Production of Nano Zinc Oxide (ZnO) by Hydrothermal Method

Duygu Yeşiltepe, Sebahattin Gürmen
Istanbul Technical University - Türkiye

Abstract

Zinc oxide (ZnO) plays an important role in current industry due to its special characteristics such as antibacteria, anti-corrosion, has low electrons conductivity and great heat resistance. In this study, ZnO particles have been produced by hydrothermal method using zinc nitrate (Zn(NO₃)₂), ammonia (NH₃) and distilled water have been used as medium. Before the heat treatment the physical properties of ZnO particles have been characterized by X-ray diffraction (XRD) and differential thermal analysis (DTA). After the calcination process at 550°C particles have been analyzed by XRD and scanning electron microscope (SEM). SEM investigation has given detailed information about particle size and morphology. ZnO nanoparticles have been successfully synthesized by hydrothermal method.

1. Introduction

In recent years, the number of applications of ZnO nanostructures has risen dramatically and this can be expected to be just the beginning [1]. ZnO is one of the most promising materials in the materials research and device fabrication technologies with a wide range of potential applications from sensors, gas sensors–biosensors, to optoelectronic devices such as UV lasers, field emitters, varistors and solar cells due to its wide band gap (3.37 eV) and large exciton binding energy (60 meV). ZnO nanostructures display unique properties including high surface to volume ratio, nontoxicity, biocompatibility, chemical and photochemical stability, optical transparency, electrochemical activity, and high electron communication features [2]. Zinc oxide particles were extensively used in fields as diverse as textile, antibacterial activity or organic coatings, due to the nontoxic nature of ZnO and the ability to block the UV radiation. In the tin can industry, ZnO has been used as a pigment in lacquers for preventing the sulphide staining [3].

Zinc oxide nanoparticles can be prepared by different methods such as hydrothermal, chemical vapor deposition, sol-gel, thermal decomposition, Pechini, electrochemical precipitation, chemical precipitation and spray pyrolysis [4,5,6]. From these methods, the hydrothermal method is more popular because of its cheapness, reliability, repeatability, and simplicity. In addition, the nanoparticles which are produced by this route, show good optical properties. But, this can be achieved only by good control of the size and morphology of the particles. That is, we have to carefully control the hydrothermal process and the growth of nuclei must be prevented [4].

In this study, ZnO particles were produced by hydrothermal method. The effects of the processing parameters on their size and crystalline structure were characterized by X-ray diffraction (XRD), energy dispersive spectrometry (EDS) and scanning electron microscope (SEM).

2. Experimental

2.1. Materials

The reagent (Zn(NO₃)₂·6H₂O) was in analytical grade and used without further purification. A water leach solution of (Zn(NO₃)₂·6H₂O) (powders were dissolved in deionized water) was used as starting material for this research. The concentration of the precursor solution is 0.1M. Solution was stirred by using a magnetic stirrer.

2.2. Experimental Procedure

ZnO nanostructure was synthesized by using hydrothermal method. In order to prepare a solution 5.95 g of Zinc nitrate was weighted using a weighting balance. Then, 200 ml of distilled water was measured by a measuring cylinder. After that, 5.95 g zinc nitrate was dissolved with a 200 ml of distilled water. The solution was stirred with a constant stirring for about five minutes. After well mixed, 5 ml ammonia (NH₃) solution was dropwised to the solution containing zinc nitrate with a constant stirring by magnetic stirrer. After the reaction, white precipitate was formed.
X-ray diffraction (XRD, RIGAKU) using Cu Kα radiation was used to examine the crystalline phase of the prepared particles. For XRD analysis, the ZnO dispersed particles were placed on a glass substrate and allowed to dry in air at room temperature. Particles were characterized by differential thermal analysis (DTA). After that, ZnO dispersed particles were done calcination process at 550°C and then particles were analyzed by XRD. Then, particles were analyzed by SEM for given detailed information about particle size and morphology. The chemical compositions of particles were analyzed by the energy dispersive spectroscopy (EDS) instrument.

3. Result and Discussion

3.1. X-ray Analysis of ZnO Nanopowder

XRD analysis determined the phase’s presence in nanopowder [7]. XRD result as shows in Fig. 1 are the resulting pattern of ZnO nanopowder of before calcination process in various profiles of peak and diffraction angle 2θ, which represent the diffraction of ZnO nano-powder using Zinc nitrate as precursor. According to the XRD patterns, the diffraction peaks of the samples were identified as Zn(OH)₂ (JCPDS Card no: 48-1066).

![Figure 1. XRD analysis of the hydrothermal ZnO nanoparticles of before calcination process.](image1)

Figure 1. XRD analysis of the hydrothermal ZnO nanoparticles of before calcination process.

XRD result as shows in Fig. 2 are the resulting pattern of ZnO nanopowder of after calcination process at 550°C. According to the XRD patterns, the diffraction peaks of the samples were identified as hexagonal wurtzite structure of ZnO particles (JCPDS Card no: 36-1451).

![Figure 2. XRD analysis of the hydrothermal ZnO nanoparticles of after calcination process at 550°C.](image2)

Figure 2. XRD analysis of the hydrothermal ZnO nanoparticles of after calcination process at 550°C.

3.2. Differential Thermal Analysis (DTA) of ZnO Nanopowder

Differential thermal analysis is a technique by which one can measures the mass loss with respect to the temperature [8]. Fig. 3 shows the weight loss of the powder sample from room temperature to 1000°C, for as-synthesized powder. The primary weight loss was observed at 100°C due to the water.

![Figure 3. DTA analysis of the hydrothermal ZnO nanoparticles of before calcination process.](image3)

Figure 3. DTA analysis of the hydrothermal ZnO nanoparticles of before calcination process.
3.3. Scanning Electron Microscope (SEM) of ZnO Nanopowder

SEM images of nanopowder ZnO particles produced at 550°C is shown in Fig. 4. There are varieties of ZnO nanostructures that had been discovered, which are in a form of nanorods, nanospheres and flower shape structures.

Figure 4. SEM analysis of the hydrothermal ZnO nanoparticles of after calcination process at 550 °C.

4. Conclusion

ZnO particles were successfully fabricated by hydrothermal method using zinc nitrate (Zn(NO₃)₂), ammonia (NH₃) and distilled water. Then, the crystal structures of the ZnO were investigated using XRD. X-ray diffraction pattern of ZnO before and after calcinations using simple solution phase method. The results indicated that ZnO particles are the wurtzite phase. The synthesized ZnO nano-powder obtained exhibit good crystallinity. ZnO could be one of the most important nanomaterials in future research and applications.

References


