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RESEARCH ARTICLE

Morphology and structure study of polygon ZnO nanorods: Biomedical applications

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Abstract: In this study, zinc oxide (ZnO) nanoparticles (NPs) were first synthesized using co-precipitation method in the presence of $Zn(NO_3)_2.6H_2O$ precursor and calcined at different temperature of 450°C and 1000°C. Samples were then characterized by x-ray diffraction (XRD), transmission electron microscopy (TEM), energy dispersive spectroscopy (EDS) and scanning electron microscopy (SEM). The XRD study revealed the hexagonal wurtzite structure for annealed samples. SEM images showed tthat he morphology of the ZnO NPs changed from sphere-like shape to polygon shape by increasing temperature. The exact size of NPs were measured by TEM analysis about 40 nm for as-prepared samples. The EDS analysis demonstrated an increasing level of Zn element from 28.5 wt% to 50.8 wt% for as-synthesized and annealed samples, respectively.

Keywords: ZnO nanorods, polygon-shaped nanoparicles, Coprecipitation synthesis, EDX

1 Introduction

In recent years, research on nanostructured has been increasingly improved to study their physicochemical properties [1–18]. Recent advanced research in nanotechnology have increased the ability to make a variety of nanoparticles, including metal NPs and metal oxides NPs [19–28]. Among metal oxide semiconductors [29–39], zinc oxide NPs has attracted a great deal of attention due to its unique properties such as direct wide band gap energy (3.37 eV) and high exciton energy (60 meV) [40–45], which makes it suitable for applications such as piezoelectric materials, solar cells, and sensors. Among zinc oxide nanostructures, zinc oxide nanorods have many applications such as catalytic activity, wastewater treatment, sensors, antibacterial and antifungal agents, cancer treatment and cosmetics due to UV light absorption compared to other metal oxides due to their very good electronic and photocatalytic properties. To synthesize zinc oxide nanoparticles, various methods have been used. Chemical methods such as hydrothermal [46], solvothermal [47] and sonochemical [48] methods have received much attention due to their simplicity, cheapness and high efficiency. These methods are not very popular due to high-temperature synthesis. In addition, in order to improve their shape and crystal phase use of chemicals such as surfactants, coating and stabilizing agents for these methods are very expensive [49, 50]. These chemicals often adhere to the surface of NPs and cause toxicity that is not environmentally friendly [51, 52]. Co-precipitation synthesis is very economic and simple synthesis to control the size and morphology of the NPs. The aim of this study is to synthesize zinc oxide NPs and investigate the structure and morphology of the them by XRD, SEM, TEM and EDS analyses at room temperature.

2 Experimental detail

ZnO NPs were successfully fabricated by coprecipitation method. All chemicals were purchased with high level of purity (99%) from Merck company. Firstly, 10 g zinc nitrate was dissolved in 100 mL deionized water under magnetic stiller. After 10 min, 10 mL ethylene glycol (EG) stabilizer was slowly added to the solution and the temperature increased to 80°C. After evaporation (2 hours), samples were heated at 450°C and 1000°C for 4 hours and they were characterized in order to study their physicochemical properties. The XRD analysis was recorded with 2θ in the range of 4-85° with type X-Pert Pro MPD, Cu-K_α: $\lambda = 1.54$ Å.

The morphology and elemental study were performed by SEM and EDS analysis with type KYKY-EM 3200, 25 kV. The exact size of the NPs were measured by TEM analysis with type Zeiss EM-900, 80 kV.

3 Result and discussion

To study the crystal structure of the samples, XRD analysis was performed. Figure 1(a) shows the XRD analysis of as-synthesized ZnO NPs and Figure 1(b) and Figure 1(c) exhibit the XRD spectra of annealed samples at 450°C and 1000°C for 4 hours, respectively. The peaks appeared at 2θ angles in 31.88, 34.56, 36.37, 47.66, 56.69, 62.98, 66.46, 68.05, 69.15, 72.70, and 77.05 degrees correlate with crystal planes of (100), (002), (101), (102), (110), (103), (200), (112), (201), (004) and (202) respectively, indicating the single crystal hexagonal wurtzite ZnO structure. The mean size of ZnO NPs has been measured from full width at half maximum (FWHM) and Debye-Sherrer formula [53]. The crystallite size of as-synthesized and annealed ZnO NPs at 450°C are calculated about 40 nm and 65 nm, respectively. The surprising result is that the intensity of crystal plane of (002) increase by increasing temperature from 450°C and 1000°C.

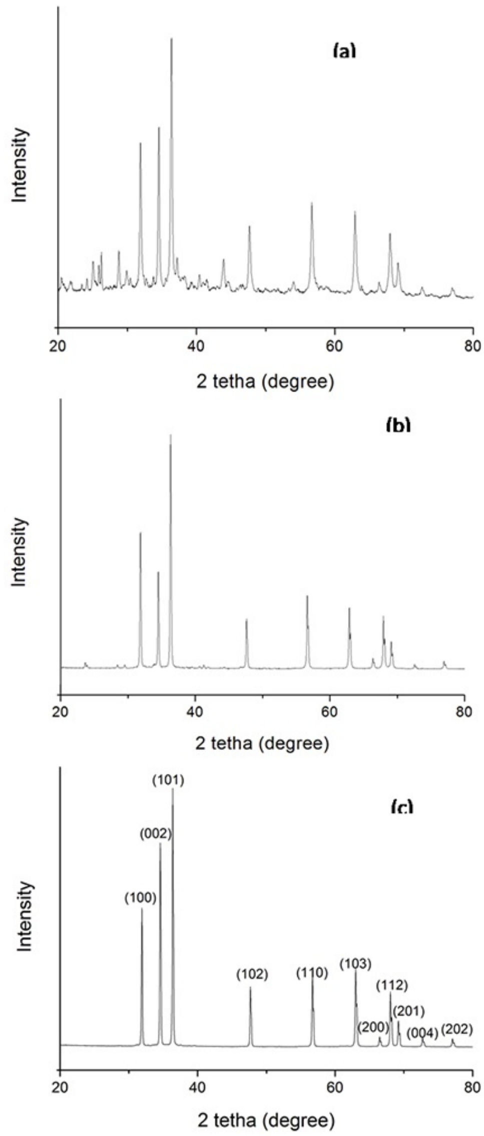

Figure 1 XRD pattern of ZnO NPs (a) as-synthesized, (b) annealed at 450°C and (c) annealed at 1000°C

Morphology studies of the samples was carried out by SEM analysis. The results show that the morphology of the NPs change from sphere-like shape to polygon shape by increasing temperature. Figure 2(a) illustrates the as-prepared ZnO NPs prepared and Figure 2(b) and Figure 2(c) reveal the annealed samples at 450°C and 1000°C respectively. In fact, the NPs are closed to each other by increasing temperature due to increasing the intermolecular and interatomic forces [54–64] which lead to change the morphology of the NPs [65–73]. In addition, samples are less agglomerated because the EG stabilizers are removed from NPs by increasing treatment [74–80].

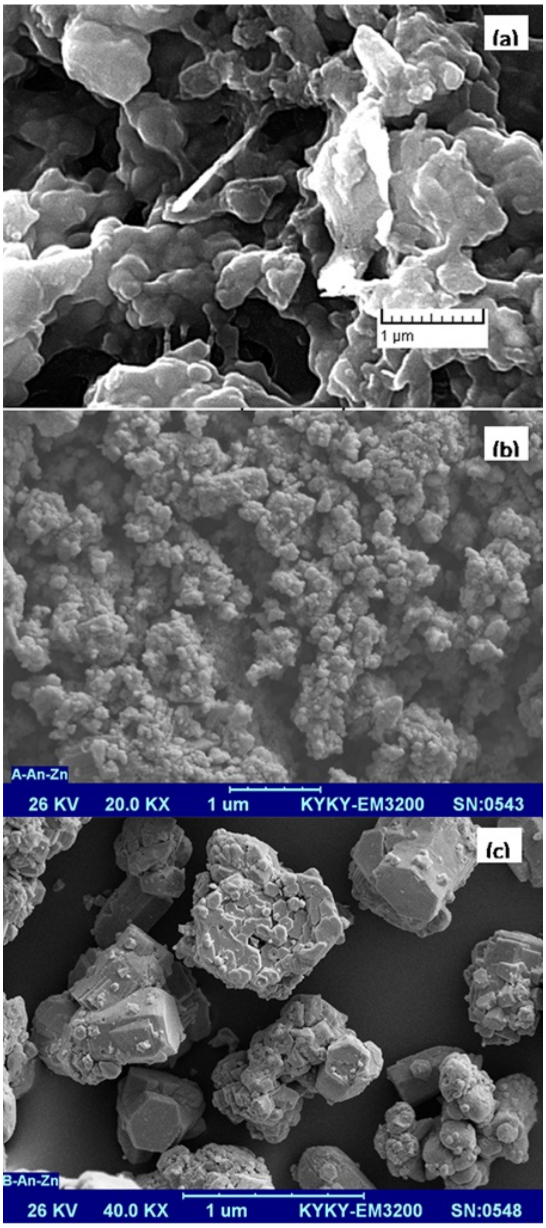

Figure 2 SEM images of ZnO NPs; (a) as-synthesized, (b) annealed at 450°C and (c) annealed at 1000°C

To calculate the exact size and shape of the NPs, TEM analysis was performed. Figure 3 demonstrates the as-prepared TEM analysis of ZnO NPs. As it can be seen from TEM, the spherical particles are formed in the range size of 20-100 nm. It seems that particles are formed as sponge shape [81, 82].

The samples was analyzed by EDS to specify the chemical composition. Figure 4 indicates the as-prepared samples which confirms the existence of Zn and O atoms with less sulfur contamination. The EDS analysis demonstrates an increasing level of Zn element from 28.5 wt% to 50.8 wt% for as-synthesized and annealed samples, respectively (not shown here). It may due to formation of more Zn atoms in chemical composition after increasing temperature to 1000°C.

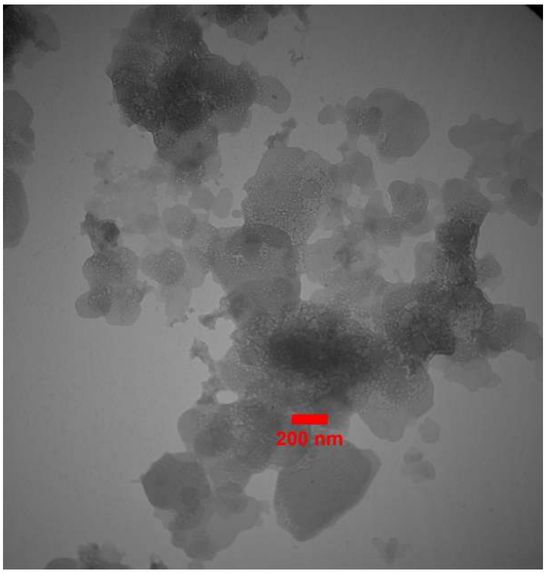

Figure 3 TEM image of the as-synthesized ZnO NPs

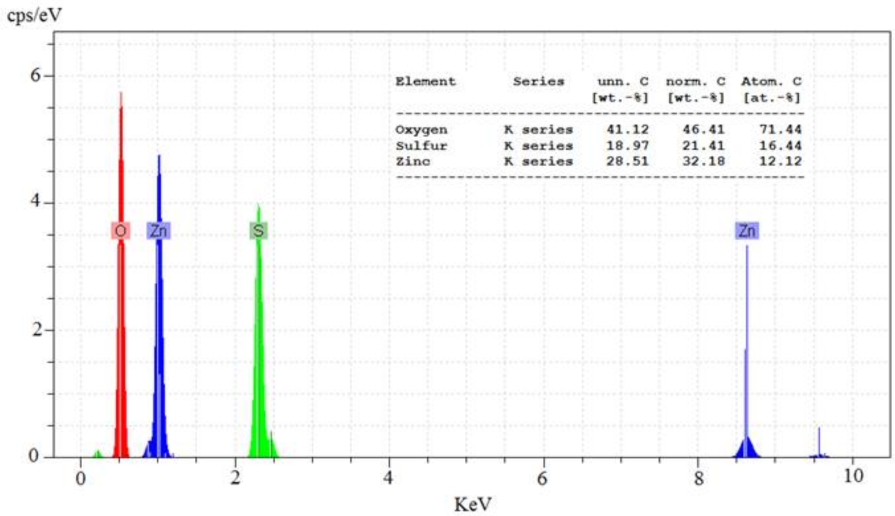

Figure 4 EDS analysis of the as-synthesized ZnO NPs

4 Conclusion

In summary, ZnO NPs were successfully fabricated by a simple coprecipitation synthesis using zinc nitrate as precursor and EG as stabilizer. The structure results revealed the single phase hexagonal wurtzite crystal structure of ZnO NPs. The SEM images, exhibited that the morphology of the samples changed from spherical shape to polygon shape by increasing temperature with less agglomeration. TEM analysis showed the mean size of as-synthesized sample was about 60 nm. Finally, EDS test demonstrated increasing level of Zn atoms by increasing temperature from 450°C to 1000°C.

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