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Structural and Optical Properties of Zno Nano Particles

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Abstract: *In the present report preliminary studies on synthesis and growth of ZnO nanocrystals have been reported. ZnO precursors were prepared by precipitation method from Zinc nitrate and Ammonia in aqueous solutions at a pH value9.0 .ZnO nanocrystals were then synthesized by heating the precursor in a muffle furnace at temp 350°C for 3 hours and allowed to cool to room temperature. The precursors and synthesized nanoparticles were characterized by X-Ray diffraction (XRD) and the results showed a single phase wurtzite structure for ZnO nanoparticles. It was found that the synthesized ZnO nanocrystals have wurtzite structures with a=b=3.214 Å and c=5.154 Å. Crystallite size was calculated using Debye_Scherrer's equation and the average crystallite size from first three peaks was found to be 55.18 nm. The morphology of prepared ZnO nanopowders was characterized by scanning electron microscope (SEM). From the compositional analysis by Energy dispersive analysis of X-ray (EDX) it was confirmed that Zinc and oxygen are present in the sample. Optical characterization was done to study other characters. Diffuse reflectance spectroscopy (DRS) results shows that the band gap of ZnO nanoparticles is 3.20eV.*

Keywords: *ZnO, nanoparticles, precipitation, XRD, SEM, band gap*

I. Introduction

In many areas of chemistry, physics and material science transition metal oxides with nano structure have attracted substantial interest during the last few years because of their novel optical and electrical properties as well as semiconductor crystals with a large binding energy [1]. Zinc oxide is an important n-type semiconductor with a direct band gap of 3.37 eV with large excitation binding energy (60 meV) at room temperature [2]. In a variety of applications, Zinc oxide nanoparticles are used as photo catalyst [3,4], antibacterial treatment [5] ,UV absorption, light emitting diodes, photo detectors, solar cells and gas sensors [6,7] .Searching of new methodology to synthesize uniform nanosized ZnO particles is of great importance both for fundamental studies and practical applications, and thus from literature survey it is seen that various methods such as hydrothermal method, sol-gel, spray pyrolysis and direct precipitation methods have been adopted for the fabrication of nano-sized ZnO particles with a uniform morphology, size and reproducibility. In this work, ZnO nanoparticles were synthesized using the cost competitive and simple precipitation process. The single step process with large scale production without unwanted impurities is desirable for cost effective preparation of ZnO nanocrystals. As a consequence, the low cost precursors such as zinc nitrate & ammonium hydroxide to synthesize ZnO nanoparticles through a simple precipitation route are used. Here we report preliminary studies on synthesis of ZnO nanocrystals using chemical method & the characterization of ZnO nanoparticles using XED, SEM, EDX & Diffused reflectance for UV-VIS study.

II. Experimental Details

Zinc nitrate, ammonium hydroxide (25% pure ammonia solution, density 0.91), purchased from Merck & de-ionized water were used without further purification. Zinc oxide nanoparticles were synthesized by wet chemical method using zinc nitrate and ammonium hydroxide precursors.

In this experiment 0.5M $Zn(NO_3)_2$ solution was kept under constant stirring using magnetic stirrer for half an hour to completely dissolve and ammonia solution was added under high speed (200rpm) constant stirring drop by drop touching the walls of the vessel to make the pH of the solution 8.0. The reaction was allowed to proceed for 45 minute at temperature 50°C. The beaker was sealed and the solution was allowed to settle for overnight and further the precipitate was filtered for several times using whatman filter. The precipitate was heated for 3 hrs with temperature 70°C to dry it completely in a hot air oven and grinded with a mortar. After 24 hrs it was heated in a muffle furnace at 350 °C for 3 hrs and allowed to cool to room temperature.

The prepared ZnO nanoparticles were characterized for their optical and nanostructure properties. X-ray diffraction pattern was recorded using Philips –X'pert Pro X-ray diffractometer operating at 40KV-30mA using CuK_α radiation of wavelength $\lambda = 0.1541$ nm in the scan range $2\theta = 20°$ to 70°. Morphology of the sample was investigated using Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Analysis (EDAX) has been used for compositional analysis of the prepared ZnO nanoparticles. The optical band gap E_g was

estimated from UV-Visible diffuse reflectance spectroscopic studies in a wavelength range from 200 nm to 700 nm.

III. Results and Discussion

3.1. XRD Analysis:

Figure 1 shows the XRD patterns of the samples. The 2θ variation was employed with a 0.02 degree step and a time step of 0.5 second. Intensity in counts/sec is plotted against 2θ in figure 1. From this figure, well crystallized diffraction peaks were observed. Also, Table 1 shows that calculated "d" values are in good agreement with those taken for JCPDS card file data for ZnO powder. That is the prepared material has crystallized in a hexagonal wurtzite structure.. Presence of several peaks indicates random orientation of the crystallites. The average crystallite size was calculated from the Debye-Scherrer formula

$$D = \frac{K\lambda}{\beta COS\theta}$$ ---------- (1) Where k denotes Scherrer constant (the shape factor) =0.9, λ= 0.154 nm is the wavelength of the incident CuK_α radiation; β represents full-width at half maximum of the respective peak and θ is the Bragg diffraction angle. The average crystallite size was found to be 55.18 nm from 1st three peaks of the XRD data.

From XRD data the lattice parameters were calculated from the following formula [8]:

$$d_{hkl} = \frac{1}{\sqrt{\frac{4}{3}\left(\frac{h^2 + k^2 + hk}{a^2}\right) + \frac{l^2}{c^2}}}$$ ---------- (2)

$$a = \frac{\lambda}{\sqrt{3}\sin\theta_{100}} \quad \text{And} \quad c = \frac{\lambda}{\sin\theta_{002}}$$ ----- (3) where λ= 1. 54 Å is the wavelength of the X-ray radiation used, d_{hkl} is the crystalline surface distance for hkl indices, θ_{100} and θ_{002} are the angles of diffraction peaks (100) and (002) respectively. The unit cell volume for hexagonal structure can be calculated by the relation

$$v = a^2 \text{ c sin } 60° = \frac{\sqrt{3}}{2} a^2 c$$--- (4).

The calculated lattice parameters are listed in Table 2.

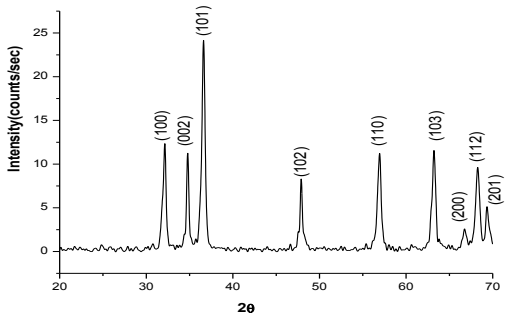

Fig1: XRD patterns of synthesized ZnO nanoparticles

Table1: Comparison between standard and observed "d" values of synthesized ZnO nanoparticles

hkl	ZnO prepared in this work		JCPDS 36-1451	
	2θ(°)	d value (Å)	2θ(°)	d value (Å)
100	32.1532	2.781	31.770	2.814
002	34.7676	2.577	34.422	2.602
101	36.6078	2.452	34.353	2.476
102	47.8697	1.898	47.539	1.913
110	56.9193	1.616	56.603	1.626
103	63.2637	1.468	62.864	1.478
200	66.7831	1.399	66.378	1.379
112	68.2939	1.371	67.961	1.360
201	69.3535	1.354	69.100	1.358

Table2: Unit cell parameters, cell volume, and c/a ratio of ZnO nanoparticles calculated from XRD data

	a (Å)	c (Å)	c/a ratio	Unit cell volume $(Å)^3$
Prepared sample	3.214	5.154	1.6036	46.105
JCPDS	3.250	5.207	1.6020	47.630

Table 3: crystallite surface distance

hkl	d (Å)
100	2.786
002	2.577
101	2.452

3.2 SEM & EDX Study:

Fig2 represents the SEM image of ZnO nanoparticles at different magnifications. These pictures confirm the formation of ZnO nanoparticles. These pictures substantiate the approximate spherical shape to the nanoparticles, and most of the particles exhibit some agglomeration. From the pictures, it also can be seen that the size of the nanoparticle is more than that calculated from the Debye-Scherrer formula indicating the agglomeration of crystallites in ZnO nanoparticles. The aggregation of particles should had been originated from the large specific surface area and high surface energy of ZnO nanoparticles [8]. The aggregation occurred probably during the process of drying [9,10]. From EDX analysis it is seen that only Zinc and oxygen are present in the sample and no other impurities are present.

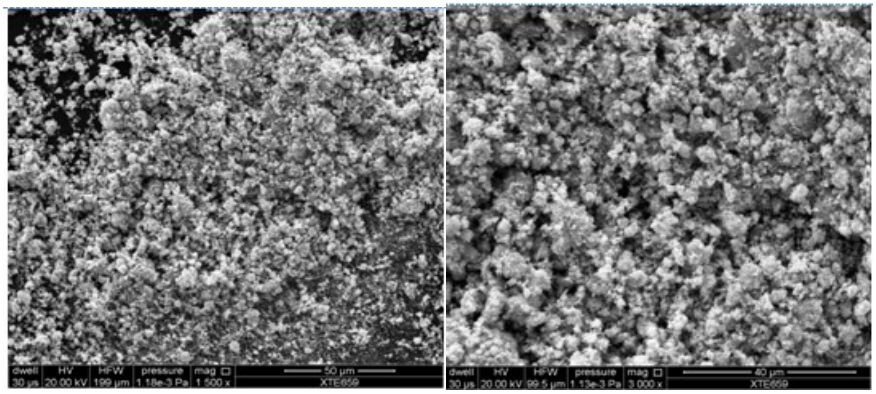

Fig2: SEM image of ZnO nanoparticles

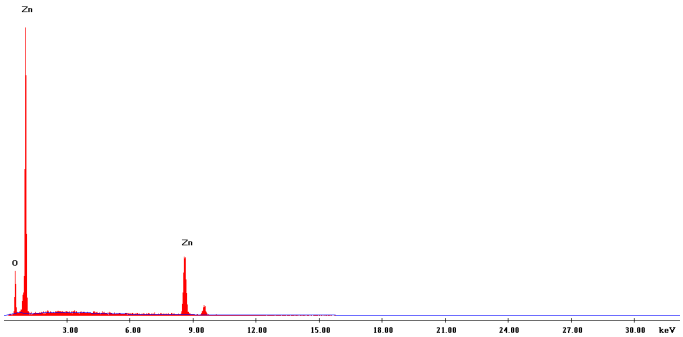

Fig3: EDX of ZnO nano particles

Table 4: Compositional analysis of the sample

Element	weight percentage	Atomic percentage
O K	18.96	48.87
Zn K	81.04	51.13
Total	100	100

3.3 Optical characterization:

It was carried out by measuring the diffuse reflectance spectroscopy in UV-Vis range. The spectrum was taken in the range of 200-700 nm. Fig 4 shows the diffused reflectance spectra (absorbance as a function of wavelength). The exciton absorbsption is at about 362 nm. The optical band gap E_g of the nanocrystals was calculated from Tauc plot as shown in Fig 5. The presence of a single slope in the plot suggests that the films have direct and allowed transition. For such transition we have $(\alpha h\nu)^2 = A(h\nu - E_g)^n$ where α is absorption coefficient, hν is photon energy, E_g is optical band gap, n is 1 for direct transition & A is a constant. The band gap energy is obtained by extrapolating the straight line portion of the plot to zero absorption coefficient. The band gap value of ZnO nanoparticles is found to be 3.20 eV. This red shift of the band gap energy is due to agglomeration of the nanocrystallites into larger crystallites as reported by various authors in different literatures [11.12].

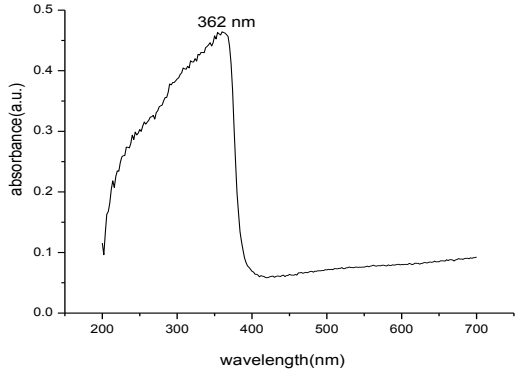

Fig4: DRS of synthesized ZnO nanoparticles

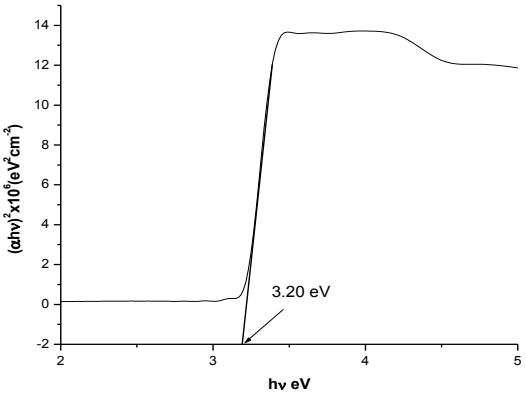

Fig 5:Tauc plot for determination of band gap

IV. Conclusion:

The ZnO nanoparticles with hexagonal structure have been synthesized by simple cost competitive precipitation method after annealing the precursor at 350°c.The prepared ZnO particles were characterized by XRD, SEM, EDAX and diffused reflectance study. XRD and SEM studies confirmed the nanostructures for the prepared ZnO nanoparticle. SEM micrographs illustrates that the particle size increases at high annealing temperature. The optical direct band gap of the synthesized nanoparticles is found to be 3.20 eV. The ZnO nanoparticles can be used in different industrial applications, namely, luminescent material for fluorescent tubes, active medium for lasers, sensors, and so forth.

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References

[1]. Hu, J.T., Odom, T.W. and Liebe, C.M. (1999) Chemistry and Physics in One Dimension: Synthesis and Properties of Nanowires and Nanotubes. Accounts of Chemical Research, **32**, 435-445. http://dx.doi.org/10.1021/ar9700365

[2]. C. M. Lieber, Solid State Commun. 66 (1998) 5309.

[3]. Annapoorani, R., Dhananjeyan, M.R. and Renganathan, R. (1997) An Investigation on ZnO Photocatalysed Oxidation of Uracil. Journal of Photochemistry and Photobiology A: Chemistry, **111**, 215-221.http://dx.doi.org/10.1016/S1010-6030(97)00170-6

[4]. Huang, W.-J., Fang, G.-C. and Wang, C.-C. (2005) A Nanometer-ZnO Catalyst to Enhance the Ozonation of 2,4,6-Trichlorophenol in Water. Colloids and Surfaces A: Physicochemical and Engineering Aspects, **260**, 45-51. http://dx.doi.org/10.1016/j.colsurfa.2005.01.031

[5]. Sánchez, L., Peral, J. and Domènech, X. (1996) Degradation of 2,4-Dichlorophenoxyacetic Acid by in Situ Photogenerated Fenton Reagent. Electrochimica Acta, **41**, 1981-1985. http://dx.doi.org/10.1016/0013-4686(95)00486-6

[6]. Preetam Singh, Ashvani Kumar, Ajay Kaushal, Davinder Kaur, Ashish Pandey, R.N.Goyal, Bull. Mater.Sci.31(3) (**2008**) 573-577

[7]. M.S.Wagh, G.H.Jain, D.R.Patil, S.A.Patil, L.A.Patil, Sensors and Actuators B 115, (**2006**) 128- 133

[8]. Davood Raoufi, Journal of Luminescence, 134 (2013) 213-219

[9]. R.Y. Hong, J.Z. Qian, J.X. Cao, Powder Technol. 163 (2006) 160.

[10]. R.Y. Hong, J.H. Li, L.L. Chen, D.Q. Liu, H.Z. Li, Y. Zheng, J. Ding, Powder Technol. 189 (2009) 426.

[11]. H.L.Hartnagel,A.L.Dawar,A.K.Jain,C.Jagadish,SemiconductingTransparentThin Films, Institute of Physics Publishing, Bristol/PA, 1995

[12]. J.I. Pankove, Optical Progress in Semiconductors, Dover, New York, 1975.