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A SIMPLE AND EFFECTIVE METHOD FOR PREPARATION AND CHARACTERIZATION OF ZINC OXIDE NANOPARTICLES

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ABSTRACT

Nanosized zinc oxide has been synthesized by using simple and efficient pericipitation method from zinc Nitrate and liquid ammonia with calcinations step at high temperature at 500^{o}C temperature for 5 hours. The nanosized ZnO were characterized by using XRD (X-ray diffraction), TGA/DTA (Thermal gravimetry analysis and Differential Thermal analysis), Scanning Electron Microscopy (SEM)/ Transmission Electron Microscopy (TEM) and magnetic measurement techniques. XRD studies showed that zinc oxide was formed as ZnO and it has hexagonal wurtzite crystal structure. The particle size of the synthesized zinc oxide was determined by TEM. TEM images showed that the size of the particle of the zinc oxide varied from 21-40 nm, which is in good agreement with the theoretically predicted size of nanomaterials. This method is cheaper, easier and effective in comparison to the known methods for the synthesis of nanomaterials. There is excellent agreement between the experimental and theoretical results.

Key words: Nanomaterials, Zinc oxide, Scanning electron microscopy (SEM), Transmission electron microscopy (TEM), XRD analysis.

INTRODUCTION

Metal oxides have attracted lots of attention over last few years due to their ability to withstand harsh process conditions. Metal oxides such as NiO and ZnO are of particular interest as they are regarded as safe materials for human beings and animals. The use of zinc oxide has been seen as a viable solution for environmental protection.

Recently, nanotechnology have emerged as the forefront of science and technologies. The intersecting fields of study that create this domain of advancement of nanotechnology. Nanotechnology is forecasted as the second industrial revolution in the world. The novel

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properties have attracted global interst across disciplines. ZnO nanoparticles exhibit bright stable photoluminisence in colloidal dispersion[1].

ZnO is a versatile semiconductor material[2]. Zno has band gap energy of 3.37 eV and it has very large excitation binding energy (60 meV) at room temperature. It is wurtzite type semiconductor[3]. Recently ZnO has been attracting attention because of its demand for its thermal stability, flexibility to form different nanostructures, commercial demand for optoelectronic devices. ZnO can form different nanostructures[4-6]. ZnO has wide application in surface acoustic wave devices[7], field emission[8], gas sensors[9], ceramics[10], solar cells[11], nanogenrators[12], biosensors[13], varistors[14], electrodeposition[16], antimicrobial textiles[17], catalysis, environmental protection, biotechnology, piezoelectric behaviours[18-21] and ultraviolet nanolasers[22].

EXPERIMENTAL

Chemicals

All chemicals used in the experiment are of analytic reagent grade. Zinc nitrate i.e. $Zn(NO_3)_2$ was purchased from Merck, India. Liquid ammonia was also purchased from Merck, India. Deionised water was used throughout the experiment.

Synthesis of ZnO

500 mL of 0.5 M solution of Zn $(NO_3)_2$ was taken and aqueous ammonia was added drop wise with constant stirring until the pH of the solution reached to 10. The precipitate thus obtained were filtered on buckner funnel and washed several times with distilled water. The precipitate were dried in oven at 70^oC for 24 hrs and were calcined at 500^oC in a muffle furnance for 5 hours. Obtained material was ground and sieved through 100 mesh size sieve.

Equipments

Equipment used

Characterization and structural education of the synthesized oxides were performed using The power X-ray diffraction patterns were recorded using Rigaku rotating anode Ru-H3R X-ray diffractometer as well as Brucker D8 high resolution diffractionate and PAN analytical X'perts Pro diffractometer employing CuKα (λ = 1.5404 Å) radiation. The crystalline size of zinc ferrite was calculated using Scherrer equation.

$$t = K\lambda / B \cos \theta$$

Where t is the average crystallite size of the phase under investigation, K is the Scherrer constant (0.89), λ is the wave length of X-ray beam used, B is the full-with half maximum (FWHM) of diffraction (in radians) and θ is the Bragg's angle.

Transmission electron micrograph (TEM) were recorded on Hitachi H7500. The samples were dispersed in ethanol and then treated ultrasonically in order dispersed individual particles over a gold grid.

The magnetic properties of the solid was measured at room temperature using a Vibrating sample Magnetometer Model 155.

The surface morphology of Nickel oxide prepared by precipitation method was investigated by using scanning Electron Microscope Quanta 200 FEG (FEI Netherlands).

Calibration of pH meter

We calibrated the pH meter using buffer solution of pH 4, pH 7 and pH 9.

RESULTS AND DISCUSSION

X-Ray studies

As is well known that properties and performance of different devices depend strongly on the surface characteristics. X-Ray diffraction of synthesized zinc oxide is shown in Fig. 1. X-ray diffraction patter of pure zinc-oxide indicated that zinc oxide in the form of ZnO hexagonal wutrzite structure Fig. 1. In X-ray diffraction, some prominent peaks were considered and corresponding d-values (2.8179, 2.6049, 2.4786……..) were compared with standards JCPDS file No 80-0075 (Table 1). X-ray diffraction shows that metal oxide is pure ZnO having ZnO is a single crystalline and exhibit hexagonal wurtzite structure. X-ray diffraction shows high degree of orientation. It is found that form is anisotropic that on average crystallites may be regarded as cylinders but they are in fact right prisms whose cross section is an irregular hexagon. Size of the crystals was also calculated using Scherrer equation and it was observed that it support TEM studies. X-ray diffraction data indicates that ZnO naoparticles have hexagonal unit cell structure. Individual nanoparticles having size 21-40 nm were found. There is excellent agreement between experimental and theoretical results.

Table 1: X-ray diffraction data of zinc oxide

S. No.	d = λ/2 Sin θ (Observed)	d = λ/2 Sin θ (Reported)	I/I_O x 100% Observed	I/I_O x 100% Reported
1	2.8179	2.8179	57.86	57.86
2	2.6049	2.6048	44.24	44.25
3	2.4786	2.4785	100	99.99
4	1.9128	1.9128	22.92	22.92
5	1.6269	1.6269	32.43	32.45
6	1.4784	1.4785	27.62	27.64
7	1.4089	1.4087	4.40	4.39
8	1.3789	1.3789	24.32	24.30
9	1.3601	1.3601	11.41	11.41
10	1.3024	1.3025	1.90	1.91
11	1.2393	1.2392	3.80	3.81
12	1.1822	1.1822	1.90	1.90

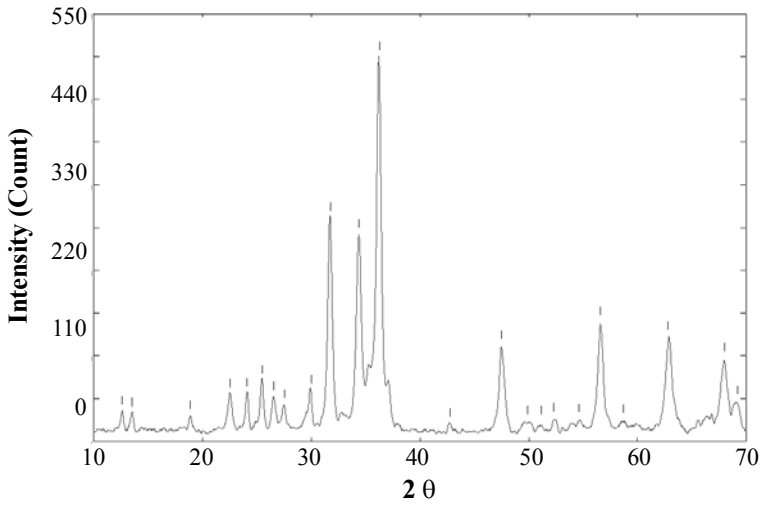


Fig. 1: X-ray diffraction patterns of calcined sample after calcinations

Thermal analysis

Thermal analysis includes a group of techniques in which a physical property of a substance is measured as a function of temperature or time while the substance is subjected

to a controlled temperature programme. The analysis involves thermogravity (TG), differential thermal analysis (DTA) and derivative Thermogravimetry (DTG). Thermal Gravimetric studies of the calcined oxides prepared were done between a temperature range of 10-1000^{o}C under N_2 atmosphere. The TGA/DTA curves of the oxides are shown in Fig. 2. The maximum total weight loss observed for Zinc oxide and their corresponding temperature is summarized in Table 2. Results showed that in the synthesized oxides shows some weight loss and oxide undergoing decomposition, dehydration or any physical change. From TGA curve we observed that Zinc oxides shows stable weight loss above 959.88^{o}C.

Table 2: Observations of weight loss for zinc oxide at corresponding temperature range

S. No.	Maximum % loss in weight	Temperature range ^{0}C
1	16.817%	35.03-425^{0}C
2	13.212%	691.58-959.88^{0}C

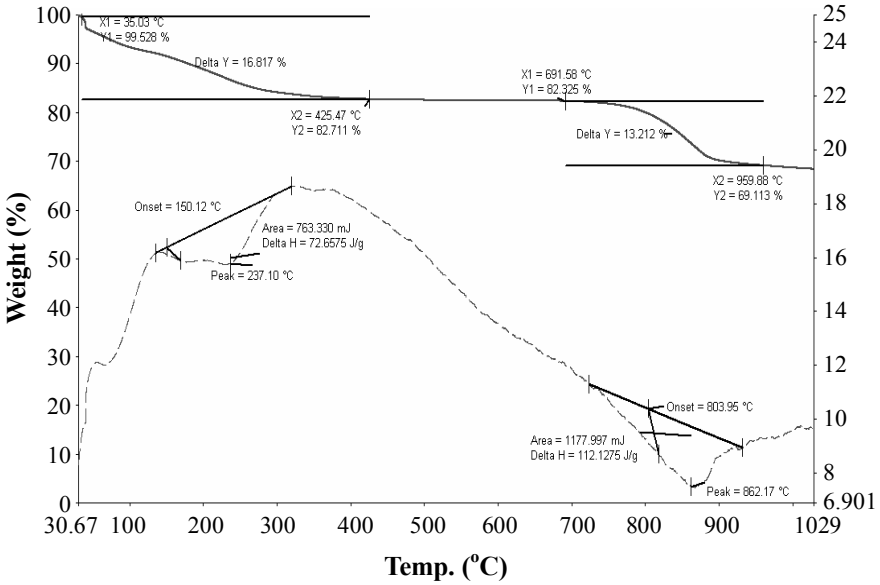


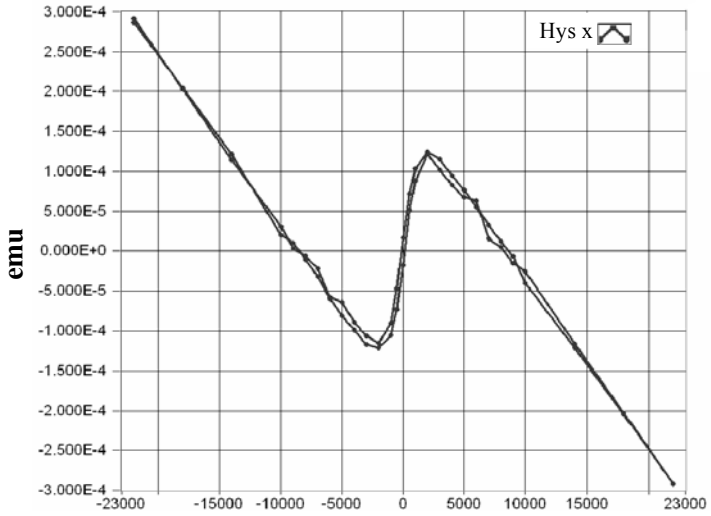
Fig. 2: TGA-DTA graph of zinc oxide

In DTA curve also, there is exothermic and endothermic peak which shows phase transition, solid state reach on any chemical reaction occurred during heating treatment.

From TGA curve we observed that zinc oxide showed stable weight loss above 959.88^{o}C (Fig. 2).

Magnetic measurements

The zinc oxide is amphoteric oxide and semiconductor at room temperature. It has excellent piezoelectric properties. It is possible to induce room temperature ferromagnetic like behavior in ZnO nanoparticles without doping with magnetic impurities but simply inducing an ferromagnetic alteration of their electronic configuration (Fig. 3).



Parameters	Upward part	Downward part	Average	Parameter definition
Hysteresis loop				Hysteresis parameters
Hc Oe	-22004.00	-22000.000	-2.000	Coercive field: Field at which M//H Changes sign
Mr emu	-17.684E-6	15.698E-6	16.791E-6	Remanent magnetization: M at H = 0
S	0.061	0.055	0.058	Squareness: Mr/Ms
S^+	1.035	1.036	1.036	1-(Mr/Hc) (1/slope at Hc)

Fig. 3: Magnetic measurements of synthesized ZnO

SEM and TEM studies

Morphology of the sample was investigated using SEM and TEM. Parts (a, b and c) of Fig. 4 shows typical SEM images of the sample. Scanning electron microscopy shows the hexagonal structure of zinc oxide Fig. 4. SEM images demonstrate that a bulk quantity of flower like bunches exist. Each bunch is gathered of closely packed nanometer scale rods.

Parts (a) and (b) of Fig. 5 shows TEM images of the sample. The flower like nanostructure is observed from part [b]. During the TEM sample preparation, flower like nanostructures were not destroyed. This indicates that the formation of flower like nanostructures is not due to aggregation.

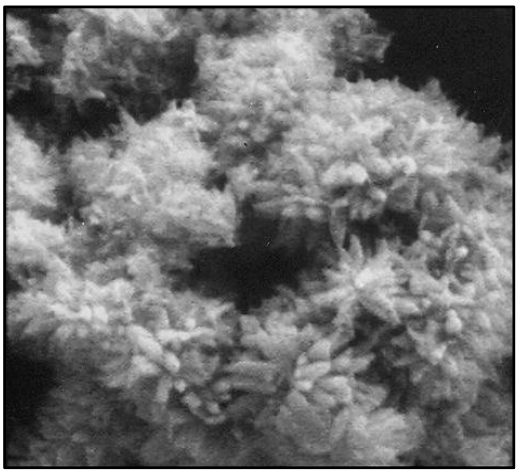


Fig. 4 (a): SEM Micrographs of zinc oxide

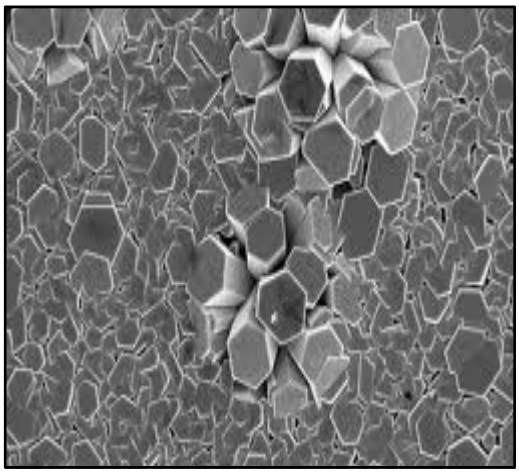


Fig. 4 (b): SEM Micrographs of zinc oxide

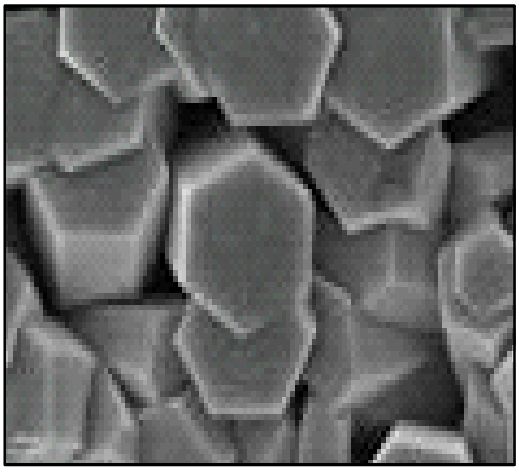


Fig. 4 (c): SEM Micrographs of zinc oxide

TEM is evidence for formation of hexagonal ZnO nanoparticles (Wurtzite structural type Fig. 5). TEM studies shows that the ZnO nanoparticle are hexagonal and size of the obtained nanoparticles is in the range 21-40 nm Table 3.

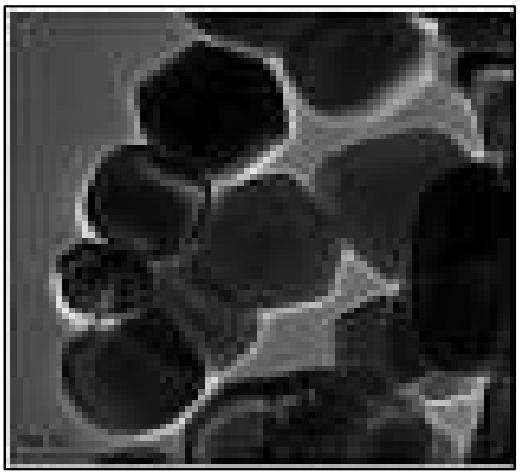


Fig. 5 (a): TEM micrographs of zinc oxide under high magnification (Scale bar is 20 nanometer)

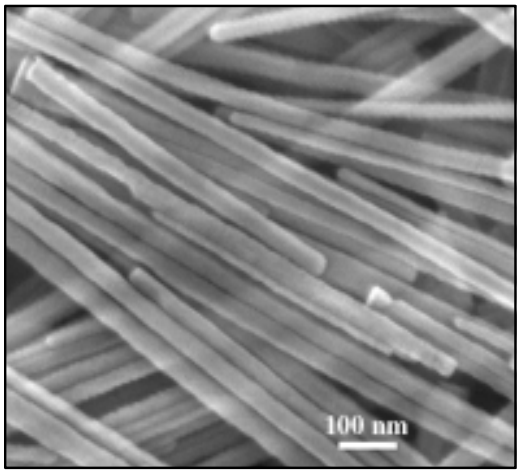


Fig. 5 (b): TEM micrographs of zinc oxide under low magnification (Scale bar is 100 nm)

Table 3: Practical size of synthesized zinc oxide at different scales

S. No.	Scale (20 nm)	Scale (100 nm)
1	39	38
2	36	37
3	35	35
4	21	21
5	37	37
6	38	38
7	37	36
8	26	25
9	40	39
10	40	40
Range	21 nm to 40 nm	21 nm to 40 nm

CONCLUSION

In conclusion, a general and facile approach has been developed to prepare nanosized zinc oxide from commercially available reagents by aqueous precipitation method. There is excellent agreement between experimental and theoretical results. There is great attention in zinc oxide nanoparticles both from the point of view of simpler, cheaper and easy handling using aqueous precipitation method and determination of fundamental properties. The results indicates that nanosized zinc oxide nanopricles with hexagonal wurtzite crystal structure. From TEM studies it is found that particle has average size of 21-40 nm. SEM studies indicate that ZnO has hexagonal structure. Magnetic measurements showed that ferromagnetic nature.This method is advantageous over the existing methods for synthesis of nanosized zinc oxide. Therfore the proposed method is very much promising and have extensive applications.

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