

## Synthesis and properties of ZnO nanoparticles at room temperature: Effect of capping agent

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### ABSTRACT

Zinc Oxide nanoparticles in aqueous phase were studied using an easy, cost effective and environmental friendly co-precipitation method. ZnO nanoparticles are of special interest due to its non-toxic nature towards health. This paper reports on structural and optical properties of polymer capped ZnO nanoparticles prepared through wet chemical method using PVP (polyvinylpyrrolidone) as capping agent and acidic level maintained by using NaOH at pH-9. Due to capping agent the dynamic of binding and unbinding and its concentration become prerequisite for reduction of particles size and formation of agglomeration. The XRD analysis confirms the growth of wurtzite phase of ZnO nanoparticles. Scanning electron microscope (SEM), energy dispersive analysis of X-rays (EDAX), photoluminescence (PL) and Fourier transform infrared (FTIR) spectrophotometers were employed to study the structural, morphological, compositional and luminescence properties.

**Keywords:** Capping agent, Chemical co-precipitation, Photoluminescence, SEM, XRD.

### I. INTRODUCTION

Among the II-VI compound semiconductors, ZnO has many applications like dye and gas sensors, bio fuels, piezoelectric transducers, photo catalysts and solar cell windows (1-7). ZnO in nano-form is one of the most promising diluted magnetic semiconductors (DNSS) oxide materials for spintronic devices (8, 9). It has also used for the application of optoelectronic devices such as photo detectors, short wavelength light emitting diode, ultraviolet(UV) lasers etc. due to its wide band gap (3.3eV) at room temperature and large excitonic binding energy(60MeV) (10, 11). On comparison with other optoelectronic materials, ZnO has some advantage of being low cost and easily synthesis to obtained nanostructure. Many theoretical and experimental works has been done on optical and electrical properties because of large optoelectronic applications of ZnO nanoparticles with different morphologies such as nanoparticles, nanobelts, nanoprisms, nanowires, etc. ZnO is an n-type semiconductor due to its presence of intrinsic defects such as oxygen vacancies and Zn interstitials.

In this paper, to study the structural and optical properties of polymer capped ZnO nanoparticles prepared through chemical co-precipitation method were investigated. Capped ZnO nano samples were prepared with different concentrations of PVP (0 gm, 0.3 gm, 0.5 gm and 0.7 gm).

### II. EXPERIMENTAL AND CHARACTERIZATION TECHNIQUES:

For the synthesis of pure and PVP capped ZnO nanoparticles, the analytical grade (AR), high purity chemical Zinc acetate dehydrated ( $ZnO_2CCH_3$ ).  $2H_2O$  and PVP (polyvinylpyrrolidone) were used. The experimental procedure for the preparation of ZnO and PVP capped ZnO is as follows: 0.1M zinc acetate hydrate was dissolved in 100ml double distilled water maintaining the acidic level at PH=9. The slow addition yields the white gel. After that 0.3 gm of PVP (polyvinyl pyrrolidone) is added as a capping agent to reduce the particle size and stirred for 4 hours at room temperature and become a precipitate. The precipitate is collected and dry for 24hr at 800c at oven. Same procedure is repeated for preparing pure, PVP (0.5 gm, 0.7 gm) capped ZnO nanoparticles.

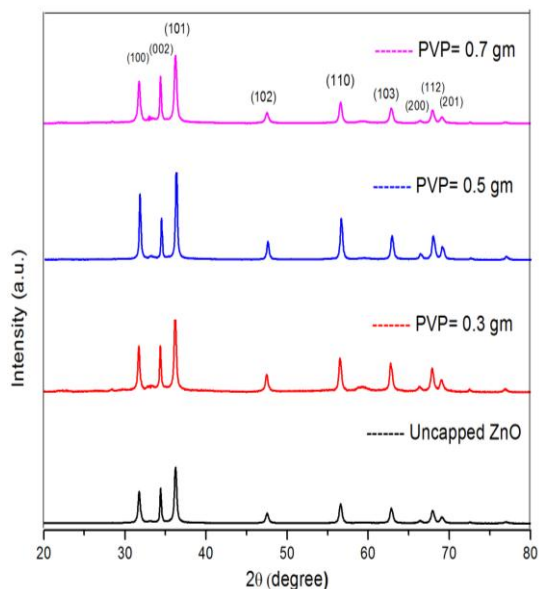
To identify the crystal structure, X-ray powder diffraction (XRD) using Cu-K $\alpha$  radiations ( $\lambda= 1.54 \text{ \AA}$ ) in  $2\theta$  range from 200 to 800 and the morphological analysis of sample was carried out on scanning electron microscope (SEM). The compositional analysis was carried out using EDAX attached with SEM. Fourier transformation infrared (FTIR) spectra of infrared spectrometer (spectrum 65, PerkinElmer) is operated in the range of 4500-400  $cm^{-1}$  with a resolution of 1  $cm^{-1}$ . The photoluminescence (PL) spectra of prepared nanoparticles were carried out between the

wavelengths ranging from 350-600 nm using FLS980 EDIN BURCH instruments at room temperature.

### III. RESULTS AND DISCUSSION

#### 3.1 Structural analysis

The The characteristic XRD spectra of the pure and PVP capped ZnO nanoparticles are shown in fig.1. The major diffraction peaks observed at  $2\theta$  values= $31.7^\circ$ ,  $34.4^\circ$ ,  $36.2^\circ$ ,  $47.5^\circ$ ,  $56.7^\circ$ ,  $62.7^\circ$ ,  $68.1^\circ$ ,  $68.8^\circ$  corresponds to (100), (002), (101), (102), (110), (103), (112), (201) reflection planes which were confirmed from (JCPDS card 80-0075). There is no impurity peak observed in the polymer capped XRD. The intensity of (101) peak is dominant over all other peaks confirming the formation of hexagonal wurtzite structure. As the concentration of PVP increases, the crystalline size of the prepared samples decreases.



**Fig. 1: XRD patterns of uncapped and PVP (0.3, 0.5 & 0.7 gm) capped ZnO nanoparticles**

The average particle size was estimated Using the Debye–Scherrer’s formula,

$$D = \frac{0.94\lambda}{\beta_{hkl} \cos \theta}$$

Where, D is the average Particle size,  $\beta_{hkl}$  is full width at half maximum of XRD peak expressed in radians and ‘ $\theta$ ’ is the position of the diffraction peak.

The average particle size of the samples calculated by Debye- Scherrer’s equation were tabulated in the below Table 1.

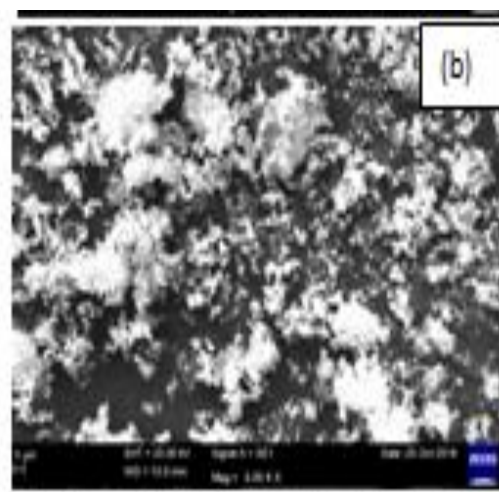
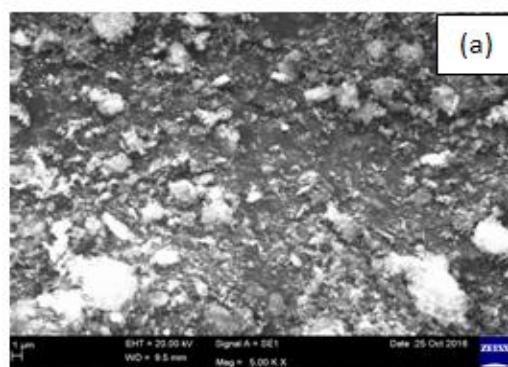
**Table 1: Average Particle size of polymer capped ZnO nanoparticles**

S.No	Capping agent concentration (at.%)	Particle size(nm)
1	Uncapped ZnO	62
2	PVP (0.3 gm)	40
3	PVP ( 0.5 gm)	33
4	PVP (0.7 gm)	36

The particles size reduces from 62 to 33 nm with increasing PVP concentration upto 0.5 gm and further increasing the PVP concentration, Particle size increases due to agglomeration. The crystal size obtained from uncapped ZnO nanoparticles is bigger than the capped crystals and hence, capping agent plays an important role in tuning the various size-dependent properties of the prepared nano samples.

#### 3.2 Morphological studies

The morphology of PVP capped ZnO nanoparticles are presented in fig.2. The spherical nanoparticles are agglomerated so as to reduce the total surface free energy. This clearly indicates the increment of grain boundaries and reduction of grain size with PVP concentration.



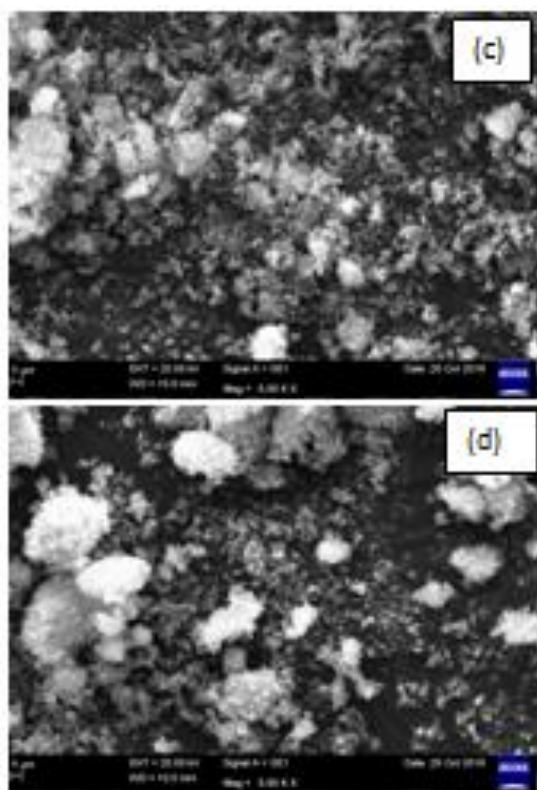


Fig.2 SEM images of (a) uncapped, (b) PVP (0.3 gm) , (c) PVP (0.5 gm) and (d) PVP (0.7 gm) capped ZnO nanoparticles

### 3.3 Compositional analysis

The EDAX profile of PVP (0.5 gm) capped ZnO is shown in Fig.3. It is evident from the EDAX Spectra that no impurities are present due to capping agent. The elemental carbon present in the EDAX profile may be due to carbon tape used during the analysis.

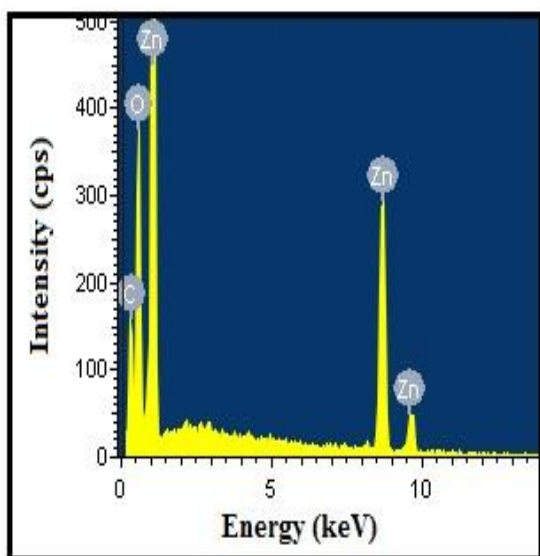


Fig.3. The EDAX profile of PVP (0.5 gm) capped ZnO nanoparticles.

### 3.4 Photo Luminescence studies

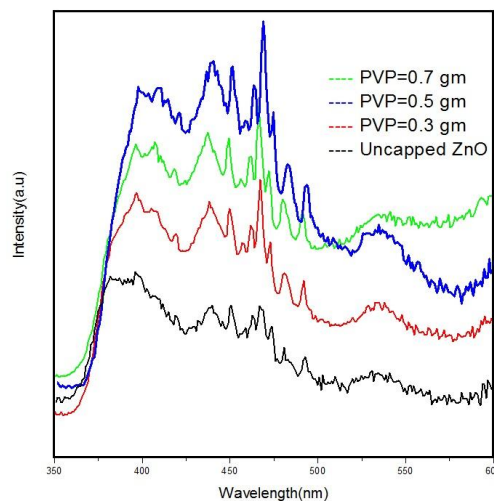


Fig.4 Photoluminescence spectra of uncapped and PVP capped ZnO nanoparticles

PL measurement has been carried out at room temperature between 350nm to 600nm. Fig.4 shows the PL spectra of uncapped and PVP capped samples at room temperature. ZnO has one sharp band associated with near band edge emission at 396 nm and another broad emission in the visible region around 523 nm [12]. Three well defined emission peaks (ultra violet, blue and green regions) are noticed in all the four samples even though the intensity position and width of peaks are different. Capped samples exhibited enhanced luminescence due to decrease in particle size and hence increase in surface to volume ratio. Among the prepared nanosamples, PVP (0.5 gm) capped sample exhibited better luminescence properties.

### 3.5 FTIR studies

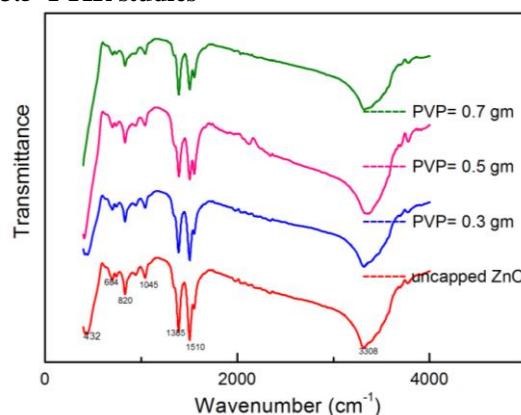


Fig.5. FTIR spectra of uncapped and PVP capped ZnO nanoparticles.

The infrared spectra of uncapped and PVP capped ZnO nanoparticles recorded in the region of 4000-400  $\text{cm}^{-1}$  is shown in fig.5. The wide intense absorption peaks position at 3308-3436  $\text{cm}^{-1}$

corresponds to O-H group stretching vibration due to the bound H<sub>2</sub>O on the surface of nanocrystalline powder sample. The absorption peaks near 1510 cm<sup>-1</sup> is attributed to the bending vibration of free water. As the doping increases it shifts from 1510-1630 cm<sup>-1</sup>. The absorption peak near 430-500 cm<sup>-1</sup> can be attributed to the Zn-O stretching mode (13, 14).

#### IV. CONCLUSIONS:

Uncapped and PVP capped ZnO nanoparticles were successfully prepared by chemical co-precipitation method maintained the acidic level with pH value 9 at room temperature. XRD data clearly shows that it has wurtzite hexagonal structure. As the PVP concentration increases the crystal size decreases which is mainly used for optoelectronic properties. The FTIR, SEM, EDAX tells about the bond structure and morphology of ZnO. From the photoluminescence studies it was observed that, 0.5 gm PVP capped ZnO nanoparticles exhibited enhanced luminescence properties.

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