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Synthesis and Characterization of $Zn(acac)_2$ One-dimensional Nanostructures by Novel Method

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In this work we have sublimated bulk Zn(acac)2 at different temperatures and the effect of sublimation temperature on the morphology of product was investigated. Nanostructures were characterized by means of SEM, XRD and FT-IR.

Keywords: $Zn(acac)_2$, Sublimation, One-dimentional nanostructures, Scanning electron microscope.

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1. INTRODUCTION

Nanomaterials have at least one dimentional from 1 to 100 nm. Recently, Preparation and characterization of one-dimensional (1D) nanostructures such as nanotubes, nanowires, nanobelts and nanorods have received much attention. This interest arises from their irregular and attractive properties and applications that are better than their bulk counterparts [1-4].

Amid the various methods developed for preparing metal oxides nanostructures, the organometallic molecular precursor approach has been regarded as one of the suitable and useful techniques, because it does not require complicated processes and rigorous preparation conditions, but homogeneity, purity, composition, phase, and microstructure of the resultant products as well as controls.

Zinc (II) acetylacetonate [$Zn(acac)_2$] due to suitable equilibrium vapor pressure and low decomposition temperature (T_{dec} = 110 °C), commercial availability and its relatively low cost compared to the costs of other β-diketonate compounds, Including the popular precursors.

2. EXPERIMENTAL AND CHARACTERIZATION

2.1 Materials and Instrumentals

Only the reagent that is [$Zn(acac)_2$] was purchased from Merck Company.

Powder X-ray diffraction (XRD) patterns were collected from a diffractometer of Philips Company with X'PertPro monochromatized Cu Kα radiation (λ = 1.54 Å). Microscopic morphology of the products was studied by SEM (LEO 1455VP). FT-IR spectra were recorded on Magna-IR, spectrometer 550 Nicolet in KBr pellets in the range of 400–4000 cm^{-1}.

2.2 Preparation of [$Zn(acac)_2$] Nanostructures

Preparation of nanotubes of $Zn(acac)_2$ from bulk $Zn(acac)_2$ was executed in a vertical quartz pipe set under vacuum condition. Each experiment was started with loading 0.03 g $Zn(acac)_2$, which would be transferred in the bottom of external pipe. Then, the system is being vacuumed by related pump. The flow of water caused to cool inner pipe, and cold wall will increase the yield of collected product. Set is heated to reach desire temperature, then, the nail-like thin depositions are seen at the external part of inner pipe, set is opened and the products are collected.

3. RESULTS AND DISCUSSION

To investigate the effect of sublimation temperature as a parameter on the morphology and size of the products, the various experiments were performed.

3.1 Fourier transform infrared spectroscopy

FT-IR spectra were recorded to show the chemical groups for obtained product. Nanomaterials have high surface-to-volume ratios and this caused that large fractions of atoms were available on surface and for this reason, surface stress is increased, so, stretching frequencies of nanomaterials are higher than bulk material, as can be seen in Fig. 1. The C=O peaks related to nano (Fig. 1a) and bulk (Fig. 1b) $Zn(acac)_2$ are at 1599.19 cm^{-1} and 1595.63 cm^{-1}, respectively. The peak shift of the carbonyl group is due to the interaction of the metal with oxygen. The weak peaks at 3440 cm^{-1} are assigned to the stretching vibration of absorption water. Residual peaks are representative of the methyl and methylene groups of $Zn(acac)_2$.

3.2 X-ray Diffraction Patterns

The XRD results of the bulk and nano samples $Zn(acac)_2$ are shown in Fig. 2a and b, respectively. Sublimation of the $Zn(acac)_2$ at 110 °C resulted in the formation of monoclinic crystalline phase, namely zinc acetyl acetonate, with cell constants a = 10.48, b = 5.37, c = 10.94, which are in agreement with JCPDS 41-1634. The average diameter size of the sample is about 17.69 nm, estimated via the Debey–Scherrer equation [5].

3.3 Scanning Electron Microscopy Images

Fig. 3 shown SEM images related to sublimated Zn(acac)2 at various temperatures. For investigating the

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effect of increasing the sublimation temperature on the Zn (acac)2 morphologies the reaction carried out in 110, 120, 130 and 140 °C. According to the Fig. 3, the morphologies of all prepared Zn (acac)2 were 1-dimensional nanostructures which their ratio of length to diameter are decreased with increasing sublimation temperature. As it is seen from, with increasing the temperature from 110 °C (Fig. 3a) and 120 °C (Fig. 3b) to 130 (Fig. 3c) and 140 °C (Fig. 3d), the obtained nanobelts were been thicker and longer. As the temperature increases to 130 °C (Fig. 3c), the irregular sticking structures were obtained with further increasing of the temperature to 140 °C (Fig. 3d) branching and disorder tubes were created. For explanation of this phenomenon can be said that due to the mechanism and particular shape of sublimation set, the products were grown in one-dimension and 1-dimentional structures were produced.

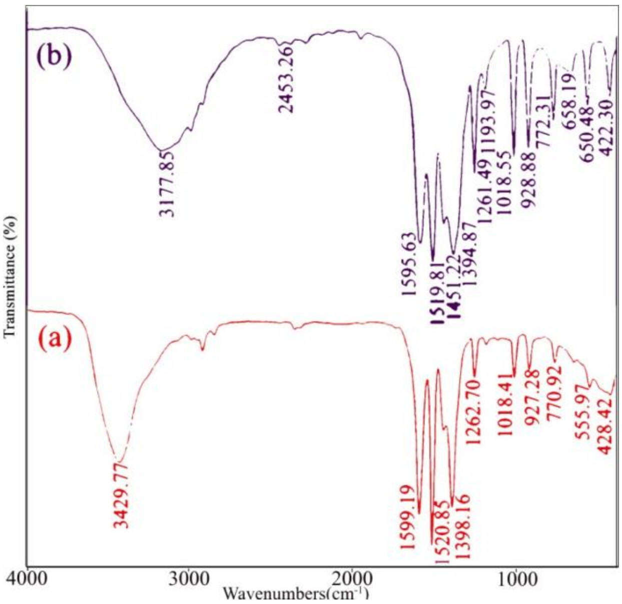

Fig. 1 – FT-IR spectra of a: sublimated $Zn(acac)_2$ at 110 □C ,b: bulk $Zn(acac)_2$.

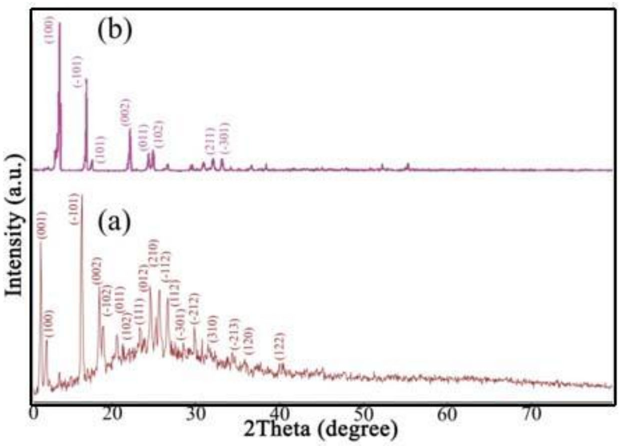

Fig. 2 – XRD pattern of a: sublimated $Zn(acac)_2$ at 110 □C ,b: bulk $Zn(acac)_2$.

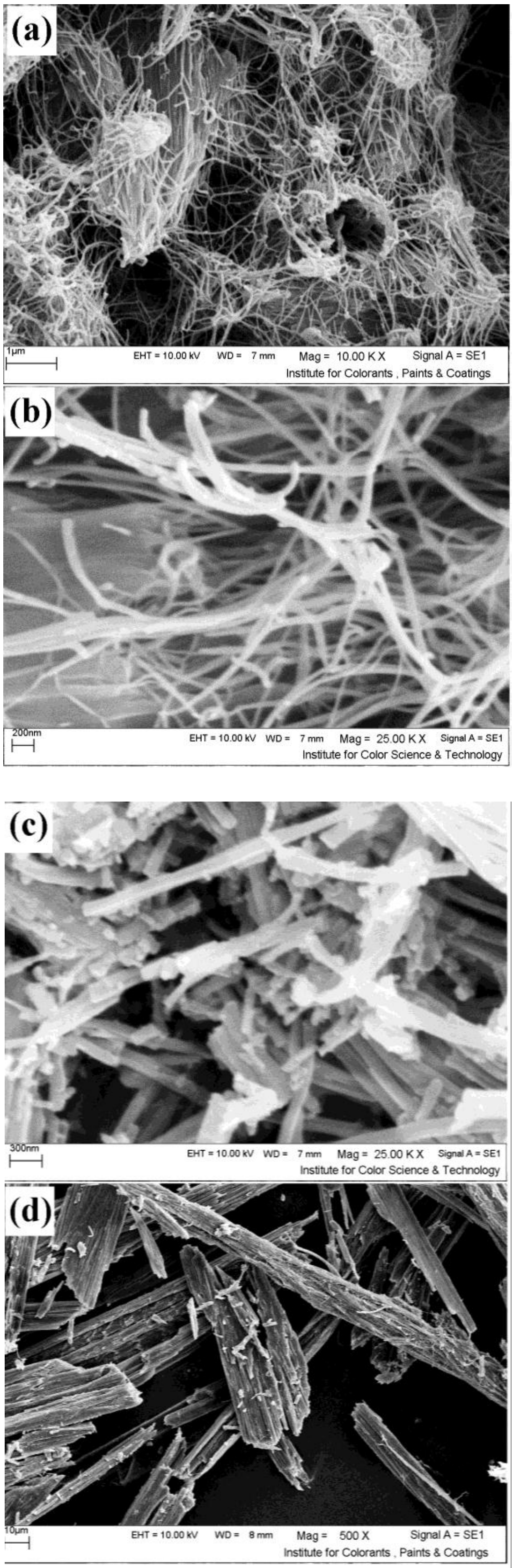

Fig. 3 – SEM images; a-d: sublimated $Zn(acac)_2$ at; 110,120, 130, 140 °C

4. CONCLUSION

Sublimation method provides an eco-friendly, economical and simple method for self-assembled 1-dimentional nanostructures of ß-diketone complexes. For the first time, we have been produced nanotubes complex via this way. Uniform $Zn(acac)_2$ nanotubes were achieved in sublimation temperature 110 °C, these tubes have length to diameter ratio about 23, this value is expressing that sublimation method can be known as particular rout for synthesis of nanowires and nanotubes of inorganic solid compounds with high vapor pressure.

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