

Synthesis and Characterizations of ZnO Nanoparticle via Chemical Wet Method

Abdullateef Toyeebah Folake and Salawu Ganiyat Abiodun

Department of Mechanical Engineering, Federal Polytechnic Offa, Kwara State, Nigeria

Abstract: The nanoparticles were produced from chemical wet method. The produced material dried at a temperature of 80°C in an oven to form a white powder. The structure of the composite was revealed by XRD with narrow diffraction peaks (002), (121), (123) (001), (110), (004) (003), (031), (132), (130) and (200) with particle size of 1.098 nm. The SEM images show porous fine-grained nanostructure ZnO nanoparticle

Keywords: ZnO, XRD, SEM, Inorganic and Chemical wet method

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I. Introduction

Zinc oxide (ZnO) is an inorganic compound which is present in the earth crust as mineral zincite. ZnO is a wide band gap semiconductor of 3.37 eV at room temperature with high optical transparency and Luminescence in visible-near ultraviolet range of spectrum. It is made up of group II-V elements [1]. This semiconductor has several unique properties that includes good transparency, high electron mobility, wide bandgap, and strong room-temperature luminescence. Zinc Oxide (ZnO) nanoparticles are having high exciton binding energy nearly 60 meV [2].

Therefore, the semiconductor is widely used in light emitting diodes, solar cells, gas sensors and transparent electrodes in liquid crystal displays. Moreover Zinc Oxide is environmental friendly and ease to synthesize [2]. Many techniques are being used to synthesize ZnO nanoparticles which include Chemical Bath Method, precipitation method, spray pyrolysis method, micro emulsion method, and hydro-thermal method. Sol gel method and simple chemical method. However, chemical wet method is used to synthesis ZnO nanoparticle in this report due to its notable advantages such as large-scale production, low cost and low synthesis temperature [3].

II. Materials and Method

Zinc oxide nanoparticles was synthesis using zinc acetate dehydrate as a precursor. 0.5M of precursor was dissolved in distilled water in which 250ml of methanol is added and stirred vigorously. 1M of NaOH was added to vary its pH. The obtain solution will be kept in oven for 5hrs at 80°C. The obtain product will be wash with methanol and dried at 60°C. This resulted in white crystalline powder.

III. Result

Structural Analysis

The x-ray diffraction measurement were carried out on sample A to F with 2θ scanning from 10° to 74° in step size 0.008° and a constant counting times of 3.17 s/step, and the generator settings of 40mA and 42kV, with CuK_α of 1.5406 Å. The results are shown in figure 1.

Table 1.0. Peaks list details of XRD pattern of ZnO powder samples prepared by solution method.

HKL	Position ($2\theta^\circ$)	Height (counts)	FWHM($2\theta^\circ$)	D- spacing (Å)	Texture coefficient	Re Int %
110	18.335	9898.71	0.1023	4.83880	2.11	23.38
022	19.8005	809.63	0.1279	4.48393	3.12	1.91
121	23.7427	2222.37	0.1535	3.74760	4.23	5.25
123	28.6325	1975.07	0.1535	3.11871	2.03	4.66
001	9.2017	42338.23	0.1279	9.61106	2.33	100.00
003	27.0513	5975.87	0.1535	3.29628	5.17	14.66
004	22.2317	543.88	0.1023	3.99877	5.01	1.28
200	35.3854	2567.63	0.1791	2.53671	3.15	6.06
031	25.1393	3951.67	0.1791	3.54248	4.14	9.33
132	31.2106	1879.95	0.1532	2.86400	3.68	4.44
130	34.5470	5618.25	0.1535	2.59633	2.41	13.27

The lattice parameters $a=b$ and c for hexagonal crystal structure of ZnO was calculated from the observed values of 2θ using d values (interplanar spacing) for the hexagonal structure

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

Where d is inter atomic spacing which can be calculated using equation 2:

$$d = \frac{\lambda}{2\sin\theta} \text{-----2}$$

a and c can be calculated using equation 3 and 4 respectively[4].

$$a = \frac{\lambda}{\sqrt{3}\sin\theta} \sqrt{h^2 + hk + k^2} \text{-----3}$$

$$c = \frac{\lambda}{2\sin\theta} l \text{-----4}$$

The average crystal size was calculated to be 1.098nm by using equation 6

$$D = \frac{k\lambda}{\beta\cos\theta} \text{-----6}$$

Where k is the crystallite shape factor (constant) with approximation to 0.9, D is the crystal size (in Å), λ is wavelength (1.54060 Å), β is full width at half maximum (FWHM in radian) and θ is the Bragg diffraction angle[5].

The preferential crystal orientation can be obtained from the texture coefficient T_c , which is given by the equation 7[6].

$$T_{c(hkl)} = \frac{I_{(hkl)}}{I_r(hkl)} \left(\frac{1}{n} \sum \frac{I_{(hkl)}}{I_r(hkl)} \right)^{-1} \text{-----7}$$

Where $T_{c(hkl)}$ is the texture coefficient, $I_{(hkl)}$ is the XRD intensity, n is the number of diffraction peaks considered and $I_r(hkl)$ is the standard intensity of the plane which is taken from reference data[7].

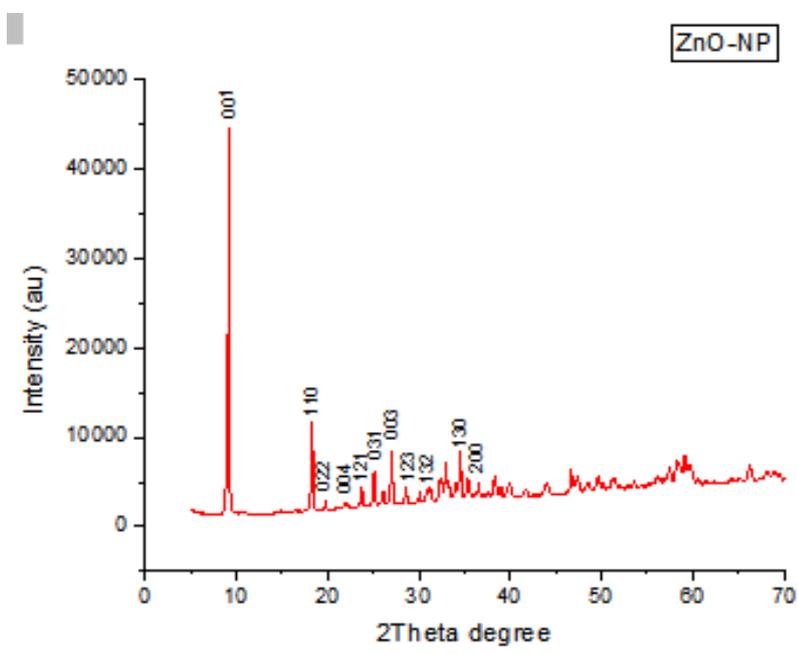


Figure 1: XRD pattern of ZnO

Morphological Analysis

The Scanning Electron Microscope (SEM) (Hitachi X650) equipped with Energy Dispersive Spectroscopy to reveal the morphological features and elemental composition of GLG structure. The instrument operated at a voltage of 20 kV while the image was captured using voltage acceleration of 5 kV to reduce charge accumulation to the sample.



Figure 2: Scanning electron microscopic image of synthesized ZnO nano structures

Optical Analysis

Avantes UV-Visible Spectrophotometer was used to determine the Absorbance A and Transmittance T at SHESTCO, Abuja, Nigeria.

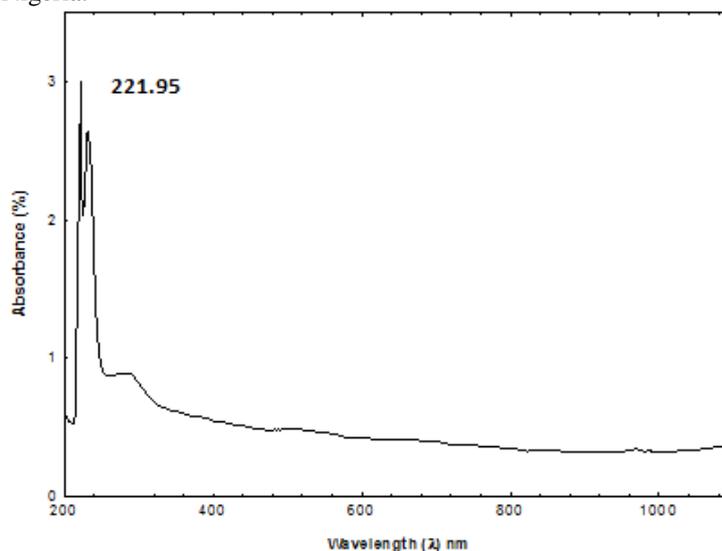


Figure 3: Absorbance vs Wavelength

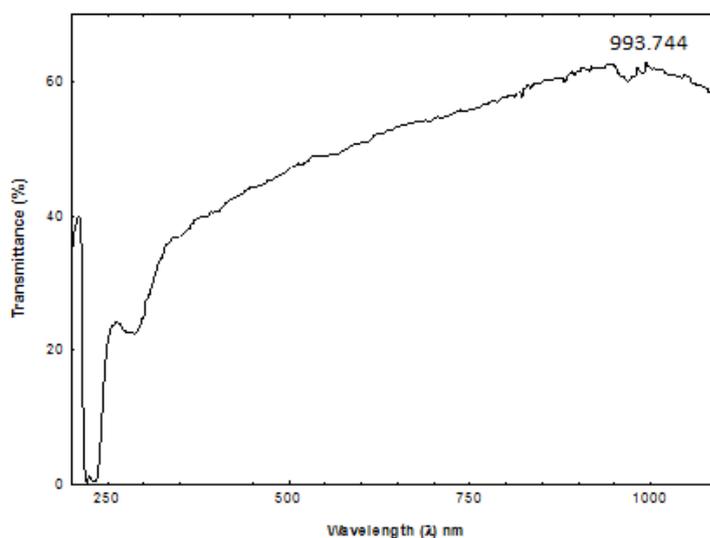


Figure 4: Transmittance vs Wavelength

IV. Discussion

XRD pattern of ZnO nanoparticle powder sample prepared by solution method is shown in figure from the XRD profile, the obtained diffraction peaks were (002), (121), (123) (001), (110), (004) (003), (031),(132), (130) and (200). The peaks confirmed the presence of ZnO nanoparticle [2]. The indexed peaks of XRD pattern of the sample prepared are given in table from this data set, the distance between two lattice planes was found to be 9.61106 \AA . The higher FWHM 0.1279 degree at 9.2017 degree indicates the increased size of ZnO nanoparticle. The relative intensity of the diffraction peak at 9.2017 degree was found to be sharp and having higher intensity which indicated that large quantity of ZnO nanoparticle were observed. The grain size of ZnO powder was calculated to be 1.098 nm using Debye's Scherrer equation [8].

If $T_{c(hkl)} \sim 1$ for all the considered planes, the particles are randomly oriented. It can be observed from table 1 that $T_{c(hkl)}$ values from all planes are more than unitary which indicates there is an abundance of grain formed along those planes [9]. This also indicates a higher degree of preferred orientation along those planes

The deviation in the $T_{c(hkl)}$ from unitary will correspond to change in atomic densities of all planes. Thus, the texture analysis of ZnO, indicates that the material is highly textured along (003) plane [8].

SEM images of the sample surface were acquired. The morphology of ZnO sample synthesized at low temperature was investigated by SEM. Figure 2 shows SEM image of ZnO nanoparticles. Typical image taken from this sample, indicating the presence of agglomerates, with a rough porous fine-grained nanostructure. The size of the grains (in the range 1.5 nm) is in agreement with the average crystallite size as estimated by XRD. This figure reveals that the ZnO nanoparticles have particle like morphology, as reported by Pal et al. for synthesis of ZnO nanostructures with different morphologies through a hydrothermal technique. The increasing agglomerates can be noted. It can be suggested that its presence is causing an increase of defects in ZnO nanostructure, enhances the surface energy of grains favoring, as a consequence, their agglomeration.

The UV visible spectrum of the sample is as shown in figure 3. The sample absorbs the radiations in the UV range up to 221.95 nm . The ZnO nanoparticles are transmitted through visible spectrum radiations

The absorption and the transmission spectra of ZnO film are shown in Figure 3 and 4 respectively. It can be seen that the absorbance of the ZnO film is high at short wavelengths ($\lambda < 380 \text{ nm}$) and low at long wavelengths ($\lambda > 380 \text{ nm}$) but decreased absorbance trend extending beyond 1000 nm of wavelength.

In Figure 4. It is shown that the transmission of ZnO film is high over large wavelengths. ZnO film exhibits a high average transmittance ($\sim 40.52\%$) in the visible region. This suggests that the produced film indicates a good optical quality due to low scatter or absorption losses [10].

V. Conclusion

ZnO nanoparticle was successfully synthesized from wet chemical method. The XRD pattern obtained confirmed the formation of hexagonal ZnO. The XRD studies of ZnO revealed that the average grain size which was calculated by Debye-Scherrer formula is 1.098 nm . The highest texture coefficient was found in (003) plane. The characterization of SEM show that the ZnO internal structure of porous fine-grained nanostructure. This study provides a new approach which can be used to produce ZnO material that will be cost effective and large-scale production.

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