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Normality Effect on Characterization of Chemically Synthesized ZnO Nanoparticles

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Abstract: *The current research activities that focus on the preparation of ZnO nanostructure material and their physical and optical properties characterization. ZnO nanoparticles were synthesized using sol-gel chemical precipitation technique with zinc acetate and sodium hydroxide in distil water as a starting materials. Stock solutions of zinc acetate and sodium hydroxide at different normality (0.10N, 0.25N, 0.50N, 0.75N and 1N) were used for preparation of ZnO's. Synthesized sample of ZnO precipitate was dried at ordinary temperature for 12 hours and calcinied at 100^0C for 2 hours and were physically characterized by using XRD (X-ray diffraction), SEM (scanning electron microscope) and FTIR spectroscopy. The XRD spectra exhibit wurtzite hexagonal crystal structure. The particle size statistically estimated from XRD data show that the ZnO samples consist of nanosize particles. The optical properties of ZnO nanoparticles was studied by using UV-visible absorption and PL (photoluminescence). The effect of normality of different precursors used in preparation of ZnO nanoparticles, on spectroscopic characterizations was investigated. UV-Visible spectra give blue luminescence near band edge. PL exhibits green luminescence that suggest surface defect due to oxygen vacancies effect in ZnO nanoparticles.*

Keywords: ZnO nanoparticles, N-normality, XRD, SEM, FTIR, UV (Visible absorption), PL (photoluminescence)

I. INTRODUCTION

In this study, ZnO is an important compound semiconductor material due to its direct energy band gap (3.37eV) and large excitation binding energy (60 MeV). It has the unique morphological and optical properties that can be used in a variety of applications, oxide coating for solar cell, gas sensor, UV photodiode, field emission device, capacitor, UV resistance coating, photo printing, electro-photography, electrochemical, catalytic, optoelectronic, photochemical, sunscreen lotion (creams), cosmetic and medicated creams.

ZnO nanoparticles have a great advantage to apply to catalytic reaction process due to their large surface areas and high catalytic activity. Since Zinc oxide shows different morphological, optical and chemical properties depending upon the size of nanoparticals, the morphological, optical and chemical properties of synthesed ZnO are to be investigated.

These wide varieties of prominent applications require the fabrication of special morphological and fictionalization of ZnO nanostructures (P.M.Aneesh *et al.,* 2007). Different methods have been used for the production of ZnO nanoparticles such as 1) Chemical synthesis 2) Hydrothermal method 3) Electrophoretic deposition 4) Co-precipitation 5) Mechanochemical-thermal synthesis 6) Chemical vapour depositions 7) Thermal decomposition 8) Sol-Gel method 9) Electrochemical depositions and 10) Anodization

The present study was aimed to i) Synthesis ZnO nanoparticle using simple Sol – Gel precipitation method by utilizing Zinc acetate and sodium hydroxide in distilled water, ii) study the morphological changes after change in normality of solution samples.

II. MATERIAL AND METHODS

Zinc acetate and sodium hydroxide were used as starting materials in chemical precipitation sol-gel method. The entire chemical used were of analytical reagent grade obtained from Merk (Mumbai) India and distil water is used for the preparation of solution.

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2.1 Chemically Synthesis ZnO

Zinc acetate and sodium hydroxide solution were prepared in distil water by stirring for 2 hours. In next step sodium hydroxide (NaOH) solution was slowly added into Zinc acetate drop-wise using a dropping funnel, this mixture was then stirred for 2 hours as a result a precipitation of the ZnO nanoparticles was formed in the solution. The synthesized precipitation was centrifuged, filtered and washed with distil water. The precipitate was dry in room temperature for 3 days and ground to fine powder using agate mortar and pestle. The obtained fine powder was calcined at 100^0C for 1 hour in oven to remove the unwanted byproducts. The prepared samples was then characterized by various techniques such as X-ray diffraction (XRD), UV visible absorption spectra, FTIR, and Scaning Electron Microscopy (SEM).

III. RESULTS AND DISCUSSION

3.1 XRD

The XRD pattern of ZnO nanoparticles grown at different Normality values (0.10N, 0.25, 0.50N, 0.75N, 1N) of reaction mixtures were presented in figure1.The XRD pattern exhibit distinct peaks attributed to hexagonal structure of ZnO nanoparticles. The pattern is well matched with standard JCPDS card no. (36-452). X-ray reflection intensity data were recorded over a wide range of 2θ = 20-80 degree. The average grain size of the samples was estimated with the help of Debeye-Scherrer equation (1) using the full width at half maxima of (100), (002), (101), (102), (110), (103), (200), (112), and (201) of the x-ray diffraction peaks. A significant change in crystalline size was obtained for the samples at different Normality.

$$D = \frac{0.94\lambda}{\beta COS\theta} \quad (1)$$

where **D** is the crystalline size nanoparticles (nm), λ is the wavelength of incident x-ray (nm), ß is the full width at half maxima and θ is the diffraction angle.

The grain size was found to be in the range of 22 nm to 33 nm depending on the growth condition. The normality of precursors was greatly influenced on the particles size and crystallinality of prepared ZnO nanoparticles. Grain size with normality was shown in table 1.

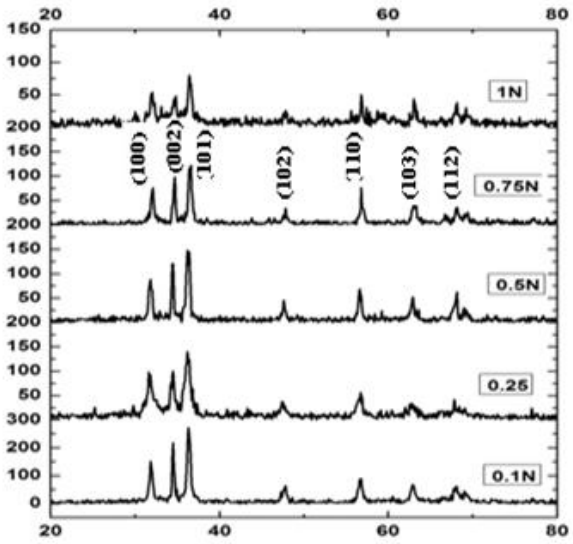

Figure 1. XRD spectra of ZnO Nano particles

Table 1. Normality and Grain Size

Sr. No.	ZnO sample with different Normality	ZnO crystalline size (D)nm
1	ZnO 0.10N	33.93
2	ZnO 0.25N	22.76
3	ZnO 0.50N	33.36
4	ZnO 0.75N	25.66
5	ZnO 1N	33.25

3.2 FTIR Study

Figure 2 shows the FTIR spectra of ZnO nanocrystals prepared at different Normality values of reaction mixture. The FTIR is powerful tool for investigation of molecular structure. The FTIR spectra exhibit very low intense peak at 3486 cm^{-1} can be assigned to O-H stretching mode for the adsorbed atmospheric moisture. The peak at 1638 cm^{-1} is attributed to OH bending mode of water and at about 1402.08cm^{-1} the -COO mode arises from the absorption of atmospheric CO2 on the surface of the nanoparticles (11). A strong band at 630.36 cm^{-1} was assigned to the stretching mode of Zn-H. The main absorption band at 495 cm^{-1} is due to Zn-O stretching vibrations. The O-H bending and –COO vibrational bands decreasing in case of 0.10N, 0.50 and 1N normality ZnO NPs.

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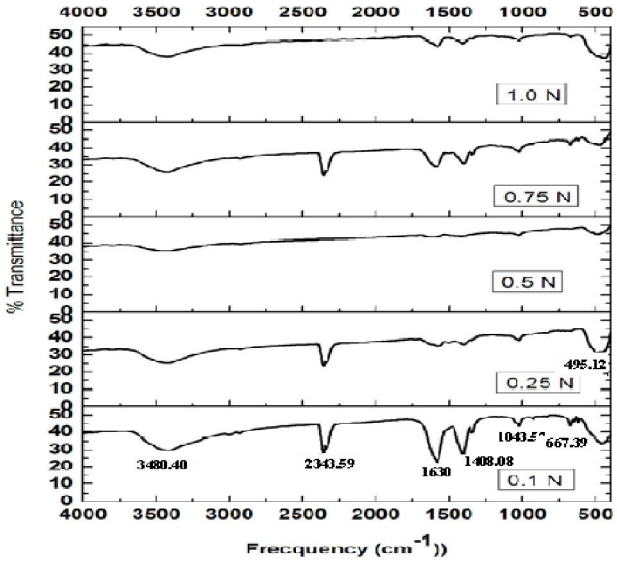

Figure 2. FTIR of ZnO Nanoparticles prepared for different Normality

Table 2. FTIR spectra of ZnO nano particles			
Sr. No.	Frequency of Vibrational Band	Intensity	Vibrational Group
1	418.57	21.769	Zn-
2	1271.49	67.331	Zn-O
3	1411.94	51.665	Zn-O
4	1566.25	49.237	ZnO-H
5	3421.83	42.281	OH

3.3 Surface Morphology

The morphology of the nanostructures obtained for prepared ZNO NPS by using Scanning Electron Microscopy tool are shown in Figures 3. The SEM micrographs exhibit different morphology depending upon Normality of reaction mixture used in preparation of ZnO NPS. The ZnO nanoparticles prepared from reaction mixture 1N exhibit excellent nanoflower. For normality value0.10N, 0.25N 0.75N the NPS exhibit small rod like crystals set in ZnO matrix. The different morphology of ZnO NPS leads to significant change in structural and optical properties.

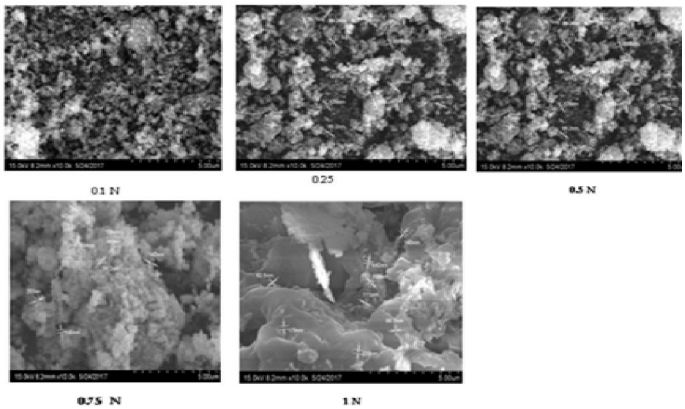

Figure 3. SEM images of ZnO nanoparticles prepared using different normality of Precursors

3.4 Optical Study

UV-visible absorption spectra of prepared ZnO nanoparticles are presented in figure 4. The optical absorption exhibits strong absorption peak by the ZnO NPs prepared from reaction mixture of normality value 0.10N and 0.50N in the UV region at 372 and 382 nm respectively. They also show intense absorption at 402 to 406 nm wavelength. The 0.25N and 0.75N sample show higher blue shifting as compared to others. Other ZnO NPs prepared from reaction mixture of normality values 0.10N, 0.50N and 1N exhibit significant absorption peaks in the UV-region. The band gap energies obtained from the spectra are tabulated in table 2, shows variation with normality values. The ZnO particles Prepared form reaction mixture of normality values0.10N, 0.5N and 1N exhibit band gap 3.28 eV and 3.34 eV.

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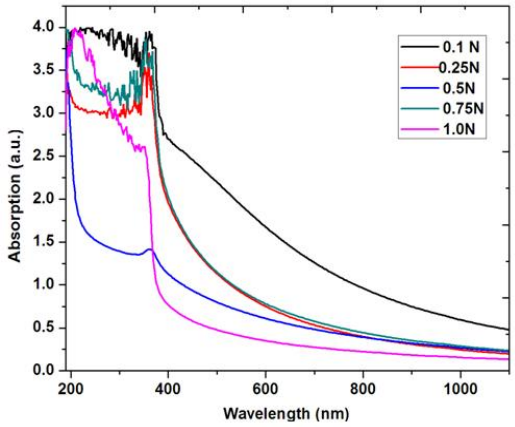

Figure 4. UV- Visible absorption spectra of ZnO Nanoparticles prepared for different Normality

Table 3. Energy band Gap and wavelength at band edge of ZnO Nano Particle

Sr.No.	Normality	Wavelength (λ nm)	Band gap energy (eV) Eg=hc/λ
1	0.10N	372	3.28
2	0.25N	402	3.42
3	0.50N	382	3.31
4	0.75	406	3.29
5	1N	398	3.34

3.5 PL Photoluminance

In PL spectra of the 1N normality, ZnO nanoparticles synthesized from the pure precursors shown in figure 5. The presence of the peak at 372nm is corresponding to the excitation emission or UV emission in ZnO Nanoparticles. The peak in the spectral range 420nm to 562nm corresponding to the visible emission of ZnO nanoparticles. The peak 420nm corresponds to the blue band and the peak at 460nm corresponds to weak blue-green band, the peak at 442nm to 562nm is due to various defects such as interstitial Zn and the presence of the acceptor and donor states in the region between the valance and conduction bands. The green band results due to the presence of slight oxygen vacancy defect in the ZnO nanoparticles.

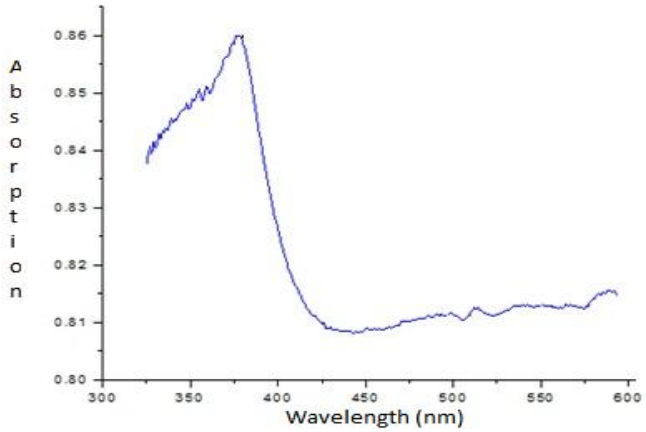

Fig. 5 PL Photoluminescence

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IV. CONCLUSION

In this study, ZnO nanorods are successfully fabricated by Sol-Gel chemical method. The XRD pattern confirms the excellent hexagonal crystallinity of ZnO nanorods prepared from reaction mixture of normality values 0.50N and 1N. The FTIR spectra exhibits strong absorption band at 477 cm^{-1} attributed to Zn-O stretching mode for normality values 0.10N, 0.50N and 1N. The sharp optical absorption peak at 398 nm and 372 nm exhibited by ZnO (0.10N, 0.50N and 1N) NPS in the UV region of electromagnetic spectrum and significant blue shifted.

From the study of XRD, FTIR SEM and UV –Visible it is concluded that for presently employed Sol-Gel preparation technique the optimized normality value of the reaction mixture may be in between 0.50N and 0.75 N to obtain good quality ZnO NPS.

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