Hydrothermal synthesis and characterization of Zinc Oxide nanoparticles of various shapes under different reaction conditions

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Abstract
Zinc Oxide (ZnO) nanoparticles were synthesized by hydrothermal method under different conditions and studied various properties. FTIR studies proved the presence of ZnO bonding and purity of the samples. Grain size was found to be decreased with the increase of reaction temperature and increased with reaction time. TEM images show formation of nanorods under same reaction temperature, also nanoflowers and nanospheres for different temperatures. Intensity of luminescence peaks is found to be changed with variation in interplanar spacing. UV–vis spectra helped to identify the increased photon absorption in particles of bigger size. Change in bandgap value is also observed due to the difference in size of nanoparticles.

1. Introduction

Nanoparticles of transition metal oxide semiconductors exhibit unique electrical, optical and mechanical properties. These nanostructures have wide applications in different fields based on their size and shape [1–3]. Nanoparticles of various morphology like- nanorods, nanosheets, nanobelts etc can be utilized for solar cells, light emitting diodes, gas sensors, and biological probes. Size, shape, lattice constant and crystallinity of nanoparticles have very important role in choosing these materials for different applications [4]. Research work in the field of renewable energy sources has already been started since energy crisis is one of the most important issues which is expected to happen in near future [5, 6]. Nanoparticles of ZnO, NiO, TiO\textsubscript{2} etc exhibit excellent optical and electronic properties which are the main requirements for the functioning of optoelectronic devices [7–9]. Structural and optical characterization help us in suggesting materials for fabrication units.

Zinc Oxide is a transition metal oxide semiconductor with wide bandgap and high exciton binding energy. Hence ZnO is one of the most preferable materials in optoelectronic research field [10–18]. It is a low-cost material and easily available in nature [19]. ZnO nanoparticles are transparent to visible part of light and absorbing UV radiations. It is less toxic, high resistant and durable material [20, 21]. Morphology of ZnO nanoparticles can be modified easily [22]. Electron mobility is high for ZnO nanostructures [23].

Many techniques like chemical vapour deposition, spray pyrolysis, sol-gel method, hydrothermal method etc are already available for the synthesis of nanomaterials. Preparation techniques play a very important role in determining the size and shape of nanoparticles [18, 24]. Hydrothermal method has been attracted many researchers because of its distinct advantages like simple equipment, low cost and mild preparation conditions [25]. It is an environment friendly technique. Size and shape of nanomaterials can be modified by hydrothermal method. Morphology of nanoparticles mainly depends on reaction time, temperature and concentration of reacting solutions. This in turn affects the physical and chemical properties of nanoparticles. Particles are choosing for various applications according to their morphology. In this work we mainly emphasize the effect of reaction parameters on structural and optical properties of nanoparticles. Here hydrothermal reaction time is varied from 1 h to 5 h and temperature from 100 °C to 150 °C. Ph of solution mixture is kept as 12 to understand...
its influence on nanomaterial growth \[22, 26–29\]. In nano level, minute change in morphology of the particles affects the structural and optical properties drastically. We can consider reaction parameters as the tuning factors for the production of nanomaterial according to our requirement.

2. Experimental details

2.1. Materials
Zinc Nitrate [Zn(NO$_3$)$_2$.6H$_2$O] and Sodium Hydroxide (NaOH) pellets from Sigma-Aldrich. All chemicals used were analytical grade without further purification. Distilled water was used throughout the experiment.

2.2. Synthesis
Hydrothermal method is opted for the synthesis of nanoparticles. 0.5 M Zinc Nitrate solutions were prepared in 30 ml distilled water under stirring for 30 min. Meanwhile 5 M Sodium Hydroxide solutions were prepared by mixing weighed pellets of NaOH in 30 ml distilled water under stirring for same duration. NaOH solution is added dropwise to former solution under continuous stirring until Ph of the reactants becomes 12 \[3, 27, 29–32\]. This solution mixture was transferred into Teflon lined sealed stainless-steel autoclaves and kept in hydrothermal oven at a temperature of 100°C for 2 h. Then the beaker was taken outside and allowed to cool naturally to room temperature. The resultant solution was filtered, then washed with distilled water and kept for drying under lamp. The experimental procedure is repeated for 125°C and 150°C by keeping 2 h duration as constant and samples were named as HTZNp1, HTZNp2 and HTZNp3 respectively. Samples were also prepared by varying the reaction time and keeping temperature as constant. Samples synthesized at a temperature of 120°C for a reaction time of 1 h, 3 h and 5 h were named as HTZNp4, HTZNp5 and HTZNp6. The method of preparation for the nanoparticles is shown in scheme 1.

2.3. Characterization details
Crystal structure and average crystallite size were measured by XRD Bruker D8 Advance. Presence of metal oxide bonding and purity of samples were confirmed by FTIR Thermo Nicolet Avtar 370 (4000 cm$^{-1}$ to 400 cm$^{-1}$). Morphology of the samples was observed by SEM Jeol 6390LA/OXFORD XMX N and HRTEM: Jeol/JEM 2100. Thermal stability was determined by TGA Perkin Elmer STA6000. UV absorption was measured by JascoV-760 spectrophotometer. Photoluminescence is observed by Fluoromax 4 spectrophluorometer.

3. Results and discussion

3.1. FTIR analysis
FTIR studies help us to analyse the possible bonding between neighbouring structures in practical applications \[18\]. FTIR measurements were carried out for a range of 4000–400 cm$^{-1}$ and are shown in figure 1. In this range bands are due to vibration of ions in crystal lattice \[33\]. The intense band near 3439 cm$^{-1}$ in all samples indicates presence of OH stretching vibration. This may be due to oscillation of water molecule \[3, 14, 18, 34, 35\]. It is
shifted towards higher wave number side and intensity is more for HTZNP2 sample. Also, this band is appeared as narrow for HTZNP2, but widened for HTZNP1 and HTZNP3. This is because water content and its energy may be more at a temperature of 125 °C. Broadening of this band occurred in HTZNP1 and HTZNP 3 may be attributed to decrease in particle size [11]. There is no shift in this wave number for samples HTZNP4, HTZNP5 and HTZNP6 which are prepared at same temperature. Band at 2925 cm⁻¹ which arises due to –CH₂ vibration is present only in HTZNP2, HTZNP5 and HTZNP6 [34, 35]. The band at 1633 cm⁻¹ shows presence of C=O functional group [34]. There is no shift in this band due to change in reaction time and temperature. But intensity of peak is observed to be more for HTZNP3. This also can be due to small particle size [11]. The bands at 1560 cm⁻¹ belongs to symmetric C=O stretching [18]. In figure 1 (a), this band is shifted to higher wave number side for HTZNP2 and in figure 1 (b), this is shifted to higher wave number side for HTZNP5. This is assigned to minute change in bond length [11]. The bands at 1507, 1383 and 831 cm⁻¹ arise due to vibration and bending modes of CO₃²⁻ [3, 14, 18, 36]. These bands are not visible in HTZNP3. Disappearance of these bands at high temperature indicates complete decomposition of precursors. Increased concentration at low temperature causes high intensity for HTZNP 1 [14, 18].

Band at 1120 is present only for HTZNP5 and HTZNP6. This may be due to O–H deformation [37, 38]. The band close to 1043 cm⁻¹ shows –CH₂ vibration. It is present in all samples except HTZNP3. Disappearance of this band at high temperature may be ascribed to change in particle size. Band near 736 cm⁻¹ is found in HTZNP1, HTZNP2 and HTZNP4 due to H–O–H bending vibration which indicates the presence of water of crystallization [13, 30]. This band is absent in other samples. Due to high temperature and more duration, changes might have happened in this band. Band at 688 cm⁻¹ is due to deformation vibration. It is present in HTZNP1 and HTZNP2, but absent in HTZNP3 [31]. Band present at 418 cm⁻¹ which belong to Zn–O stretching mode is shifted to higher wave number region as temperature is increased [39]. This is attributed to increase in stretching energy due to increased temperature. Reaction temperature influence vibration of various functional groups present in samples. High temperature may affect morphology of nanoparticles which in turn causes intensity variation of bands. Increase in temperature causes increase in vibrational energy of Zn–O bond and hence a shift in band towards higher wavenumber side [39].

3.2. XRD analysis
Crystal structure of the samples were analysed by using XRD spectrometer. XRD is helpful for determining crystallite size, sample purity and lattice strain due to dislocation. Peaks are observed at 2θ values of 31.52, 34.15, 35.95, 47.28, 56.28, 62.52, 66.11, 67.68, 68.83, 72.22 and 76.84. The intense peaks appeared at different 2θ values indicate hexagonal wurtzite structure of ZnO nanoparticles. Peaks and intensities are matching with literature values and are shown in figure 2 [18, 30, 35, 38, 40, 41]. Intensity of (101) plane is found to be maximum for all six samples. Intensity of peak corresponding to (100) plane is found to be increased for samples synthesized at same temperature under different reaction time. Absence of other peaks indicates purity of samples.

In figure 2(a), intensity of (101) is appeared as lowest for HTZNP3. Diffraction angle is found to be right shifted for this sample. This can be assigned to stress developed and defects formed inside the material. In figure 2(b), it was observed that intensity is less for HTZNP5. Sharpness of diffraction peaks corresponding to
(100), (002) and (101) planes are observed to be increased for nanoparticles formed at same temperature which indicates good crystallinity of samples [42–44].

Average grain size of ZnO is determined by using equation (1)-Debye–Scherrer formula

\[ D = \frac{0.89\,\lambda}{\beta\,\cos\,\theta} \]  

Where D is the crystallite size, \( \lambda \) is the incident radiation wave length, \( \beta \) is the full width at half- maximum of the ZnO (101) line and \( \theta \) is the diffraction angle [3, 12, 18, 26, 35, 45]. Grain size was determined for various samples. When temperature is increased, crystallite size is found to be decreased. When reaction time is increased, grain size is increased. Grain size and FWHM of samples are shown in table 1.

### 3.3. SEM analysis

SEM images of hydrothermally prepared samples are shown in figure 3. Nanostructures with various size and shape are obtained under different reaction parameters. It is observed that reaction temperature and duration influence morphology very much and hence the structural properties of nanoparticles. By varying reaction parameters 0D, 1D, 2D and 3D structures are obtained. SEM reveals the flower like structure (3D) to first two samples (figures 3(a), (b)), spherical granular shape (0D) to HTZNP3 (figure 3(c)) and nanorod (1D) like morphology to HTZNP4, HTZNP5 and HTZNP6 (figure 3(d)–(f)). HTZNP1 (2D) has nanosheets joined to form flower structure [2, 3, 29, 39, 46, 47]. Aloe Vera like pattern with more uniformity of flower like pattern is very clear in HTZNP2.

From FTIR analysis it is observed that intensity of peak 1383 cm\(^{-1}\) is appeared to be maximum for smaller rods. This is because carboxylate group exists at the surface. As size of the particle increases intensity of the peak decreases. Vibration bands due to CO\(_3^2\) and –CH\(_2\) groups and deformation vibration bands are absent in nanoparticles of spherical shape [35].

Sharpness of XRD peaks corresponding to (100), (002) and (101) planes are found to be increased for nanorods [42–44, 46]. Position of diffraction angle shift, intensity variation in XRD peaks and presence/absence of some bands in FTIR also predicted possibility of change in morphology and size of nanoparticles.

### Table 1. Grain size and FWHM of samples prepared.

| Sample name | FWHM | Grain size (nm) |
|-------------|------|----------------|
| HTZNP1      | 0.312| 46.19          |
| HTZNP2      | 0.346| 41.66          |
| HTZNP3      | 0.415| 34.74          |
| HTZNP4      | 0.322| 44.76          |
| HTZNP5      | 0.270| 53.31          |
| HTZNP6      | 0.184| 78.21          |

![Figure 2. XRD patterns of (a) HTZNP1, HTZNP2 and HTZNP3 (b) HTZNP4, HTZNP5 and HTZNP6.](image)
3.4. TEM analysis

TEM images of hydrothermally grown samples are shown in figures 4–9. Flower shaped ZnO nanoparticles were obtained for 100 °C and 125 °C. Length of flower petal is obtained as 413 nm and diameter 78 nm for HTZNP1. Length is 495 nm and diameter 131 nm for HTZNP2. When temperature is increased to 150 °C nano particle is changed to spherical in shape. Average size of HTZNP3 is obtained as 40 nm. Among the three samples
synthesized for same duration, size of HTZNP3 at high temperature is found to be less. Nanostructures synthesized at same temperature are having same morphology [32]. Length of HTZNP4 is obtained as 312 nm and diameter as 160 nm. For HTZNP5, length is found to be 160 nm and diameter 70 nm. HTZNP6 is seen with a length of 216 nm and diameter 112 nm. From XRD graphs it is clear that intensity of plane (100) is increased for nanoparticles of rod morphology. Also, intensity of peak corresponding to (101) plane is found to be

![Figure 5. TEM images and SAED image of HTZNP2.](image)

![Figure 6. TEM images and SAED image of HTZNP3.](image)
maximum for nanorods of bigger size and peak intensity is minimum for smaller rods. Intensity of all peaks is found to be minimum for particles of smaller size. Interplanar spacing is found to be 0.23 nm for HTZNP1, HTZNP3, HTZNP4 and HTZNP5 and 0.25 nm for HTZNP2 and HTZNP6. Interplanar spacing is matching with literature [1, 26, 37, 44, 46]. From SAED pattern it is clear that HTZNP1 and HTZNP2 are polycrystalline and other samples are single crystalline in nature [48].
3.5. TGA analysis

Thermogravimetric analysis (TGA) helps us to study thermal behaviour of samples. In figure 10(a), it is shown that first decomposition started at a temperature of 210 °C for HTZNP1 and at 270 °C for HTZNP2 and HTZNP3. It is observed that sample synthesized at high temperatures decayed at higher temperature. Above 80% of material was existing even after heating up to 700 °C. Thermal decomposition of rod-shaped nanoparticles is shown in figure 10(b). Only 5% of the material is decomposed and it is started only at 200 °C. After 250 °C material was almost stable. All nanorods are showing same type of thermal behaviour. Less than 2% weight loss happened below 200 °C in all samples is due to desorption of physically adsorbed water present in samples [18, 29, 36]. Thermal degradation of poly crystalline samples HTZNP1 and HTZNP2 is more comparing to other samples. Weight loss is observed to be large in Poly crystalline nanoparticles than single crystalline particles. Hence single crystalline materials are thermally stable as observed in literature [13].
3.6. PL analysis

Physical and optical properties of semiconducting particles will change due to quantum size effects [48]. Photoluminescence spectroscopy (PL) provides information about extrinsic and intrinsic defects. It gives information about different energy states existing in between valance band and conduction band [46, 49]. PL spectra of ZnO nanoparticles exhibit two emission bands: one is in UV and one is in visible region [29, 44]. PL spectra were observed by using spectrofluorometer with an excitation wave length of 350 nm at room temperature. Peaks appeared in PL emission spectrum in the range from 370 nm to 500 nm indicates transition of electrons from bottom of conduction band to zinc vacant energy levels. UV emission peak appeared in various samples from 378–387 nm corresponds to near band edge emission of ZnO. This is due to radiative recombination of free excitons [9, 29, 33, 50]. Different peaks spread between 430 to 500 nm can be assigned to recombination of photogenerated holes with singly ionized state related to defects [9, 14]. Blue emission band appeared near 477 nm is caused by transition of electron from conduction band to zinc interstitial oxygen defects present in the sample [51]. Violet band observed near 416 nm in all samples is attributed to recombination of electron at the zinc interstitial and a hole in the valance band. Other violet peak present near 405 nm is referred to transition from Zinc interstitial to Zinc vacancy and peak at 382 is due to near band edge emission [52]. Oxygen vacancies, Zinc vacancies, Oxygen interstitials and Zinc interstitials are main defects which are attributed to visible emission [53].

In figure 11(a), we can see that when temperature is increased from 100 to 125 °C, intense peak present in UV region is slightly shifted to shorter wave length region. This can be interpreted as increase of exciton energy due to decrease in grain size when reaction temperature is increased. Different behaviour of HTZNP3 graph is caused by its spherical nature. Higher UV emission intensities observed for HTZNP1 and HTZNP2 can be assigned to larger grain size and flower like structure. Intensity of this band is comparatively less for HTZNP3.

Excited electrons lose their energy mainly by two ways—radiative recombination and nonradiative recombination. Non radiative trapping rate of excited charge carriers may be high for spherical nanoparticles. This can be the reason for less photoluminescence intensity in UV region. Maximum intensity peaks observed for HTZNP2 confirmed the formation of large number of defects in the sample [46]. In figure 11(b), all three graphs are similar in nature due to same shape. There is a red shift for peak in UV region from HTZNP4 to HTZNP6. This is ascribed to the increase in crystallite size from HTZNP4 to HTZNP6 as shown in XRD analysis. One broad band centred at 456 nm can be seen in all the three samples. Intensity of this peak is found to be increased when reaction time is increased. This is due to increase in grain size. All results are matching with literature [46, 47, 54]. Blue emission band is observed at 428 for HTZNP1, 431 for HTZNP4 and HTZNP5 and at 432 for HTZNP6. This can be explained as near band edge emission due to free exciton recombination. The emission band occurred at about 486 nm can be referred as deep level emission. Presence of intrinsic and extrinsic defects which arises due to electron recombination in Oxygen vacancy with hole in valance band is the main reason for this band [55]. Small peaks at 476, 486 and 496 nm are attributed to the presence of more surface defects, Zinc and Oxygen interstitials in these three samples. Here also intensity is maximum for 5 h and minimum for 1 h. This can be assigned to the formation of more surface defects, Zinc and Oxygen interstitials due to more reaction time [27, 46]. From figure 11(a), it is clear that luminescence observed for HTZNP2 in UV

![Figure 11. Photoluminescence spectra of (a) HTZNP1, HTZNP2 and HTZNP3 (b) HTZNP4, HTZNP5 and HTZNP6.](image-url)
and visible regions is more than other two samples. Among samples prepared under same temperature a slight increase in luminescence is observed for HTZNP6 and is shown in figure 11(b). TEM analysis is showing an interplanar spacing of 0.25 nm for these two samples. Other samples are appeared with an interplanar spacing of 0.23 nm. High luminescence of HTZNP2 and HTZNP6 may be due to structural difference occurred as a result of variation in interplanar spacing. Full-width at half maximum is only few nm for the peaks. So, size of the particle is in nano range [56].

3.7. UV analysis
UV absorption spectra is analysed for all samples from 200 nm to 800 nm. Peaks observed in the absorption spectra are due to transition of electrons between valance band, conduction band and intrinsic defect levels [46]. In figure 12(a), an intense broad peak is observed for HTZNP1 and HTZNP2 from 360–380 nm. This is the characteristic absorption peak of ZnO which is ascribed to intrinsic band-gap absorption of ZnO. But this is widened for HTZNP3. This may be due to its spherical shape and smaller size. Absorption is found to be maximum for HTZNP2. This may be referred to its larger size and flower like 3D structure.

In figure 12(b), peak in UV region is found to be at 242 nm for HTZNP4 and HTZNP5. But for HTZNP6 it is at 259 nm. This red shift is due to increase in grain size. Here also an intense peak is observed at 360–380 nm. Maximum absorption is observed for HTZNP4. Size of nanorod is found to be maximum for HTZNP4. When particle size increases visible absorption increases. This may be due to the increase in number of Oxygen vacancies [22, 54, 57].

UV–vis spectra for determination of bandgap is given in figures 13 and 14. From figure 13, bandgap of HTZNP1 is found to be 3.14 eV, 3.12 eV for HTZNP2 and 3.18 eV for HTZNP3. Bandgap of HTZNP2 is found to be minimum. In figure 14, bandgap of HTZNP4 is obtained as 3.13 eV, 3.11 eV for HTZNP5 and 3.10 eV for HTZNP6. HTZNP6 is having minimum bandgap. PL intensity of HTZNP2 and HTZNP6 is found to be maximum. Bandgap value is minimum for these samples. Interplanar spacing is found to be maximum for these two samples. Variation in bandgap can also be occurred due to size difference [29, 41].

3.8. Strain calculations from W-H plot
Strain is developed in nanoparticles due to crystalline imperfections and distortions [11]. High intensity of photoluminescence peaks, smaller bandgaps and large interplanar spacing observed in HTZNP2 and HTZNP6 may be caused by this induced strain. Figures 15 and 16 show W-H plots which are used to determine the strain. Negative slope of the fitted line found in HTZNP2, HTZNP4, HTZNP5 and HTZNP6 is referred to compressive strain and positive slope obtained for HTZNP1 and HTZNP3 indicates tensile strain [11]. W-H equation for determination of strain is given by equation (2)

\[ \varepsilon = \frac{\beta \cos \theta}{4 \sin \theta} \]  

Here, \( \theta \) is the x-ray diffraction angle and \( \beta \) is full width at half maximum obtained from XRD analysis. \( \varepsilon \) is the slope of the fitted line which indicates strain in the sample [11]. From figure 15 strain present in HTZNP1 is
Figure 13. Bandgap determination from UV–vis absorption spectra of (a) HTZNP1, (b) HTZNP2 and (c) HTZNP3.

Figure 14. Bandgap determination from UV–vis absorption spectra of (a) HTZNP4, (b) HTZNP5 and (c) HTZNP6.

Figure 15. W–H plot of HTZNP1, HTZNP2 and HTZNP3.
obtained as 0.093, 0.302 for HTZNP2 and 0.116 for HTZNP3. Strain calculated in HTZNP2 is more than other two samples. Figure 16 shows a strain of 0.115 in HTZNP4, 0.120 in HTZNP5 and 0.152 in HTZNP6. Here HTZNP6 is showing maximum strain. Micro strain is related to lattice misfit, which causes changes in d-spacing [46].

Enhanced optical properties (High intensity of photoluminescence peaks, smaller bandgaps) and morphological changes (large interplanar spacing) observed in HTZNP2 and HTZNP6 may be caused by this high value of strain developed in these samples.

4. Conclusions

ZnO nanoparticles were synthesized by hydrothermal method under different reaction conditions. It is observed that reaction parameters play a very important role in determining size and shape of nanoparticles. FTIR study revealed the effect of reaction parameters on vibrations of various functional groups. From TGA it is clear that all samples started to decompose only after 200 °C and increase in reaction temperature pushed this value to a higher temperature 270 °C. XRD and TEM analysis proved the change in structural properties of nanomaterials with respect to the change in reaction time and temperature. When reaction temperature is increased, crystallite size is reduced and a blue shift is observed in PL analysis. It is observed an increase in grain size as a result of increase in reaction time and hence a red shift in photo luminescence. A decrease in bandgap value is observed for samples with more interplanar spacing. PL intensity is appeared maximum for these samples. Strain calculated from W-H plots is maximum for HTZNP2 under same reaction temperature and HTZNP6 under same reaction time.

Hydrothermal synthesis can be chosen as a suitable method for synthesis of different nanostructures with considerable optical properties. Here nanoflowers and nanorods are showing excellent photoluminescence, UV absorption and suitable bandgap. Nanorods are thermally stable than nanoflowers. Optical properties and thermal stability are the key factors for solar cell materials. Based on their optical properties and thermal stability we can propose these hydrothermally synthesised nanorods and nanoflowers for different layers of solar-cell.

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