



Synthesis and Structural properties of Zinc Oxide Nano Particles (ZnO NPs): A Review

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ABSTRACT: ZnO (Zinc Oxide) is generally recognized as safe (GRAS) material by FDA (Food and Drug Administration, USA) that is why it is used as food additive and in various medicine. The FESEM (Field Emission Scanning Electron Microscopy) of hydrothermally synthesized ZnO NPs showed that ZnO NPs are spherical in shape with a diameter of 20-30 nanometer. The XRD (X-rays Diffractometry) and TEM (Tunneling Electron Microscopy) studies of ZnO nanopowder synthesized by ultrasonic spray pyrolysis (USP) technique showed that the crystalline size increased with an increase in the pyrolysis temperature and also exhibit direct band gap in the range 3.37 – 3.40 eV. Also the band energy gap of bulk ZnO crystals was approximately 3.3 eV. The SEM (Scanning Electron Microscopy) micrographs of ZnO NPs prepared by chemical method using PVP (Polyvinyl Pyrrolidone) as capping agent showed spherical shape of nanoparticles, having band gap energy of 3.4 eV and these nanoparticles also showed lattice contraction due to high electrostatic interaction between Zn^{2+} and O^{2-} ions. ZnO is an important member of semiconducting material of II-VI group. Due to unique properties, ZnO is the richest family of nano structures among all semiconducting materials and thus used in the number of application like gas sensor, varistors and low voltage phosphor. ZnO is only semiconductor material extensively investigated after Si/Ge. Also nano cluster of ZnO, which exists as nanoparticles, nanobelts, nanorods, nanotubes, nanowires, nanoflowers as well as colloid and bulk nanoparticles also have shown potential/excellent luminescence properties.

Keywords: ZnO, GRAS, FDA, NPs, XRD, TEM, USP, SEM, PEG, CTAB, MEA, EG.

INTRODUCTION

ZnO is also known as “Lu-Gan Stone” in china and has been used in medical Treatment from number of years ago¹⁴. In 1966 Damen and Porto studied the Raman Effect study of ZnO, Which leads to the identification of ZnO phonon energies¹⁵. ZnO NPs has been employed as a powerful Heterogeneous catalyst during several organic transformations^{16, 17, 18, 19, 20 & 21} with the increase in concentration of ZnO NPs, there is a decrease in the microstructural parameters (such as lattice strain and crystalline size)²². ZnO nanocrystals showed potential novel applications including photocatalysis (acts as photo catalyst)²³, transparent conductive oxides²⁴, ultraviolet (UV) light absorbers²⁵, photocatalysis (i.e. highly active free radicals decomposing or dissociates organic molecules) were also useful as organic Pollutant scavenging and antifouling²⁶. Which also helps in enhancing the photo catalytic properties of semiconductor nanoparticles (Such as ZnO NPs)^{27, 28, 29, 30}. In ZnO NPs when polymeric matrix was added, there was strongly change in physical mechanical, optical, thermal and other properties of a material^{31, 32}. Due to the weakened intermolecular Vander Waal’s forces between the ZnO NPs. Also due to the versatile properties of ZnO NPs as biodegradability and biocompatibility³³, ZnO NPs were used as most efficient catalyst in the Knoevenagel condensation and synthesis of coumarins in High yield, through the Reaction between various ortho-hydroxy benzaldehydes with 1, 3 dicarbonyl compounds under microwave and thermal conditions³⁴. The recent literature survey of nano ZnO³⁵ as Heterogeneous catalyst received many versatile features because of its inexpensive, non toxic catalyst and has eco-friendly nature (such as low corrosion, minimum wasting, easy transportable recycling of the catalyst and easily disposal nature). The various organic ligands such as polyethylene glycol (PEG)³⁶, cetyltrimethyl ammonium bromide

(CTAB)³⁷, olic acid³⁸ and gelatin³⁹ acts a polymerization agents that had been used to control the morphology and size of ZnO NPs and also helps in order undergoing spontaneous growth and aggregation. ZnO synthesized by using Zinc Chloride and sodium hydroxide as precursors of different temperatures revealed that the size of the particles is directly proportional to the reaction temperature as well as ionic strength (i.e. Base concentration). The possible mechanism or reason for these above two observations is that the particles may acquire the shape and size in such a way so as to minimize the Gibb's free energy⁴⁰. In contrast to physicochemical approach, Biological approach of formation of ZnO NPs is a simple and cost effective⁴¹. The formation of ZnO NPs biologically by using *C. Procera* (calotropis Procera), which is a desert plant known as madar in Grreco Arab medicine (Latex at Room Temperature) and is widely distributed in Tropical and subtropical Africa and Asia^{42, 43}. In addition to this, for the synthesis of spherical shaped ZnO NPs milky latex of calotropis procera had been used as a reducing material as well as surface stabilizing agent⁴⁴. In the recent studies in the field of biology^{45, 46, & 47}, many Researchers reported that ZnO NPs used in the treatment of Cancer^{48, 49, 50, 51 & 52}. Also the antibacterial properties of these ZnO NPs against *Escherichia coli* and *staphylococcus aureus*⁵³ were also studied. Furthermore ZnO NPs have good biocompatibility to human cell⁵⁴. The possible mechanism for ZnO antibacterial activity is the release of Zn²⁺ ions which can damage the cell membrane and interact with interacellular contents⁵⁵.

*Wide range of nanostructures of ZnO, makes it best suited for nanoscale optoelectronics (M.H. Huang et al, 2001)*⁵⁶ and piezoelectric nanogenerators (J. Song et al, 2006)⁵⁷. As Nano zinc oxide has antibacterial, antifungal properties it is been considered as efficient material in Medicine and Biotechnology field (Z.L. Wang et al; J.Sawai et al, 2004)^{58, 59}. Unique optical, electrical and chemical properties of nano ZnO have increased its importance in electronic sector, solar cells, gas sensors, and photocatalytic degradation of chemicals (J. B. Baxter et al, 2005; Z. Ling et al, 2001; C. Y. Hsu et al, 2008; Y. Wang et al 2008)^{60, 61, 62 & 63}.

For the past decade, scientists have been involved in the development of new synthetic routes enabling the precise control of the morphology and size of the nanoparticles. In addition, nanoparticle synthesis can be possible *via* liquid (chemical method)^{64, 65, 66, 67, 68 & 69}, solid, and gaseous media^{70, 71, 72 & 73}, but due to several advantages over the other methods, chemical methods are the most popular methods due to their low cost, reliability, and environmentally friendly synthetic routes, and this method provides rigorous control of the size and shape of the nanoparticles^{74, 75 & 76}. In general, nanoparticles with high surface-to-volume ratio are needed, but the agglomeration of small particles precipitated in the solution is the main concern in the absence of any stabilizer. In this regard, preparations of stable colloids are important for nanoparticle growth. In addition, nanoparticles are generally stabilized by steric repulsion between particles due to the presence of surfactant, polymer molecules, or any organic molecules bound to the surface of nanoparticles. Sometimes vander Waals repulsion (electrostatic repulsion) also plays important role in nanoparticles stabilization. With all the issues related to nanoparticle synthesis, there are various types of nanoparticles reported in the literature, e.g., metal nanoparticles, metal oxide nanoparticles, and polymer nanoparticles. Among all these, metal oxide nanoparticles stand out as one of the most versatile Materials, due to their diverse properties and functionalities. Most preferentially, among different metal oxide nanoparticles, zinc oxide (ZnO) nanoparticles have their own importance due to their vast area of applications, e.g., gas sensor, chemical sensor, bio-sensor, cosmetics, storage, optical and electrical devices, window materials for displays, solar cells, and drug-delivery^{77, 78, 79, 80 & 81}.

Nanostructures are of requisite claim owing to their incomparable dimension dependent properties and promising applications as building blocks in electronics, optoelectronics^{82, 83 & 84}, sensors & actuators and in bioimaging^{85, 86}

Among them all, II–VI direct band gap semiconductors have engrossed substantial consideration due to their applications in light-emitting diodes^{87, 88 & 89}, photo-detectors, field emitters and full colour displays.^{90, 91} The wide band gap and notably large binding energy make them an ideal choice for inorganic passivation shells in a variety of semiconductor core/shell nanocrystals and also an attractive host for the development of doped nanocrystals.^{92, 93} In recent years, metal oxides and their nanostructures have emerged as an important class of materials with a rich spectrum of properties and great potential for

device applications, e.g., transparent electrodes, high-electron mobility transistors, gas sensors, photovoltaic and photonic devices and hydrogen storage applications.⁹⁴ Of their class, wide-band gap oxide semiconductors have occupied the forefront in the past decade because of an increasing need for short wavelength photonic devices, high-power, high-frequency electronic devices. Although research on the wide bandgap oxides has been focused on a plethora of oxides e.g. TiO₂, CuO₂, SnO₂ and ZnO etc. among them, zinc oxide an II–VI semiconductor with a wide gap of 3.37 eV and sufficiently large exciton binding energy of 60 meV at room temperature making it suitable as a material for blue and ultraviolet light-emitting devices, has been of growing interest due to their scientific and technological importance. ZnO has a key of nanotechnology and well-recognized functional material suitable for various applications such as optoelectronic, the solar cell and so on.^{95, 96, 97 & 98.} Nano-structured ZnO have been synthesized by various methods, such as chemical vapor deposition,⁹⁹ laser ablation,¹⁰⁰ molecular beam epitaxy¹⁰¹ or a simply just by heating Zn powders with or without catalyst.¹⁰² Because of the ability to produce high yield, easy scalability and low cost, the combination of thermal evaporation and vapor transport method has emerged as a promising route to grow ZnO nanostructures.^{102, 103} In addition to the conventional nanowire and nanobelt structures, a diverse group of novel ZnO nanostructures has been discovered recently, such as nanocages,¹⁰⁴ anorings,¹⁰⁵ nanohelices/nanosprings,¹⁰⁶ nanobows,¹⁰⁷ nanodiscs,¹⁰⁸ nanotetrapods¹⁰⁹ and nanocombs¹¹⁰ etc. These novel nanostructures indicate that ZnO contains probably the richest family of nanostructures among all materials, in both structure and properties, and also provides valuable models for understanding the nanoscale crystal growth. This is in addition to the high potential ZnO has for fabricating novel nanoelectronics and optical devices with enhanced performance. It is now well known that the doping of nanocrystals can be achieved.^{111, 112 & 113}

OBJECTIVE OF THE STUDY

From literature survey it is revealed the main objectives of this study as:

- 1) Versatile applications in industries at higher level.
- 2) In biology and medical sciences (in the treatment of cancer).
- 3) To aware the population about the utility of ZnO nanoparticles.

MATERIAL AND METHODS

ZnO NPs synthesized via sol-gel method in starch media were calcined at different temperatures exhibited a hexagonal (wurtzite) structure, ranging sizes from 30 to 50 nm¹³⁴.

The nanofuilds were synthesized by dispersion of ZnO NPs in EG (Ethylene glycol) solution using an ultra sonicator¹³⁵.

ZnO – SnO₂ (Zinc oxide-tin dioxide) nanocomposites were prepared from ZnO and SnO₂ NPs by using homogeneous precipitation route, in which Zinc acetate dehydrate [Zn (CH₃COO)₂ .2H₂O] and tin (II) chloride dehydrates (SnCl₂, 2H₂O) have been used as precursor, monoethariolamine (MEA) was used as a sol stabilizer¹³⁶.

ZnO flower like and brush pen like nanostructure can be formed on large scale by a hydrothermal decomposition route¹³⁷.

Chu et al recently reported a solution based high yield synthesis of co-doped ZnO nanorods with Room Temperature ferromagnetism¹³⁸.

Xu et al. reported on the high yield synthesis of single crystalline ZnO Hexagonal Nano plates¹³⁹.

Zinc oxides and indium ion doped ZnO Particles with different morphology (flower and prism like structure) were prepared in aqueous medium by the so-called hydrothermal method¹⁴⁰.

Colloid chemical synthesis method opens up ways to prepare nanoparticles with controlled size high specific surface area and phrases with various degree of crystallinity.

Amor Sayari in 2013, synthesized the nanocrystalline ZnO flakes via simple reaction process by using zinc acetate dehydrate dimethyl amine and sodium hydroxide under refluxing at 85°C for 4h¹⁴¹.

A variety of methods such as thermal oxidation of metallic zinc, hydrothermal method, plasma chemical synthesis, laser ablation and vapor condensation^{142, 143 & 144} have been used to synthesized uniform nanosized ZnO nanoparticles and control their size and morphology.

Pure nanocrystalline ZnO compound was synthesized by chemical co-precipitation method. The composite of polyaniline with nanosized prepared by in situ chemical oxidation. Polymerization method with ammonium per sulfate as an oxidant in aqueous HCL acid under constant stirring at 0-4°C in the presence of nitrogen atmosphere¹⁴⁵.

Kim Leang Khan et. Al in 2013 studied that ZnO nanoparticles were synthesized in an ethanolic medium and then mixed with chitosan solution prepared in 1% acetic acid. The result composite was used as a seed layer for the fabrication of vertically aligned ZnO nanorods¹⁴⁶.

P.K. Giri et al. synthesis low cost low temperature method with high yield (>90 %) of synthesizing ZnO nanoparticles by using two different catalysts (acetic acid and Trifluoroacetic acid)

Structural studies: The XRD and FTIR studies of synthesized ZnO NPs by sol-gel method revealed that all of the Zinc Oxide nanoparticles calcined at 400°C, 500°C and 600°C exhibited the hexagonal wurtzite structure.

X -ray diffractometer (XRD) study of synthesized ZnO-SnO₂ nanocomposites by homogeneous precipitation route showed that there are preferred crystal orientation and particle size of thin film.

ZnO nanorods with 30-40 nm diameter and 600-800 nm length were synthesized by a hydrothermal route and ZnO nanowires which had a diameter of 20-30 nm and length of 1-2 micro meter were prepared by an electrodeposition method¹⁴⁷.

The XRD, IR, SEM and TEM investigation of Nanocrystalline ZnO sensors prepared by chemical precipitation method showed that nano ZnO having wurtzite hexagonal structure and the average particle size of prepared ZnO sensors increased with increasing the sintering temperature and decreased with CeO₂ addition¹⁴⁸.

The FESEM measurement of ZnO nanoparticles synthesized by novel gas condensation process revealed that all the nano particles are in the structure of a Hexagonal prism and its average particle size is 20 nm. Also ESCA (electronic spectra for chemical analysis) study of this method confirmed the chemical elemental compositions and also confirmed that powder produced by this system is really ZnO¹⁴⁹.

The XRD spectrum of ZnO flakes prepared by a simple solution process indicates that these nanosized ZnO particles are highly crystallized in structure and the entire diffraction peaks matched well with Bragg reflection of the standard wurtzite type ZnO structure^{150, 151}. The Uv-visible absorption of this process showed that ZnO nano particles has blue shift compare to that of ZnO bulk (375 nm) and demonstrated that this blue shift depends upon the nano crystal size¹⁵².

The FTIR spectrum shows a characteristic significant spectroscopic band at around 521cm⁻¹¹⁵³ and also confirmed the high purity.

The ZnO nanoparticles incorporated HPMC films were prepared by the solution casting method revealed that there are decrease in 30% tensile strength, 37% in young's modulus and 24% in percentage elongation when the ZnO concentration is 0.04%¹⁵⁴.

P.K. Giri et al. confirmed that XRD patterns of ZnO nanoparticles shows excellent crystallinity. SEM and TEM images shows hexagonal cross section of the nanoparticles and nanorods and they also exhibit a strong Uv-absorption peak at ~378 nm and strong Uv-emission peak at ~380nm¹⁵⁵.

REVIEW OF APPLICATIONS

Zinc Oxide nanoparticles (ZnO NPs) have extensively used in cotton and wool fabrics for Uv-shielding as well as to impart sunscreen activity to the treated textiles¹¹⁴.

Zinc Oxide nanoparticles are commonly applied in Pollutants Removal and disinfectants, because of its high chemical stability, oxidation- reduction capability and poison less characteristics.

Zinc Oxide nanoparticles are used as a suitable candidate for Luminescent materials due to its unique properties such as wide band gap (3.37 eV), large binding energy (60 meV) and Radiation hardness nature^{115, 116}.

Zinc oxide nanoparticles can also be used to increase the wash fastness, by dipping fabrics in a solution containing a specific binder¹¹⁷.

In the decade of 1970, ZnO NPs was used in the manufacturing of simpler Zinc oxide devices like ceramic varistor and piezoelectric Transducer¹¹⁸.

In comparison to bulk material, zinc oxide nanoparticles having a large surface area to volume ratio, which significantly helps increasing the effectiveness in blocking Uv-radiation¹¹⁹.

ZnO has versatile applications including sunscreen food additives, pigments (paint pigment) Rubber manufacture and electronic materials.

Robust ZnO has important applications in the field of biosensors, solar cell window, piezo actuator devices surface acoustic devices and in gas sensors^{120, 121}.

In the Knoevenagel condensation, ZnO NPs used as a most efficient catalyst for the synthesis of coumarins. In addition to this Knoevenagel condensation is one of the most useful and widely employed methods for carbon-carbon formation which helps in the synthesis of the fine chemical¹²² hetero Diels Alder reaction¹²³.

ZnO NPs show high Piezo Electricity because it has melting point 1800°C and symmetrical (Hexagonal wurtzite) as well as not having symmetrical center.

Biological approach for the formation of ZnO NPs has versatile applications in biosensing electronics and photonics¹²⁴.

ZnO NPs is a biocompatible material with antiseptic properties.

ZnO NPs also used in the treatment of cancer biologically.

ZnO NPs can be used to produce vulcanisates with high tensile strength, tear resistance and hardness.

For the carboxylated elastomers, apart from the scorch problems, Zinc-oxide is used as a very effective and common cross linking agent¹²⁵.

Even DNA (Deoxyribonucleic acid) and RNA (Ribonucleic acid) damage has been raising industrial and academic concerns for the safe use of ZnO as effective Uv-shielding agent.

ZnO is used as an additive in the manufacture of concrete and as a coating agent in various paints¹²⁶.

ZnO NPs are utilized in electronic and electrical devices, image recorder, attenuation of light, High Temperature lubricant gas turbine engines, and also used as a demilitarization of chemical biological warfare agent¹²⁷. ZnO NPs also exhibits various catalytic antibacterial, anticorrosive, antifungal and Uv-shielding/filtering properties.

Co-doped ZnO and Mn-doped ZnO would have a potential to be used as the durable Uv-shielding agents for the protection of plastics, textiles and other organic matters from Uv-rays.

ZnO NPs are used in the various commercial products such as cosmetics, sunscreen and also used in various antibacterial activities¹²⁸.

Nano ZnO in future is also used in Auto Industry. Nano ZnO is used in hydrogen fuel engine, in engine oil reduces friction, ZnO LED's used in head light (more efficient less power usage and longer life). Nano ZnO is also acts as an activator and accelerator in vulcanized rubber tyre, having longer working life¹²⁹.

Most sunscreen lotions that protects human skin from dangerous Uv-rays of sunlight whereby inorganic NPs (Titania and Zinc Oxide) as photo catalyst perform better than organic photo catalyst.

ZnO has been widely used in various filed and serves applications such as in catalysis¹³⁰ Gratzel type solar cells¹³¹ and short wavelength light emitting devices^{132, 133}.

SCOPE OF FUTURE RESEARCH

Nano ZnO in future widely used in auto industry, due to its low cost ,reduces engine oil friction nature,in headlights of more efficiency, less power usage & longer life. Furthermore also extensively used in textiles industry & in various cosmetics or sunscreen lotions that protects human skin from dangerous Ultraviolet – rays.

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