



Role of NaOH concentration on Structural, Morphological and Optical Properties of ZnO Nanopowders Synthesized by Sol-gel process

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Abstract : In the Present work, Structural, Morphological, Compositional and optical properties of ZnO Nanopowders synthesized by sol-gel process at different concentrations of NaOH were reported. The synthesized Nanopowders were further analyzed using XRD, SEM, FTIR, UV-Visible optical absorption and PL characterizations. Crystalline size and Lattice strain determined from XRD spectra. Morphology of Nanopowders viewed from SEM images observed at different magnifications. The presence of Functional groups analyzed from FTIR spectra. Optical properties of absorption and emission were resolved using optical absorption and PL characterizations. From the results it was very clear that particles synthesized with different NaOH concentrations played a vital role on Structure, Surface morphology, optical properties of Nanopowders. As synthesized Nanopowders can further utilized in Fabrication of Various optoelectronic devices including solar cells, LED etc

Keywords: FTIR ,Nanoparticles,SEM,XRD,Zinc Oxide

Introduction

Nanotechnology is an emerging field of science and technology and it has been applied to various fields' right from medicine, textile and education to defense and manufacturing. The Concept of miniaturizing devices to the ultimate atomic scale became dominant technological development for the last few years. Nanostructured materials are objects of intermediate size between microscopic and molecular structures. They include Nanorods, nanowires, nanopores, nanosheets, nanoparticles etc. Nanoparticles considered as particular interest in applications of optoelectronic devices [1]. Among these nanomaterials, the metal oxide nanostructures have become of particular interest to scientists for the development of different optical, biochemical and biomedical nanodevices [2]. Metal oxide nanoparticles show fast electron transfer properties as they have high surface area to volume ratio, low toxicity, are environment-friendly, have chemical stability and biocompatibility [3]. They rapidly helps in improving the performance of Nanomaterials. Among metal oxide Nanoparticles ZnO nanoparticles due to their wide energy band gap of 3.37 eV, biocompatibility, high electron mobility, fast electron transfer rate, environmental friendly, high melting point, these are used to fabricate sensitive and precise nanodevices based on nanomaterials for the application of sensing, optical absorption and luminescence emission [4,5]. Researchers reported ZnO Nanoparticles could enhance light-trapping for solar energy technology and LEDs (Yeong Hwan Ko et al 2014), ZnO Nanostructures considered as excellent material for fabrication of highly sensitive and selective gas sensors (Rajesh kumar et al 2015) ZnO nanoparticle dispersed PANI is a promising material for emissive layer in polymer light-emitting diodes. (J.Kavyatri et al 2013) ZnO nanoparticles coating with PVA is a good material for small-signal, visible blind, and wavelength selective UV detection. (Shayla Sawyer et al 2011).

As seen from literature Various growth methods were reported for synthesis of Nanoparticles **Swaroop K. and H.M. Somashekarappa*2015** reported on Effect of Ph values on surface Morphology and Particle size variation in ZnO Nanoparticles Synthesised by co-precipitation Method. **Numan salah et al 2011** reported High-

energy ball milling technique for ZnO nanoparticles as antibacterial material. Ozlem Altintas *et al* 2010 reported on Nanoparticle synthesis by microemulsion method. J.Tamil Illakkiya 2014 reported on Characterization of ZnO Nanoparticles synthesized by wet chemical method. Hamid Reza Ghorbani *et al* 2005 reported on Synthesis of ZnO Nanoparticles by Precipitation Method. Yan chu *et al* 2007 reported on Preparation of pure ZnO nanoparticles by a simple solid-state reaction method

From literature It has also been observed that sol -gel method has several advantages because of low temperature (<100°C) processing, cheap, environment- friendly etc. Scientists synthesized ZnO Nanoparticles by solgel process. Riyadh M. Alwan *et al* 2015 Synthesis of Zinc Oxide Nanoparticles via Sol – Gel Route and Their Characterization, M. Shayani Rad *et al* 2012 presented on Synthesis and structural, optical and antibacterial characterization of ZnO and ZnO:Ag nanopowders prepared via modified sol-gel method, T.V.Kolekar *et al* reported on Synthesis By Sol-gel Method And Characterization Of Zno Nanoparticles. M. Ebrahimzadeh Abhirashmi *et al*, 2010 reported on synthesis and structure of pure and Mn doped ZnO Nanopowders by solgel process.

In the present work ZnO Nanopowders were synthesized with different concentrations of NaOH by Sol-gel method. These Nanopowders further characterized to determine the influence of NaOH concentration on structural, morphological, compositional and optical properties of ZnO Nanopowders.

Experimental Procedure

Chemicals Required

Zinc Nitrate $\text{Zn}(\text{NO}_3)_2$, Sodium Hydroxide NaOH, Ethanol, All chemicals utilized were of Analytical grade. All chemicals are used without further purification.

Experimental Procedure

The nanopowders were synthesized by using the following process .First aqueous solution of Zinc Acetate 0.2 M was prepared by dissolving zinc Acetate in ethanol 60 ml with continuous stirring of Zinc Nitrate using a magnetic stirrer for 2 hrs .Aqueous solution of sodium hydroxide was prepared in the similar way with continuous stirring with magnetic stirrer for 2 hrs. When the chemicals dissolved the prepared aqueous sodium hydroxide solution added to Zinc acetate solution and resultant mixture so farmed kept under vigorously stirred for 3 hours till white precipitate obtained within the solution. The resultant precipitate centrifuged and allowed to stayed to digest for 24 h at room temperature. During this time, OH⁻ and Cl⁻ ions were diffused through the medium and white gel-like precipitate of $\text{Zn}(\text{OH})_2$ was formed. The remaining solution centrifuged for 10 min and the precipitate was removed. The obtained precipitate kept in an oven around 70°C till the solution dries .During drying $\text{Zn}(\text{OH})_2$ is completely converted to into ZnO. In the final step the particle was grinded to obtain Powder [6,7,8]

In a similar process Nanopowders were prepared at different concentrations NaOH 0.3 M, 0.4 M .The synthesized ZnO nano powders labeled as

Sample 1: ZnO nano powder with NaOH-0.2 M

Sample 2: ZnO nano powder with NaOH-0.3 M

Sample 3: ZnO nano powder with NaOH-0.4 M

Results and Discussions

XRD

Structural properties of Nanopowders were analyzed from XRD. Figure 1.1,1.2 and 1.3 represent XRD spectrum of ZnO Nanopowders synthesized using different concentrations of NaOH = 0.1,0.2 and 0.3 respectively. Various diffraction peaks were observed in the spectra. Figure 1.1 diffraction peaks appear at scattering angles (2θ) = 31.774°, 34.400°, 36.248°, 47.499°, 56.724°, 62.771°, 66.290°, 67.866°, 69.019°, 72.56°, 76.85°, 81.259°, 89.470°, 92.63°, 95.147° and 93.487° respectively. In Figure 1.2 diffraction peaks appear at 31.480°, 34.454°, 36.237° (0.388), 47.47°, 56.605°, 62.742°, 66.33°, 67.81°, 68.99°, 76.65°, 81.17°, 89.47° and

95.09° corresponding to lattice planes respectively. In Figure 1.3 18.358, 25.114, 29.318, 29.446, 68.34, 82.38, 31.95, 34.47, 35.44, 36.23, 39.007, 42.644, 46.610, 48.005, 48.43, 55.679, 56.58, 58.16, 59.84, 61.64, 62.368, 63.592, 68.34 and 82.38 respectively. The XRD patterns of the samples reveal that all peaks correspond to the characteristic peaks of the hexagonal wurtzite structure of ZnO with space group P6₃mc and lattice parameters of $a = b = 0.3250$ nm and $c = 0.5207$ nm according to the JCPDS database 36-1451. The crystalline size calculated using Scherer's formula $D = 0.89\lambda / b \cos\theta$, where λ is the X-ray wavelength (0.15406 nm), θ is the Bragg diffraction angle, and b is the full width at half maximum (FWHM) of the peak diffraction is found to be 37.87 nm, 39.33 nm and 55.173 nm for NaOH 0.2 M, 0.3 M and 0.4 M respectively. No diffraction peaks from impurities and residues were detected, indicating that the synthesized products are pure ZnO NPs. Agglomeration is observed at higher concentrations of NaOH. From spectrum it was clear that increased concentration leads to significant lowering of crystallinity [9,10,11,17].

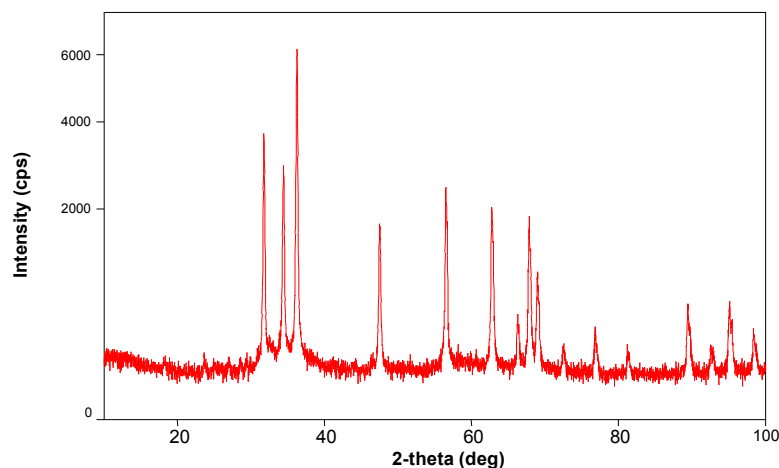


Figure 1.1: XRD spectrum of ZnO Nanoparticles synthesized with 0.2 M

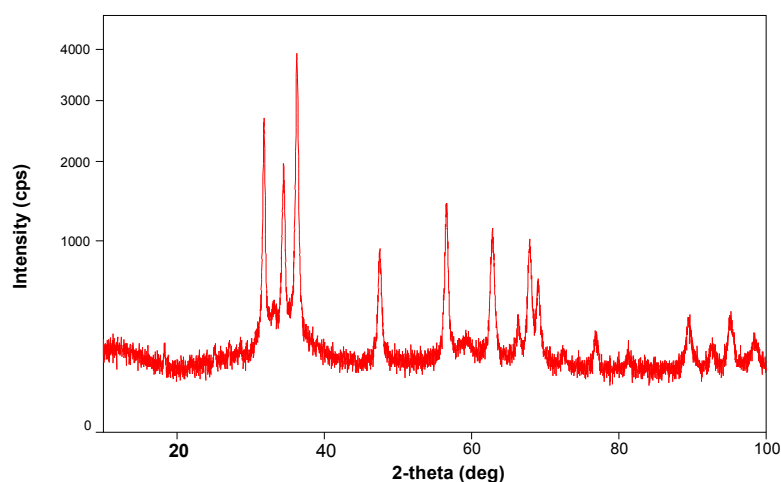


Figure 1.2: XRD spectrum of ZnO Nanoparticles synthesized with 0.3 M

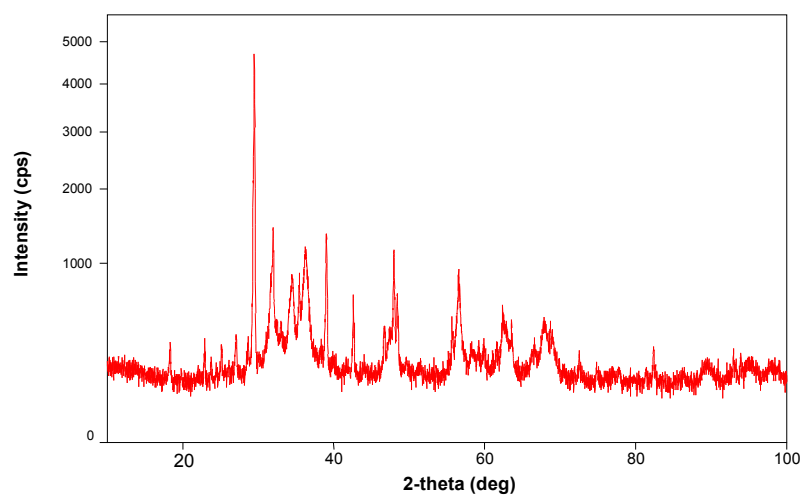


Figure 1.3: XRD spectrum of ZnO Nanoparticles synthesized with 0.4 M
Scanning Electron Microscopy

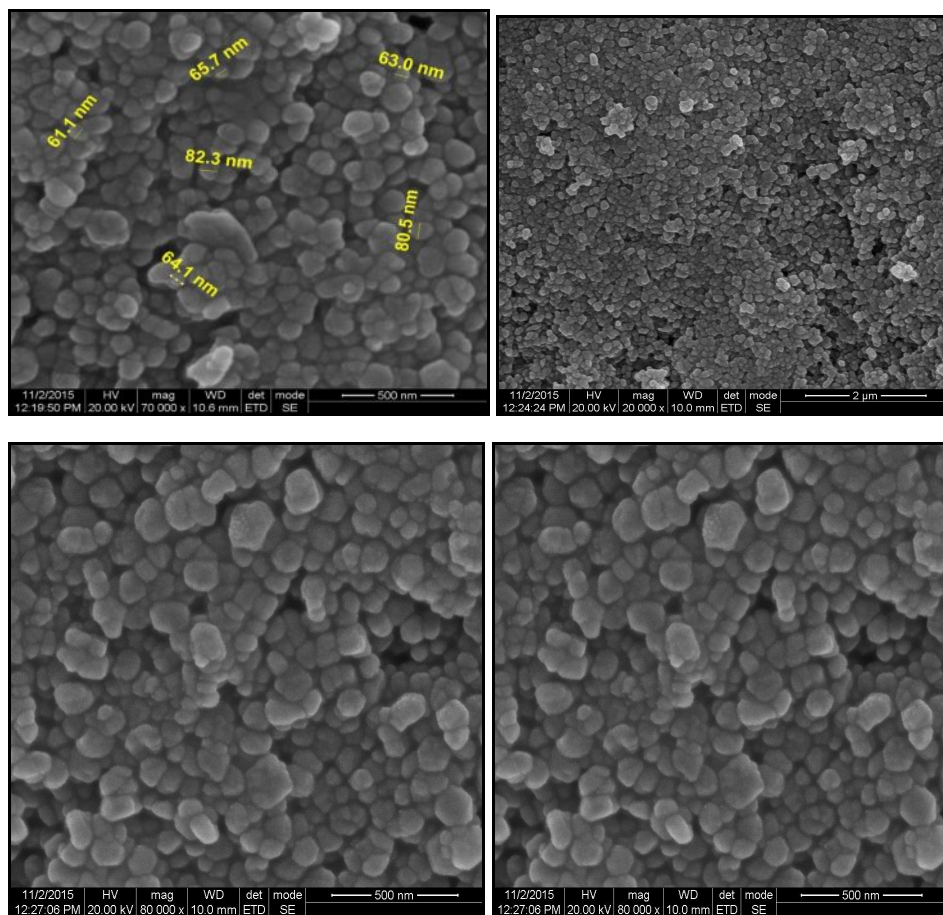


Figure 2.1: SEM images of ZnO nanoparticles at 0.2 M concentration.

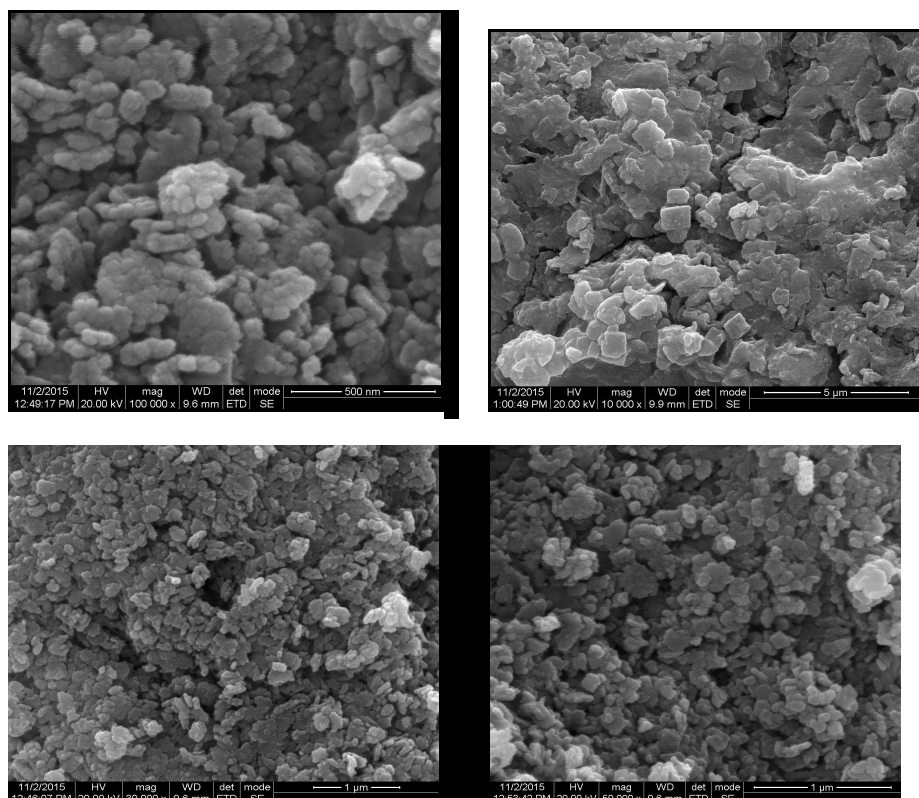


Figure 2.1: SEM images of ZnO nanoparticles at 0.3 M concentration.

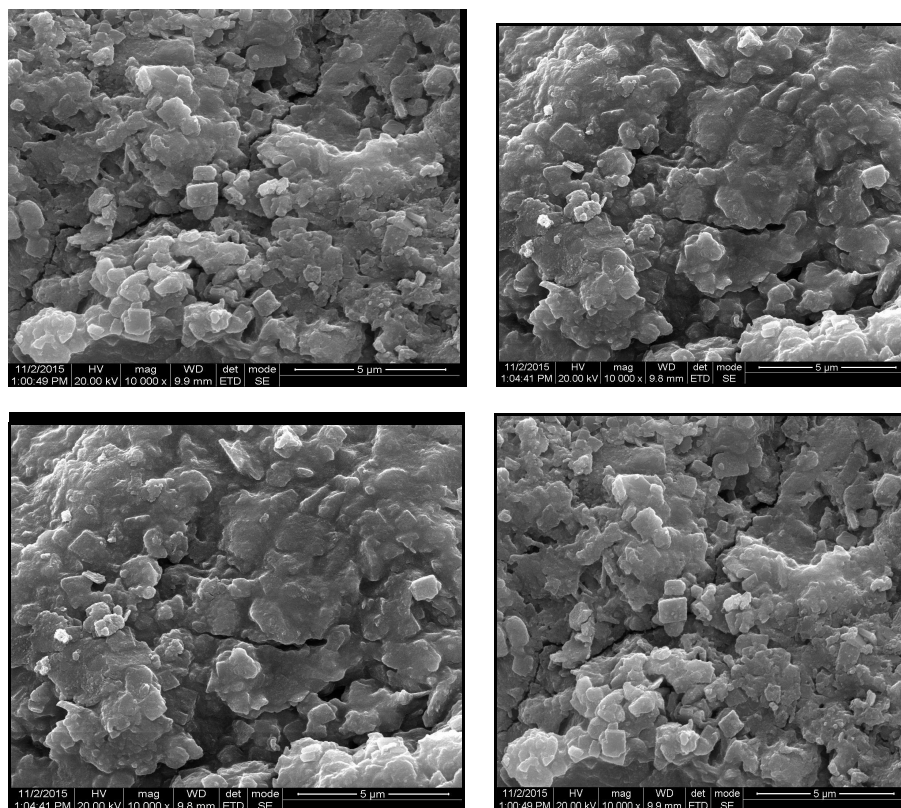


Figure 2.1: SEM images of ZnO nanoparticles at 0.4 M concentration.

Morphological Properties of powders were analyzed from SEM micrographs. Figure 2.1, 2.2 and 2.3 represent SEM spectra of ZnO Nanopowders observed at different magnifications. The images clearly represent formation of Nanoparticles. From images it was observed that, Sample synthesized at NaOH -0.2 M composed

of monodispersed and spherical shaped nanoparticles as concentration of NaOH is further increased to 0.3 M the shape of the particle changes and particle size also increases. The morphology observed in the samples show fine grains of ZnO converted in to particles when observed at different magnifications. Particles changed to random morphology with irregular particle size distribution were observed at high concentration of NaOH-0.4. From spectra it was clear that particles synthesized at 0.1 M concentration of NaOH show the zinc oxide is in pure form and particles are beautiful white colored spherical nanoparticles [12,13,16]

Fourier Transform Infrared spectroscopy

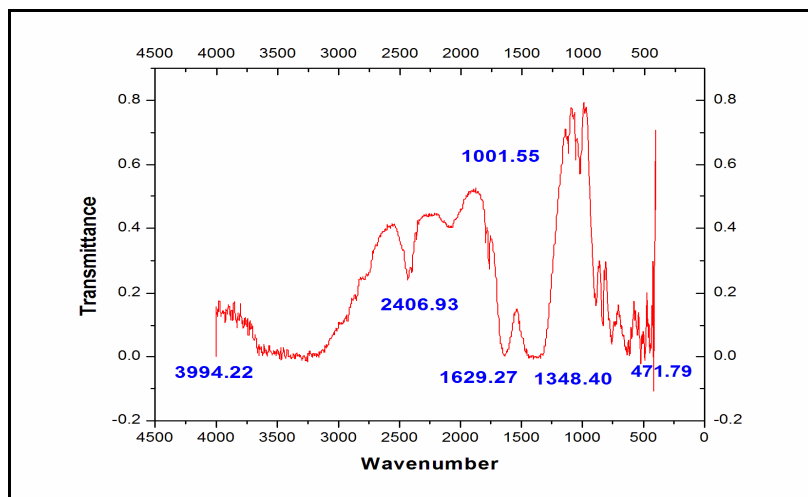


Figure 3.1 :FTIR spectrum of ZnO Nanoparticle of 0.2 M

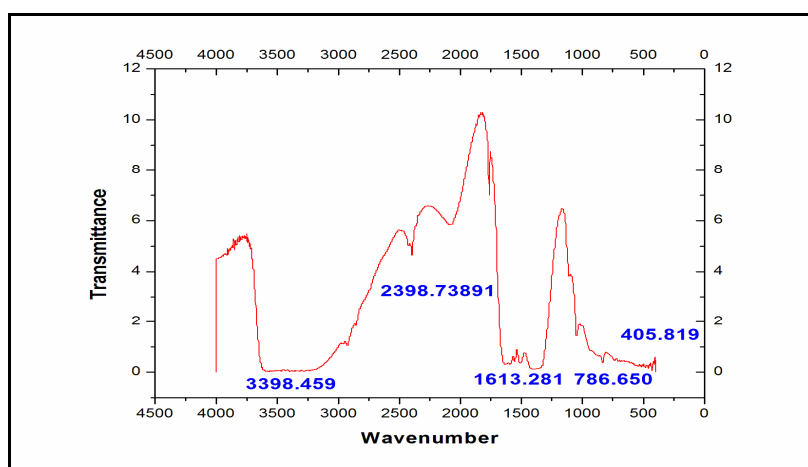


Figure 3.2 :FTIR Spectrum of ZnO Nanoparticle at 0.3 M

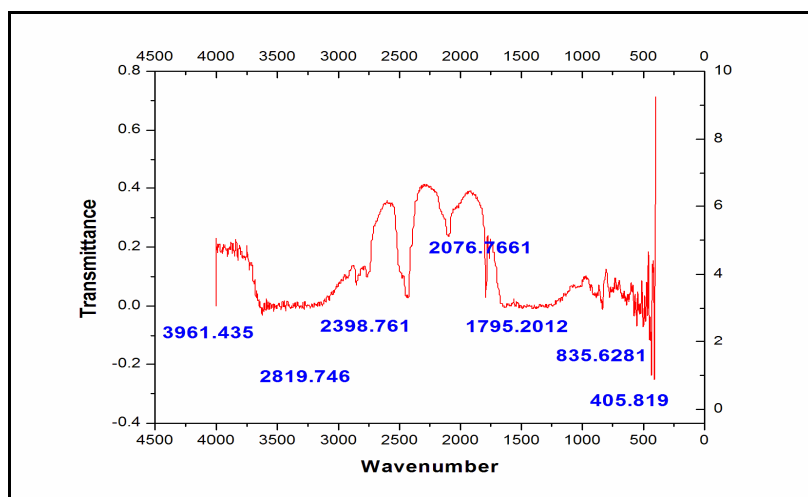


Figure 3.3 :FTIR Spectrum of ZnO Nanoparticle at 0.4 M

The formation of wurtzite structure was further confirmed from FTIR spectra. Figure 3.1, 3.2 and 3.3 represent FTIR spectra of ZnO nanopowders recorded in the range 4500 -500⁻¹ synthesized for different concentrations of NaOH =0.1, 0.2 and 0.3 respectively. Various bands were observed in the FTIR spectra. The position and number of absorption bands not only depend on crystal structure and chemical composition but also on crystal morphology. The broad band observed around 3500 cm⁻¹ and 1600 cm⁻¹ corresponds to O-H stretching vibration due to the absorbed water on the surface of the samples. The absorption around 2500 cm⁻¹

is because of the presence of CO₂ molecules in the environment. The intense absorption peak at ~400 cm⁻¹ is

related to the stretching vibrations of Zn-O bond. The carbonate stretches in samples are observed in samples at 1540 and 1480 cm⁻¹. From spectra it was observed that the peaks at high concentration of NaOH possess peaks leads to agglomeration and also possess with the lower crystallinity [14,15]. The results further confirm XRD results of the spectra

UV-Visible Optical Absorption Spectroscopy

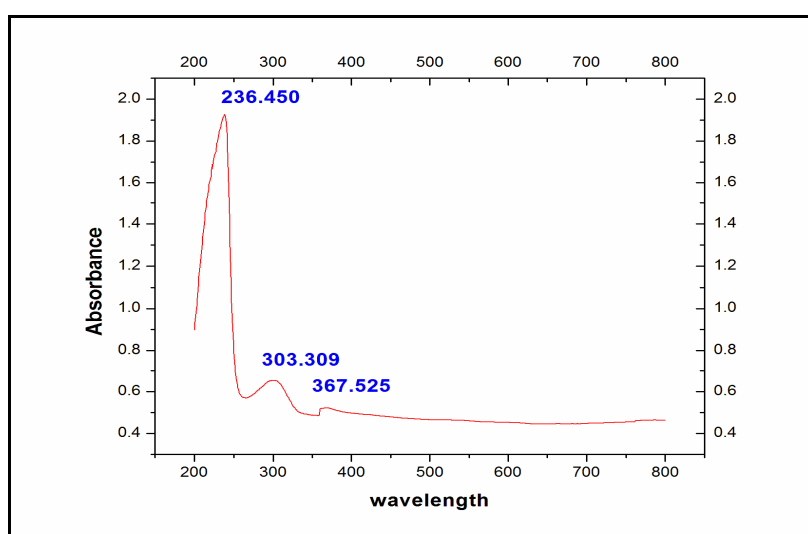


Figure 4.1 : UV-Visible Absorption spectrum of ZnO Nanoparticle synthesized using 0.2 M

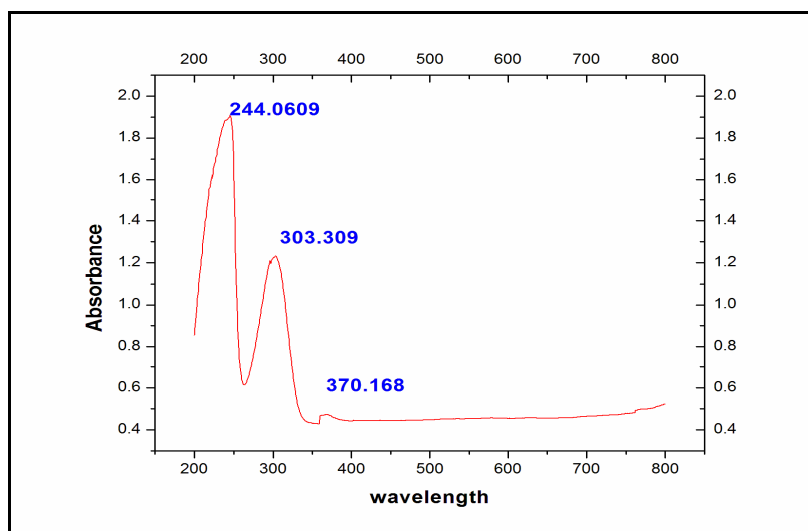


Figure 4.2 : UV-Visible Absorption spectrum of ZnO Nanoparticle synthesized using 0.3 M

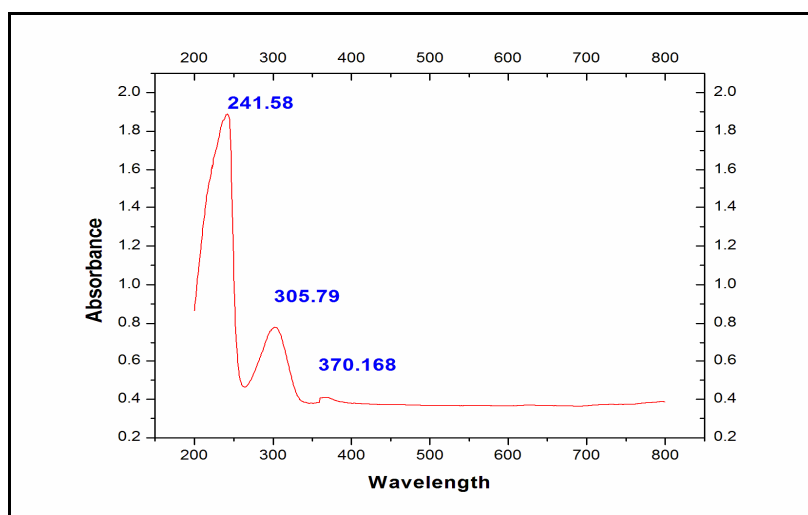


Figure 4.3 : UV-Visible Absorption spectrum of ZnO Nanoparticle synthesized using 0.4 M

Optical properties of absorption were analyzed from UV-Visible optical absorption spectra. Figure 4.1, 4.2 and 4.3 represent optical absorption spectra of three ZnO nanopowders recorded in the range 200-800 nm. Spectra show three absorption peaks around 240 nm, 300 nm, 370 nm, which corresponds to the Ultraviolet region of the spectra. Slight shift in the absorption peak with increased intensity was noticed due to increased NaOH concentration. From spectra, it was also clear that disappearance of absorbance peak in the visible regions can also be shown [18,19].

Photoluminescence spectroscopy

Optical properties of emission were analyzed from PL spectroscopy. Figure 5.1, 5.2 and 5.3 represent PL spectra of three ZnO nanopowders at different concentrations of NaOH. Spectra exhibit emission bands around 360 nm, which corresponds to UV emission. Three bands also were recorded around 400-500 nm, which attributes to blue luminescence, 500-600 nm, which corresponds to blue green emission in the visible region of PL spectra. From spectra, it was observed that the concentration of NaOH plays a vital role in increasing the luminescence intensity as NaOH concentration increases. Intensity in emission also increases. [18,19]

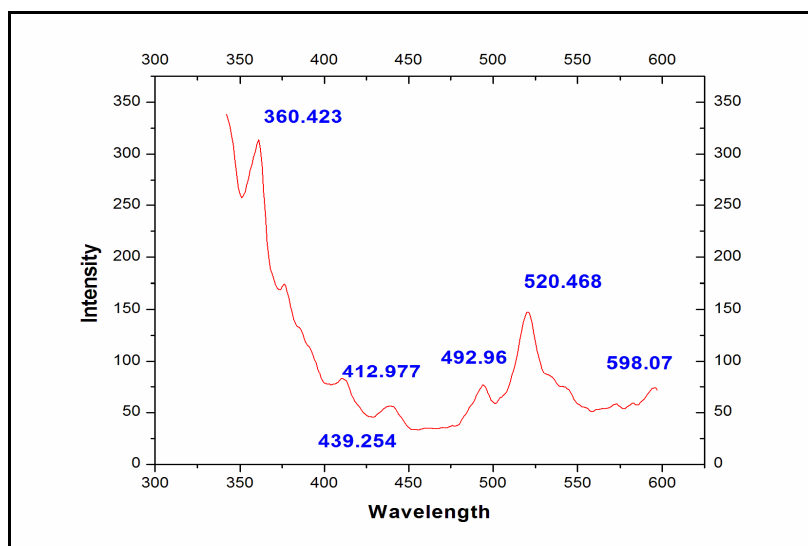


Figure 4.1: PL Spectra of ZnO Nanoparticles synthesized at NaOH-0.2 M

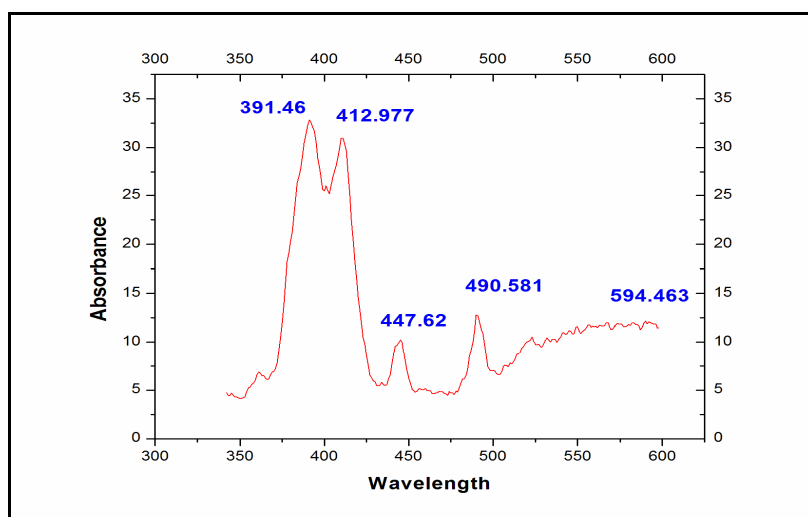


Figure 4.2: PL Spectra of ZnO Nanoparticles synthesized NaOH- 0.3 M

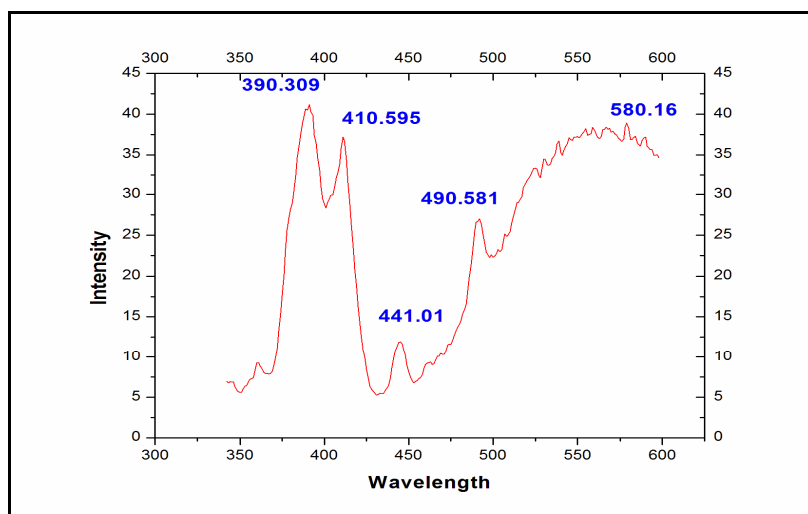


Figure 4.3: PL Spectra of ZnO Nanoparticles synthesized NaOH-0.3 M

Conclusion

In the present work ZnO nanopowders were synthesized at different concentrations of NaOH and powders were further analyzed to determine the influence of NaOH concentration on crystalline size, morphology, compositional, optical absorption and Luminiscence emission properties of ZnO Nanopowders. XRD confirms formation of Wurtzite structure of ZnO. The Decrease in crystallinity of Nanopowders was observed with increasing NaOH concentration. SEM confirms formation of nearly spherical images at low concentration. The structural properties of Nanopowders were further confirmed from the FTIR spectra. Various functional groups were also analyzed from FTIR spectra. Optical absorption reveals absence of absorption band in the visible region in all the samples. PL spectra shows Luminiscence blue and green emission of ZnO Nanopowders with intensity with increase in NaOH. The results clearly show Concentration of NaOH plays a vital role in controlling the size, morphology and optical properties of Nanopowders. The fabrication method utilized in this work is simple, low cost. As synthesized nanopowders can be used in fabrication of optoelectronic devices solar cells, LED's with blue and green luminiscence etc.

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