

## SYNTHESIS OF ZnO NANOPARTICLES USING SOL-GEL METHOD

Veronica Deekala<sup>1</sup>, Jyothsna Pragathi Yazala<sup>1</sup>, Anitha Kowthalam<sup>2</sup> and Ramesh Raju Rudraraju<sup>1\*</sup>

<sup>1</sup>Department of Chemistry, University of College of Sciences, Acharya Nagarjuna University, Guntur, 522510, India.

<sup>2</sup>Department of Chemistry, Sri Krishnadevaraya University, Ananthapur, 515003, India.

\*Corresponding Author: Ramesh Raju Rudraraju

Department of Chemistry, University of College of Sciences, Acharya Nagarjuna University, Guntur, 522510, India.

Article Received on 08/04/2019

Article Revised on 29/04/2019

Article Accepted on 20/05/2019

### ABSTRACT

ZnO nanoparticles are synthesized by using SOL-GEL method. In this method, the solution was maintained at PH-11 and calcination temperature at 600<sup>0</sup>C. The structure, morphology and nanoparticle size of ZnO were investigated. The structural analysis confirmed the Cubic structure of ZnO was formed without impurity. Morphological and Elemental Analysis reveal the ratio of Zinc and oxyzen of ZnO particles. From D.C Conductivity and Photo-Catalytic studies, we confirmed that these particles also acts as Conductor and Photo-catalyst respectively.

**KEYWORDS:** Zinc Oxide, SOL-GEL Method, Nanoparticle, Conductor, Photo-Catalyst.

### INTRODUCTION

Nanotechnology involves the creation and Manipulation of materials at the Nanometer(nm) scale either by scaling up from single groups of atoms or by refining or reducing bulk materials. Bulk materials possess relatively constant physical properties regardless of their size, but nanoparticles exhibit size dependent physical and chemical properties. Nanoparticles posses the unique size dependent property as 'Specific Surface Area', which is the ratio of surface area to volume. The production of nanoparticles can be achieved through two approaches known as 'top-bottom' approach and 'Bottom-Up' approach. In the Top-Bottom approach, the bulk material is broken down into particles at Nano-scale through different processes like grinding, milling etc. In the Bottom-Up approach, the atoms/molecules self assemble to form new nuclei, which grow into a particle of Nano-Scale. A number of specific methods have been developed amongst them those which are broadly in use like Co-Precipitation method, Sol-Gel method, Micro emulsion technique, Ball-Mill method and Green chemical synthesis. The Sol-Gel techniques having better advantages such as homogeneity compared to the traditional methods, high purity, lower processing temperature and more uniform phase distribution in multi component systems, better size and morphological control, the possibility of preparing new crystalline and nanocrystalline materials.

#### Zinc Oxide Nanoparticles (ZnO NPs)

Zinc oxide is non-toxic material and is listed as 'Generally Recognized As Safe'(GRAS) by the united states Food and Drug Administration. Due to the

antimicrobial properties ZnO has been used as linings and coating in food containers. ZnO nanoparticles owing to their small size and large specific surface exhibit enhanced antimicrobial activities. Zinc oxide possesses a high radiation, chemical and thermal resistance, it is widely used in creation, of various instruments in particular to form transparent contacts of solar cells. Due to its unique optical, acoustic and electrical properties ZnO finds use in Gas sensors, varistors and generators of surface acoustic waves. The crystal structure, lattice parameters, band gap, melting point and density of Zinc Oxide is given in table-1.

**Table 1.**

S.no	Properties	Zincoxide
1	Preferred phase	Wurtzitee
2	e parameters 'ALattice'	A=3.250 Å.
3	Lattice parameters 'C'	C= 5.207 Å
4	C/A	1.602
5	Thermal conductivity	0.6 , 1-1.2 μ Siemen
6	Static dielectric constant	8.656
7	Band gap	3.37 eV @ 300K direct
8	Refractive index	2.008, 2.029
9	melting point	1975 <sup>0</sup> C
10	Density (solid)	5.6 g/cm <sup>3</sup>

The present work focus on the synthesis nanocrystals of ZnO by Sol-Gel process. These nanoparticles are characterized by XRD diffraction and morphology of Nanocrystalline were investigated by Scanning Electron Microscopy(SEM), Energy Dispersive Spectrum(EDS), Fourier Transform Infrared Spectrum(FT-IR).

## EXPERIMENTAL

### Preparation of Zinc Oxide Nanoparticles

Zinc nitrate dihydrate of 90% purity, KOH and distilled water are used in this synthesis of Zinc Oxide. 8 gms of KOH is dissolved in 15 mL of distilled water which is taken in 50ml of burette. One mole of zinc nitrate is taken and dissolved in 100ml of deionised water. To maintain the PH value of the zinc nitrate solution at 12 KOH solution is added dropwise through burette. The solution is stirred for 2 h at 300°C using Magnetic Stirrer. Until the solution becomes transparent. The reacted solution is dried at 60°C overnight to yield milky-white ZnO nanoparticles, which are finally calcined at 100°C for 1 h and preserved in air-tight vials for further studies. The calcinated powder is grinded for 2hrs in motor. The chemicals, organic solvents and the reagents used in the study are of analytical reagent (AR) quality and the media are obtained, Chennai and Himedia Ltd. Mumbai.

### Mixing and grinding

Zinc Oxide material is sensitive to impurity elements. So, we used high purity chemicals with 99% purity or above. Stoichiometric quantities of precursor materials Zn(NO<sub>3</sub>)<sub>2</sub>, KOH are taken and intimately mixed. This serves several goals, to reduce the atomic diffusion distance so that the reaction can be complete, to break up large grains and to achieve chemical homogeneity.

Grinding process is done for 6 hours using above mentioned raw materials in Stoichiometric amounts. Usually grinding cannot be done too fine to reach 0.1 nm mean particle size where colloidal properties may play a role in subsequent forming operations. In the present work, grinding is accomplished using Agate mortar and pestle. Sufficient amount of methanol is used to prevent selective sedimentation of reagents by forming slurry.

### Calcining

After mixing the raw powders in Agate mortar, the fine powder need to be heated to effect solid state reaction among the reagents to form desired solid solution and to remove the gases of the reaction to form final powder. Calcining below 600°C in Lisicon materials results in loosing of CO<sub>2</sub>, Na, and H<sub>2</sub>O and causes complete the reaction. Here, we calcined the raw powder in Alumina crucible which can withstand higher temperatures.

### Pelletization

The calcined powder is pressed into circular discs with diameter 13mm and thickness 3-4mm at a pressure of 3 tons/sq. inches. The powder should be added with a

binder i.e. 5% of Poly Vinyl Alcohol (PVA) in order to bind the particles during the pressing process.

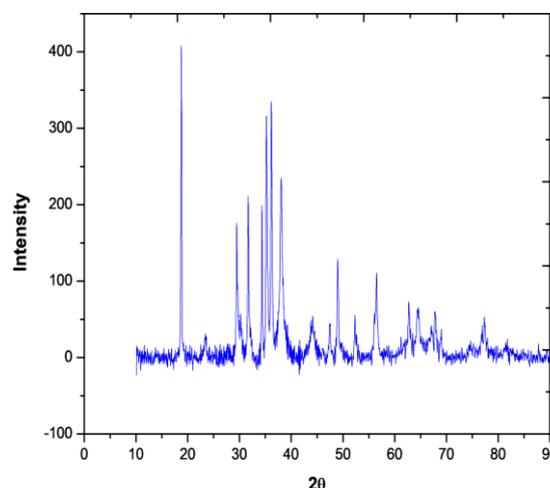
### Forming and Sintering

Sintering is needed for calcined powders in order to eliminate the unwanted peaks other than our desired phase. Intermediate compounds formed during calcinations may be eliminated by heating at proper sintering temperature. Calcined powder is sintered and used for the purpose of XRD, and FT-IR Spectroscopic studies. The pressed discs are then placed on platinum foil and sintered in silicon carbide heat furnace in air at 650°C for 2 hours. The sintering temperature alters the properties and morphology of the compound.

## RESULT AND DISCUSSION

### X-ray Diffraction Analysis

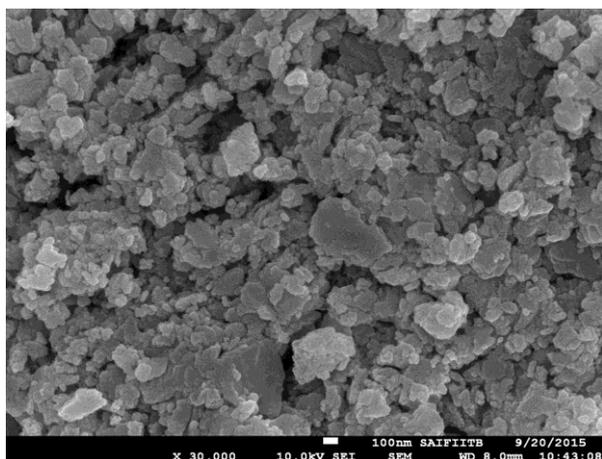
The diffractogram obtained complexes has been given in figure and the observed diffraction data, with the help of the data obtained from the powder XRD, the particle size calculations are performed using Scherer equation.



The diffraction pattern is recorded and radius 2θ values of 36.088 and 18.768 are observed, which corresponds to Bragg reflections of hexagonal wurzite structure which are in agreement with JCPDS File No. 36-145.

### Field Emission-Scanning Electron Microscopy (FE-SEM):

FE-SEM enables us to obtain chemical information from the specimen by using various techniques, including the X-ray energy dispersive spectrometer (EDS). The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface, topography, composition and electrical conductivity.

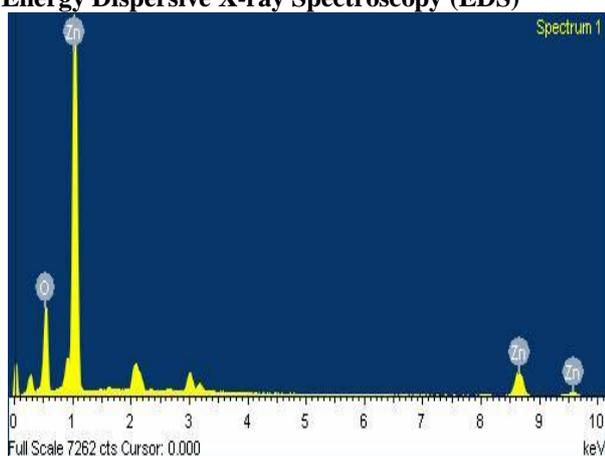


FE-SEM Morphology for Zinc Oxide

Table 2: The elemental composition of Zinc and Oxygen in Zinc Oxide NP.

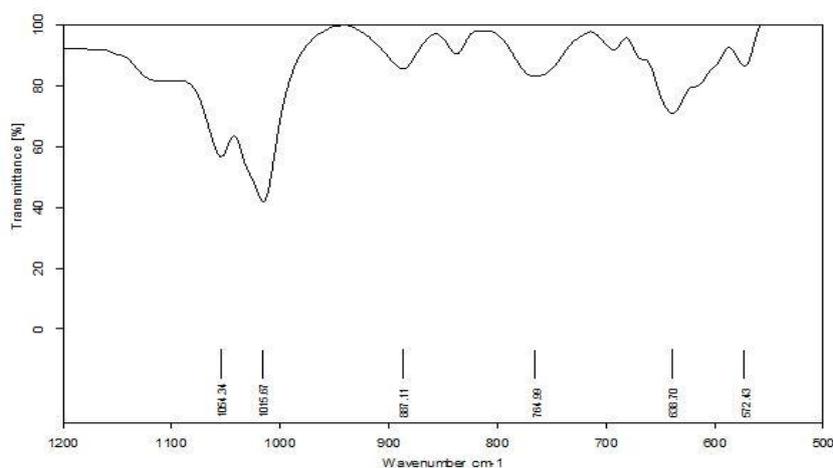
Element	Weight%	Atomic%
O	22.31	53.98
Zn	77.69	46.02
Total	100.0	100.0

## Energy Dispersive X-ray Spectroscopy (EDS)



EDX of ZnO for chemical synthesis

## Fourier Transform Infrared Spectroscopy

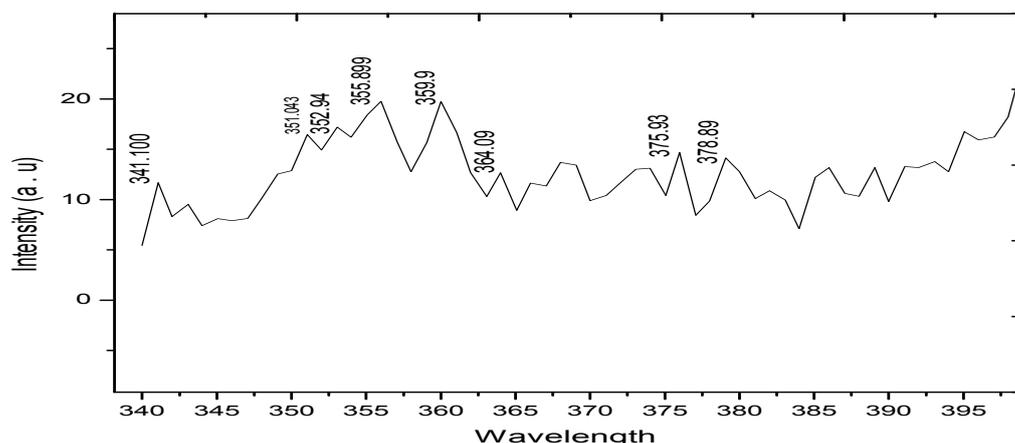


FTIR for Zinc Oxide(Chemical Synthesis)

## Peak Assignments of Zinc Oxide(Chemical Synthesis).

S. No	Wave number (cm <sup>-1</sup> )	Peak Assignments
1	1054,1015	O-C Stretching
2	882	CH <sub>2</sub> Stretching
3	630	P-O Stretching
4	572	C-H Bending

**Photoluminescence:** The Energy band gap for ZnO is in good agreement with standard value(3.37-3.80 eV).



PL spectra for ZnO (Chemical Synthesis)

### Photo-Catalysis

By Passing the UV light on the Zinc Oxide (Catalyst) using the photo-catalysis, the solution becomes transparent i.e. its indication of the photo-reduction of the solution. Therefore the zinc oxide acts as photo-catalytic material. The exposure of UV light to the ZnO, decolorized the dye, the decolorization signifies the catalytic activity of ZnO nanoparticle. It is vastly using in inorganic dye industry. So it is industrial catalyst for photo reduction.

### CONCLUSION

The prepared ZnO NP's are Characterized by using spectroscopic, microscopic, conductivity, photoluminescence and photo degradation studies such as XRD, FE-SEM, FT-IR and Photo catalysis.

- From the XRD results, the crystal size of Green Synthesis method ZnO is less than the Chemical method ZnO.
- FE-SEM results revealed the rod like morphology of the Green Synthesis ZnO, the Chemical method ZnO is Spears shaped morphology.
- From the Energy Dispersive Spectroscopy (EDS) the atomic percentage and weight percentage of Zinc and Oxygen of ZnO particle.
- From the Photo-catalytic Studies, we found that the ZnO NP's are also acts as a photo catalyst.

### REFERENCES

1. A.GLeyva, P.Stoliar, M.Rosenbusch, V.Lorenzo, P.Levy, Albonetti, M.Cavallini, F.Biscarini, H.E.Troiani, J.C. uriale, R.D.Sanchez. Microwave assisted synthesis of manganese mixed oxide nano structures using plastic templates. Journal of solid state chemistry vol.117, pp. 3949-3953, 2004.
2. T. suresh and Annadurai[2013] synthesis, characterization and photo catalytic degradation of malachite green dye using titanium dioxide Nanoparticles. International journal of research in Environmental science and Technology .Vol.3,no.3,pp.71-77 ,2013
3. Harish kumar, Manisha and Poonam Sangwan synthesis and characterization of MnO<sub>2</sub> Nanoparticles using Coprecipitation Technique. International journal of chemistry and chemical engineering vol. 3, no.3, pp. 155-160., 2013
4. E.Kumar, P.Selvarajan, D.Muthuraj. Synthesis and characterization of CeO<sub>2</sub> nanocrystals by Solvothermal route. Material Research vol.16 no.2, pp1-6, 2013. No.3 PP.71-77.
5. Askarinjad, Azadeh, Alavi, Mohammad Arvin, Morsali. Sonochemical Synthesis of ZnO nano Particles A Novel Direct method. Iran J. Chem Eng. vol.30, no. 3, pp 75-81, 2011.
6. H.A Wahab & A.A salama et al. (2013). Optical, structural and morphological studies of ZnO nano-rod thin film using sol-gel. 3, 46-51.
7. C. Chai (2012). The Global Market for Zinc Oxide Nanopowders 2012. New Report on Global Zinc Oxide Nanopowder Market, 135-140.
8. K Yung, H. Ming, C. Yen & H.Chao, (2012). Synthesis of 1D, 2D and 3D ZnO Polycrystalline Nanostructures Using Sol-Gel Method. Journal of Nanotechnology, 1-8.
9. A.R.Bari, M.D.Shinde, D.Vinita & L. Patil (2009). Effect of Solvents on the Particle Morphology of nanostructured ZnO. Indian Journal of Pure & Applied Physics, 47, 24-27.
10. H.Morkoç, & ÜÖzgür. (2009). Chapter 1 General Properties of ZnO. In Zinc Oxide: Fundamentals, Materials and Device Technology. Wiley-VCH.