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SYNTHESIS AND CHARACTERIZATION OF ZnO NANOPARTICLES AT DIFFERENT CALCINATION TEMPERATURES BY SOL-GEL METHOD

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Abstract

In this work, zinc oxide nanoparticles were synthesized by using sol-gel method at calcination temperatures of 400˚C and 500˚C. Sol-gel method is the simplest method and has the ability to control the particle size and morphology through systematic monitoring of reaction parameters. Zinc oxide nanoparticles were synthesized via sol-gel method using zinc acetate dihydrate [$Zn(CH_3COO)_2.2H_2O$] as a precursor and methanol as a reagent. Distilled water was used as solvent medium and ammonia was used to adjust the pH value. And then the obtained samples were characterized by X-ray Diffraction (XRD) to confirm the formation of ZnO phase, which showed that it had this type of hexagonal wurtzite structure. In the comparison of the crystalized sizes of the samples, the sample calcinated at 500˚C have smaller crystalized size than the sample calcinated temperatures at 400˚C. The Scherrer's equation was used to calculate the crystalized size of the ZnO powder. And then, scanning electron microscopy (SEM) was used to observe the surface morphology of obtained ZnO nanoparticles.

Keywords: Sol-gel method, Zinc acetate dehydrate, ZnO nanoparticles, XRD, SEM

Introduction

Nanotechnology represents one of new sciences that promise to provide a broad range of novel uses and improved technologies for numerous applications. One important reason behind the intense interest is that nanotechnology permits the controlled preparation of nanomaterials where at least one dimension of the structure is less than 100 nm. Schematic illustration of structural dimensionality of nanomaterials with expected properties is shown in Figure 1. Synthesis of metal nanoparticles with specific properties is a newly established research area that attracts a great deal of attention. There are several methods for synthesis of metal nanoparticles. Among the metal nanoparticles, Zinc Oxide is the semiconductor material with high chemical stability, strong photo sensitivity and non-toxicity property and is widely used in antibacterial materials . Compared with ordinary Zinc Oxide powder, Zinc Oxide nanoparticles have a large specific surface area and small size effect and show wide applications . Several different methods can be used to control the size distribution of semiconductor and metal nanoparticles to obtain monodisperse samples. Common synthetic methods of ZnO nanostructures are shown in Figure 2. Most of the ZnO crystals have been synthesized by traditional high temperature solid state method which is energy consuming and difficult to control the particle properties. ZnO nanoparticles can be prepared on a large scale at low cost by simple solution-based methods such as chemical precipitation, sol-gel synthesis and solvothermal / hydrothermal reaction. So in the present work, ZnO nanoparticles are synthesized by using sol-gel method. And the obtained ZnO nanoparticles were characterized by X-ray Diffraction (XRD) and scanning electron microscopy (SEM).

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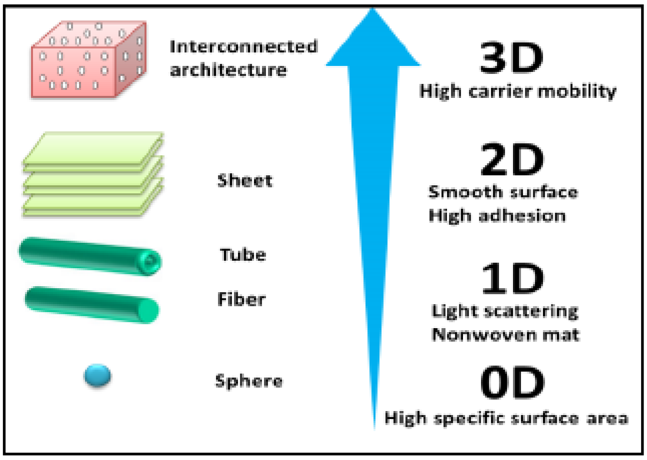
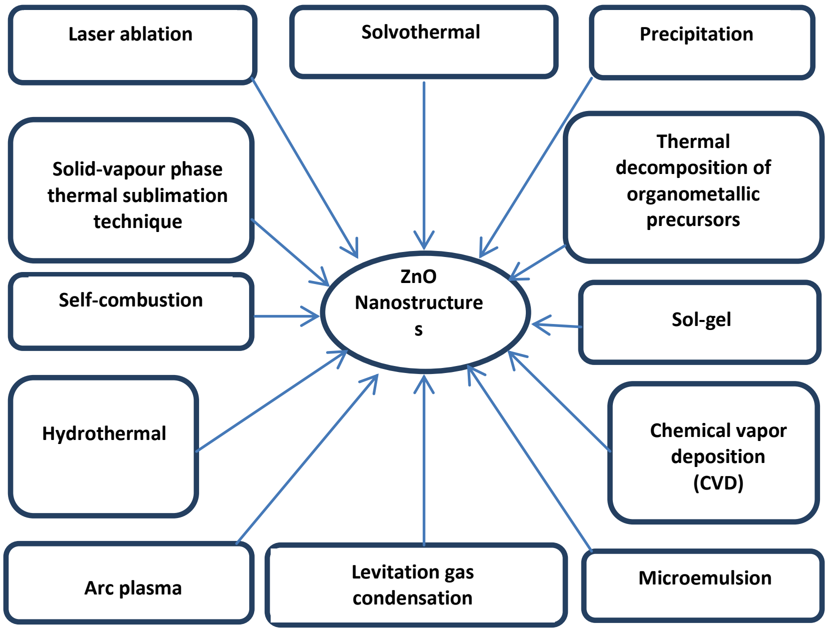


Figure 1 Schematic illustration of structural dimensionality of nanomaterials with expected properties



[Shaheed, M. A, et. al, J. Environ. Anal. Chem, 2015]

Figure 2 Common synthetic methods of ZnO nanostructures

Materials and Methods

(1) Synthesis of ZnO Nanoparticles

Synthesis of zinc oxide by sol-gel is one of the most successive methods for producing large-area and good quality ZnO nanostructures with suitable precursor. The main parameters for ZnO synthesis by sol-gel are precursors, reagents and experimental parameters. Most commonly used ZnO precursors are zinc acetate dihydrate, zinc nitrate hexahydrate and zinc acetylacetonate hydrate. Synthesis of ZnO using sol-gel consists of three processes: stirring, heating and annealing.

Synthesizing ZnO nanoparticles via sol-gel technique includes the use of materials such as zinc acetate dihydrate [$Zn(CH_3COO)_2.2H_2O$], methanol (CH_3OH) and distilled water. Zinc acetate dihydrate was used as precursor and methanol was used as a reagent. Distilled water was used as a solvent medium and ammonia (NH_3) was used to adjust pH value of solution between 9 and 11. The materials used in synthesizing ZnO nanoparticles were shown in Figure 3.

In order to prepare a solution, methanol (1 M) is made into 0.6 M to dilute and 13 g of zinc acetate dihydrate was weighed using a weighing balance. After that, 13 g of zinc acetate dehydrate ($Zn(CH_3COO)_2.2H_2O$) was dissolved with a 100 ml of methanol (0.6 M) under continuous stirring for 15 min. The mixed solution was stirred with a magnetic stirrer at 80˚C for 10 min to form transparent solution; its pH was adjusted by adding dropwise 5 ml of ammonia per 5 min. After 23 ml of ammonia have been added to the solution, pH value was 10. After adjusting the pH, the solution was heated at 100˚C for 30 min under stirring and then the temperature was increased up to 150˚C until gelation occurred. And white precipitate was formed after the solution evaporated. The precipitates were washed four times with methanol. And then the washed precipitates were dried at 190˚C for 15 min and annealed at 500˚C and 400˚C for 3 hrs in furnace. Finally, zinc oxide powder was obtained. Figure 4 shows the flow scheme of preparation of ZnO nanoparticles and Figure 5 shows the procedures of ZnO nanoparticles.

Figure 3 The materials used in synthesizing ZnO nanoparticles

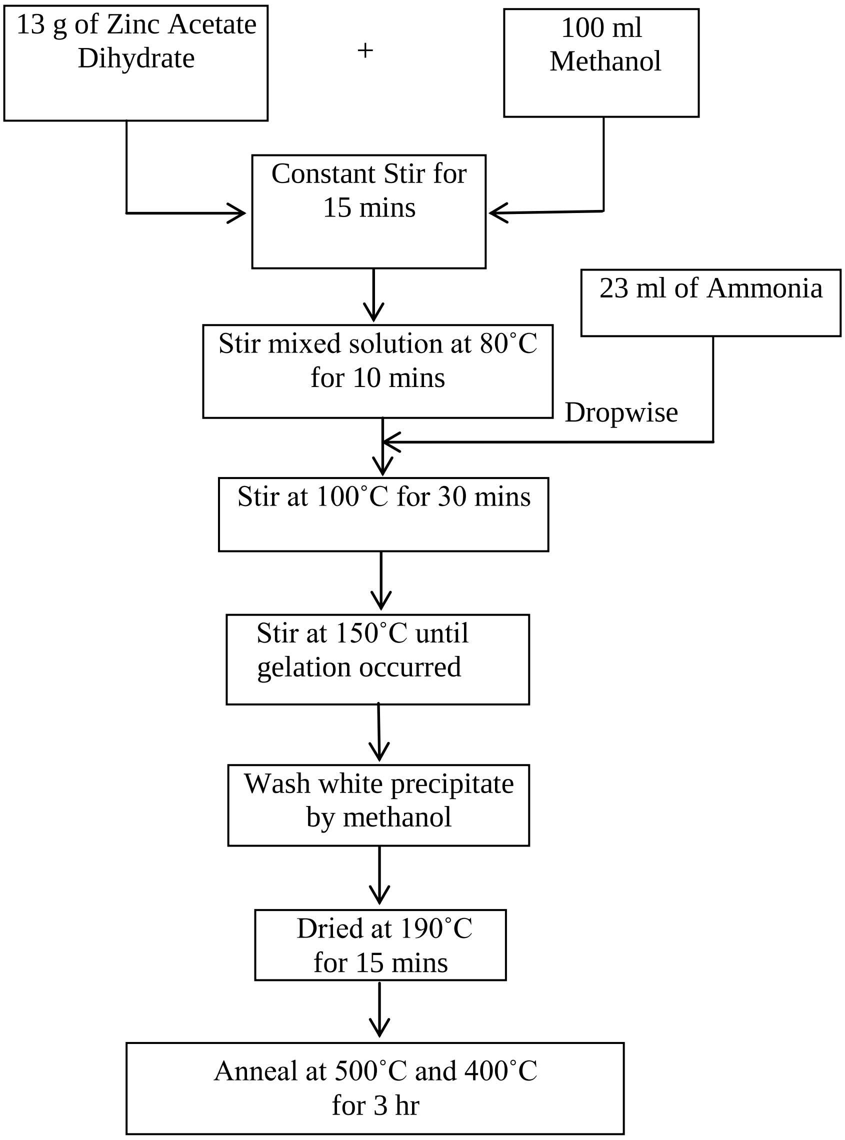


Figure 4 The flow scheme of preparation of ZnO nanoparticles

Figure 5 The procedures of ZnO nanoparticles

(1) X-ray Diffraction (XRD)

The synthesized ZnO powder was characterized by powder X-ray diffraction (XRD: RIGAKU-RINT 2000 X-ray Diffractometer). The X-ray diffraction data were recorded by using CuK_{α} radiation (λ = 0.154056 nm). The average crystallite size of the sample was estimated with the help of Scherrer's equation using the diffraction intensity of the peak.

$$D = \frac{0.94\lambda}{B\cos\theta}$$

where λ is the wavelength, B is the full width at half- maximum (FWHM) and θ is the diffraction angle.

(2) Scanning Electron Microscopy (SEM)

Scanning Electron Microscope (SEM) [JEOL - 6000] was used to observe the surface morphology of obtained ZnO nanoparticles. This SEM is scanning electron microscope with a high resolution. The scanning electron microscope is a type of electron microscope that creates various images by focusing a high energy beam of electrons onto the surface of a sample and detecting signals from the interaction of the incident electrons with the sample's surface. The type of signals gathered in a SEM varies and can include secondary electrons, characteristic X-rays, and back scattered electrons. In a SEM, these signals come not only from the primary beam impinging upon the sample, but also from other interactions within the sample near the surface. The SEM is capable of producing high-resolution images of a sample surface.

Results and Discussion

(1) XRD Analysis

The XRD pattern of the ZnO powder was recorded in the diffraction angle range 10˚ to 70˚. The Bragg's reflections in Figure 6 and 7 which were corresponded to the (100), (002), (101), (102), (110), (103), (200), (112) and (201) planes, respectively, after comparing them with the data in the JCPDS card 65-3411. They have been investigated that the orientation of (101) plane was favorable in hexagonal wurtzite structure for 500 ˚C and 400˚C. The standard lattice parameters 'a' and 'c' were a = 3.2 Å and c = 5.2 Å. But the lattice parameters 'a' and 'c' calculated from XRD were found to be a = 3.2475 Å and c = 5.2045 Å for 500˚C and a = 3.2462 Å and c = 5.2066 Å for 400˚C.So, both standard and experimental lattice parameters are in very good agreement for two samples. The average crystallite size of ZnO nanoparticles for two temperatures were presented in Table 1.

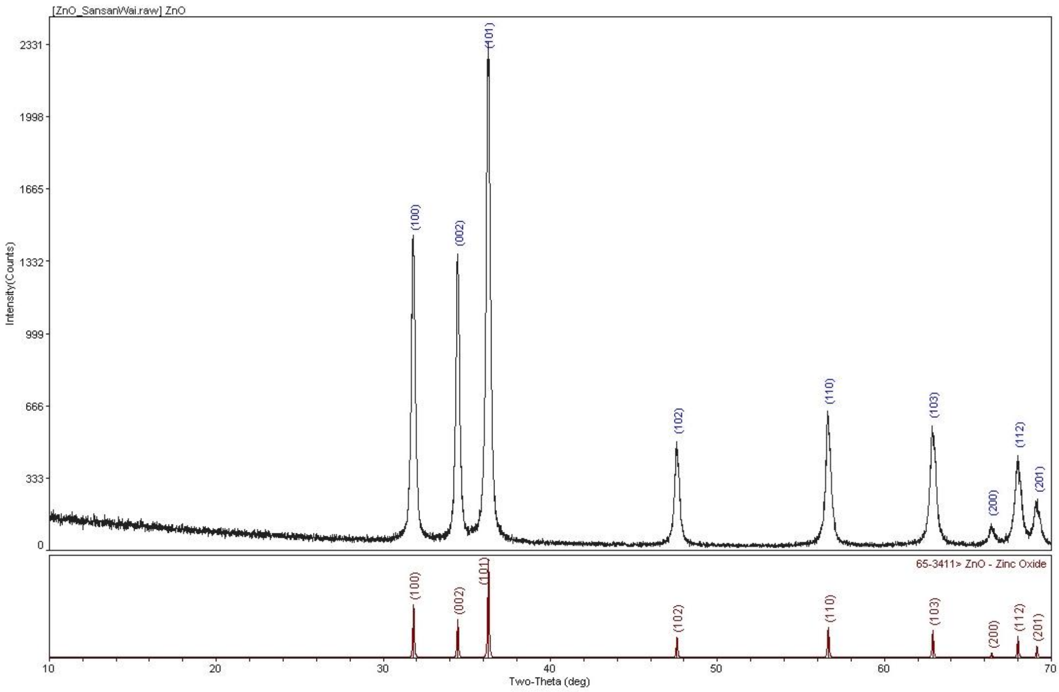


Figure 6 XRD pattern of ZnO nanoparticles powder at calcination temperature 500˚C

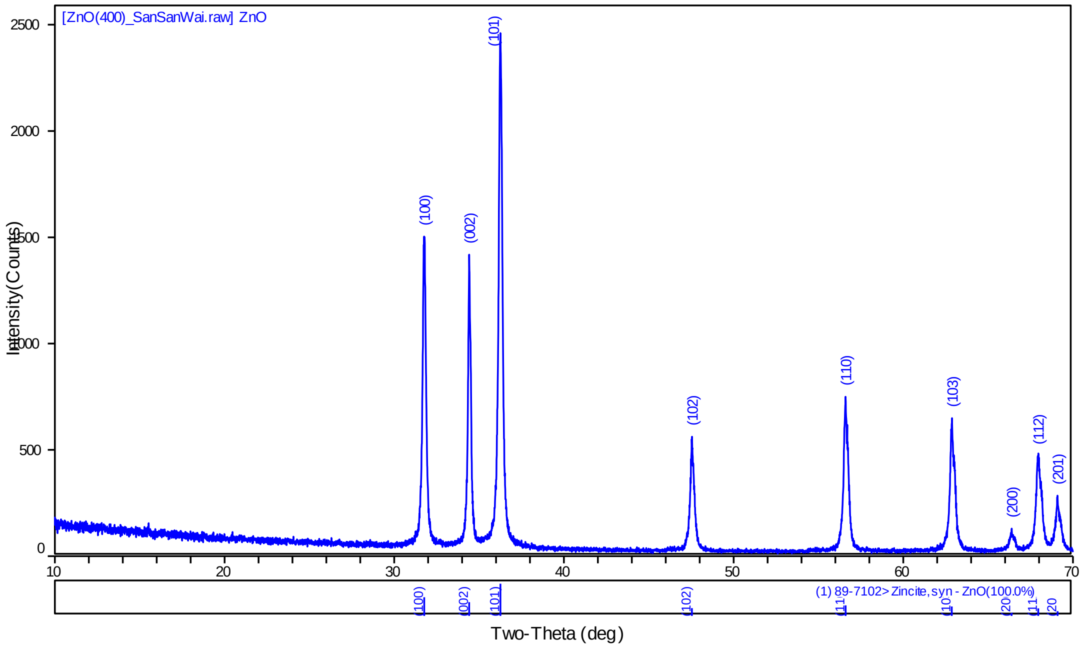


Figure 7 XRD pattern of ZnO nanoparticles powder at calcination temperature 400˚C

Table 1 The average crystallite size of the obtained ZnO powder

Sample	Average crystallite size, D (nm)	lattice parameter 'a'	lattice parameter 'c'
At 500˚C	26.61	3.2475 Å	5.2045 Å
At 400˚C	35.07	3.2462 Å	5.2066 Å

(2) SEM Investigation

SEM images also confirmed the formation of ZnO nanoparticles. The SEM micrographs of the synthesized ZnO powder were shown in Figure 8 and Figure 9. In Figure 8, the average grain size of ZnO nanoparticles at 500°C was 261 nm. It was found that the minimum grain size of ZnO nanoparticles was 117 nm and the maximum grain size of ZnO nanoparticles was 444 nm. In Figure 9, the average grain size of ZnO nanoparticles at 400°C was 31.533 µm. It was found that the minimum grain size of ZnO nanoparticles was 12.649 µm and the maximum grain size of ZnO nanoparticles was 124.724 µm.

The images showed most of the particles were spherical in shape with smooth surface, however some agglomerated particles were also presented. Agglomeration was understood to increase linearly with annealing temperature and hence some degree of agglomeration at this temperatures (500°C and 400°C) appears unavoidable.

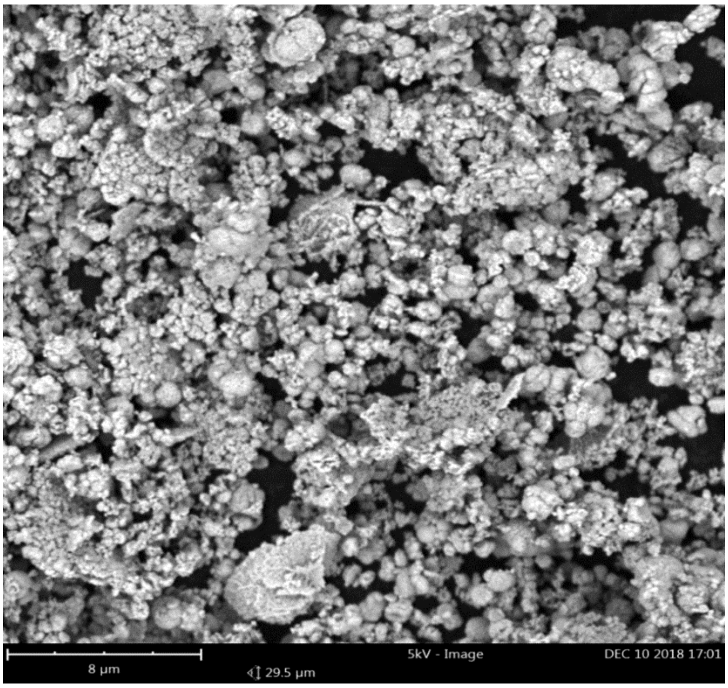


Figure 8 SEM micrograph of the synthesized ZnO powder at 500˚C

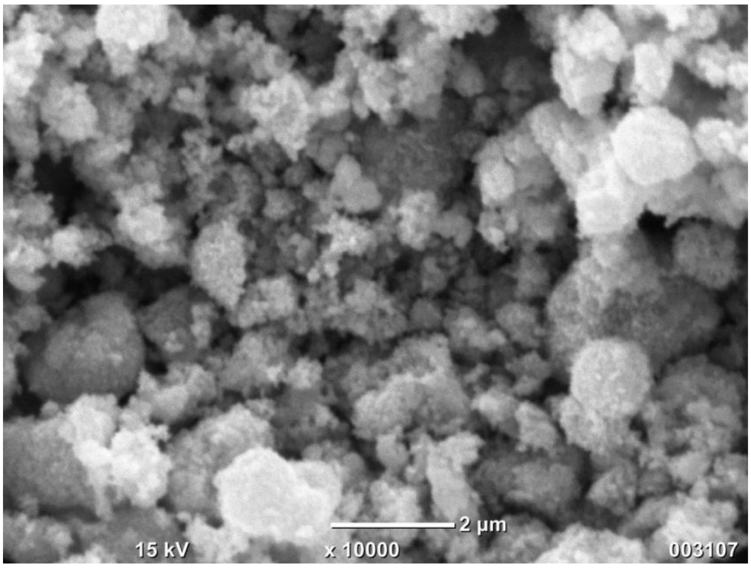


Figure 9 SEM micrograph of the synthesized ZnO powder at 400˚C

Conclusion

ZnO nanoparticles were successfully synthesized by sol-gel method using the materials such as zinc acetate dihydrate [$Zn(CH_3COO)_2.2H_2O$], methanol (CH_3OH), ammonia and distilled water. The obtained samples were characterized by using XRD for phase analysis. From the XRD profiles for the samples, it confirmed the presence of hexagonal wurtzite structure. The average lattice parameter 'a' and 'c' of ZnO nanoparticles were found to be 3.2475 Å and 5.2045 Å. So, both standard and experimental lattice parameters are in very good agreement. The average crystallite size of the sample was estimated with the help of Scherrer's equation using the diffraction intensity of the peak. It was found that the average crystallite size of ZnO nanoparticles was 26.61 nm for 500˚C and 35.07nm for 400˚C. The temperature increases, the crystallite size decreases. The morphological properties of the obtained samples were also characterized by using SEM. According to the SEM micrographs, the average grain size were 261 nm for 500˚C and 31.533 µm for 400˚C. It was found that ZnO nanoparticles were spherical in shape with smooth surface. Most of the nanoparticles were well-dispersed and some aggregates exhibited nanoscale features.

Future Plan

In the area of water purification, nanotechnology offers the possibility of an efficient removal of pollutants and germs. Today nanoparticles, nanomembrane and nanopowder used for detection and removal of chemical and biological substances. Nanomaterials reveal good result than other techniques used in water treatment because of its high surface area (surface/volume ratio). So, the antibacterial behavior of ZnO nanomaterials will be studied. They can be developed as antibacterial agents to improve the water quality. Then, ZnO nanomaterials will be applied in water and waste water treatment processes.

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