



Original Research article

Rapid Synthesis of a Nano-sized Copper(II)Oxide by Calcination of the Cu(II)Schiff base Complex

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ABSTRACT

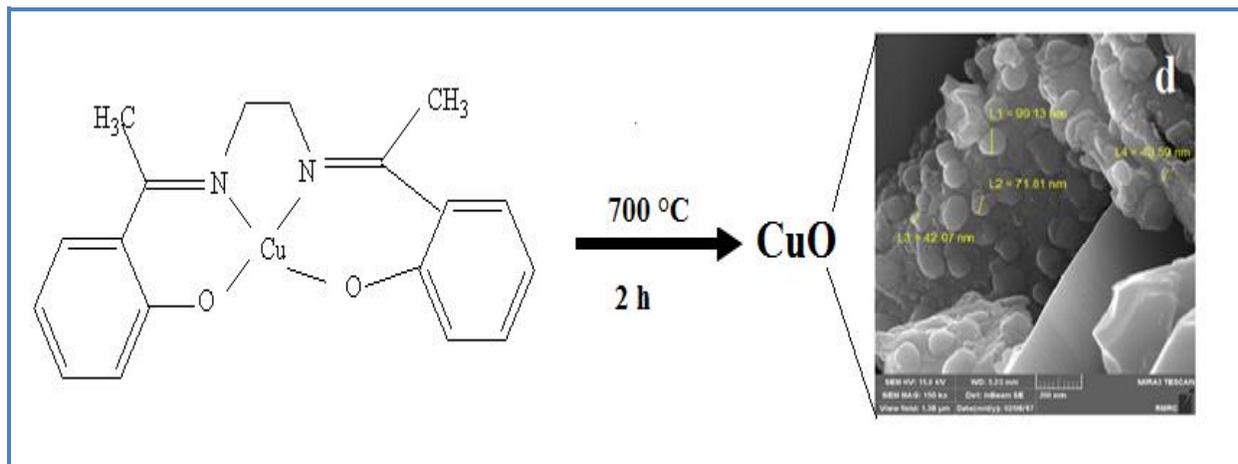
In this study, the Cu Schiff base complex was successfully synthesized through a facile and rapid method. Then, the complex was calcinated at 700 °C for 2 h under air atmosphere leading to CuO nanoparticles. The synthesized CuO nanoparticles were characterized by FT-infrared spectroscopy (FT-IR), scanning electron microscopy (SEM), X-ray diffraction (XRD), and energy dispersive spectroscopy (EDS). The XRD analysis confirmed that the samples were crystallized in the monoclinic CuO phase. The average crystallite size of the nano-sized of CuO nanoparticles was obtained at about 68 nm as determined by Debye- Scherrer equation from the data of X-ray diffraction.

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Graphical Abstract



Introduction

Schiff base complexes have a remarkable effect as catalysts in oxidation reactions such as oxidation of alkenes, styrene oxidation, and oxidation of sulfides to sulfoxide. They also are important because of many features and abundant applications. Schiff base complexes could be easily synthesized leading to various structures and useful activities especially biological ones [1,2]. In addition, they have various applications in medicine such as antimicrobial, anti-tumor, antiviral, sedative, anti-cancer and antioxidant compounds. Furthermore, these compounds show significant biological activities because of their metal complexation ability[3].

Among Schiff base complexes, copper Schiff base complexes have attracted considerable attention for their applications [4,5]. Copper Schiff base complexes have been synthesized to study the non-enzymatic oxygenation reactions and also to understand the oxygenate-catalyzed reasons [6].

Moreover, metal oxide nanoparticles have particular importance because of the fact that the reduction in particle size to nanometer scale results in fascinating physical and chemical properties which are different from the bulk analogues. In addition, nanodimensions cause changes in electronic structure of materials that show fascinating physical and chemical properties which are in turn different from the bulk materials such as the mechanical, optical, and magnetic properties[7, 8].

Metal oxides, due to low toxicity, high chemical / thermal stability and easy synthesis, play an important role in many applied technological fields such as the manufacture of microelectronic circuits, sensors, piezoelectric devices, fuel cells, surface coatings Corrosion and as the catalyst [9, 10].

During recent years, synthesis of materials in nanoscale has been increased based on the fact that the reduction in particle size to nanometer scale results in high surface to volume ratio. Accordingly, we wish to report on the preparation of a new nano copper Schiff base complexes, where the ligand is 2-((E)-1-(2-((E)-1phenylethylideneamino)ethylimino)ethyl) phenol. Furthermore, the copper (II) complex is used as a precursor to produce pure CuO nanoparticles using thermal decomposition method.

The structure and morphology of synthesized nanoparticles were investigated by the means of Fourier transformed infra-red (FT-IR), field emission scanning electron microscopy (FESEM), X-ray diffraction (XRD), and energy dispersive spectroscopy (EDS).

Material and methods

Materials and instrumentation

All the chemical reagents used in experiments were of spectroscopic grade and used as received without further purification. Fourier transform infrared (FT-IR) spectra were recorded on a Shimadzu system FT-IR 8400 spectrophotometer using KBr pellets. Product X-ray diffraction (XRD) data were recorded by a Rigaku D-max C III and X-ray diffractometer using Ni-filtered Cu K_{α} radiation. The sample was characterized with a field emission scanning electron microscope (FESEM) (Hitachi S-4160). Energy dispersive spectroscopy (EDS) was recorded on Oxford instrument.

Synthesis of Cu Schiff base complex

CuCl₂ (5 mmol, 0.85g) was added drop-wise to a methanolic solution of 2-((E)-1-(2-((E)-1phenylethylideneamino)ethylimino)ethyl) phenol (5 mmol, 1.489g) with constant stirring. The mixture was then refluxed for 3 h resulted in a deep green precipitate which was then separated by filtering, washed with cold methanol and dried at 100°C for 2 hours. The structure of the complex was confirmed by some common spectroscopic methods. Figure 1 shows the synthesized copper Schiff base complex.

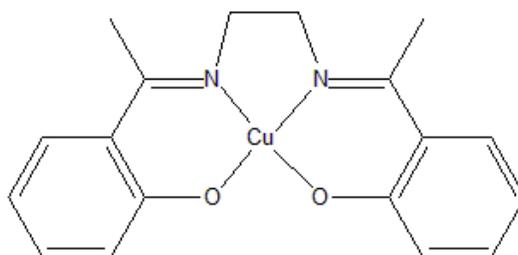


Figure 1. The synthesized Cu(II) Schiff base complex

Synthesis of CuO nanoparticles

The title complex was calcinated at 700 °C for 2 h under air atmosphere leading to nanosized CuO and then its structure was characterized by some spectroscopic methods such as: XRD, SEM, FT-IR and EDS.

Result and Discussion

IR study

The infrared spectra of the copper Schiff base complex and CuO nanoparticles are shown in Figure 2. For copper Schiff base complex the strongest bonds in 1579 cm^{-1} are assigned to the vibrations of azomethine groups $\nu_{(\text{C}=\text{N})}$. The bands at 2962 and 3038 cm^{-1} were assigned to the aliphatic C-H bonds of the azomethine group. In addition, the Cu-N bands of copper were also predicted by the bands observed at 659 cm^{-1} where the Cu-O band of the copper complex was also predicted by the bands observed at 690 cm^{-1} [11].

Figure 2**(b)** represents the FTIR spectrum recorded for the CuO nanoparticles in the range of 500 to 3500 cm^{-1} . The characteristic bands observed at 426 cm^{-1} , 507 cm^{-1} , and 619 cm^{-1} can be assigned to the A_u mode, B_u mode, and the other B_u mode of CuO [12]. The high-frequency mode at 619 cm^{-1} may be attributed to the Cu-O stretching along the (-101) direction, while the peak at 507 cm^{-1} can be assigned to the Cu-O stretching vibration along the (101) direction [13]. Moreover, no other IR active mode was observed in the range of 619 to 700 cm^{-1} which totally rules out the existence of another phase, i.e., Cu_2O . The broad absorption peak at around 3400- 3500 cm^{-1} is caused by the adsorbed water molecules since the nanocrystalline materials exhibit a high surface to volume ratio and thus absorb moisture.

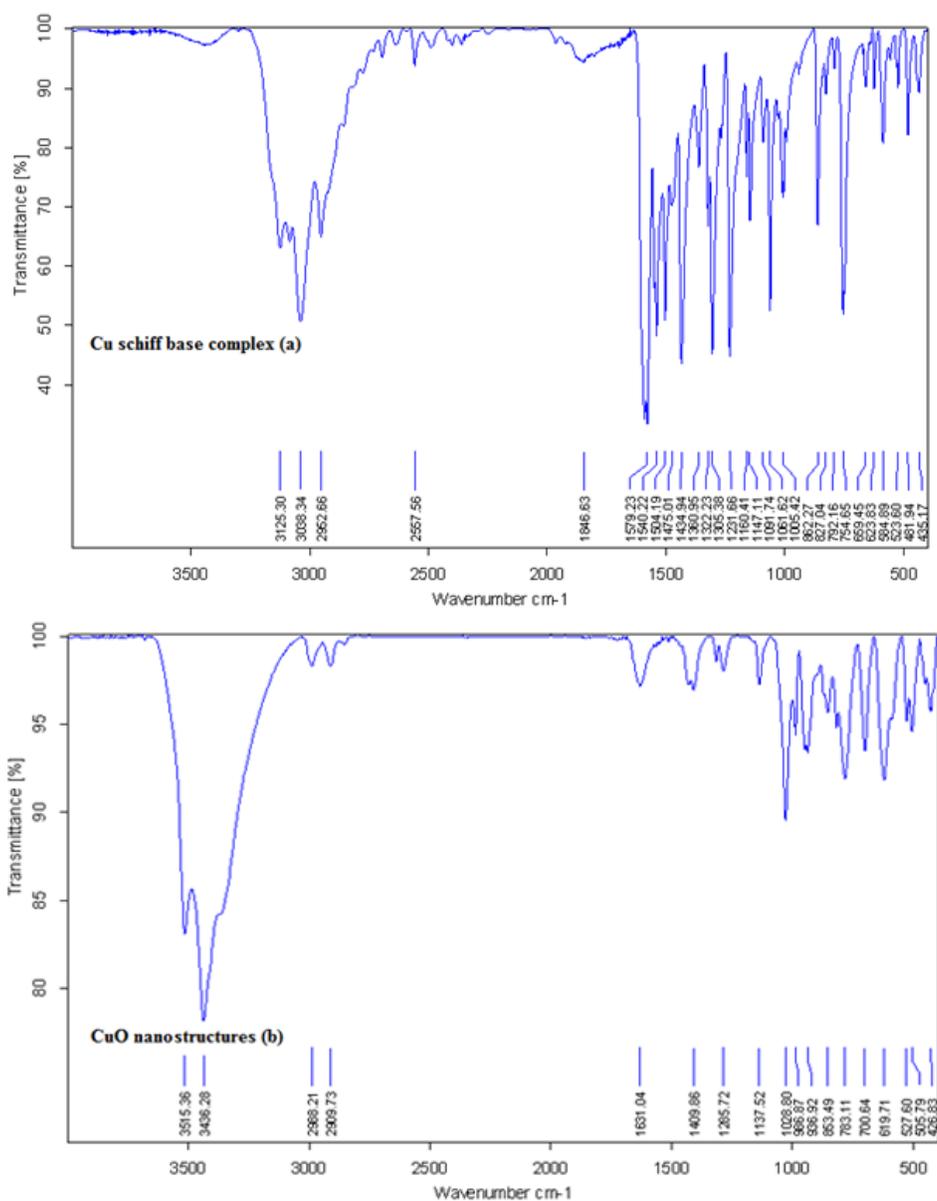


Figure 2. Infrared spectra of (a) copper (II) Schiff base complex and (b) CuO nanoparticles

XRD analysis

Figure 3 shows the XRD pattern of the as-prepared CuO nanoparticles. All the peaks of the prepared CuO can be assigned to monoclinic symmetry of CuO (space group $C2/c$; JCPDS Card no. 80-1916). 15 No other peaks are observed indicating the high purity of the as-prepared samples. The broad XRD peaks also indicate that the sample is composed of CuO nanocrystallites.

The average particles size were calculated 33.07 by applying a full width at half maximum (FWHM) and the value of 2θ of the characteristic peak of the XRD pattern using the Debye– Scherrer equation [14].

$$D = k\lambda/\beta\cos\theta$$

Where D is the average size, K is a constant (ca. 0.9), k is the wavelength of Cu K_{α} , β is the full width at half maximum (FWHM) of the diffraction peak and θ is the Bragg' angel.

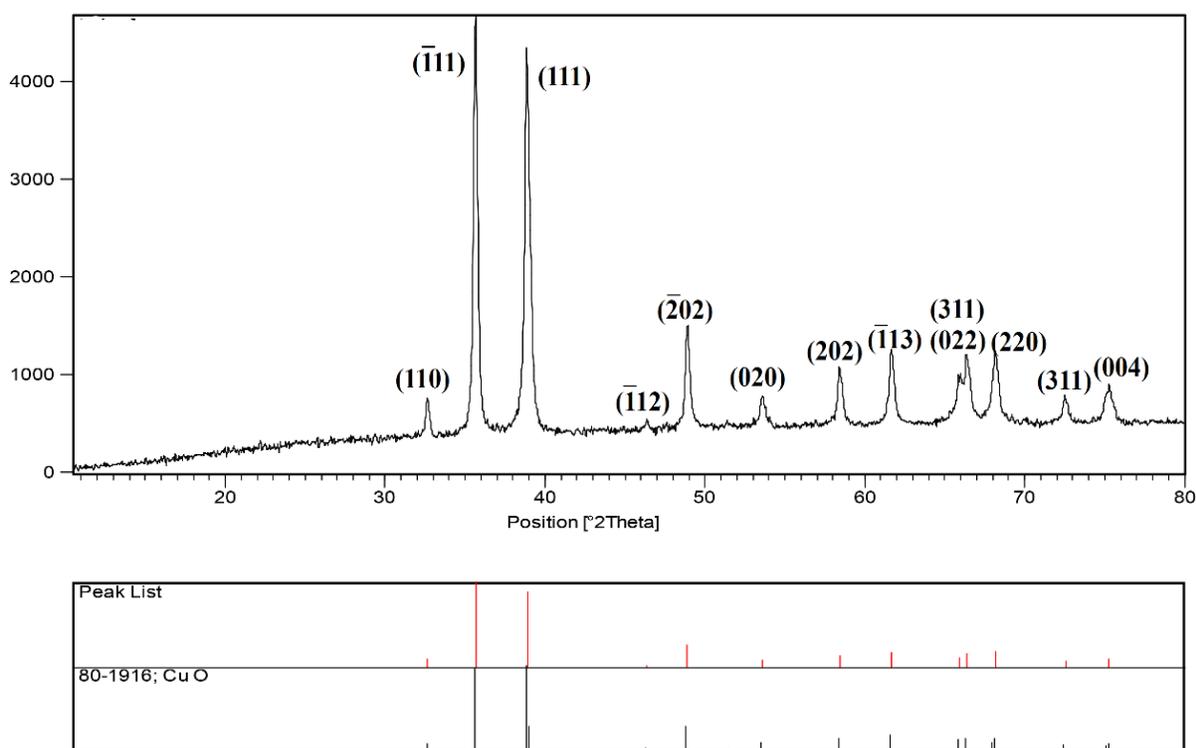


Figure 3. The XRD pattern of the prepared CuO nanoparticles

FESEM micrographs

The surface morphology and element distribution of the CuO nanoparticles were revealed through the FESEM and map line image shown in Figure 4. SEM map lining of as-synthesized material shows a homogeneous distribution of spherical particles of the prepared CuO nanoparticles. In Figure 4(a) and (b) map line of oxygen and Cu elements are demonstrated respectively by the yellow and red colors and Figure 3 (c) shows the map line analysis for CuO nanoparticles. FESEM of synthesized nano particles is shown on Figure 4(d); as can be seen, nanoparticles are synthesized properly.

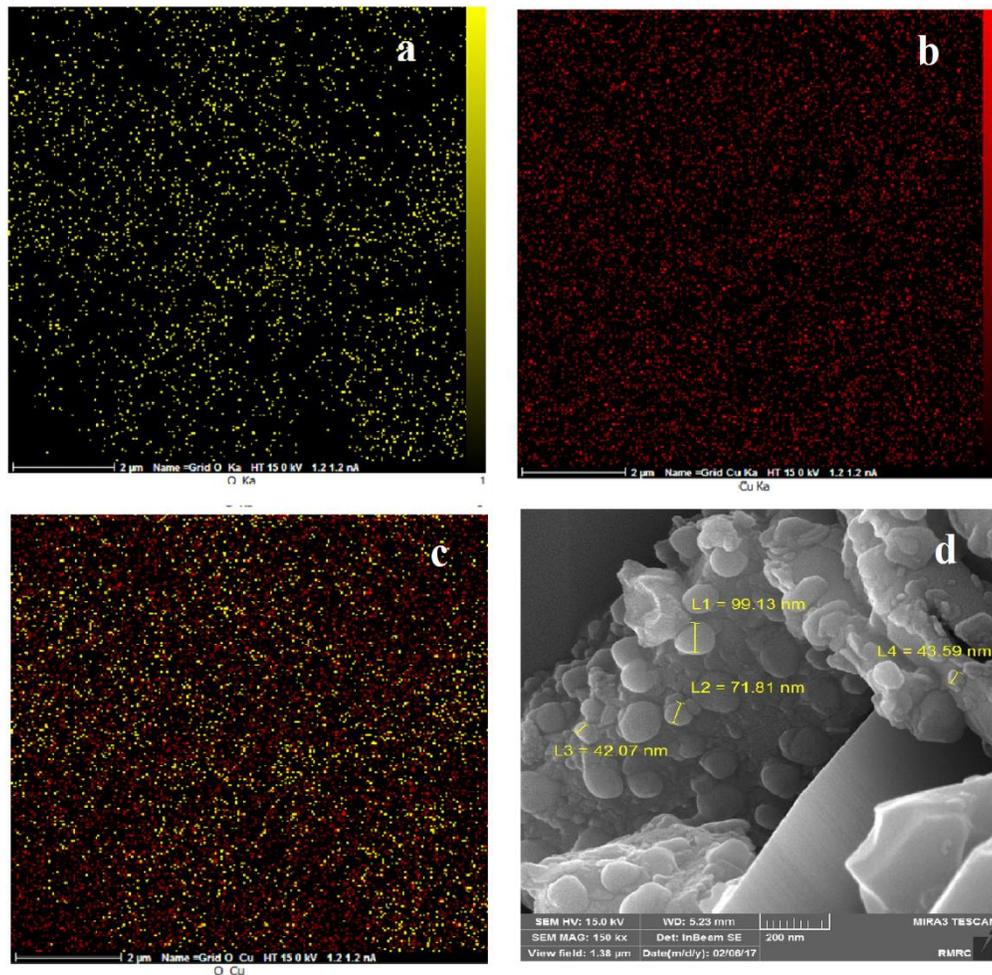


Figure 4. Map line analysis (a) oxygen signal; (b) copper signal; and (c) reveal the chemical composition of both elements Cu, and O (d) FESEM image of the CuO nano particles.

EDS analysis

Figure 5. shows EDS of as-synthesized CuO nanoparticles. As can be seen, EDS spectrum results confirm the presence of Cu and O in the synthesized nanostructure. It is clearly displayed that no other peak related to any impurity has been detected in the EDS. Accordingly, it confirms that the nanoparticles are pure. The EDS results of the synthesized nanoparticles are reported on Figure 5.

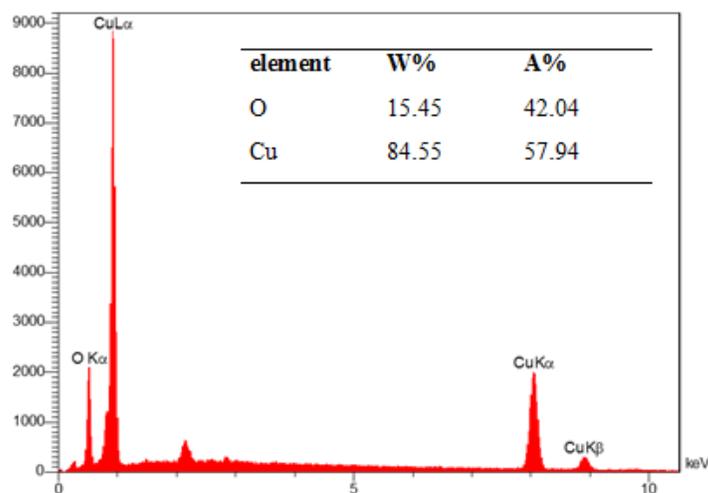


Figure 5. The EDS spectra of as-synthesized CuO nanoparticles

Conclusion

CuO nanoparticles were synthesized via a simple method by thermal decomposition of Cu(II) Schiff base complex. The nanoparticles were characterized by FT-IR, XRD, SEM, and EDS techniques. XRD analysis confirmed that the samples were crystallized in monoclinic CuO phase. Using Scherer equation, the size of nanostructures was estimated about 68 nm. SEM images showed that the morphology of particles is sphere, and the size of the particles is within the nanoscale.

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