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## Synthesis of nano ZnO Material by Chemical Route and its Physical Characterization

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### Abstract:

Nanostructured ZnO material was prepared by chemical route using Zinc acetate as precursor solution and Stearic acid as a surfactant. In synthesis 0.5 M Zinc acetate ( $Zn(CH_3COO)_2 \cdot 2H_2O$ ) was prepared in 30ml methanol and 0.5 M Stearic acid ( $CH_3(CH_2)_{16}COOH$ ) was prepared in 70 ml methanol. These solutions were added under the vigorous magnetic stirring. At room temperature; aqueous 25% NaOH was added drop by drop to reach pH 8.5. This was then placed in a magnetic stirrer for 2hr. After completion of reaction, the white precipitate formed was washed thoroughly with distilled water followed by ethanol to remove the impurities. The precipitate was dried over 60°C for 6 h in order to remove water molecules. The formation of ZnO nanoparticles were confirmed by X-ray diffraction (XRD), transmission electron microscopy (TEM), selected area electron diffraction (SAED) and UV studies. XRD analysis confirmed the powder to be ZnO with wurtzite structure, with crystallite size ranging from 6 to 27 nm. Observation from TEM images confirmed that the grains were nearly hexagonal rod type in nature with sizes from 22 to 56 nm.

**Keywords:** Chemical route, Zinc acetate, Stearic acid, Nano ZnO, Particle size.

### Introduction

The Nanosized particles of semiconductor materials have gained much more interest in recent years due to their desirable properties such as specific surface area and morphology has significant role for applications in different areas such as catalysts [1], sensors [2], photoelectron devices [3,4], highly functional and effective devices [5]. These nanomaterials have novel electronic, structural, and thermal properties which are of high scientific interests in basic and applied fields.

ZnO is a polar inorganic crystalline material with many applications due to its unique combination of interesting properties such as non-toxicity, good electrical, optical and piezoelectric behavior, stability in a hydrogen plasma atmosphere and low price[6-9]. ZnO is a well- known semiconductor with a wide direct band gap (3.37 eV) and a large exciton binding energy of 60 meV at room temperature [10,11] and it has a wide range of applications such as solar cells, luminescent, electrical and acoustic devices, gas and chemical sensors, coatings, catalysts, micro lasers, memory arrays and biomedical applications[9,12].

Over the past few years, considerable attention has been devoted to the synthesis of ZnO nanocrystalline, and remarkable achievements have been made. Up to now, several routes are available in reports for the synthesis of ZnO nanocrystalline, such as sol-gel [13], spray pyrolysis[14] chemical route[15] and solvothermal / hydrothermal reaction[16,17].



Chemical route technique has number of advantages including easy process ability at ambient conditions, very easy to control the particle size, use of simple equipment, large area uniform production, environmental friendliness and less hazardous. The low reaction temperatures make this method an attractive one for microelectronics and plastic electronics. Nanostructured ZnO has many applications in the industrial arena; hence the preparation of nanostructured ZnO powder is an important issue. conference website.

## Experimental

### Material Preparation

Nanostructured ZnO material was prepared by chemical route method using Zinc acetate as precursor solution and Stearic acid as a surfactant. In synthesis 0.5 M Zinc acetate ( $Zn(CH_3COO)_2 \cdot 2H_2O$ ) was prepared in 30ml methanol and 0.5 M Stearic acid ( $CH_3(CH_2)_{16}COOH$ ) was prepared in 70 ml methanol. These solutions were added under the vigorous magnetic stirring.

At room temperature, aqueous 25% NaOH was added drop by drop to reach pH 8.5. This was then placed in a magnetic stirrer for 2hr. After completion of reaction, the white precipitate formed was washed thoroughly with distilled water followed by ethanol to remove the impurities. The precipitate was dried over 60°C for 6 h in order to remove water molecules.



### Physical Characterizations

#### X-Ray Diffraction

In order to understand the structural properties of nano ZnO powder, the X-ray diffraction study was undertaken. The X-ray diffraction analyses of ZnO powder material was carried in the 20-80° range using  $Cu\alpha$  radiation.

Fig. 1 shows an XRD pattern of ZnO material plotted  $2\theta$  (deg.) verses intensity having several peaks of Zinc Oxide indicating random orientation for the polycrystalline nature. There are no other characteristic impurities peaks were present in the XRD pattern. It confirms that the product obtained is in pure phase. The observed diffraction peaks of ZnO are corresponding to the hexagonal wurtzite structure of ZnO and well matched with the JCDPS (76-0704) reported data of ZnO. The higher peak intensities of an XRD pattern is due to the better crystallinity with preferred orientation along the (101) direction.

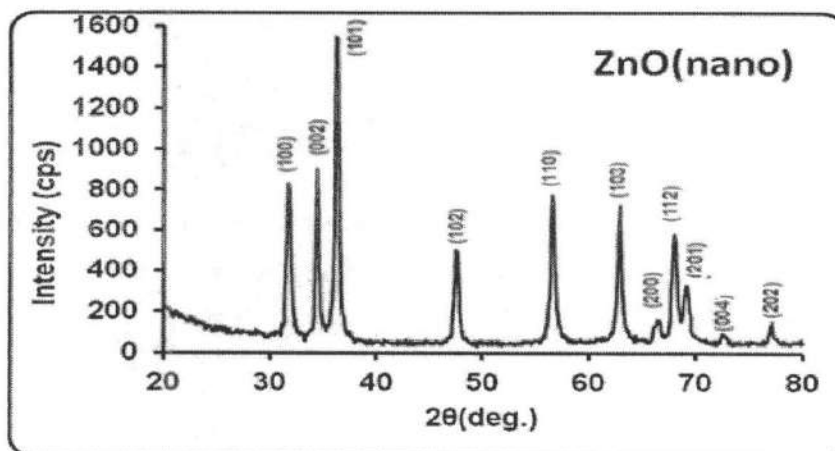


Fig. 1 XRD pattern of nanocrystalline ZnO material

Table I Xrd Data of Nanocrystalline ZnO.

(2θ) (deg.)	observed d-spacing(Å)	FWHM(nm)	Crystallite size d (nm)	Plane(hkl)
31.70	2.8204	0.475	19	100
34.40	2.6049	0.336	27	022
36.20	2.4794	0.428	22	101
47.50	1.9088	0.456	21	102
56.60	1.6248	0.488	21	110
62.90	1.4764	0.473	22	103
66.40	1.4049	1.141	9	200
68.00	1.3775	0.558	19	112
69.10	1.3583	1.055	10	201
72.60	1.3012	2.036	6	202

The average crystallite size was calculated from XRD data and using Debye Scherer's formula [18].

$$D = 0.9\lambda / \beta \cos\theta \quad (5)$$

Where, D- Average crystallite size,  $\beta$  - Broadening of the diffraction line measured at half-maximum intensity (FWHM),  $\lambda$  -Wavelength of the X- ray radiation and  $\theta$  -Bragg's angle.The d-spacing, FWHM and crystallite size of the nano ZnO material are listed in Table 1.

The good agreement of d-values with the standard JCPDS data indicates that the prepared material was ZnO with hexagonal wurtzite structure. The average crystallite size was found to be in the range of 6-27 nm.



From the XRD pattern, the lattice constants of hexagonal ZnO phase can be calculated using the (6) and (7) [19],

$$\frac{1}{d_{(hkl)}^2} = \frac{1}{a^2} \left( \frac{h^2 + k^2 + k^2}{3} \right) + \frac{1}{c^2} \quad (6)$$

Where d - is interplanar distance,

a and c - are lattice constants(hexagonal structure)

$$\frac{c}{a} = \sqrt{\frac{3}{2} \frac{d_{(101)}^2}{d_{(110)}^2}} \quad (7)$$

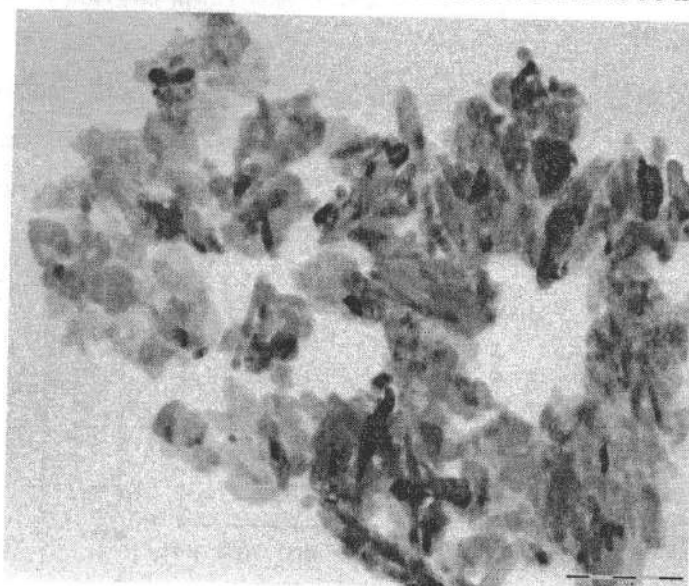
Table 2 Lattice Constants Nanocrystalline ZnO

Phase	Plane(hkl)	2θ	d (Å)	a (Å)	c (Å)
ZnO	101	36.20	2.479	3.546	5.791

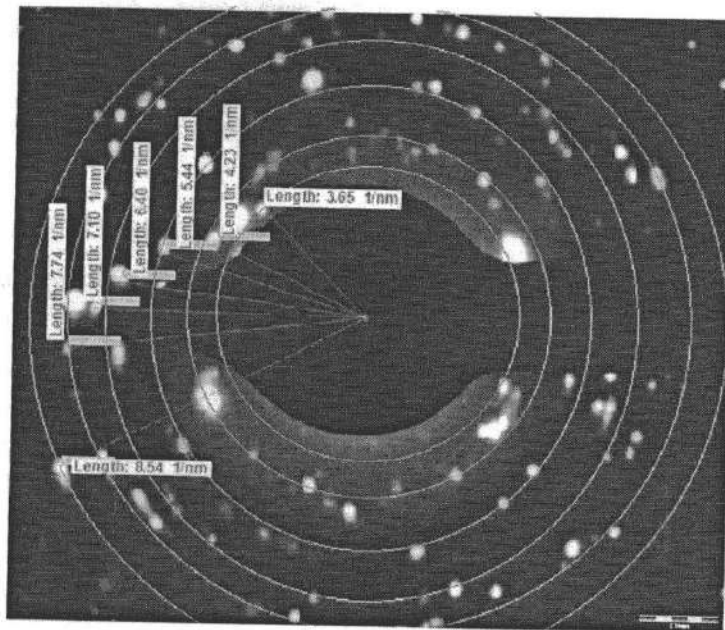
From Table 2, it has been observed that the lattice parameters calculated are also in agreement with the reported values from JCPDS data (a = 3.25 Å, c = 5.207 Å.)

#### Microstructural Analysis by TEM

TEM image and the corresponding selected area electron diffraction (SAED) pattern for nanocrystalline ZnO powder are shown in Fig. 2 (a) and (b). The morphology and microstructure of nanopowder were investigated from TEM images. All the sample was scanned in all zones before the picture was taken. The micrographs showed that the some particles were nearly hexagonal rod shape and some particles were pendant shape randomly oriented in different directions with sizes from 22 to 56 nm.



(a)



(b)

Fig. 2 (a) TEM image (b) SAED pattern of nanocrystalline ZnO material

In selected area electron diffraction (SAED) pattern, the bright diffraction spots confirm the high crystallinity of these particles with wurtzite structure. Moreover, these spots arranged in rings also indicate the presence of plentiful well-defined nanocrystallines in these particles. Every ring in SAD pattern corresponds to a group of lattice planes of the same miller index family in the polycrystalline sample. While the sample containing large number of small randomly distributed particles results in continuous rings. That is because these particles all contribute to the formation of the diffraction pattern.

#### Optical Properties by UV Spectroscopy

Fig.3, shows the variation of absorbance of nanocrystalline ZnO powder with wavelength. The band gap energy of the sample calculated from the absorption edges of the spectra was 3.36 eV. This value is well matches with the reported value of 3.37 eV [20].

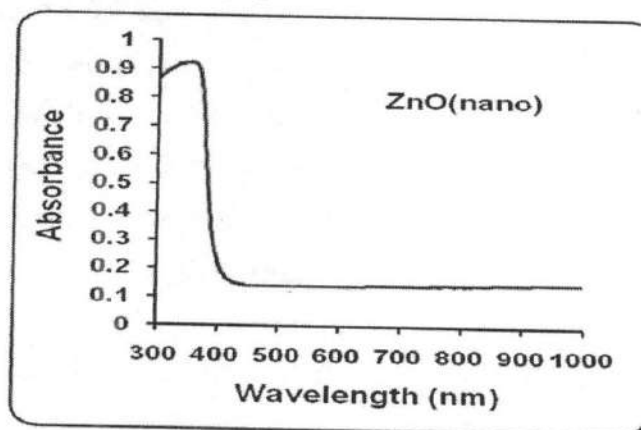


Fig. 3. Absorption spectra of nanocrystalline ZnO



### Conclusions

- i. The Nanostructured ZnO material was synthesized by chemical route.
- ii. XRD analysis confirmed the prepared material was pure ZnO with the crystallite size varying from 6-27 nm.
- iii. Observations from TEM images confirmed that the some nano particles were nearly hexagonal rod shape and some particles were pendent shape oriented randomly with sizes varying from 22 to 56 nm.

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