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Zinc oxide nanoparticles via an aqueous acetate-succinate gelation method

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Abstract

The synthesis of ZnO nanoparticles via an aqueous acetate-succinate gelation method using the solution of zinc acetate dihydrate and succinic acid as a precursor. Samples of the precursor form the preparation were calcined at 400, 500, 600 and 700°C for 2 h and calcined at 400°C for 4, 6 and 8 h to complete. The structure was characterized by x-ray diffraction (XRD). XRD patterns showed that ZnO nanoparticles were formed to a crystallites of wurtzite at 400°C. From JCPDS file no. 36-1451, it can be noticed that the samples were highly crystalline, and all peaks can be confirmed to be a hexagonal structure that is more interesting for application. The analysis of different functional groups present in precursor and zinc oxide were carried out by fourier transform infrared spectroscopy (FT-IR) in the range 200-4000 cm⁻¹. The mean particle size of zinc oxide nanoparticles were determined by scanning electron microscopy (SEM). SEM image of ZnO nanoparticles exhibit a spherical morphology with a narrow and uniform size distribution.

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Keywords: ZnO nanoparticles; aqueous acetate-succinate gelation method

1. Introduction

ZnO semiconductor nanoparticle is very important in the ongoing research activity across the world. Because of its high chemical stability, low dielectric constant, large electromechanical coupling coefficient and high luminous transmittance. ZnO materials have been widely used as dielectric ceramic, pigment, catalyst and sensing material [1-3], solar cells [4-5], varistors [6]. Chemical methods give the ability to produce powders with an exceptionally small size in the nanometer range [7], Many authors already reported the production of fine powders via different synthesis routes: precipitation routes [8], micro-emulsion [9], a combustion synthesis route [10], an adapted Pechini process, etc [11,12]. In this presentation we report the production of nanosized ZnO using an aqueous carboxylate gelation method. This method already turned out to be successful in the syntheses

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of superconductors [13], and other multimetallic ceramics [14,15]. The advantages of the aqueous method used are its simplicity and the fact that dopants can easily be introduced as salts. The process reported here is potentially cheaper, simple and useful.

2. Materials and Methods

2.1 Synthesis of ZnO nanoparticles

The following reagents were used: zinc acetate dehydrate ($C_4H_6O_4Zn \cdot 2H_2O$), succinic acid ($C_4H_6O_4$) and ammonia solution, 25-30% analytical grade without further purification.

A flow chart of the synthesis procedure is shown in Fig. 1.

The synthetic procedure was as follows: 0.5 M zinc acetate dihydrate and 0.5 M succinic acid were dissolved to 100 mL in deionized water. Then succinate solution was added and stirring the mixing solution in order to coordinate the metal ions present. The ratio of the acid to the metal concentration was 1:1. The pH of the solution was increased up to 8.5 by addition of a concentrated ammonia solution. Finally, the resulting precipitates were filtered and dried after several washings at 60 °C for 12 hr, a solid precursor gel results.

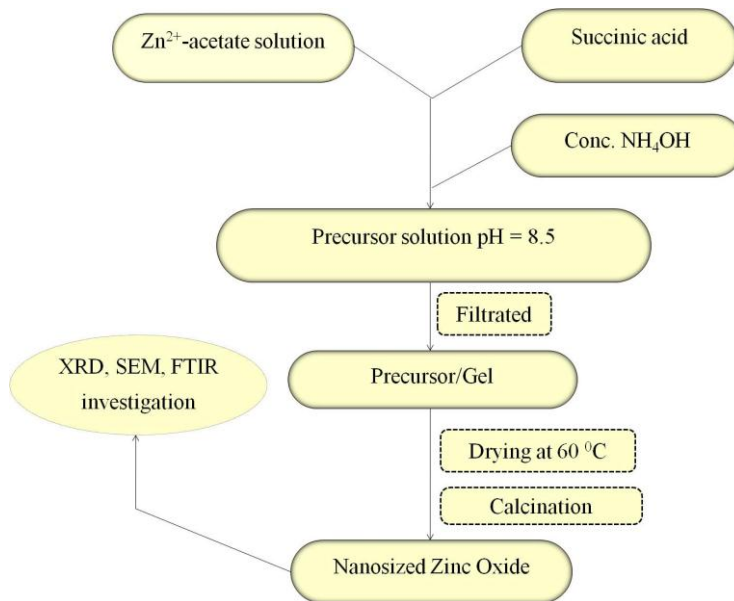


Fig. 1. Schematic overview of the synthesis procedure

2.2 Characterization

The powder X-ray diffraction (XRD) analyses were performed for phase identification of succinate gelation products in as-prepared (dried at 60 °C for 12 hr) and after calcination treatments. A Rigaku D/Max-2200PC diffractometer was employed in XRD analyses, using Cu-K α radiation ($\lambda = 1.54 \text{ \AA}$) and X-ray source operating voltage of 40 kV in the 2θ range of 10–80° at a rate of 2°/min.

The morphology and size of the ZnO nanoparticles were examined using a Hitachi SU-1500 model SEM. Samples were formed using a suspension of ZnO nanoparticles dispersed in isopropanol by ultrasonic treatment. A few drops of dilute dispersion were deposited onto a carbon film taped to an aluminum grid and dried in air.

Fourier transform infrared spectroscopic (FT-IR) studies are carried out in a Perkin Elmer Spectrum 100 FT-IR Spectrometer, using pellets prepared with KBr.

3. Results and Discussion

3.1 XRD Analysis

X-ray powder diffraction patterns of ZnO nanoparticles are illustrated in Fig. 2. All peaks can be well indexed to the zincite phase of ZnO (JCPDS No. 36-1451). No. peaks from any else phase of ZnO and impurities were observed, which indicates the high purity of the obtained zincite ZnO nanoparticles. The diffraction peaks were sharper with the increase of the calcination temperature, which implies that the crystalline structure tends to more integrity and the average particle size increases with the increase of the calcination temperature. The decomposition of intermediate succinate phase gave highly monodispersed ZnO nanoparticles, exhibiting pure hexagonal crystal phase.

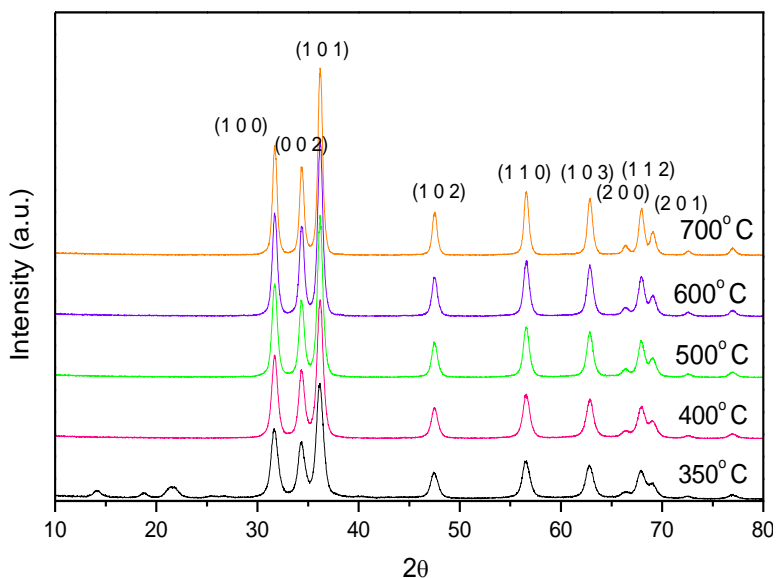


Fig. 2. XRD patterns of ZnO nanoparticles calcined at 350 °C, 400 °C, 500 °C, 600 °C and 700 °C for 2 hr

3.2 FT-IR Analysis

Fig. 3. shows the FT-IR spectra of the precursor dried at 60 °C for 12 hr and ZnO nanoparticles were calcined at 350 °C, 400 °C, 500 °C, 600 °C and 700 °C for 2 hr. From the curve of precursor, the peak at 3376 cm^{-1} is the stretching vibration of O–H in crystal water. Symmetric and asymmetric stretching of CH_2 are located around 2930 cm^{-1} . The most important bands in the present work are those stretching bands of the COO^- functional groups at ca. 1530 cm^{-1} ($\nu_{\text{as}} \text{COO}^-$) and 1389 cm^{-1} ($\nu_{\text{s}} \text{COO}^-$). FT-IR spectra of ZnO nanoparticles exhibits a rather broad and moderately strong band at 520 cm^{-1} , owing to Zn–O vibrational mode when increase of the calcinations temperature. The band at 3457 cm^{-1} corresponds to O–H mode of adsorbed moisture. There was slightly evidence of free precursor in the sample after decomposition of the sample at 400–600 °C for 2 hr, because stretch vibration of C=O and C–O disappeared.

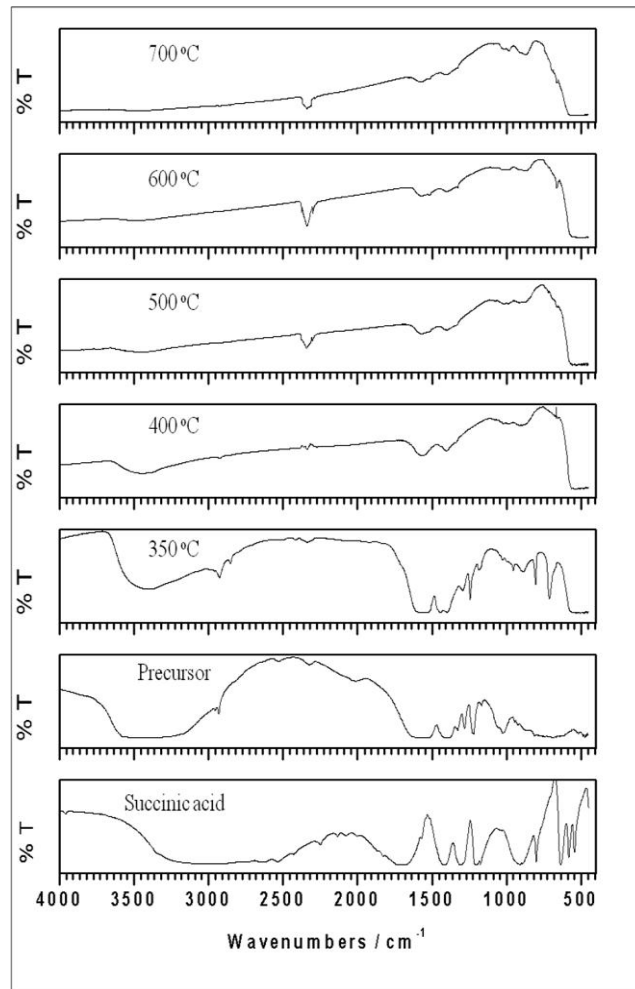


Fig. 3. FT-IR spectra of the precursor and ZnO nanoparticles calcined at 350 °C, 400 °C, 500 °C, 600 °C and 700 °C

3.3 Particle morphology by SEM

Fig. 4. shows SEM micrographs of ZnO particles after calcinations for 2 hr at 350, 400, 500, 600 and 700 °C. ZnO nanoparticle exhibit a spherical morphology with a narrow and uniform size distribution. The average particle size varies in the range of 60–100 nm and increases with calcination temperature within the temperature range of the study.

EDS spectra of ZnO nanoparticles after calcination for 2 hr at 350, 400, 500, 600 and 700 °C were analyzed. The signal of EDS spectrum corresponded to Zn and O element were clearly confirm the ZnO nanoparticles. The element compositions are shown in Table 1. However, the amount of element composition as slightly different depending on the different selected area of EDS analysis.

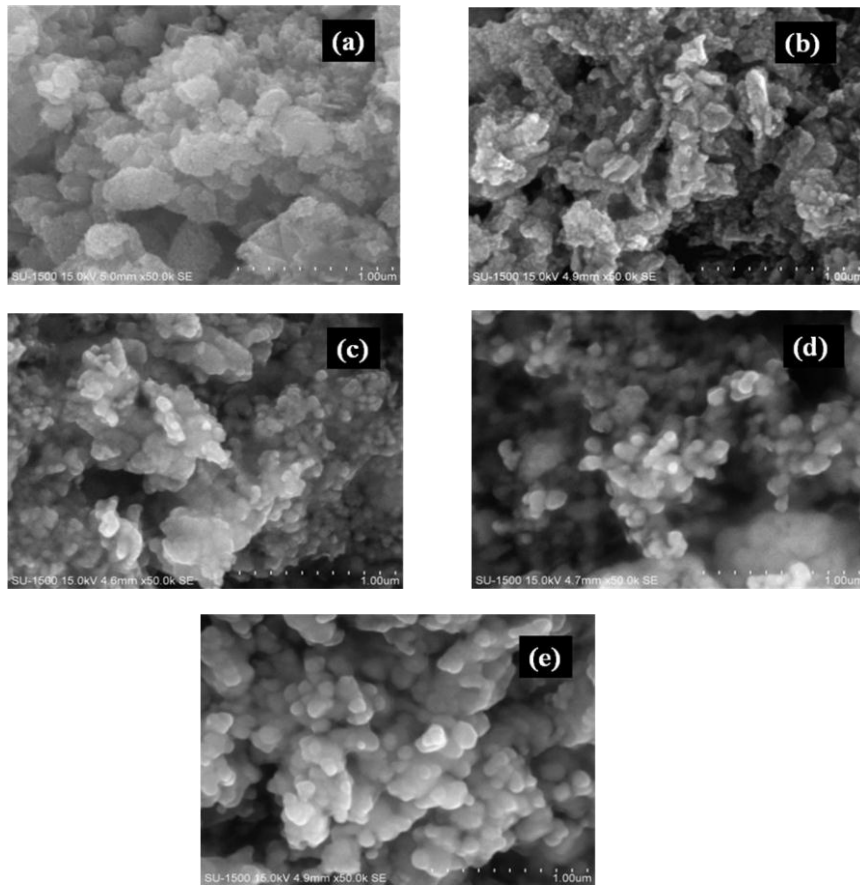


Fig. 4. SEM images of ZnO nanoparticles calcined at (a) 350°C (b) 400°C (c) 500°C (d) 600°C (e) 700°C

Table 1. The element composition of ZnO nanoparticles after calcinations for 2 hr at 350, 400, 500, 600 and 700 °C

Temp. (°C)	% atomic of Zn	% atomic of O
350	53.27	22.59
400	56.08	19.29
500	61.04	20.05
600	58.65	19.33
700	57.74	16.76

4. Conclusions

ZnO nanoparticles were successfully synthesized via an aqueous acetate-succinate gelation method. The synthesized product were characterized by X-ray diffraction method. The XRD patterns showed that the hexagonal structure of ZnO. JCPDS file No. 36-1451, which confirms the obtained ZnO posses wurtzite structure that more interested for application. The mophology were analyzed and proved by SEM. ZnO nanoparticle exhibit a spherical morphology with a narrow and uniform size distribution. The average particle size varies in the range of 60-100 nm.

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