process, condensation remains within the localized solution alongside the gel network precipitate, which reduces porosity and growths the thickness between colloidal particles **(Liu et al., 2002).** Finally, Drying and Calcination steps. The drying step is considered a complicated step because water and organic compounds are separated to form gels. There are atmospheric, thermal and freeze-drying. Calcination removes residues and water molecules from the sample. The calcination temperature is a very important factor influencing the material's pore size and the density **(Hench & West, 1990; Niederberger & Pinna, 2009).**

Precipitation as a chemical method is one of the most widely used methods for nanoparticle synthesis because of its simplicity and low cost. The precipitation method was employed to synthesize ZnO nanoparticles, which are based on chemical reactions between raw materials (zinc nitrate solution and sodium hydroxide) **(Kahouli et al., 2015)**.

The Leidenfrost method considers green nanotechnology focuses on developing simple, novel and environmentally friendly ways to make nanoparticles **(Abdelaziz et al., 2013a).** The Leidenfrost is a phenomenon in which a liquid is approached with a hot plate whose temperature is greater than the boiling point of the liquid, making an isolating vapor layer that saves the liquid from boiling fast **(Abdelaziz et al., 2013a; Alimohammadian & Sohrabi, 2020; Sobac et al., 2014)**. The metal oxide formed when a drop of water is sprinkled on a hot surface at a surface temperature above the boiling point of the water, where the water molecules ionized inside the droplet into $H^+$ and $OH^-$ (Fig 1) because of the heating to temperatures above the boiling point of water. There is a negative charge inside the droplet due to the large number of hydroxyl ions, outside the droplet there is a positive charge due to the formation of hydronium ions. Metal ion reacts with hydroxide ion forming a metal hydroxide which ends up in metal oxide **(Elbahri et al., 2017; Moghazy, 2023).**

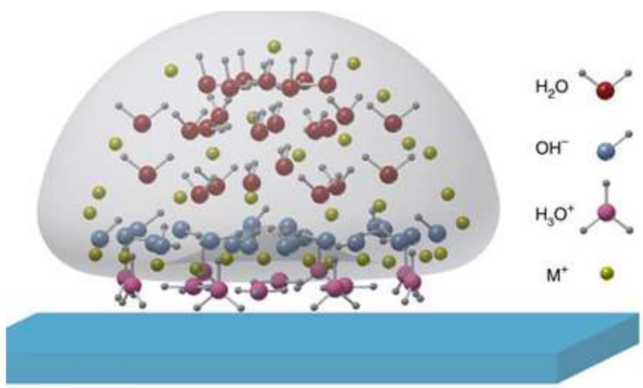



Fig 1: Leidenfrost Phenomenon **(Abdelaziz et al., 2013b).**

The work aims to synthesize ZnO by three different methods (green and chemical methods) to achieve high purity with a small crystallite size.

## 2. Materials and Methods

### 2.1. Materials:

Zinc nitrate-hexahydrate ($Zn(NO_3)_2.6H_2O$), Polyethylene glycol (PEG), sodium hydroxide (NaOH) and ethanol.

### 2.2. Synthesis of ZnO:

#### 2.2.1. So-gel Method:

The sol-gel method illustrated in Fig 2. 10ml of 0.6M $Zn(NO_3)_2.6H_2O$ aqueous solution was mixed with 8g of polyethylene glycol in a flask. The obtained solution was heated under stirring at 70°C for 20 min to obtain a homogenous gel solution. The obtained gel was calcinated at (500 °C) for one hour **(Pavani & Kumar, 2015).**

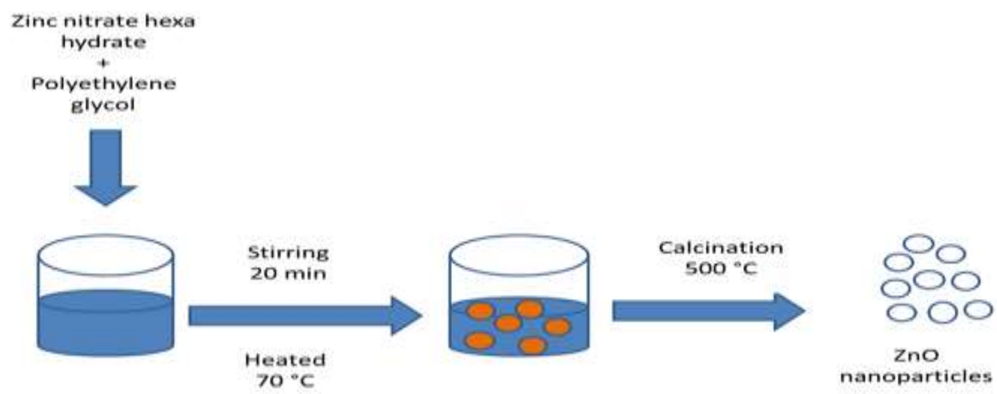



Fig 2: ZnO Synthesized Using Sol-gel Method.

### 2.2.2. Precipitation Method:

To a 0.1M $Zn(NO_3)_2.6H_2O$ solution, 0.9 M NaOH was added drop by drop with constant and high-speed stirring (Fig 3). The resulting precipitate was allowed to settle overnight and the supernatant was washed with distilled water and ethyl alcohol. The same washing procedure was made several times to remove the residual impurities present in the solution. The obtained white powder was dried at 60°C for 12 hours to obtain ZnO **(Dass et al., 2019).**

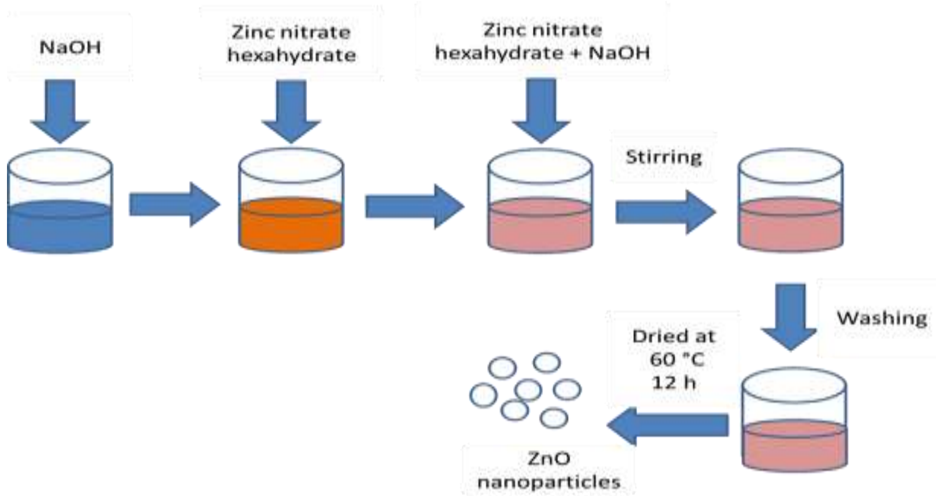



Fig 3: ZnO Synthesized Using Precipitation Method.

### 2.2.3. Leidenfrost Method:

To a hot beaker (300°C), a 50 mL of 10 mM solution of Zinc nitrate hexahydrate was added from a burette drop by drop to simulate the Leidenfrost effect by forming a vapour layer. Rapid vaporization processes take place in a very short time to create ZnO nanoparticles by heat convection, providing the required activation energy for the nucleation of nanoparticles **(Abdelaziz et al., 2013a).**

# 3. Results and Discussion:

## 3.1. Characterization:

## 3.2. X-ray Diffraction Analysis (XRD):

The XRD pattern of ZnO nanoparticles prepared by the three methods is shown in Figure (4). All 2θ values correspond to the crystal planes of (100), (002), (101), (102), (110), (103) and (112), respectively.

The XRD pattern of ZnO nanoparticles prepared by the Sol-gel method shows a diffraction peak at 2θ = 31.8°, 34.3°, 36.2°, 47.5°, 56.5°, 62.8° and 67.9°. The Crystallite size of the ZnO nanoparticles is determined to be 17.2 nm **(Abushad et al.).**

The XRD pattern of ZnO nanoparticles from the Precipitation method shows seven diffraction peaks at 2θ values of 31.7°, 34.3°, 36.2°, 47.5°, 56.6°, 62.8° and 67.9°. The Crystallite size of the ZnO nanoparticles is observed as 27 nm **(Moharram et al., 2014).**

Diffraction peaks at 2θ= 31.8°, 34.4°, 36.2°, 47.5°, 56.6°, 62.8° and 67.9 are observed in XRD patterns of ZnO nanoparticles from the Leidenfrost method. The Crystallite size of the ZnO nanoparticles is detected as 65 nm.

In comparison between the three methods (Table 1), all studied methods give multi-crystal ZnO NPs in pure form but with different crystalline sizes and intensities. The smallest crystallite size resulted from the sol-gel method compared with the two other methods. In contrast, the Leidenfrost method shows the highest intensity (2496.8) and sharpest peaks compared with soil-gel (372.7) and precipitation methods (366.8).

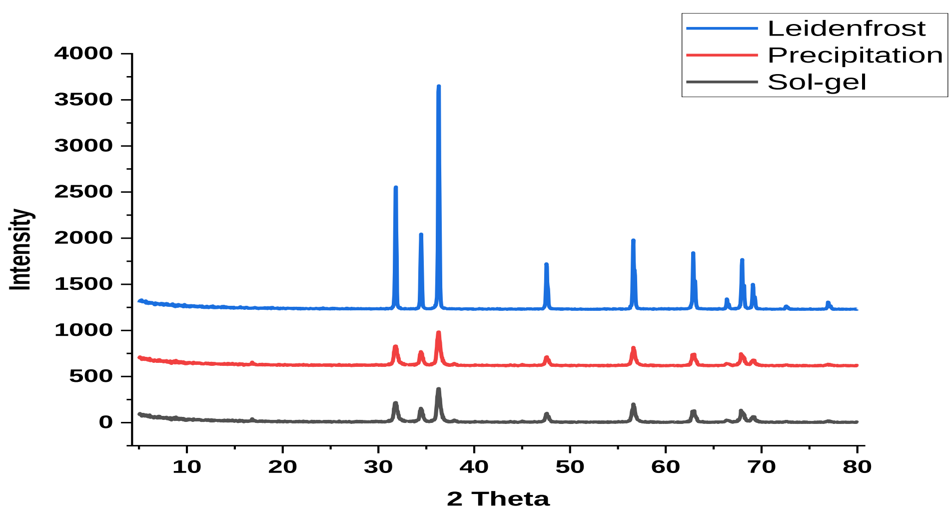



Fig. 4: XRD patterns of ZnO nanoparticles Using Sol-gel, Precipitation and Leidenfrost Methods

Table (1): Summarize The Crystallite Size (nm) and Intensity of ZnO Synthesized by Sol-gel, Precipitation and Leidenfrost Methods:

| **Method** | **Crystallite size (nm)** | **Intensity** |
|---|---|---|
| Sol-gel | 17.2 | 372.7 |
| Precipitation | 27 | 366.8 |
| Leidenfrost | 65 | 2496.8 |

### 3.3. Fourier Transform Infrared Spectrometer (FTIR):

FTIR analysis helped to identify functional groups existing in the synthesized samples. Figure (5) showed the FTIR spectra of the synthesized ZnO nanoparticles in the range of 4000–400 $cm^{-1}$. The FTIR spectrum of ZnO prepared using the sol-gel method shows a broad band at 3429 $cm^{-1}$ related to the O-H stretching, while the peak at 430 $cm^{-1}$ refers to the stretch vibrational of ZnO **(Salman et al.)**. The peak at 1624 $cm^{-1}$ was attributed to C=C stretching vibration because of the presence of polyethylene glycol **(Thirugnanam, 2013).**

For the precipitation method, there are two peaks at 1333 and 1500 $cm^{-1}$ due to the O–H bending of water, as well as a band at 3525 $cm^{-1}$, which is the stretching vibration of the O–H group. The peak at 445 $cm^{-1}$ is attributed to the Zn–O stretching **(Suntako, 2015).**

In the case of the Leidenfrost method, the O-H stretching is responsible for the peak at 3414 $cm^{-1}$. The formation of a ZnO nanoparticle is shown at the peaks at 686 and 452 $cm^{-1}$, which reflected the stretching vibration between the metal and oxygen (M+, O) of the Zn-O lattice **(Ashok et al., 2019; Haghighatzadeh et al., 2021).**

Comparing the three methods, the Leidenfrost method shows ZnO and O-H vibration bands only because of the low chemicals used in the synthesized method. On the other hand, in the two chemical methods (sol-gel and precipitation) various peaks were observed in addition to the ZnO peak due to the different chemicals used in the synthesized methods.

The results are similar to the previously reported works**, (Alamdari, et al., 2020)** found that the FTIR spectrum of the ZnO NPs showed a sharp and intense band at 546 $cm^{-1}$ which was attributed to metal-oxygen vibration **(Alamdari et al., 2020)(Shamhari, et al., 2018)** found a peak at 414 $cm^{-1}$ that attributed to Zn-O stretching vibration **(Shamhari et al., 2018)(Handore, et al., 2014)** observed the presence of a broad peak at 545 and 457$cm^{-1}$ corresponding to ZnO stretching vibration **(Handore et al., 2014)**. **(Dinesh, et al., 2014)** found a band at 473 and 481 $cm^{-1}$ confirms the formation of ZnO nanoparticles **(Dinesh et al., 2014).**

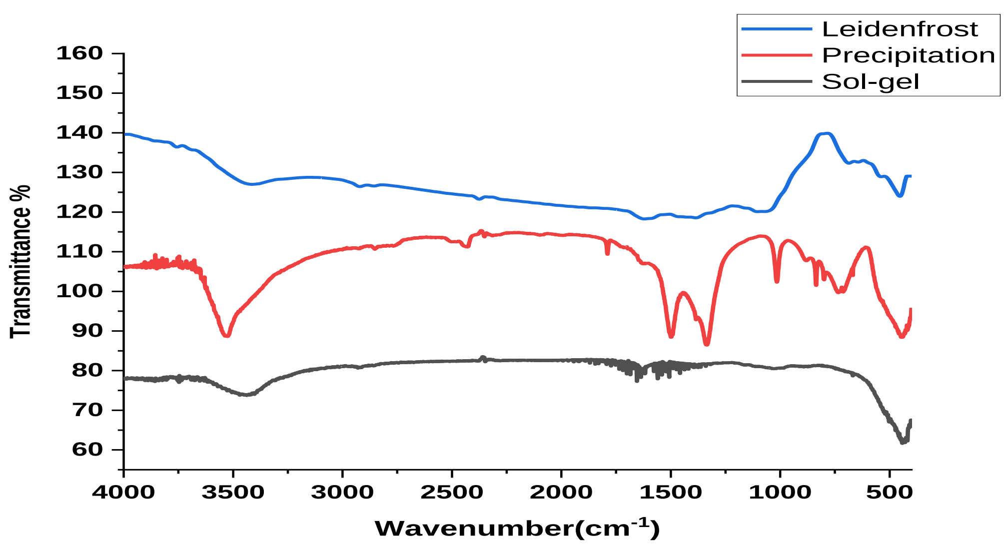



Fig 5: FT-IR Spectrum of Synthesized ZnO Nanoparticles; Sol-gel, Precipitation, Leidenfrost Method

### 3.4. Spectral Characterization:

The UV-Visible study was carried out to obtain information about the optical properties of the synthesized zinc oxide nanoparticles through band gap energy calculation. The band gap is the energy gap between the valence band ($E_v$) and the conduction band ($E_c$) **(Choi et al., 2017)**. The UV spectra were studied in a range of 200–1000 nm. The UV-visible absorption spectrum of zinc oxide nanoparticles obtained from various preparation methods was shown in Figure 6 (a& b& c). Figure 6 (a) shows the spectrum analysis of ZnO nanoparticles synthesized using the sol-gel method. There are significant bands at 390 and 968 nm. While for ZnO nanoparticles synthesized using the precipitation method, Figure 6 (b), there are two peaks at 295 and 374 nm. For the Leidenfrost method, maximum absorption bands were observed at wavelength 370 and 968 nm.

The UV-Vis spectrophotometer absorption spectra for the suspended solution of ZnO were used to calculate the optical band gap. Fig. 7 shows the optical band gap of the ZnO nanoparticles synthesized by different methods using Tauc's equation (eq. 2) which is normally used to indicate the band gap energy of the semiconductor materials **(Tauc & Menth, 1972; Usha & Christy, 2016; Vishwakarma et al., 2016)**:

$$(h\upsilon\alpha)^n = A(h\upsilon - E_g)\ldots\ldots\ldots\ldots..(2)$$

Where, h, $\upsilon$, $\alpha$, A, $E_g$ and n are the Plank constant, light frequency, absorption coefficient, proportional constant, energy gap and power index which depend on the nature of the transition, respectively. As ZnO is a direct band gap semiconductor material, the value of n=2.
By substituting h and c with their values, the equation becomes:

$$(\alpha \frac{1240}{\lambda})^2 = \left(\frac{1240}{\lambda} - E_g\right)\ldots\ldots\ldots\ldots\ldots(3)$$

$\alpha$ is the absorption value in the UV-vis spectrum that could be detected by a UV-vis spectrophotometer, while $\lambda$ is the detection wavelength. The $E_g$ was calculated by the intersection of linear fits of $(\alpha h\upsilon)^2$ versus $h\upsilon$ plots.

The data in Table (2) shows the band gaps of synthesized ZnO nanoparticles by the three methods. The band gaps of the ZnO nanoparticles are found to be 3.3 eV, 2.03 eV and 3.2 eV for sol-gel, precipitation and Leidenfrost method, respectively. It is observed that these values are lower than that of the bulk ZnO (3.37 eV) **(Jafarova & Orudzhev, 2021; Mang & Reimann, 1995)**. The variation in band gap energy for the three synthesized methods explained by the variation of crystallinity and crystallite size cause the band gaps of synthesized ZnO lower than the bulk ZnO (3.37 eV) **(Al-Ariki et al., 2021; Davis et al., 2019).**

In comparison with previous works **(Dass, et al., 2019)** found that the band gap of ZnO nanoparticles prepared by a chemical precipitation method was 3.19 eV **(Dass et al., 2019)**. **(Rauf, et al., 2017)** found that the band gap of ZnO nanoparticles was 3.27 eV**(Rauf et al., 2017)**. **(Vishwakarma, et al., 2016**) found that the band gap of ZnO nanoparticles was 3.003 eV **(Vishwakarma et al., 2016). (Pavani and Ajay, 2015)** found a band gap of 3.41 eV of ZnO nanoparticles **(Pavani & Kumar, 2015).**

Table (2): Band Gap of ZnO Nanoparticles for The Sol-gel, Precipitation and Ledienfrost Methods

| | **Sol-gel** | **Precipitation** | **Leidenfrost** |
|---|---|---|---|
| **band gap, eV** | 3.3 | 2.03 | 3.2 |

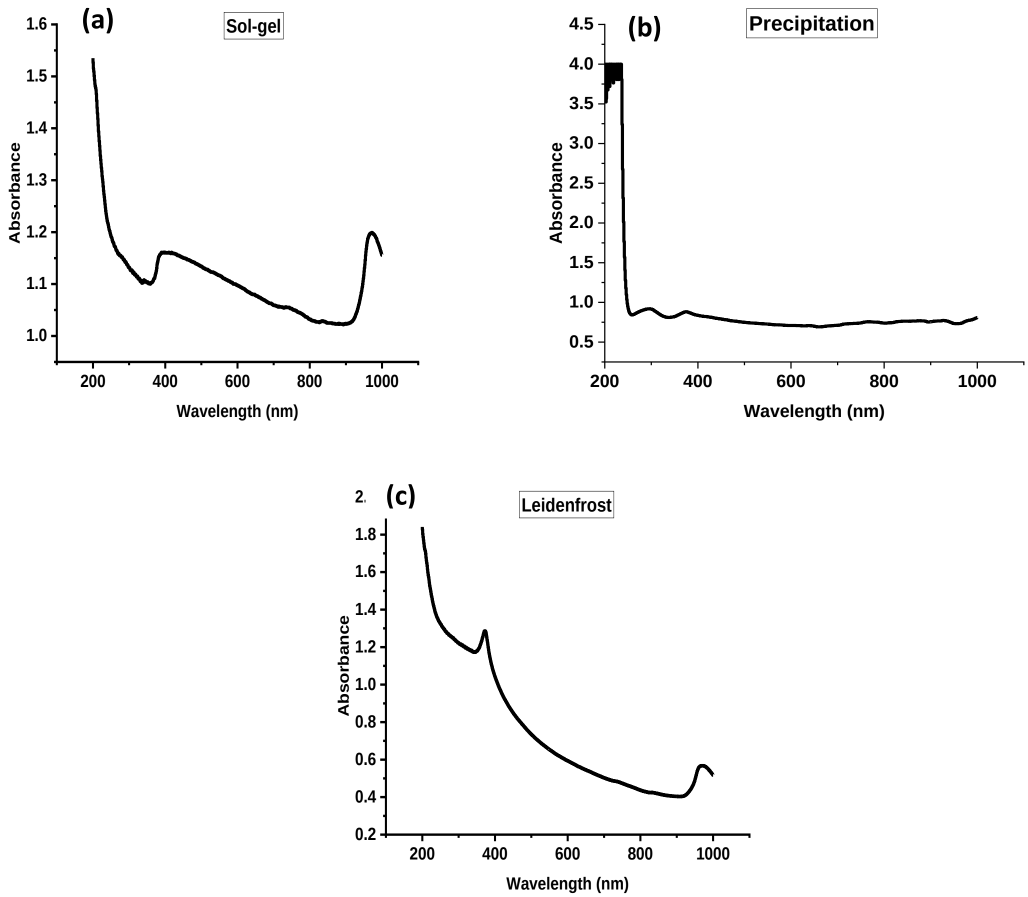
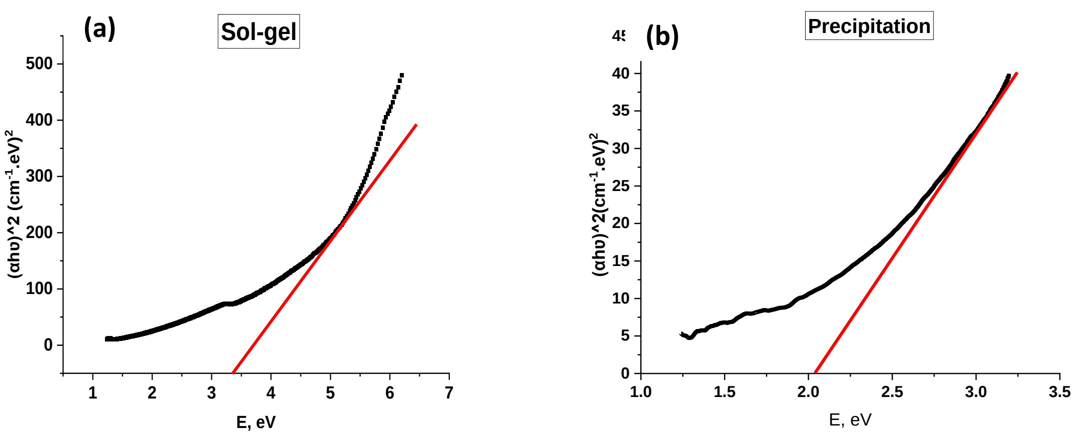



Fig 6: UV-Vis Spectra of Synthesized ZnO Nanoparticles; (a) Sol-gel, (b) Precipitation, (c) Leidenfrost Method



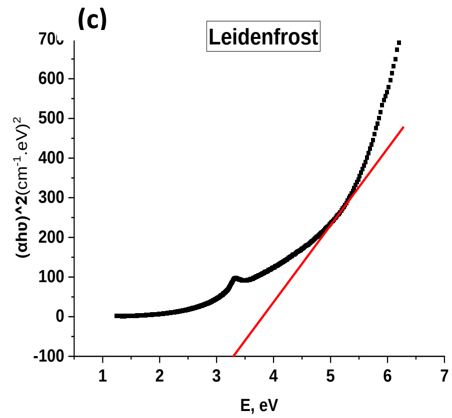



Fig 7: Tauc's Plot of The Absorption Spectra of Synthesized ZnO Nanoparticles; (a) Sol-gel, (b) Precipitation, (c) Leidenfrost Method

## 4. Conclusion:

Zinc oxide nanoparticles were prepared by different methods such as sol-gel, precipitation as chemical methods and Leidenfrost as a green synthesis method. The prepared nanoparticles were characterized by XRD. The crystallite size of the prepared nanoparticles was 17.2, 27 and 65.4 nm for Sol-gel, Precipitation and Leidenfrost methods, respectively, which show preferred growth orientation along the (101) plane. Comparing the three methods, chemical methods (sol-gel and precipitation) form ZnO nanoparticles with small crystallite size compared with the green one (Leidenfrost method) while XRD analysis shows that the green method gives the sharpest and highest intensity peaks. FTIR analysis showed the functional groups of ZnO nanoparticles and UV-VIS studies indicated the optical properties of the synthesized ZnO of the three methods, where the band gap of ZnO is 3.3, 2.03 and 3.2 eV for sol-gel, precipitation and Leidenfrost, respectively.

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