

## Preparation of ZnO nanoparticle by sol gel method and their characterization

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### Abstract

ZnO is the formula of an inorganic compound Zinc oxide. It is a whitish powder that isn't soluble in water. Various products and materials like rubbers, cosmetics, plastics, cement, ceramics, ointments, lubricants, adhesives, foods, first-aid tapes, and food supplements have ZnO as an additive in them. Naturally, zinc oxide occurs as the 'zincite' mineral, while most ZnO is synthetically produced. In this research zinc oxide (ZnO) synthesized via way of means of the use of sol- gel techniques that have been very natural in generating small sized nanostructures ( $<100$  nm). ZnO nanoparticles had been effectively synthesized via way of means of easy Sol-Gel approach. The organized ZnO nanoparticles have been characterised by the use of XRD, FT-IR.

**Keywords:** ZnO, Synthesis, properties, XRD and FT-IR

### Introduction

The properties of ZnO nanoparticles depends on how they are synthesized. Naturally, Zinc oxide occurs as 'zincite,' a coarse-grained mineral. A different number of chemical, biological, and physical methods are used to synthesize nanoparticles of Zinc oxide. Extractions of plants and other microorganisms are employed in biological methods, and they are relatively new. Physical methods are such as thermal evaporation method, arc plasma method, vapor deposition, etc., while biosynthesis, sol-gel process, precipitation, combustion methods, hydrothermal techniques, and wet chemical synthesis are some of the chemical methods that are used to synthesize nanoparticles of ZnO. The solid-state pyrolytic method is a rapid, simple, and cost-effective method that gives controllable size.

ZnO is the formula of an inorganic compound Zinc oxide. It is a whitish powder that isn't soluble in water. Various products and materials like rubbers, cosmetics, plastics, cement, ceramics, ointments, lubricants, adhesives, foods, first-aid tapes, and food supplements have ZnO as an additive in them. Naturally, zinc oxide occurs as the 'zincite' mineral, while most ZnO is synthetically produced. Zinc oxide nanoparticles have less than 100 nanometers of diameter. As compared to their high activity as a catalyst and size, Zinc oxide nanoparticles have a large enough surface area. The way they are synthesized modulates the exact chemical and physical properties of nanoparticles of Zinc Oxide.

For numerous applications in biomedicine, Nanotechnology is the best suitable technique for the last few decades. Nanoparticles of ZnO has remarkable ultraviolet (UV)-absorbing characteristics and visible light transparency, turning these Nanoparticles into remarkable sunscreen agents. On a daily basis, Zinc oxide is exposed to humans, and it originates from numerous sources, for example, cosmetics, intake of food, and many other products. In the crust of the earth, zinc is an important and 5th common element, and in high concentration, zinc is dangerous to humans. Inside the body, ZnO dissolves only one time. To prevent the deficiency of zinc in humans, a form of zinc, Zinc acetate (E650) is added into foodstuffs. ZnO is less

toxic, less expensive, and more biocompatible than Nanoparticles of other metal oxides. They are shown to have high photocatalytic activity.

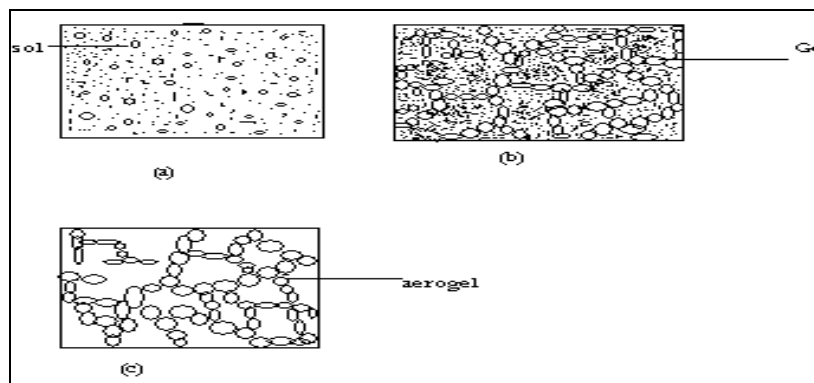
There are several methods that have been put forward for synthesis of these materials, namely chemical vapour condensation, arc discharge, hydrogen plasma-metal reaction, and laser pyrolysis with in the vapour phase, The other methods include Sol-gel synthesis, micro-emulsion, microbial processes which occur in liquid state, ball milling which is conducted in solid state<sup>1-2</sup>. The synthesis process of nanoparticles defines largely their properties. The range of metal oxide is great and their range of properties and possible applications appear to be enormous. Sol-gel process has several advantages. Solitary sol gel installation can yield materials at ultra-temperatures, synthesize nearly any substance, co-synthesize two or more substances with one other, exactly observance the microstructure of the top outputs, and punctually dominance the physical mechanical, and chemical characterizes of the ultimate outputs.

### Sol-Gel Route

As the name suggest sol-gel involves two types of materials or components, 'sol' and 'gel' are known science the time when M. Ebleman synthesized them in 1845. First of all sol-gel process is usually a low temperature process. This means less energy consumptions and less pollution too, it is a desired process. Although sol-gel process generates highly pure and well controlled ceramics as compares with other process like CVD or metalloorganic vapors derived ceramics. The choice of course depends upon the product of interest, it size instrumentation available, case of processing, etc.

In some cases sol-gel can be an economical route, provided precursors are not very expensive. Some of the benefits like getting unique material such as aero-gel, zeolites order porous solids by organic-inorganic hybridization are unique to sol-gel process. It is also possible to synthesize nanoparticles, nanorodes, nanotubes, etc. using sol-gel technique.

Sols are solid particles shown in fig. bellow they are thus a sub class of collides.



**Fig 1:** (a) sol, (b) gel and (c) monolithic solid Oaertogel

Gels are nothing but a continuous network of particles with porous filled with liquid (or polymers containing liquids). A sol-gel process involves formation of sols in liquid and then connecting the sol particles to form a network. By drying the liquid, it is possible to obtain powders, thin films or even monolithic solids. Sol-gel method is particularly useful to synthesize ceramics or metal oxides although sulphides, borides and nitrides also are possible.

## Experimental Part and method

### Synthesis of ZnO Nanoparticles

#### Chemicals

Zinc Nitrate,  $\text{H}_2\text{O}_2$  (Hydrogen peroxide), Double distilled water, ethanol.

#### Equipments

Beaker, Magnetic Stirrer, Centrifuge, Dehydrator, muffle furnace, motal & pistal.

#### Synthesis

The synthesis of ZnO nanoparticles via way of means of sol-gel technique was as followed: in this process 15.7 gm of zinc Nitrate was added to 500 ml of double distilled water with non-stop magnetic stirrer till Zinc Nitrate dissolved completely in distilled water. Then the solution was heated to  $50^\circ\text{C}$  and 700 ml of alcohol (ethanol) was added slowly with non-stop stirring, ethanol used as solvent and reductant for manufacturing of virtuous nano-particle with narrow particle size distributions and to adjust the pH of sol. The pH of solution was near about 5.4. After this, add 6 ml of  $\text{H}_2\text{O}_2$  in drop wise manner to the above solution and mixed it by using a magnetic stirrer until to get a clear solution. After adding  $\text{H}_2\text{O}_2$  the pH of sol from 5.4 to 9 (Alkaline Condition) of the solution. Let settle down and discard the upper layer. Transfer the gel into Centrifuge tubes and centrifuges at 10,000 rpm for 5 min. Discard the supernatant liquid and wash the sample with distilled water. Repeat the above step 2–3 times. The sample allowed to dry at  $80^\circ\text{C}$  until the samples becomes dry then annealed the sample at  $500^\circ\text{C}$  for 1 h in muffle furnace, crushed the annealed sample of CuO nanoparticles with Motal and Pistal. Figure 1 shows the Flow Diagram of synthesis of ZnO nanoparticles by sol- gel method.

#### Characterization of ZnO nanoparticles

The characterization of synthesized nanoparticles was done by following analytical techniques, such as Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD).

FT-IR (Fourier Transform Infrared) spectroscopy is a technique which analyses Infra-red spectrum of a sample by absorption. The sample may be solid, liquid or in gaseous state. Magnesium oxide nano-particles show different peaks at different levels in FT-IR measurement, which correspond to different functional groups, and stretches.

XRD (X-ray Diffraction) is used for identification of phase of a sample possessing crystallinity. It also provides information on unit cell dimensions. The homogeneity and bulk composition is ascertained, based on Bragg's law which relates the wavelength of radiation with the diffraction angle and lattice spacing in crystalline sample

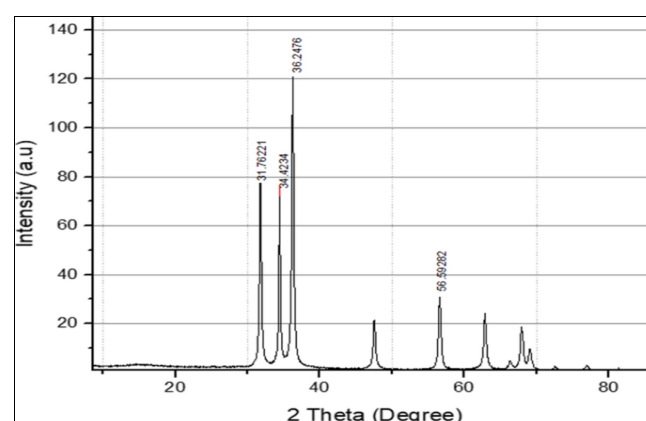
## Results and Discussion

### X-Ray diffraction analysis

The crystal structure and size of ZnO nano-particles were analysed by XRD. The sharp peak of Fig.1 indicates that the nature of nano-particles was crystalline. X-Ray Diffraction Analysis or X-Ray Crystallography was used to identify the atomic or molecular structure of particles in which the crystalline atoms cause a beam of incident X-rays to diffract into many specific directions. The crystal structure of ZnO nano-particles prepared by sol-gel route has been showed in Figs. 4 at  $\text{CuK}\alpha$  ( $\lambda = 1.54056 \text{ \AA}$ ) in the Bragg reflection  $2\theta$  range from  $10^\circ$  to  $70^\circ$  and  $10^\circ$  to  $80^\circ$  respectively. Sizes of the crystallite particles were calculated using Scherrer's equation.

$$D = K\lambda / \beta \cos\theta$$

where, D is the crystallite size of the particles, shape factor is 'K' (0.9 in the present work). ' $\lambda$ ' is the incident X-ray wavelength (1.54056  $\text{\AA}$ ,  $\text{CuK}\alpha$ ),  $\theta$  is the diffraction angle and  $\beta$  is the full width half maximum.



**Fig 1:** XRD Pattern of synthesized ZnO Nanoparticles.

The peak width and intensity the small particle size is and good crystallinity of ZnO can be easily observed and seen in fig.1. The higher intensity peaks in XRD pattern of the prepared ZnO sample was observed at  $2\theta$ - 31.7621, 34.4234, 36.2476 (Fig. 1). XRD pattern of ZnO shows sharp peaks, referring to the crystalline nature. The synthesised ZnO nanoparticles showed a single phase with clear diffraction peaks according to the information provided by reported data (JCPDS card no 36-1451). Average particle size of the ZnO nanoparticles was determined as 21.82 nm at highest peak ( $36.2476^\circ$ ) according to the Debye–Scherrer's equation. The crystallinity of ZnO refers to a wurtzite hexagonal structure which was result of penetration of two hexagonal lattices.

### FT-IR

FT-IR spectroscopy analysis has been done to observe the chemical and structural nature of the particles. The Infrared absorption band identifies the various functional group of the molecule. Both synthesized nano-particles ZnO has been scanned from 4000 to  $400\text{ cm}^{-1}$ . The region from 4000 to  $1500\text{ cm}^{-1}$  was functional group region and region from 1500 to  $667\text{ cm}^{-1}$  was fingerprint region. The various phase and functional group in nanoparticles were studied by using FT-IR technique

FTIR spectra of the synthesized ZnO in the range of 4000– $400\text{ cm}^{-1}$ . FTIR spectrum of pure ZnO nanoparticles, the peak at  $594.56\text{ cm}^{-1}$  was the characteristic absorption of Zn–O bond and the broad band peak at  $3506.9\text{ cm}^{-1}$  can be attributed to the characteristic absorption of O–H group. These data were similar to the results observed by others<sup>5</sup>. The peak at  $594.56\text{ cm}^{-1}$  was allotted to  $\text{Zn-O}^{1-2}$ . Absorption peak between 507 and 597 indicates the presence of C–I stretching which belongs to halo compounds. Bending Peak at 903 and 1001 shows the presence of C = C group belong alkene class.

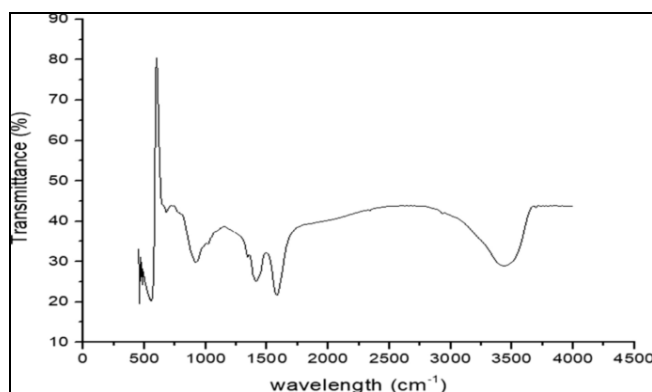


Fig 2: FTIR Spectroscopy of synthesize ZnO Nanoparticles

### Conclusion

In this research zinc oxide (ZnO) synthesized via way of means of the use of sol- gel techniques that have been very natural in generating small sized nanostructures ( $<100\text{ nm}$ ). ZnO nanoparticles had been effectively synthesized via way of means of easy Sol–Gel approach. The organized ZnO nanoparticles have been characterised by the use of XRD, FT-IR. Zinc Nitrate [ $\text{Zn}(\text{NO}_3)_2$ ] has been used in synthesis process of ZnO with spherical shape via Sol–Gel Method EDX analysis confirm the present of ZnO nanoparticles with less impurities and nearly about stoichiometry These ZnO nanoparticles provide super

capability in future packages of food packaging and growing the shelf existence of the product.

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