STRUCTURAL AND MORPHOLOGICAL PROPERTIES OF ZINC OXIDE NANOPARTICLES SYNTHESIZED BY SOL-GEL METHOD

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Abstract

Zinc oxide (ZnO) nanoparticles were generated by simple approach of sol-gel method with zinc acetate as precursor and methanol used as solvent. The structure and morphology of as-prepared ZnO nanoparticles were characterized by X-ray diffraction (XRD) and transmission electron microscopy (TEM). XRD results indicated that the product was highly pure well-crystallized hexagonal phase with space group P63mc of zinc oxide particles. TEM images showed that the product powder consisted of dispersive quasi-spherical particles with an average size around 50 nm.

Keywords: X-Ray Diffraction, Transmission Electron Microscopy, Hexagonal Phase, Space Group, Quasi-Spherical Particles.

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Introduction

Nanostructured materials exhibit more attractive properties compared with the bulk materials, such as very small particle size, large exposed surface areas and high surface energy. These properties can reduce the diffusion distance of Li ions in solid state, enlarge the contact area between the active particles, enhance the electrochemical reaction rate, as well as results in the agglomeration of nanosized particles (Bhat et al. 2010).

Among various nano-oxide particles, zinc oxide nanoparticles have a wide band gap semiconductor of 3.3eV with large exciton binding energy of 70 meV (Asmar et al. 2006; Ding et al. 2005; Alpaslan et al. 2010). This nano-oxide has broad range of applications in the manufacturing of magnetic materials, alkaline battery anodes, dye-sensitized solar cells, semiconductors, solid oxide fuel cells (SOFC), anti-ferromagnetic layers, p-type transparent conducting films, electrochromic films, heterogeneous catalytic materials and gas sensors. ZnO has superior advantage such as facile preparation, morphologic diversity and high chemical stability, prolonged cycle life, safe operation, and high specific energy. Therefore, research towards finding new materials for various applications has been accelerated. This research focuses on structural and morphological properties of zinc oxide nanoparticles which offers promising candidature for many applications such as solar thermal absorber, catalyst for O₂ evolution, photo electrolysis and electrochromic device. Most attracting features of ZnO are: (1) excellent durability and electrochemical stability, (2) low material cost, (3) promising ion storage material in terms of cyclic stability, (4) large span optical density, and (5) possibility of manufacturing by variety of techniques (Kamat and Williams 2009).

Over decades, rapid increase in number of research has been marked. The great attention towards ZnO due to its unique physical and chemical properties especially wide band gap energy which offers electronic transition to occur down to visible region. Also, the binding energy persist the event of excitonic absorption and recombination between electrons and holes even at room temperature. This process is enhanced with the nature of direct type band structure that improved the efficiency of photo generated electron transfer (Rao et al.2003).

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There are several methods that have been put forward for synthesis of zinc oxide nanoparticles such as combustion method, microwave irradiation, hydrothermal/solvothermal method, sol–gel process, chemical spray pyrolysis, sonochemical method, and so on (Badraghi et al. 2009; Chang and Wei 2008). Sol-gel method offers a good technique and control for tailoring the structures, the compositions and the morphological features of nanomaterial's.

Materials and methods

Zinc acetate (99%), Methanol and acetone were purchased from Merck. Other supplement chemicals were of AR grade. All solutions were prepared with deionized water.

Synthesis of ZnO nanoparticles

Flow chart for synthesis of ZnO by using sol gel method is shown in figure 1.t Zinc oxide sol was prepared by mixing 0.2M zinc acetate dehydrate [Zn (CH₃COO)₂. 2H₂O] with 50 ml of methanol at room temperature. The solution was stirred for 1 hour until solution has transformed into milky white slurry. The resulted white slurry was stirred for another 1 hour to allow a homogeneous mixing. After that, the sample was subjected to gelation. Filtration process was the carried out to obtain the white precipitate and further dried in an oven at 100°C for 24 hours. The dried samples were ground with mortar and pestle to yield ZnO powder. Finally, the powder was calcined at 600°C in normal air to produce a well crystallize ZnO nanoparticles (Bitenc et al. 2008).



Fig.1: Flow chart for synthesis of ZnO by using sol gel method

Results and Discussion

X-ray diffraction (XRD) pattern was obtained using Philips PW1710 automatic X-ray diffractometer with Cu-K α radiation (λ =1.5404A°) and scanning speed of 10°min⁻¹. Transmission electron microscopy image was obtained using Jeol/JEM-2100 TEM with resolution 2.3Å.

X-Ray Diffraction

Fig. 2 shows the XRD pattern of the zinc oxide nanoparticles. XRD pattern of zinc oxide shows diffraction peaks situated at 31.78°, 34.45°, 36.28°, 47.57°, 56.63°, 62.90°, 66.41°, 67.98°, 77.13° corresponds to (hkl) values of (100), (002), (101), (102), (110), (103), (200), (201) and (202) planes with hexagonal phase space group P6₃mc matches well with JCPDS data (36-1451)(JCPDS 1977). The average crystallite size of the ZnO was found to be 25nm which is calculated from FWHM of more intense peak associated with (101) plane located at 36.28° with the help of Debye Scherrer formula. XRD peaks shows line broadening which indicates that synthesized material consist of nanoscale range particles (Asadabad et al. 2011).



Fig. 2: XRD Spectra of ZnO

Transmission Electron Microscopy:

TEM image of ZnO nanoparticles are shown in Fig.3. TEM study was carried out to understand size and morphology of ZnO nanoparticles(Batdembere et al. 2022; Brzezinska et al. 2018). TEM images of ZnO nanoparticles confirmed that they are having spherical morphology with slight variation in its thickness (Geetha et al. 2016). The average particle size was found to be 25nm. The particle size determined from TEM analysis is close to that of the <u>XRD</u> analysis (Chin et al. 2018).



Fig. 3: TEM image of ZnO nanoparticles

Conclusion

ZnO nanoparticles were successfully synthesized by Sol-gel method. Hexagonal phase space group of synthesized ZnO nanoparticle was confirmed by XRD analysis. TEM analysis revealed that ZnO nanoparticles were in nanoscale range with spherical morphology. This study offers ZnO nanoparticles as a demanding material with many applications like catalyst for O_2 evolution, supercapacitor, gas sensor, photo electrolysis and electrochromic device.

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