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Synthesis and Characterization of Polyvinyl Pyrrolidone (PVP) Capped Zinc Oxide Nanoparticles

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Abstract—*Zinc oxide nanoparticles (ZnO-NPs) have been grown by simple chemical precipitation method using Zinc nitrate hexa hydrate [Zn $(NO_3)_2$.$6H_2O$] and sodium hydroxide (NaOH) in the molar ratio 1:2 as source materials. Polyvinyl Pyrrolidone (PVP) is used as the capping agent in the synthesis of ZnO-NPs. The reaction temperature is maintained at 60^0C during the growth process. The as-grown ZnO-NPs have been investigated by X-ray Diffraction (XRD) study, Scanning Electron Microscopy (SEM), Ultra Violet-Visible (UV-Vis) absorption spectroscopy and Photoluminescence (PL) spectroscopy respectively. The XRD result reveals that as-grown ZnO-NPs exhibit good crystallinity with hexagonal wurtzite crystal structure and the average crystallite size calculated from XRD pattern is found to be 15nm. The SEM image shows that the grown ZnO-NPs are nearly spherical in shape with slight agglomeration. The UV-Vis absorption spectrum exhibits a blue shift in wavelength as compared to bulk ZnO and the PL spectrum shows strong emission in the UV range with the excitation wavelength 320nm.*

Keywords*: ZnO-NPs, chemical precipitation, blue shift, photoluminescence.*

1. INTRODUCTION

Zinc Oxide (ZnO) is one of the most important II- VI semiconductors which have drawn tremendous attention in the field of research because of its unique properties. A wide band gap (3.37eV) and large exciton binding energy (60meV) at room temperature makes ZnO a promising material in optoelectronics application such as laser diodes, light emitting diode (LED), etc [1]. ZnO is also known to possess good chemical stability, good conductive, piezoelectric and non toxic properties, and is highly transparent to visible light [1,2].These properties make it suitable for a variety of applications such as gas sensors, biosensors, chemical sensors, transparent electrodes in solar cells, varistors etc.[2,3,4]. ZnO crystallizes into three forms: hexagonal wurtzite, cubic zinc blende and cubic rock salt. The hexagonal wurtzite in ambient condition is the most common and stable structure of ZnO [5, 6] with lattice parameters a= 0.325nm and *c* =0.521nm [7]. Different methods have been developed for the synthesis of Zinc Oxide nanoparticles (ZnO-NPs) such as hydrothermal method [5, 8], sol-gel method [4, 7, 9], thermal decomposition method [10], chemical precipitation method [2, 11, 12] etc. The chemical precipitation method is one of the best methods which is simple, inexpensive and does not need any sophisticated instruments. Also, the different synthesis conditions like pH, reaction temperatures, precursors concentration etc. can be varied which lead to different size and shape of the so obtained nanoparticles. Several researchers have adopted the chemical precipitation method to synthesize ZnO nanostructures. Kumar *et al* .synthesized ZnO-NPs by chemical precipitation method and investigated the correlation of the optical properties of ZnO with morphology and crystallite size [12]. Bagheri *et al.* used direct precipitation method to synthesize nano-sized ZnO particles and studied the structural and optical properties [11]. Goswami *et al.* studied the structural and optical properties of non annealed and annealed ZnO-NPs prepared by chemical precipitation technique. [13]. Moazzen *et al.* also synthesized ZnO- NPs by chemical precipitation method and studied the structural and optical properties [14].

In this study, we have grown ZnO-NPs by simple chemical precipitation method at a temperature of 60^0C and investigated the structural and optical characteristics of the ZnO-NPs by means of various characterization techniques.

2. EXPERIMENTAL

2.1 Materials

All the chemicals Zinc nitrate hexa hydrate [Zn $(NO_3)_2$.$6H_2O$] (Merck, Mumbai, India, MW= 297.49 gm/mol, 99.0 % purity), Sodium hydroxide (NaOH), (Merck, Mumbai, India, MW=40gm/mol, 97% purity), and Polyvinyl Pyrrolidone (PVP, Loba Chemie, MW= 40,000) were of analytical grade and used directly without further purification. Double distilled water was used throughout the experiment.

2.2. Synthesis

0.1M of Zinc nitrate hexa hydrate [Zn $(NO_3)_2$.$6H_2O$] solution was prepared by dissolving in 100ml distilled water under constant stirring for one hour. 0.2 M of sodium hydroxide (NaOH) solution prepared in 100ml distilled water was added drop wise into the solution of Zinc nitrate hexa hydrate under stirring, till the solution becomes milky white. 2 wt% of Polyvinyl Pyrrolidone (PVP) used as the capping agent, was then added to the solution. The final solution was stirred continuously at 60^0C for one hour and then kept overnight for stabilization. A white precipitate so obtained was filtered and washed several times with distilled water to remove any impurities present in it. The precipitate was dried at 90^0C for several hours and then subjected to annealing at 400^0C for two hours.

2.3Characterization Techniques

The prepared sample was systematically characterized by using different instrumentation techniques. The structural property was studied by a Philips X-ray Diffractometer (XPERT PRO) with Cu Kα1 radiation (λ=1.5406Å) at a voltage of 40 kV and current of 30mA with a scan rate of 0.02^0/sec in terms of intensity versus2θ plots,with2θranging from 20^0 to 80^0. The surface morphologystudies were done using Scanning Electron Microscopy (model FEI Quanta 250) and Energy dispersive X-ray (EDAX) spectrometer attached to SEM. UV-Vis absorption study of the sample was recorded in the wavelength range 200nm to 600nm at room temperature using HITACHI model U-3210 double beam spectrophotometer. Photoluminescence (PL) measurement was done at room temperature usingHITACHI model F-2500 fluorescence spectrophotometer.

3. RESULTS AND DISCUSSION

3.1XRD analysis

Figure1 shows the X-ray diffraction pattern of the synthesized sample. Comparing with the standard data (JCPDS -36-1451), all the diffraction peaks obtained for the sample can be indexed to the hexagonal wurtzite structure of ZnO. No extra peaks corresponding to any other phases are observed indicating that prepared sample is of high purity. Further, it is noticed that the peaks are sharp, which confirm the formation of good crystallinity of ZnO product. The crystallite size (D) of the sample is estimated using Debye-Scherrer formula [11]

$D= K\lambda/ (\beta Cos\theta)$ …… (1)

Where K is a constant (K=0.9 for spherical shape),λ is the wavelength (=$1.5406A^0$) of Cu $K_{\alpha 1}$ radiation, βis the full-width at half-maximum (FWHM) of the diffraction peak in radians and θ is the Bragg's diffraction angle respectively. Using the above equation, the average crystallite size calculated from the most intense diffraction peak along (101) plane is found to be 15 nm.

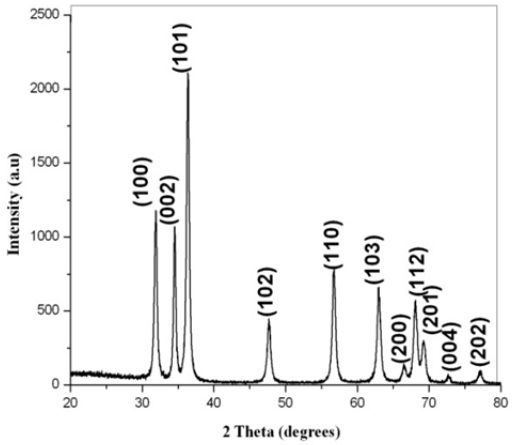

Fig. 1: XRD pattern of the ZnO-NPs

3.2 SEM analysis

Fig. 2 shows the SEM image of the sample synthesized by chemical precipitation method. It is clear from the SEM image that the particles are nearly spherical in shape with little agglomeration.

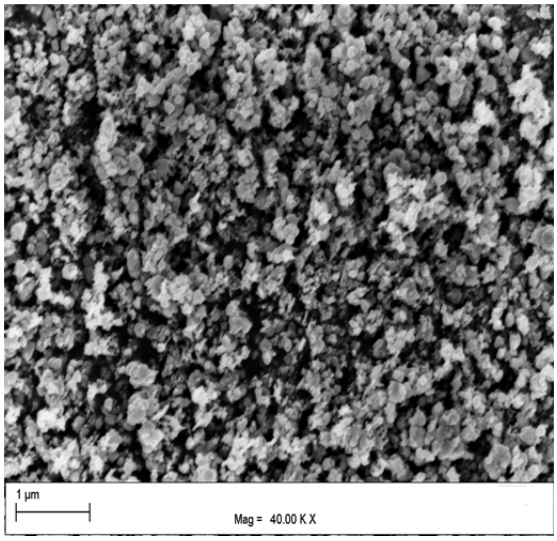

Fig. 2: SEM image of ZnO NPs capped with PVP.

To check the chemical constituents present in the as-grown ZnO sample, Energy dispersive X-ray analysis (EDAX) was done. Fig. 3 shows the EDAX spectrum of the sample which indicates the presence of Zn and O only. The absence of any foreign element clearly depicts that the sample is highly pure.

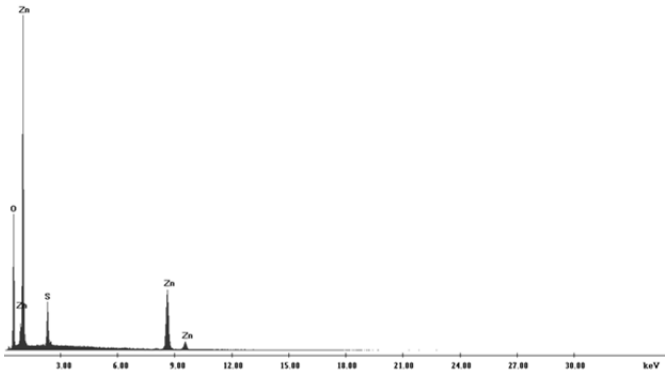

Fig. 3: EDAX spectrum of the ZnO-NPs

3.3 UV-Vis spectroscopy

Fig. 4 shows the UV-Visible absorption spectrum in the wavelength range 200nm to 600nm at room temperature. The absorption peak is observed at wavelength 326 nm which clearly indicates the blue shift of synthesized ZnO sample.

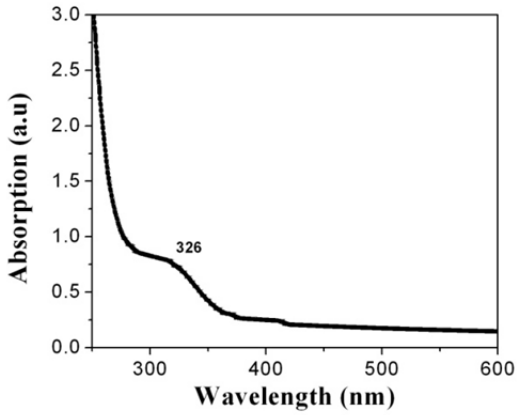

Fig.4(a) UV-vis absorption spectrum of ZnO- NPs

The optical band gap Eg is determined by using relation [6] which is given as

$\alpha = A (h\nu - E_g)^n$

Where α is the absorption coefficient, A is a constant, hν is the energy of an incident photon, E_g is the band gap. The value of n depends on nature of transitions. For direct and indirect transitions n=1/2 and 2 respectively. The band gap energy of the sample is estimated by plotting hν versus $(\alpha h\nu)^2$ graph. On extrapolating the linear portion of the curve, the intercept on the energy axis $(\alpha h\nu)^2 = 0$ gives the value of band gap energy as 3.45eV. This clearly indicates that band gap of the synthesized sample has enhanced as compared to bulk ZnO (3.37eV) which is due to confinement of particle size [16].

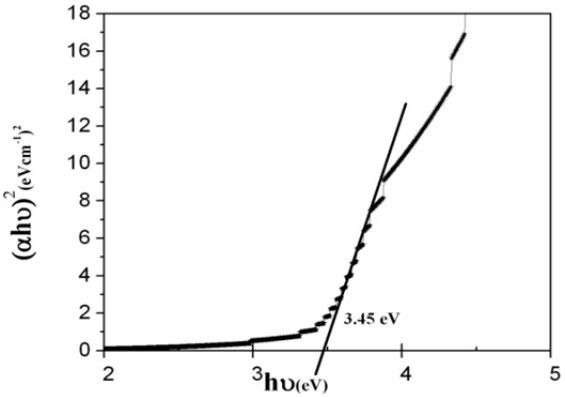

Fig.4 (b) Plot of linear portion of E_g vs $(\alpha h\nu)^2$ for ZnO-NPs

3.4 Photoluminineschence spectroscopy

The room temperature PL spectrum of the as-grown ZnO-NPs, excited at wavelength 320nm is shown in fig 5. A sharp UV emission peak positioned at wavelength 390nm is observed which corresponds to the near band-edge emission resulting from the excitonic recombination of ZnO [15]. A weak peak at wavelength 469nm i.e. blue emission is also observed which is because of zinc interstitials defects in the as-grown ZnO sample [10, 16].

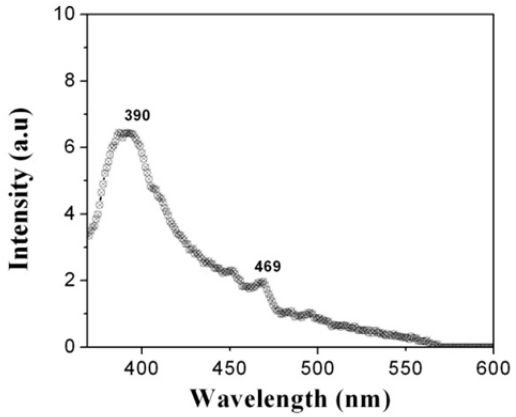

Fig.5 Room temperature PL spectrum of ZnO-NPs

4. CONCLUSION

We have successfully grown PVP capped ZnO-NPs by chemical precipitation method using Zinc nitrate hexahydrate and sodium hydroxide as source materials. XRD result reveals that the prepared sample is of high purity and has hexagonal wurzite structure of ZnO-NPs. SEM image indicates that particles are almost spherical in shape with slight agglomeration.UV-Vis absorption spectrum shows a

absorption peak at wavelength 326nm which is blue shifted. The PL spectrum shows a sharp UV emission and a weak blue emission.

5. ACKNOWLEDGEMENT

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