X- Ray Studies of Zinc Oxide (ZnO) Nanoparticles grown by Hydrothermal Technique

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Abstract: *In this study Zinc Oxide Nanoparticles was grown by Hydrothermal Technique using the solution of zinc acetate dehydrate ($Zn(CH_3COO)_2.2H_2O$) (0.1M in 50ml methanol) and Sodium Hydroxide (NaOH). We get the white solid powder from the stock solution by maintaining temperature 180°C for 12 hours into Teflon lined sealed cylindrical flask autoclaves,The crystal structure of the samples and the grain size (D) of the particles was determined from the X-ray Diffraction.*

Key Words: *Zinc Oxide, Hydrothermal, X-ray Diffraction.*

1. INTRODUCTION:

Zinc Oxide is the one of the most important n-type semiconductor materials with a 3.37 eV band gap at room temperature and 60 meV excitation binding energy that is in the UV region and makes this nanoparticle as an efficient UV absorber.[1] Semiconductor nanomaterials have been received great attentions. Among these various semiconductor nanomaterials zinc oxide is a versatile material because of its physic-chemical properties such as mechanical, electrical, optical, magnetic and chemical sensing properties.[2] Zinc oxide a chemical compound found naturally in the mineral called zincite has attracted much attention in recent times due to its low cost and because it can be obtained by simple techniques.[3] Chemical synthesis is one of the most important techniques which can be performed by using a range of precursors and different conditions like temperature, time, concentration of reactants, and so forth. Variation of these parameters leads to morphological differences in size and geometries of resulting nanoparticles. Among the nanoscale metal oxides, zinc oxide is a common host material that has been widely used due to its excellent chemical and thermal stability, low cost and environmental-friendliness.[4] In this study, ZnO particles have been produced by Hydrothermal Method using Zinc Acetate Dehydrate ($Zn(CH_3COO)_2.2H_2O$) (0.1 M), Sodium Hydroxide (NaOH), Methanol. Nanoparticles structure of the samples and the grain size (D) of the particles was determined from the X-ray Diffraction.

2. MATERIALS:

The chemicals used in this experiment are Zinc Acetate Dehydrate ($Zn(CH_3COO)_2.2H_2O$) (0.1 M), Sodium Hydroxide (NaOH), Methanol. All the Chemicals for the experiment were highly pure and there was no delay for their utilization for the experiment after receiving them.

3. METHOD:

In order to synthesize the ZnO nanoparticles, stock solutions of zinc acetate dehydrate ($Zn(CH_3COO)_2.2H_2O$) (0.1 M) was prepared in 50ml methanol under stirring. To this stock solution 25ml of Sodium hydroxide NaOH (varying from 0.2 M to 0.5 M) solution prepared in methanol was added under continuous stirring in order to get the pH value of reactants between 8 and 11. These solutions was transferred into Teflon lined sealed cylindrical flask autoclaves and maintained at temperature 180 °C for 6 to 12hour under autogenous pressure. It was then allowed to cool naturally to room temperature. After the

reaction was complete, the resulting white solid products were washed with methanol, filtered and then dried in air in a laboratory oven at 60°C.[5]

4. RESULT AND DISCUSSION:

We get the white solid powder from the stock solution by maintaining temperature 180°C for 12 hours, It turns lemon yellow on heating and reverts to white on cooling. Particles Chemical purity and stochiometry of the sample were examined using EDS. EDS conform that the developed particles has atomic percentage of 40.76 of Zinc and 59.24 of Oxygen. The elemental constitution of ZnO nanoparticles was also investigated. Two major peaks were found to have weight percentage of 73.77 of Zinc and 26.23 of Oxygen.[14]

X-ray Diffraction (XRD)

The crystal structure of the samples was investigated by analyzing the XRD data. The X-ray diffraction patterns of hydrothermal synthesized samples were shown in Fig.1. XRD spectra depicts the characteristic peaks corresponding to reflection planes (100), (002), (101), (102), (110), (103), (112) and (201) of wurtzite structure of ZnO.

The mean grain size (D) of the particles was determined from the XRD line broadening measurement using Scherrer equation. [5]

$$D = 0.94\,\lambda / (\beta \cos\theta)$$

Where, λ is the wavelength (Cu Kα),
β is the full width at the half- maximum (FWHM) of the ZnO (101) line
θ is the diffraction angle.

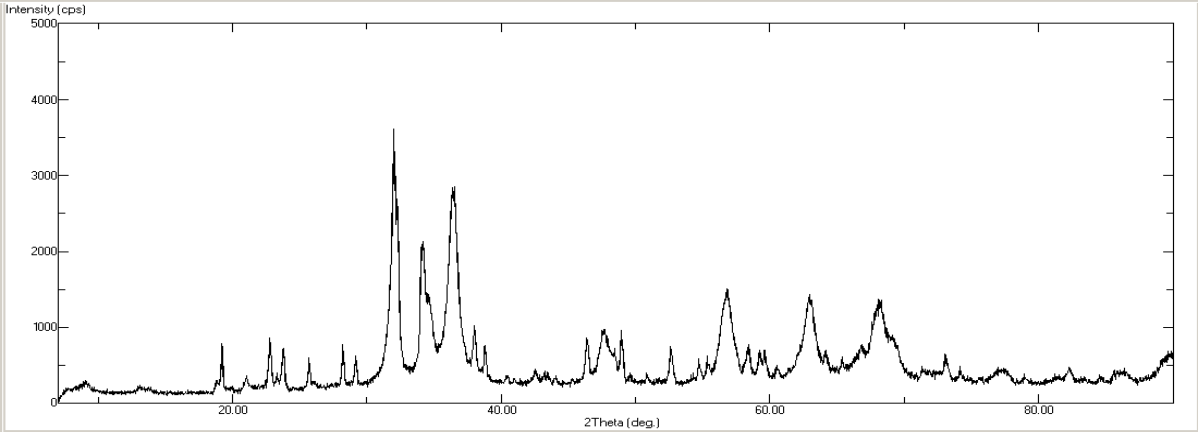

Figure 4.1: XRD patterns of ZnO nanoparticles

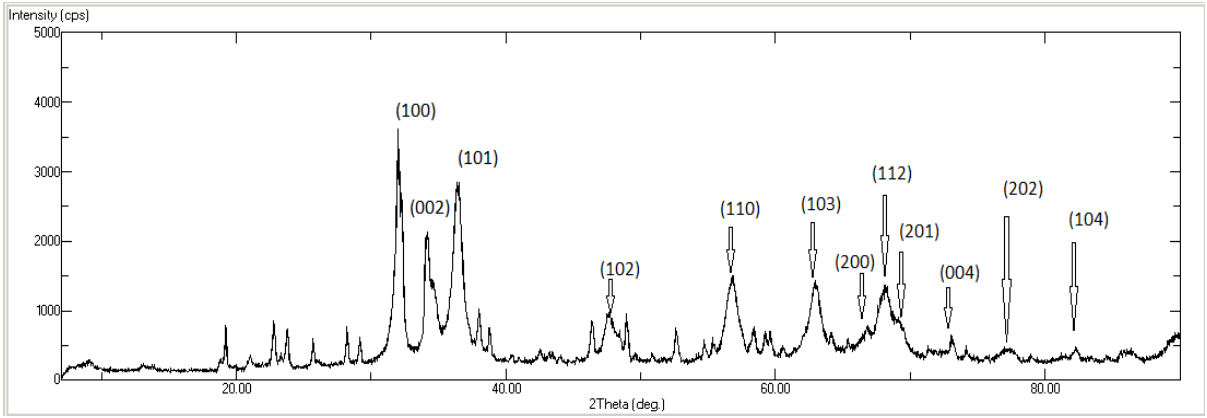

Figure 4.2: XRD patterns of ZnO nanoparticles with Plane numbering

No.	2θ	d-value	FWHM (β) (in degree)	hkl	a	c	$D=\frac{k\lambda}{\beta cos\theta}$ In nm
12	32.00	2.79	0.26	(100)	3.22	-	33.2158
14	34.12	2.63	0.40	(002)	-	5.26	21.7091
16	36.46	2.46	0.56	(101)	3.22	5.22	15.6076
23	47.64	1.91	0.49	(102)	3.21	5.25	18.5195
31	56.82	1.62	0.52	(110)	3.24	-	18.1505
36	63.02	1.47	0.68	(103)	3.30	5.13	14.3196
39	66.76	1.40	0.21	(200)	3.23	-	47.3406
42	68.20	1.37	0.56	(112)	3.21	5.25	17.9023
44	69.50	1.35	0.24	(201)	3.22	5.18	42.0982
46	73.08	1.29	0.28	(004)	-	5.16	36.9018
49	77.48	1.23	0.42	(202)	3.30	5.13	25.3407
51	82.28	1.17	0.24	(104)	3.23	5.15	45.9297

Table 4.1: Calculation of lattice constant and size of ZnO nanoparticles from Scherrer equation[7].(Where, λ= 1.54060 A˚, k = 0.94)

A definite line broadening of the diffraction peaks is an indication that the synthesized materials are in nanometer range. The grain size was found to be approximately 28 nm depending on the growth condition. The lattice parameters calculated were also in agreement with the reported values.

5. CONCLUSION:

ZnO nanoparticles was successfully synthesised by the hydrothermal method. The crystalline size of the prepared nanoparticles where determine by scherrer's equation and it was in the range of 28 nm. From the XRD, The diffraction peaks are at 2θ values of 32.00, 34.12, 36.46, 47.64, 56.8, 62.82, 66.76, 68.20, 69.50, 73.08, 77.48 and 82.28 were identified to originate from (100), (002), (101), (102), (110), (103), (200), (112), (201), (004), (202) and (104) planes. Based on the Scherrer equation the average crystallite size of the nanoparticles is observed as 28 nm. The main diffraction peak is observed at 2θ value of 32.00. This peak is identified to originate from (100) planes of the ZnO. All the peaks are indexed and found to be well matched to wurtzite structure of ZnO having hexagonal phase, which is in good agreement with the standard JCPDS Card No 75- 1526.

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