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# CuO Nanoparticls Thermally Synthesized From a Since-Wheel Copper(II) Complex

**Manel TAFERGUENNIT**
University of Sciences and Technology Houari Boumediene

**Noura KICHOU**
Mouloud Mammeri University of Tizi-Ouzou

**Nabila GUECHTOULI**
Mouloud Mammeri University of Tizi-Ouzou

**Zakia HANK**
University of Sciences and Technology Houari Boumediene

**Abstract**: The broad properties of nanostructured materials based on transition metal oxides have received great attention from researchers. Copper (II) Oxide (CuO) due to its excellent physical and chemical properties, has numerous potential applications. Among the different methods previously reported to prepare CuO nanoparticles (CuO NPs) such as sonochemical, sol-gel, and electrochemical techniques, this study proposes an easeful, simple, and cost-effective method to synthesize CuO NPs by thermal decomposition of a coordination complex i.e., a binuclear copper (II) carboxylate complex.

**Keywords:** Nanostructured materials, Combustion, Copper oxide, XRD, SEM.

## Introduction

Copper acetate (Figure 1) is probably the first binuclear metal complex in human hands and one of the best-known metal complexes in the world, and has been the subject of research by numerous researchers. Four acetate ions bridged two copper(II) ions and two water molecules were axially coordinated at both ends. The absence of Cu-Cu bonding results in a square pyramidal configuration around the copper (Mikuriya, 2021). Such a cage-shaped binuclear cluster is not only found in copper acetate, but almost all carboxylate leading to thousands of studies on copper carboxylates.

The results of thousands of studies on Binuclear copper carboxylate have been published (Mikuriya, 2021)**.** On an author hand, there has been an increasing interest in the use of these complexes to get well dispersed copper oxide nanoparticles (CuO NPs) by thermal decomposition(Nordin,2015).Among the different methods previously reported to prepare CuO NPs such as sonochemical, sol-gel, and electrochemical techniques. Thermal decomposition is simple, rapid, and cost-effective. However, the major challenge with this method is the accurate control of specific uniform morphologie for CuO nanostructures (Bhattacharjee, 2021).

In view of the above information, a copper–carboxylate complexwas synthesized in green conditions. Then, CuO NPs were prepared from the complexusing the combustion method. The two compounds were subject of various characterization techniques characterized, including SEM, EDX, UV-Visible, IR analysis and further powder XRD.

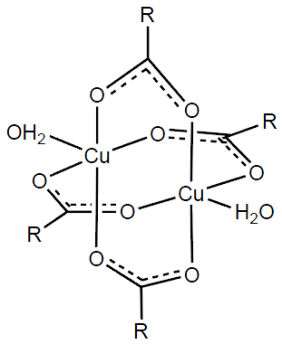


Figure 1. Representation of copper acetate complex.

## Synthesis and Characterization

### Synthesis of $[Cu_2(S)_4(H_2O)_2]$

The present copper (II) carboxylate$[Cu_2(S)_4(H_2O)_2]$was synthetizedby direct synthesis method in aqueous solution.

### Characterization of $[Cu_2(S)_4(H_2O)_2]$

**Scanning Electron Micrographs (SEM) and Energy-Dispersive X-ray (EDX) Spectroscopy:**

The determination of C, O, Cu contents in the prepared complex was conducted with a scanning electron microscope coupled with energy-dispersive X-ray (EDX) spectroscopy, the parameters were set at 10 A voltage and an acceleration of 5.0 kV. The dried sample were placed on a brass holder under vacuum, The EDX analysis was performed at many zones on the surface of the complex in order to obtain relevant chemical contents. The results are shown in Figure 2.

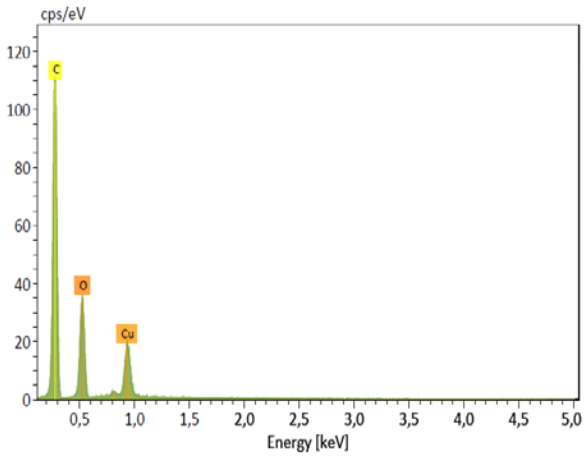


Figure 2. EDX analysis performed on the red zone on the surface of the complex

The atomic percentage of Cu, O and C elements obtained from EDX analysis were compared to those of the proposed structure and depicted in Table 1. As it is clear from Table 1, EDX analysis values support the formula of the complex$[Cu_2(S)_4(H_2O)_2]$.Furthermore, the calculated fraction of O/Cu is a clear evidence that the copper ion is coordinated to 8 oxygens, which is the equivalent of 4 ligand S, as it can be seen from Figure 3 and 4.

Table 1. Chemical contents data.

| Empirical formula | Dispersive X-Ray Analysis Atom % found (calc.%) | | |
|---|---|---|---|
| | C | O | Cu |
| $Cu_2(S)_4(H_2O)_2$ | 69,31(70.58) | 24.39(23.52 | 6,29(5.88) |

**Fourier Transform Infrared Spectroscopy:**

The Fourier Transform Infrared Spectroscopy, also known as FTIR Analysis was used to confirm the existence of the main structural groups in the prepared complex. The FTIR spectra of both the ligand and its copper complex are illustrated in Figure 3, and the main frequencies are assigned in Table 2.

Table 2. FTIR data.

| Assignments | Observedfrequencies ($cm^{-1}$) | |
|---|---|---|
| | ligand | Cu-ligand |
| ν OH | -- | 3450 |
| $\nu_{asym}COO$ | 1555[1], | 1561 |
| $\nu_{sym}COO$ | 1387 [1], | 1411 |
| ν(Cu–O–H) | -- | 869 |
| ν(Cu–O) | -- | 458[5] |

ν: stretching, sym symmetric, asym: asymmetric

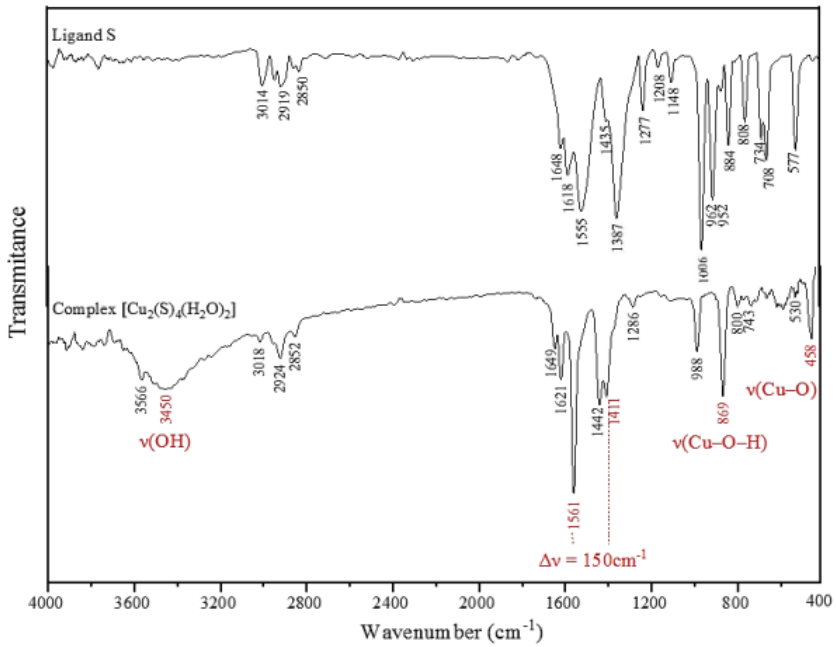


Figure 3. FTIR spectra.

**Synthesis of CuONPs**

Combustion of the copper complex$[Cu_2(S)_4(H_2O)_2]$ wasperfomed in a furnace from the room temperature to 400°C, it gave rise to a black powder predicted to be copper oxide CuO, according to the following proposed combustionequation (I):

$$Cu_2(C_5H_7COO)_4(H_2O)_{2(s)} + 28O_{2(g)} \rightarrow 2CuO + 16H_2O_{(g)} + 24CO_{2(g)} \ldots (I)$$

**Characterization of CuO NPs**

**XRD Powder and Crystal Structure**

The structure and phase composition ofsynthetized product was examined by X-ray powder diffraction. The XRD pattern (Figure 4) is well matched with the monoclinic phase of CuO (tenorite) nanoparticles and well consistent with the COD card (no card : 96-901-5925).

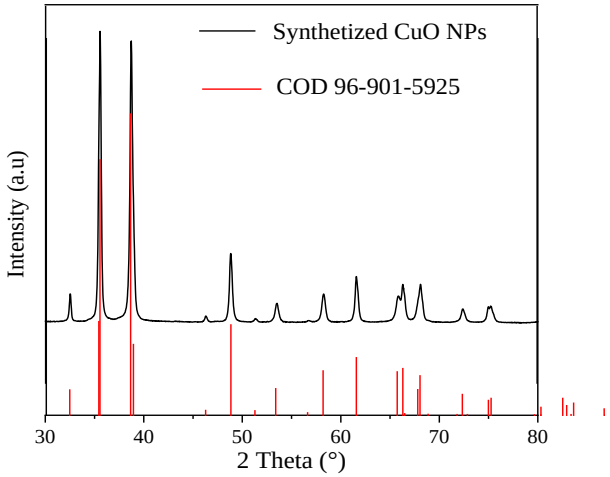


Figure 4. XRD pattern of CuO NPs.

Moreover, the crystallite size was calculated by using Debye Scherrer equation (II) [6]:

$$D = 0.9\lambda / \beta Cos\theta \ldots (II)$$

Where λ is the X-ray wave length, β is the line broadening at half the maximum intensity in radians, θ is the Bragg angle.The average size of the synthetized CuO NPs crystallites is estimated to be equal to 22nm.

**Scanning Electron Micrographs (SEM) and Energy-Dispersive X-ray (EDX) Spectroscopy:**

The surface morphology and the chemical composition of the prepared CuO nanoparticles were revealed through the SEM and EDX analysis (Figure 5and 6), the parameters were set at 10 A voltage and an acceleration of 5.0 KV, As shown from Figure 5, the surface of the CuO displays a homogeneousdistribution of nonmetric particles with a size of 100 nm. Furthermore, the EDX spectrum supports the existence of CuO.

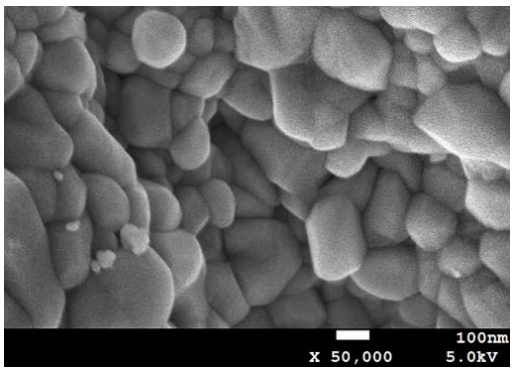


Figure 5. SEM image

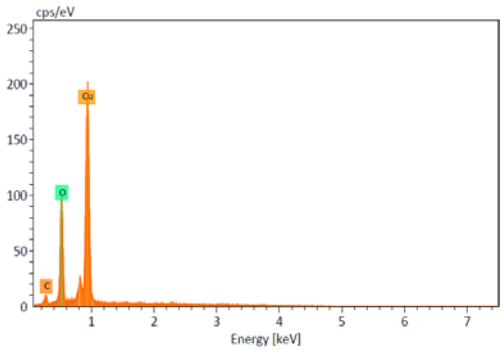


Figure 6. DX spectrum of CuO NP

**Optical Absorption Analysis**

The band gap value of the synthetized CuO NPs was analyziedbu UV-Visible spectroscopy, the absorption data were extracted using Tauc's plot method.

The method consist of tracing the absorption coefficient $(\alpha h\nu)^n = f(h\nu)$. Indeed, the relation of the absorption coefficient (α) to the incidental photon energy (hv) depends on the type of electronic transitions. When in this transition, the electron momentum is conserved, the transition is direct, but if the momentum does not conserve this transition is indirect (Radhakrishnan, 2014).

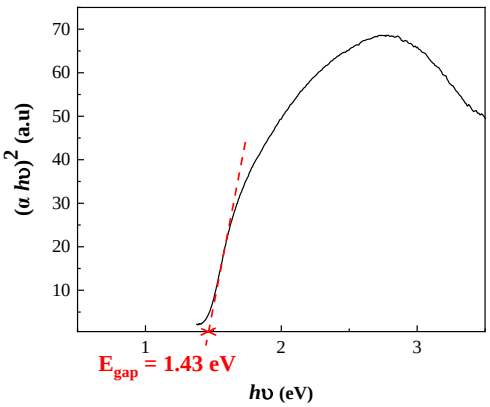


Figure 7**:**Tauc's plot for direct electronic transition

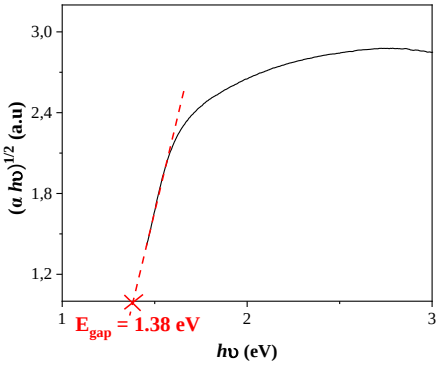


Figure 8. Tauc's plot for indirect electronic transition

By extrapolating the straight line portion of the $(\alpha hv)^{1/2}$ versus (hv) graphs to the (hv) axis until $(\alpha hv)^{1/2}$ =0. Optical analysis shows that the direct band of CuO NPs is located at 1.43eV(Figure 7), While, the indirect band gap of CuOnano particles is at 1.38 eV(Figure 8).This result is similar withprevious reports made on CuO nanoparticles (Radhakrishnan, 2014).

## Conclusion

Nanosized CuO particles were successfully synthetized using a binuclear copper carboxylate complex. The CuO NPs was characterized using several techniques. The phase composition was done using XRD analysis. The morpholy of the prepared CuO revealed homogenic nanoparticles sized. And furthermore, the absorption analysis displayed that the $E_{gap}$ value of the prepared CuO NPs is equal to 1.37 eV. Indicating that the nanoparticles could be used in semiconductor devices.

## Scientific Ethics Declaration

The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

## Acknowledgements or Notes

* This article was presented as an oral presentation at the International Conference on Basic Sciences and Technology ( www.icbast.net ) held in Antalya/Turkey on November 16-19, 2022.

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## Author Information

**Noura Kichou**
Mouloud Mammeri University of Tizi-Ouzou
Tizi-Ouzou, Algeria.
Contact E-mail: *noura.kichou@ummto.dz*

**Nabila Guechtouli**
Mouloud Mammeri University of Tizi-Ouzou
Tizi-Ouzou, Algeria

**Manel Taferguennit**
University of Sciences and Technology Houari Boumediene
Bab Ezour Algeria

**Zakia Hank**
University of Sciences and Technology Houari Boumediene
Bab Ezour Algeria

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