*Journal of Nano- and Electronic Physics*
Vol. **4** No 3, 03005(4pp) (2012)

*Журнал Нано- та Електронної Фізики*
Том **4** № 3, 03005(4cc) (2012)

# Preparation and Characterization of CuO Nanoparticles by Novel Sol-Gel Technique

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(Received 26 June 2012; revised manuscript received 07 July 2012; published online 30 October 2012)

Recent developments of nanosize materials of metal and metal oxide particles are intensively pursued because of their prominence in different fields of applications. Among all the transition metal oxides, CuO is a potential candidate for the application of magnetic storage devices, solar energy transfer, sensors, and super capacitors etc. Moreover CuO nanoparticles act as a good catalyst in some of the chemical reactions. CuO nanoparticles were prepared by novel sol-gel method. In this technique $CuCl_2.6H_2O$ is added with acetic acid and heated to 100 °C with continuous stirring. To control the ph of the above solution, NaOH is added to the solution till ph reached desired value. The color of the solution changed from blue to black with precipitation. The black precipitation was washed 3-4 times with distilled water. Finally the solution was centrifuged and dried in air for one day. The CuO nanoparticles were characterized by studying their structure with X-ray diffraction and composition by energy dispersive X-ray analysis. The size of the nanoparticles is estimated by particle size analyzer and transmission electron microscopy. The optical studies were carried out with Uv-Vis spectrophotometer.

**Keywords:** CuO nanoparticles, Sol-gel method, Catalytic activity.

PACS numbers: 81.20.Fw, 81.16.Hc

## 1. INTRODUCTION

In general there are number of metal oxides are available in nature but some of the metal oxides are most useful in accordance with their applications in day to day life in science and technology. In the periodic table transition metals are large in number and have number of applications in different fields of applications. Some transition metal oxides like ZnO, $TiO_2$, and $Fe_3O_4$ etc. proved as potential candidates for so many applications. In the same way CuO is also one of the useful metal oxides and which has so many applications in various fields. The uniqueness of CuO nanoparticles is even though they are metallic in bulk they behave like semiconductors when they are in nanosize. Semiconducting materials have been particularly interesting because of their great practical importance in electronic and optoelectronic devices, such as electrochemical cell [1], gas sensors [2], magnetic storage devices [3], field emitters [4], high-tc super conductors [5], nano fluid [6], and catalysts [7] etc. Due to the potentiality of CuO, it acts as a catalyst; whereas all metal oxides are not useful for the catalytic activity. In the fabrication of super capacitors also CuO is very useful and in nano range it has the wide band gap nearly equal to ZnO. The favorable band gap of CuO around 2.6 eV makes it useful for solar energy conversion and it can be used as solar cell window material. It is very well established fact that the application of nanofluids acts as a coolants in refrigerators. These nanofluids mixed with carrier liquid then it enhance the energy transfer comparatively carrier liquid alone. CuO can be used as coolant material and it can control temperature effectively like other coolants $TiO_2$, alumina and silver particles etc. The properties of nanomaterials will depend mostly on the nanopowders size, morphology and specific surface area of the prepared materials. Some aspects are strongly depends on the preparation methods. Our present work on CuO nanocrystals shows distinct structural, chemical bonding and electrical characteristics. Lattice strain and structural disorder can change the properties of materials in the applications of different fields in science and technology. There are number of methods for the fabrication of CuO nanoparticles. Among those we have chosen sol-gel method for the preparation of CuO nanoparticles.one that is more preferable and widely used in the synthesis of nano particles. The size of the particles from the various preparation methods listed as fallows. In case of MOCVD [metallo-organic chemical vapor deposition] CuO nanoparticles size is 0.05 to 8 μm [8], sonochemical method is around 10 nm to several microns [9]. Some results shown in some references is in the range of 1-10 nm CuO nanoparticles in sol-gel method [10]. The average diameter of the CuO nanoparticle in solid state reaction is reported in the range of 15-20 nm [11]. Smallest CuO nanoparticles were prepared by electro chemical method and the size is around 4 nm [12].

## 2. EXPERMENTAL DETAILS

CuO nanopowders were prepared by sol-gel method. The aqueous solution of $CuCl_2.6H_2O$ (0.2 M) was prepared in cleaned round bottom flask. 1 ml of glacial acetic acid was added to above aqueous solution and heated to 100 °C with continuous stirring. 8 M NaOH is added to above heated solution till pH reached to 7. The color of the solution turned from blue to black immediately and the large amount of black precipitate is formed in the bottom of the flask. The precipitate was centrifuged and washed 3-4 times with deionized water. The obtained precipitate was dried in air for 24 h. This CuO powder was used for the characterization of the material.

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The crystalline phases of CuO nanoparticles were determined by X-ray Diffractorometer (XRD, Brukerd8 Advance, Germany) using CuKα as radiation source (40 kV, step size 0.020, scan rate 0.50 $min^{-1}$, $200 \leq 800$) and composition analysis was done by Energy Dispersive X-ray Analysis (EDAX). The nanoparticle size and zeta potential is estimated by particle size analyzer (SZ-100 nanoparticle, Horiba, Germany) data. The sizes of the particles are estimated by using transmission of electron microscopy. The morphology of the prepared nanoparticles particle were carried out by scanning electron microscope (SEM, Hitachip-SEM S-3400n, Germany).

## 3. RESULTS AND DISCUSSION

### 3.1 Structural analysis

Figure 1 shows the XRD pattern of prepared CuO nanoparticles. All the peaks in diffraction pattern shows monoclinic structure of CuO, and the peaks The average grain size calculated by using Debay-Scherrer formula is approximately 18.0967 nm are comparable with Jcpdf card no .[89-5895]. The lattice parameters were calculated from XRD data is as follows. $a = 0.466$ nm, $b = 0.343$ nm, $c = 0.507$ nm. The average grain size calculated by using Debay-Schrer formula as given below is approximately 18 nm.

Debye Scherrer formula

$$D = 0.9\lambda / \beta\cos\theta,$$

where $\beta$ is full width at half maxima of the peak in XRD pattern, $\theta$ – angle of the peak, $\lambda$ – wavelength of X-rays.

Elastic strain is also calculated from XRD results. The strain results suggested that if the particle size is less than 20 nm than they have more strain and greater than 20 nm particles have less strain. It clearly shows that the smaller particles have high strain and bigger particles have less strain. These values are in agreement with the literature values [13]. Morphology index was calculated from XRD full width at half maxima values and it is found that if the size of the particle increases, then the morphology index is also increases.

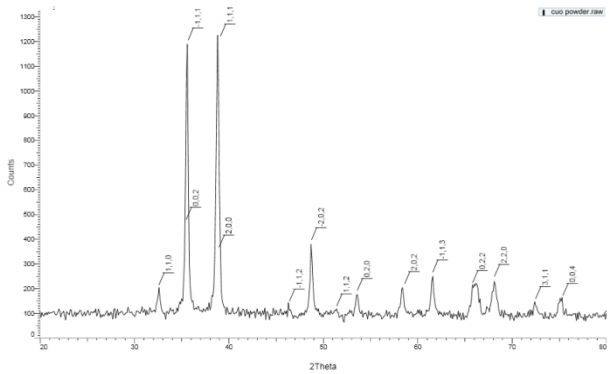


**Fig. 1** – XRD pattern of CuO nanoparticle

### 3.2 Compositional analysis

Figure 2 shows Energy Dispersive X-ray Analysis of CuO nanoparticles and the data indicates that the nanopowders are nearly stoichiometric. The wt. % of copper and oxide are calculated from EDAX data is as follows: O 9.03wt. % and Cu 90.97wt. %. There are no traces of other impurities like carbon etc. We can conclude from the EDAX data that the formation of pure CuO nanoparticles.

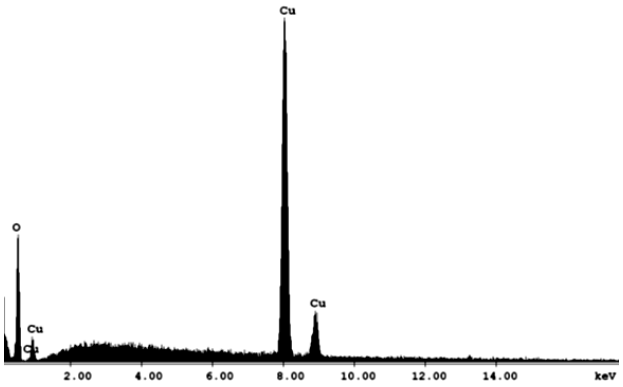


**Fig. 2** – EDAX of the CuO nanoparticles

### 3.3 Size analysis

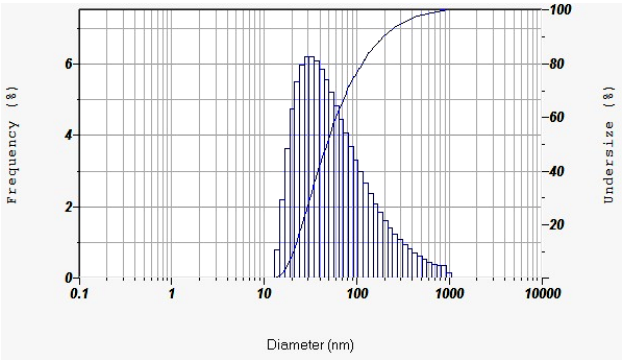


**Fig. 3a** – Histogram of particle size analyser

**Table 1** – Caliculations of particle size analyser

| | |
|---|---|
| Count Rate | : 3356 kCPS |

**Calculation Results**

| Peak No. | S.P.Area Ratio | Mean | S. D. | Mode |
|---|---|---|---|---|
| 1 | 1.00 | 86.7 nm | 109.4 nm | 32.9 nm |
| 2 | 0.00 | 921.5 nm | 52.3 nm | 910.7 nm |
| 3 | --- | --- nm | --- nm | --- nm |
| Total | 1.00 | 90.7 nm | 123.4 nm | 32.9 nm |

**Histogram Operations**

| | |
|---|---|
| Size (Median) | : 47.9 nm |
| % Cumulative (2) | : 10.0 (%) - 20.8 (nm) |
| % Cumulative (6) | : 50.0 (%) - 47.9 (nm) |
| % Cumulative (10) | : 90.0 (%) - 199.2 (nm) |
| Mean | : 90.7 nm |
| Variance | : 15236.9 $nm^2$ |
| S. D. | : 123.4 nm |
| S.P.Area | : --- |

**Cumulant Operations**

| | |
|---|---|
| Z-Average | : 0.1 nm |
| PI | : 0.304 |

The size distribution of the nanopowders in this present study is estimated with nanoparticle size analyser (SZ 100). The size distribution of nanoparticles is estimated from Fig. 3, Table 1 & is around 47.9 nm. Fig. 4 shows TEM micrograph of CuO nanoparticles. The actual size of nanoparticles is estimated from TEM micrograph. Most of the nanoparticles have size around less than 50 nm and which is in correlation with the particle size analyzer data. The TEM graph is also showed that the copper oxide nanoparticles are spherical in shape and nearly uniform in size.

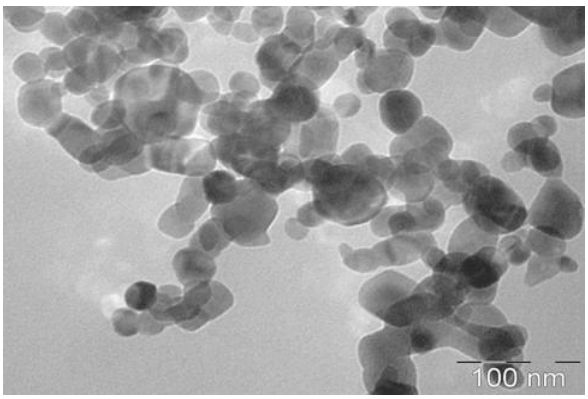


**Fig. 4** – Transmission electron microscope image of copper oxide nanopowder

### 3.4 Calculation of Zeta Potential

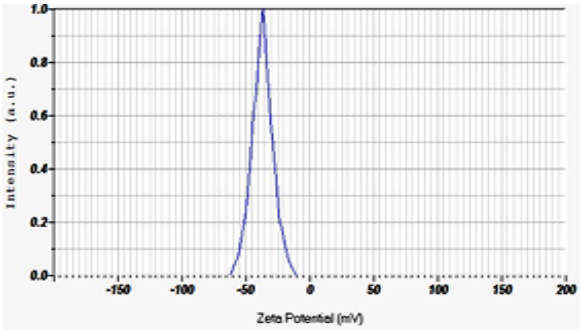


**Fig. 5** – Zeta potential of CuO nanoparticles

**Table 2** – Data of zeta potential of CuO nanoparticles

**201202241142000.nzt**

**Measurement Results**

| | |
|---|---|
| Date | : Friday, February 24, 2012 11:42:39 AM |
| Measurement Type | : Zeta Potential |
| Sample Name | : CuO Zeta |
| Temperature of the Holder | : 25.0 °C |
| Dispersion Medium Viscosity | : 0.895 mPa·s |
| Conductivity | : 0.074 mS/cm |
| Electrode Voltage | : 3.8 V |

**Calculation Results**

| Peak No. | Zeta Potential | Electrophoretic Mobility |
|---|---|---|
| 1 | -36.8 mV | -0.000285 cm²/Vs |
| 2 | — mV | — cm²/Vs |
| 3 | — mV | — cm²/Vs |

| | |
|---|---|
| Zeta Potential (Mean) | : -36.8 mV |
| Electrophoretic Mobility Mean | : -0.000285 cm²/Vs |

The Figure 5 and Table 2 show the variation of Zeta Potential with intensity. Fig.5 shows maximum at – 36.8 mV. The negative value of zeta potential suggests that the copper oxide particles are negative particles and moderately stable [14]. The negative nature of zeta potential prevents the particle coming together. Due to that nature there is a good dispersion stability in CuO nanoparticles and powder attains stable condition. Good dispersion and stability made CuO nanoparticles as good catalyst in chemical reactions. The catalytic nature of CuO is also tested in some chemical reactions and it proved as good catalyst [15].

### 3.5 Optical properties:

The optical analysis was done with UV-Vis spectrophotometer. Fig. 6 shows the wavelength vs. absorption. CuO nanoparticles showed good absorption at wavelength 472 nm. The energy gap estimated from absorption studies is 2.6 eV and in close agreement with literature values.

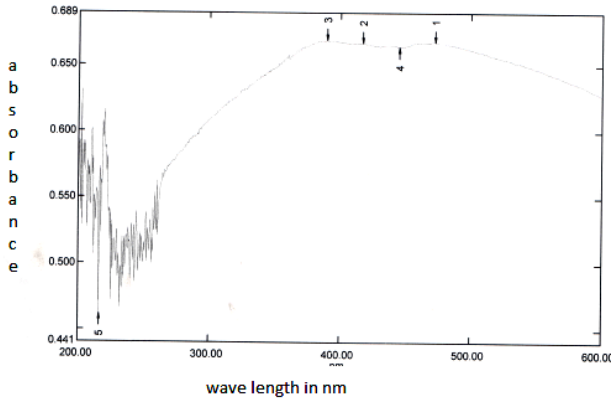


**Fig. 6** – UV-Vis spectrum of CuO nanopowder

### 3.6 Morphology studies

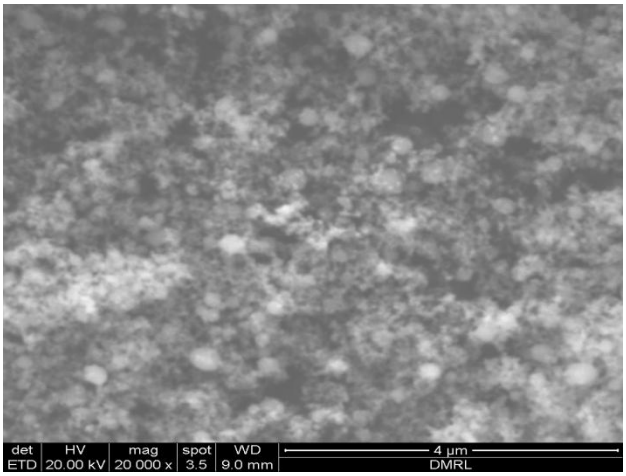


**Fig. 7** – SEM image of copper oxide nanoparticles

Fig. 7 represents SEM image of CuO nanoparticles prepared by sol-gel method. SEM data revealed that the tendency of agglomerations of CuO nanoparticles.

## 4. CONCLUSIONS

Structural analysis revealed that the copper oxide nanoparticles are monoclinic in nature. Stain of the prepared CuO nanoparticles calculations concluded that the small particles have high strain and big particles have low strain. EDAX analysis shows that the copper oxide nanoparticles are pure, free from impurities and nearly stoichiometric. The average particle size determined from Particle Analyzer is given by 47.6 nm. TEM image of CuO nanoparticles are spherical in nature and size of the CuO is also in good agreement with particle analyzer data. Zeta Potential represents good dispersion and stability nature of CuO nanoparticles. The absorption wave length of CuO nanopowder shows the blue shift region and there is enhancement in the b band gap value of CuO nanoparticles when compared with bulk copper oxide. SEM micrograph represents agglomeration of some of CuO nanoparticles.

## REFERENCES


1. P. Poizot, S. Laruelle, S. Grugeon, L. Dupontl, J.M. Tarascon, *Nature* **407**, 496 (2000).
2. V.R. Katti, A.K. Debnath, K.P. Muthe, M. Kaur, A.K. Dusa, S.C. Gadkari, *Sensor. Actuat. B: Chem.* **96**, 245 (2003).
3. H.M. Fan, L.T. Yang, W.S. Hua, X.F. Wu, Z.Y. Wu, S.S. Xie, *Nanotechnology* **15**, 37 (2004).
4. C.T. Hsieh, J.M. Chen, H.H. Lin, H.C. Shih, *Appl. Phys. Lett.* **83**, 3383 (2003).
5. M.K. Wu, J.R. Ashburn, C.J. Torng, P.H. Hor, R.L. Meng, L. Gao, *Phys. Rev. Lett.* **58**, 908 (1987).
6. M.H. Chang, H.S.Liu, C.T. Tai, *Powder Technol.* **207**, 378 (2011).
7. C.L. Carnes, K.J. Kalbunde, *J. Mol. Catal. A: Chem.* **194**, 227 (2003).
8. G.G. Condorolli, G. Malandrino, I.L. Fragala, *Chem. Vapor Depos.* **5**, 237 (1999).
9. R. Vijay Kumar, R. Elgamiel, Y. Diamanty, A.G. Edanken, *Langmuir* **17**, 1406 (2001).
10. A.A. Eliseev, A.V. Lukashin, A.A. Vertegel, L.I. Heifets, A.I. Zhirov, Y.D. Treytyakov, *Mater. Res Innov.* **3**, 308 (2000).
11. D.Z. Jia, J.Q. Yu, X. Xia, *Chinese Sci. Bull.* **43**,571 (1998).
12. K. Brgohain, J.B. Sing, M.V.R. Rao, T. Shripathi, S. Mahumani, *Phys. Rev. B* **61**, 11093 (2000).
13. A.L. Ortiz, L. Shaw, *Acta Mater.* **52**, 2185 (2004).
14. D.W. Fuerstenau, Pradip, *Adv. Colloid Interfac.* **114**, 9 (2005).
15. M. Nagaraju, K. Mukkanti, R. BuchiReddy, Y. Aparna, *NCONSEA* (*National Conference on Nano Science Nano Engineering and Applications*), op025,31 (2012).