

## Solid-State Thermal Decomposition Method for the Preparation of CuO Nanoparticles

A. Dehno Khalaji<sup>a,\*</sup>, K. Jafari<sup>b</sup>, S. Maghsodlou Rad<sup>a</sup>

<sup>a</sup> Department of Chemistry, Faculty of Science, Golestan University, Gorgan, Iran

<sup>b</sup> College of Chemistry, Shahrood University of Technology, Shahrood, Iran

### Article history:

Received 10/12/2012

Accepted 22/2/2013

Published online 1/3/2013

### Keywords:

Nanoparticles

Schiff base

Copper oxide

Thermal decomposition

### Abstract

In this paper, CuO nanoparticles have been synthesized via solid-state thermal decomposition using copper(II) Schiff base complexes as new precursors at 600°C under air atmosphere for 3 h. Surface morphology of the products were characterized by Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD) and scanning electron microscopy (SEM).

### \*Corresponding author:

E-mail address:

alidkhalaji@yahoo.com

Phone: +98 171 22445882

Fax: +98 171 2245964

2013 JNS All rights reserved

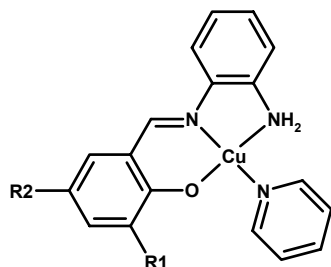
## 1. Introduction

Recently, the preparation of transition metal oxides nanoparticles with specific size and morphology have been an increasing interest due to their properties and applications [1-7]. CuO is a well known p-type semiconductor with a narrow band gap of 1.2 eV. It has a wide range of applications as catalysis, batteries, gas sensors and transistors [8-13]. Until now, various different methods have been developed to preparation of CuO nanoparticles including hydrothermal

reaction, electrochemical, thermal evaporation and decomposition [8-17]. Currently, the solid-state thermal decomposition of Schiff base complexes as new precursors is being used more and more [19-21], and as compared to conventional methods, it is much faster, economical and cleaner. Different shapes of CuO nanosized such as nanorods, nanosphere and nanowire have been synthesized by various methods [8-17].

In this study, we decided to used simple, low-cost, green and reproducible process for the

preparation of CuO nanoparticles from unsymmetric copper(II) Schiff base complexes as new precursors (Scheme 1).



R1 = H	R2 = H	(1)
R1 = CH <sub>3</sub> O-	R2 = H	(2)
R1 = H	R2 = Br	(3)

**Scheme 1.** Chemical structure of Cu(II) Schiff base complexes

## 2. Experimental

### 2.1. Materials and characterization

All reagents and solvents for synthesis and analysis were commercially available and used as received without further purifications. Fourier Transform Infrared spectra were recorded as a KBr disk on a FT-IR Perkin–Elmer spectrophotometer. X-ray powder diffraction (XRD) pattern of the complex was recorded on a Bruker AXS diffractometer D8 ADVANCE with Cu-K $\alpha$  radiation with nickel beta filter in the range  $2\theta = 10^\circ$ – $80^\circ$ . Scanning electron microscopy (SEM) images were obtained on Philips XL-30ESEM. All complexes were synthesized from Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O following a published procedure [22,23].

### 2. 2. Synthesis of [Cu(saliph)(Py)]NO<sub>3</sub> (1)

An aqueous solution of Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O (2.63 gr, 0.01 mol in 2 mL) was added to stirred solution of salicylaldehyde (1.22 gr, 0.01 mol) in methanol (30 mL) followed by pyridine (0.02 mol). The mixture was stirred at 50°C for 1 h, and then 1.08 g (0.01

mol in 15 mL methanol) of 1,2-phenylenediamine was dropwise added. The mixture was stirred for 12 h in air at room temperature. The dark-red solid was obtained after the solvent was evaporated slowly for several days at room temperature, then the products were collected by filtration, and dried in vacuum. FT-IR (KBr pellet, cm<sup>-1</sup>): 3320, 3275 (NH<sub>2</sub>), 1611 (C=N).

### 2. 3. Synthesis of [Cu(MeO-saliph)(Py)]NO<sub>3</sub> (2)

This dark-red solid was prepared by the some procedure using 3-methoxysalicylaldehyde (1.05 g, 0.01 mol). FT-IR (KBr pellet, cm<sup>-1</sup>): 3323, 3274 (NH<sub>2</sub>), 1615 (C=N).

### 2. 4. Synthesis of [Cu(Br-saliph)(Py)]NO<sub>3</sub> (3)

This dark-red solid was prepared by the some procedure using 5-bromosalicylaldehyde (2.01 g, 0.01 mol). FT-IR (KBr pellet, cm<sup>-1</sup>): 3321, 3276 (NH<sub>2</sub>), 1613 (C=N).

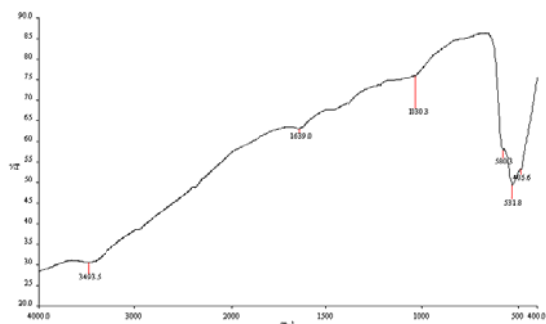
### 2. 5. Preparation of CuO nanoparticles

The complexes were loaded in to a crucible and then were placed in oven and heated at a rate of 10°C/min in air. Nanoparticles of CuO were synthesized at 500°C after 3 h, washed with ethanol and dried at room temperature. The synthesized CuO nanoparticles were characterized by FT-IR, XRD and SEM.

## 3. Results and discussion

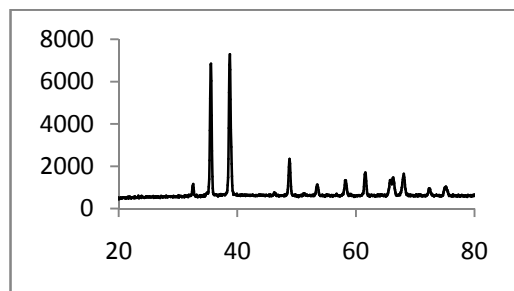
Fig.1 shows FT-IR spectra of CuO nanoparticle were formed via solid-state thermal decomposition of **1** at 500°C. The peak at 531 cm<sup>-1</sup> assigned to

Cu-O stretching [12]. Existence of free precursor is ruled out due to the absence of stretching vibrations of CH, C=N and other groups of the ligand.

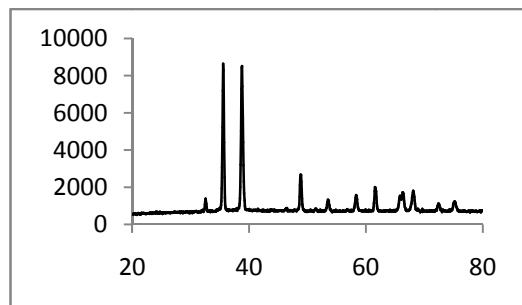


**Fig. 1.** XRD pattern of as-synthesized sample.

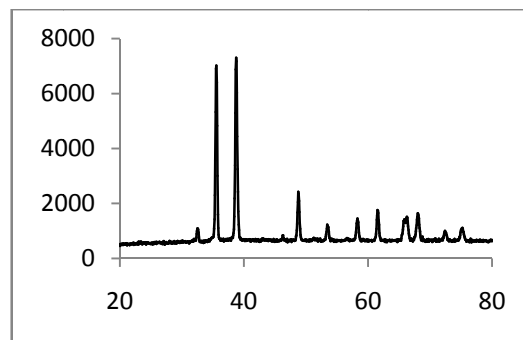
Figs. 2-4 show the XRD pattern ( $10 < 2\theta < 80$ ) of the CuO nanoparticles obtained from copper(II) complexes **1-3**, respectively. All the diffraction peaks can be indexed to pure CuO monoclinic phase [12,18]. No other impurities were detected by XRD analysis, indicating the phase purity of CuO nanoparticles.



**Fig. 2.** XRD pattern of CuO prepared from 1.

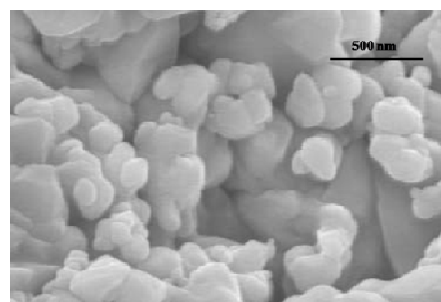


**Fig. 3.** XRD pattern of CuO prepared from 2.

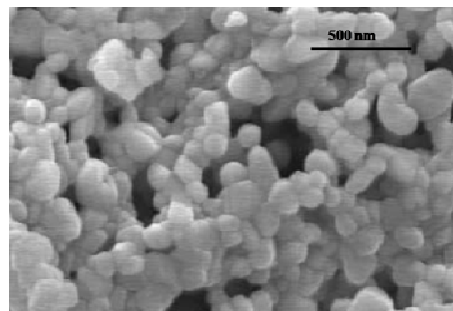


**Fig. 4.** XRD pattern of CuO prepared from 3.

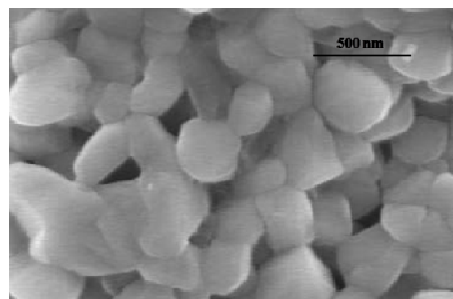
The SEM images of the CuO nanoparticles were taken (Figs. 5-7, respectively) and confirm the spherical morphology of CuO nanoparticles.



**Fig. 5.** SEM image of CuO prepared from 1.



**Fig. 6.** SEM image of CuO prepared from 2.



**Fig. 7.** SEM image of CuO prepared from 3.

#### 4. Conclusion

In summary, we have successfully prepared spherical CuO nanoparticles by solid-state thermal decomposition. This method is facile, inexpensive, nontoxic and can be extended for preparation of other transition metal oxide nanoparticles.

#### Acknowledgment

A.D. Khalaji is grateful to the Golestan University for partial support of this work.

#### References

- [1] A. Tadjarodi, M. Imani, H. Kerdari, *J. Nanostruct.* 2 (2012) 127-138.
- [2] R. Karimian, M. Zandi, N. Shakour, F. Piri, *J. Nanostruct.* 1 (2012) 39-43.
- [3] R. Jalajerdi, F. Gholamian, H. Shafie, A. Moraveji, D. Ghanbari, *J. Nanostruct.* 2 (2012) 105-109.
- [4] A. Kodge, S. Kalyane, A. Lagashetty, *Int. J. Nano Dimens.* 3 (2012) 53-57.
- [5] H. Bakhtiari, Q.S. Manuchehri Naeini, S. Haghighi, E. Emamzadeh, *Int. J. Nano Dimens.* 3 (2013) 185-190.
- [6] S. Imani, A.M. Zandi, M. Saadati, H. Honnari, B. Maddah, *Int. J. Nano Dimens.* 2 (2011) 129-135.
- [7] M. Ghane, B. Sadeghi, A.R. Jafari, A.R. Paknejhad, *Int. J. Nano Dimens.* 1 (2010) 33-40.
- [8] S. Jadhav, S. Gaikwad, M. Nimse, A. Rajbhoj, *J. Clus. Sci.* 22 (2011) 121-129.
- [9] W. Jia, E. Reitz, P. Shimpi, E.C. Rodriguez, R.-X. Gao, Y. Lei, *Mat. Res. Bull.* 44 (2009) 1681-1686.
- [10] W. Wang, O.K. Varghese, C. Ruan, M. Paulose, C.A. Grimes, *J. Mater. Res.* 18 (2003) 2756-2759.
- [11] X. Jiang, T. Herricks, Y. Xia, *Nano Lett.* 2 (2002) 1333-1338.
- [12] A. El-Trass, H. Elshamy, I. El-Mehasseb, M. El-Kemary, *Appl. Sur. Sci.* 258 (2012) 2997-3001.
- [13] A.K. Srivastava, P. Tiwari, A. Kumar, R.V. Nandedkar, *Curr. Sci.* 86 (2004) 22-23.
- [14] J. Safei-Ghomi, M.A. Ghasemzadeh, *J. Nanostruct.* 1 (2012) 243-248.
- [15] R.S. Razavi, M.R. Loghman-Estarki, *J. Clust. Sci.* 23 (2012) 1097-1106.
- [16] H.-Q. Wu, X.-W. Wei, M.-W. Shao, J.-S. Gu, M.-Z. Qu, *Chem. Phys. Lett.* 364 (2002) 152-156.
- [17] N.V. Suramwar, S.R. Thakare, N.T. Khaty, *Int. J. Nano Dimens.* 3 (2012) 75-80.
- [18] S. Sabbaghi, H. Orojloou, M.R. Parvizi, S. Saboori, M. Sahooi, *Int. J. Nano Dimens.* 3 (2012) 69-73.
- [19] A.D. Khalaji, *J. Clust. Sci.* 24 (2013) 209-215.
- [20] A.D. Khalaji, *J. Clust. Sci.* 24 (2013) 189-195.
- [21] A. Khansari, M. Enhessari, M. Salavati-Niasari, *J. Clust. Sci.* 24 (2013) 289-297.
- [22] L. Rigmonti, F. Demartin, A. Forni, S. Righetto, A. Pasini, *Inorg. Chem.* 45 (2006) 10976-10989.
- [23] L. Rigmonti, A. Cinti, A. Forni, A. Pasini, O. Piovesana, *Eur. J. Inorg. Chem.* (2008) 3633-3647.