



ORIGINAL ARTICLE

Synthesis and characterization of ZnO nanoparticles by thermal decomposition of a curcumin zinc complex



Mutasim I. Khalil ^{a,*}, Maha M. Al-Qunaibit ^a, Aisha M. Al-zahem ^a,
Joselito P. Labis ^b

^a Chemistry Department, King Saud University, Riyadh 11451, Saudi Arabia

^b King Abdullah Institute for Nanotechnology, King Saud University, Riyadh, Saudi Arabia

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Abstract ZnO nanoparticles were generated by thermal decomposition of a binuclear zinc (II) curcumin complex as single source precursor. Thermal behavior of the precursor showed a considerable weight loss at about 374 °C by an exothermic reaction with a maximum weight loss rate of 14%/min. Complete decomposition of precursor was observed within 49 min with a heating rate of 10 °C/min. Synthesized nanoparticles have been characterized by X-ray diffraction, Fourier transform infrared spectroscopy, transmission electron microscopy and selected area electron diffraction microscopy. Results revealed monodispersed hexagonal zincite structure with an average size of 117 ± 4 nm.

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1. Introduction

Metal oxides are particularly attractive with respect to applications in catalysis, sensing, energy storage, conversion optics, electronic devices (Purica et al., 2001; Aoki et al., 2000), memory arrays, biomedical application (Ayudhya et al., 2006; Wu et al., 2006) and acoustic wave devices (Gorla et al., 1999). Zinc oxide nanocrystals are one of the most intensively studied materials because of their versatile optoelectronic applications.

Their large band gap of 3.37 eV and large excitation binding energy of 60 meV ensure an efficient UV-blue emission at room temperature (Joo et al., 2005; Vafaei and Ghamsari, 2007; Kim et al., 2005). Because of this, ZnO has a great potential in several applications such as room temperature UV-lasers (Huang et al., 2001; Arnold et al., 2003), light emitting diodes (Park et al., 2002; Jo et al., 2003), solar cells (Rensmo et al., 1997) and sensors (Kind et al., 2002; Senoussaoui et al., 2004).

Various synthetic methods have been developed to fabricate ZnO nanocrystals including vapor phase growth (Sun et al., 2004), vapor liquid–solid processes (Gao and Wang, 2004), soft chemical method (Vayssieres, 2003), electrophoretic deposition (Liu et al., 2003), sol–gel processes (Zhang et al., 2003), solvothermal processes (Wang et al., 2006), and thermal decomposition techniques (Baskoutas et al., 2008; Niassari et al., 2005; Liewhiran et al., 2006; Xu et al., 2002). For example, ZnO nanocrystals with various shapes were synthesized

* Corresponding author. Tel.: +966 1 75973; fax: +966 1 75992.

E-mail address: mkhalil@ksu.edu.sa (M.I. Khalil).

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using a variety of surfactants (Joo et al., 2005), by the thermal decomposition of zinc alginate at 800 and 450 °C (Baskoutas et al., 2008), or by using bis(acetylacetonate) zinc(II) as a precursor (Niassari et al., 2005). Nano-sized ZnO powders (10–60 nm) were synthesized by thermal decomposition of zinc acetate (Liewhiran et al., 2006). Using urea and zinc nitrate as raw materials, Liu et al., 2007, prepared zinc oxide nanocrystals by a homogeneous precipitation method followed by thermal treatment at 500 °C for 10 min. On the other hand, ketones have been used as an oxolation source for the synthesis of titanium-oxo-organo crystals (Steunou et al., 1999; Garnweitner et al., 2004). Analogously, the β -diketone curcumin and curcuminoids (1,7-diaryl-1,6-heptadiene-3,5 diones) which are a group of naturally occurring 1,3-diketones with a good ligating ability for metal ions (Tennesen and Greenhill, 1992; Krishnankutty and John, 2003) is an attractive area for exploring the potential of its metal complexes for the synthesis of metal oxides nanoparticles. Curcumin is a renewable resource, its metal complexes are easily prepared that can be used to prepare metal oxides nanoparticles with low cost. In the mean time, there is no published data over the synthesis of metal oxides nanoparticles using curcumin metal complexes as a single source precursor. In this study, we describe the synthesis of ZnO nanoparticles by the thermal decomposition of a binuclear zinc hydroxido curcumin complex as a precursor prepared by the reaction of $\text{Zn}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$ and curcumin in a 1:2 mol ratio (Khalil et al., 2013). The thermal behavior of the precursor metal complex was studied by thermogravimetric analysis (TGA), and differential thermal analysis (DTA). The synthesized ZnO nanocrystals were characterized by Fourier transform infrared (FTIR) spectroscopy, powder X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and selected area electron diffraction (SAED).

2. Experimental

2.1. Chemicals and materials

All the chemicals and solvents were of reagents grade and used without further purification as purchased from Sigma–Aldrich,

New Jersey, USA. Zinc nitrate (BDH, Poole, England) laboratory reagent was used.

2.2. Analytical instruments

The FTIR spectrum was recorded on a Shimadzu FTIR-8400S, Prestige-21 spectrophotometer in a KBr matrix. TGA and DTA curves were recorded using a TA instrument model-SDT-Q600. XRD patterns were obtained by an Ultima IV X-ray diffractometer by using monochromatized Cu K α 1 radiation under the acceleration voltage of 40 kV and a current of 40 mA. The morphology of the ZnO nanocrystals was examined by a JEOL JSM-6380 LA SEM and a JEOL TEM 2100F TEM with an acceleration voltage of 200 kV.

2.3. Preparation of zinc (II) curcumin complex

(Bis(1,7-bis[4-hydroxy-3-methoxyphenyl]-1,6-heptadiene-3,5-dionato)hydroxide zinc(II), $[\text{Zn}(\text{OH})(\text{curc})]$) was prepared by a previously reported method (Khalil et al., 2013). An ethanolic mixture solution of zinc nitrate and curcumin in a 1:2 mol ratio was refluxed for 1 h and the resulting product was filtered and washed several times by de-ionized water, dried in vacuum and used as a precursor to synthesize ZnO nanoparticles.

3. Results and discussion

3.1. Thermal decomposition of zinc (II) curcumin complex precursor

Our investigation revealed that such a complex lends itself as a potential precursor for zinc oxide nanoparticles synthesis through thermal decomposition. The thermal properties of precursor were determined by thermogravimetric analysis (TGA) and differential thermal analysis (DTA). Sample was heated in an aluminum pan from room temperature to 600 °C at a heating rate of 10 °C/min. The TGA and DTA analyses (Fig. 1) revealed that the complex loses 6% of its weight at about 200 °C attributed to desorption or removal of moisture and solvents, and 50% weight loss of its total

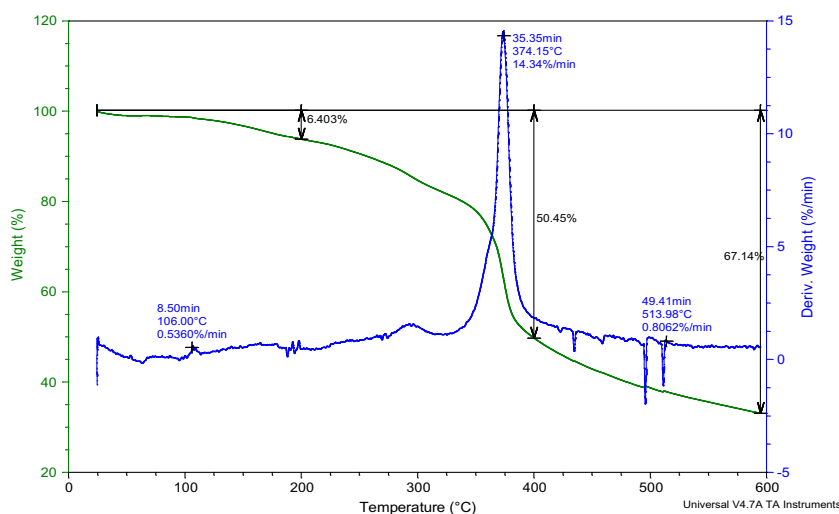


Figure 1 Thermal behavior of $[\text{Zn}(\text{OH})\text{C}_{21}\text{H}_{20}\text{O}_6]_2$.

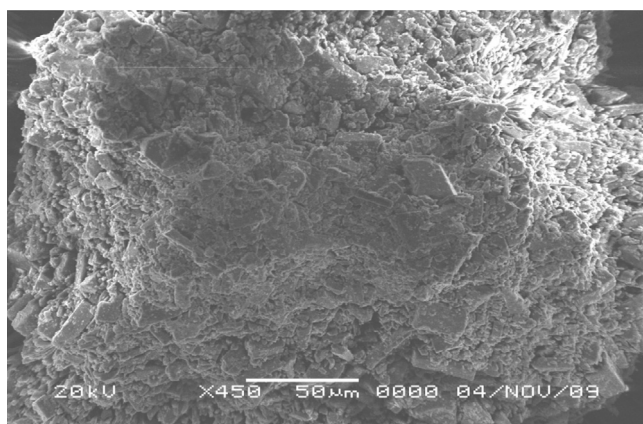


Figure 2 SEM image of $[\text{Zn}(\text{OH})\text{C}_{21}\text{H}_{20}\text{O}_6]_2$ precursor.

weight at 374°C with a maximum weight loss rate of $14\% \text{ min}^{-1}$ indicating a fast rate of degradation of curcumin into volatile combustible products. The DTA curve has a large

exothermic peak at that temperature which may be attributed to the fact that volatile organic moieties generated by the dissociation of precursor react with O_2 to form CO_2 and H_2O . This is confirmed by the weight loss observed in the temperature region $400\text{--}600^\circ\text{C}$ in the TGA curve.

The morphology of the synthesized micro particles of the zinc complex precursor shown in the SEM image of Fig. 2 was converted into hexagonal nanoparticles upon thermal treatment at 500°C .

The phase and crystallinity of the synthesized sample were investigated by X-ray diffraction patterns shown in Fig. 3. The patterns are in accord with the typical zincite structure ZnO diffraction (hexagonal phase, space group $P6_3mc$, with lattice constants $a = 3.24982 \text{ \AA}$, $c = 1.6021 \text{ \AA}$, $Z = 2$, JCPDS No. 361451) (Bigdeli et al., 2010; Liu et al., 2007).

A most intense peak at $2\theta = 36.67^\circ$ is obtained along (101) orientation. The sharpness, strong intensity and narrow width of ZnO diffraction peaks in the XRD pattern indicate that the synthesized ZnO sample is well crystallized. The FTIR spectrum of ZnO nanoparticles exhibited two strong vibrational bands at 640 and 605 cm^{-1} assigned to the stretching modes

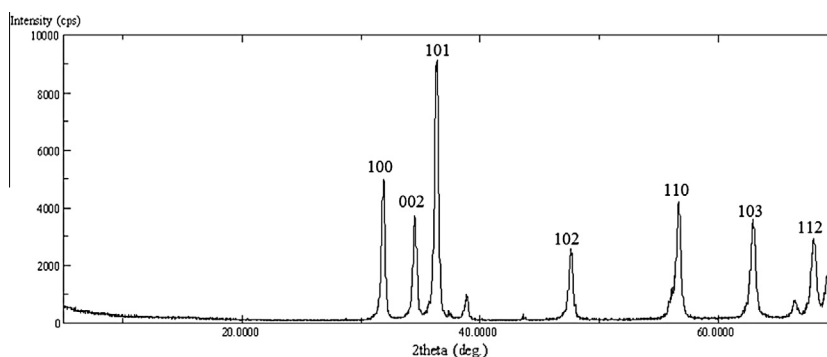


Figure 3 X-ray diffraction pattern of ZnO nanoparticles.

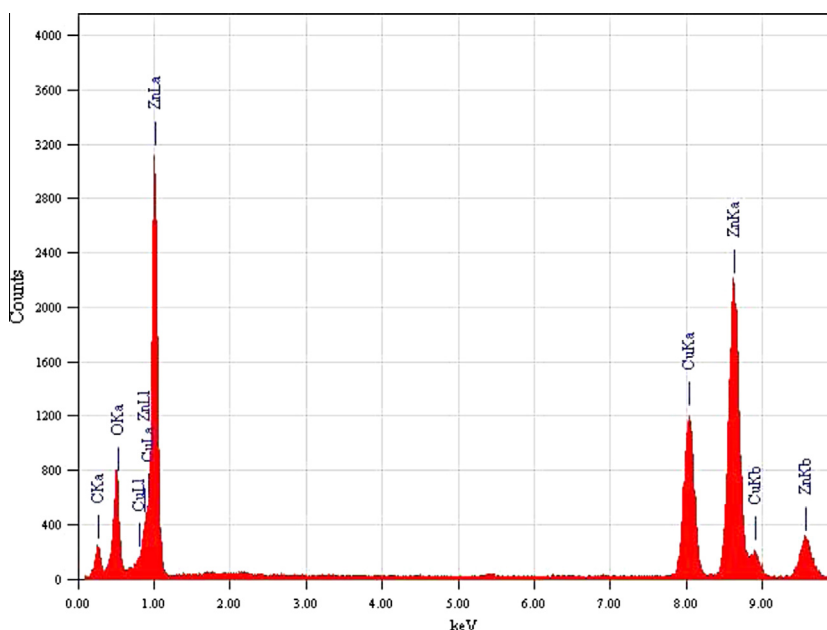


Figure 4 EDS pattern of the ZnO nanoparticle.

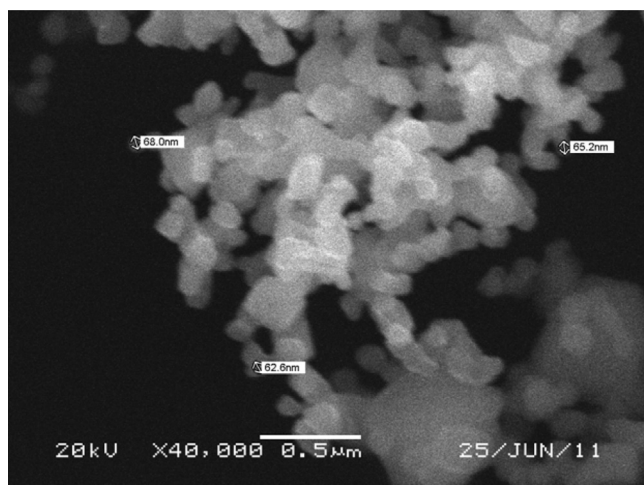


Figure 5 SEM image of ZnO nanoparticles.

of Zn–O and the weak bands at 3500 and 1040 cm^{-1} are probably attributed to the presence of water in the KBr matrix (Bigdeli et al., 2010; Chen et al., 2004). The EDS pattern of the ZnO nanoparticles is shown in Fig. 4. The Cu and C signals were from the Cu grid and carbon film coated on the support formvar. No other signals were detected within the detection limits of EDS which confirm the purity of the ZnO nanoparticles.

The scanning electron microscopy (SEM) image of ZnO sample is shown in Fig. 5. This image reveals that the ZnO is in nano scale.

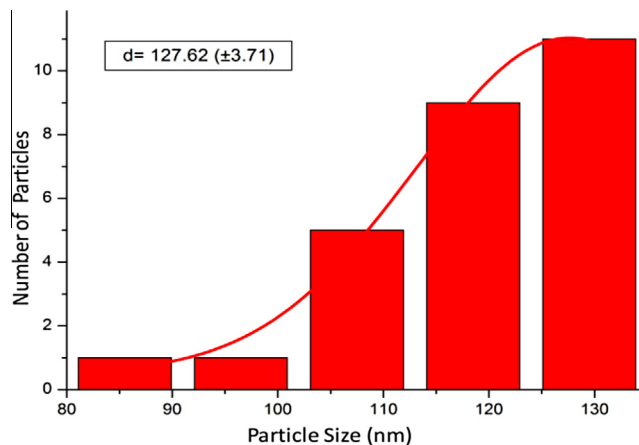


Figure 7 Histogram (average size) of ZnO nanoparticles.

The fine powder of ZnO nanocrystals was dispersed in ethanol on a carbon-coated copper grid and the high resolution transmission electron microscopy (HRTEM) images (Fig. 6) were obtained with ultra high resolution at an accelerating voltage of 200 kV. The selected area electron diffraction (SAED) pattern shown in insert (a) of Fig. 6 has sharp spots indicative of polycrystalline nature with symmetrical orientation of ZnO nanoparticles in the sample. The crystallinity of the synthesized nanoparticles was also supported by the observed lattice fringes of 0.25 nm in HRTEM image shown in insert (b) of Fig. 6. This value is very near the $0.26 \pm 0.05\text{ nm}$ spacing for ZnO corresponding to (101) plane of ZnO (Yan et al., 2000).

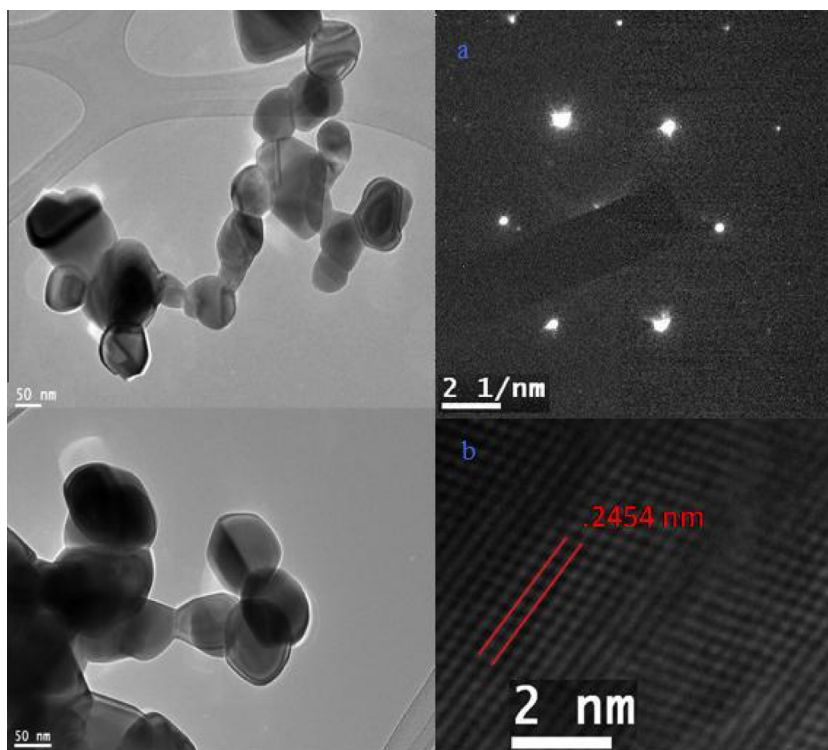


Figure 6 TEM image of ZnO nanoparticles, SAED (insert a) and high scanning resolution (insert b).

The average crystal size is estimated by considering the few number of crystals shown in the TEM image, Fig. 6, and found to be about 127 ± 4 nm presented by a histogram in Fig. 7.

4. Conclusion

The synthesis of zinc oxide nanoparticles from a curcumin zinc complex by a thermal decomposition technique was studied in this paper. Low heat energy was applied to degrade the organic moiety. Nanoparticles with an average size of 117 ± 4 nm were obtained from an easily prepared organic moiety containing metal complex precursor. Such a type of precursors have potential for synthesizing metal oxide nanoparticles.

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