Synthesis, Spectroscopic Investigation and Biological Activities of Synthesized Copper and its Oxide Nanoparticles

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Abstract : Among the various metal nanoparticles, copper (Cu) and its oxide nanoparticles have attracted considerable attention because copper is one of the most important metals in modern technology and is readily available. Copper nanoparticles have been much attractive because these are easily available, cost effective and have conducting nature. Copper Nano particles were synthesized using Copper(II) maleate by microwave synthesis. Conventional method. Copper Nanoparticles so obtained were characterized by UV- Visible spectroscopy, Fourier Transform Infrared Spectroscopy and X-Ray diffraction analysis. X-Ray diffraction analysis proved the formation of Copper Nano particles. SEM and AFM analyses showed the presence of nanoparticles. The Copper Nano particles thus formed were subjected to anti Microbial activity.

Keywords: Copper Sulfate, UV –Visible, XRD, FT-IR and Antimicrobial activity.

Key words: Copper nano particles; biological activities;

I. INTRODUCTION

Recently, research has been directed towards the synthesis and application of metal nanoparticles in view of their unique properties compared to the bulk metals.[1, 2] Among various metal nanoparticles, copper and gold nanoparticles have received considerable attention because of their unusual properties and potential applications in diverse fields. [3] The various synthetic procedures for their synthesis include micro-emulsion,[4] reverse micelles,[5] reduction of aqueous copper salts,[6] UV light irradiation,[7] physical vapor deposition,[8] and impregnation,[9] microwave heating,[10],[11] . The core shell particles are of great interest due to their potential applications in diverse fields including catalysis, drug delivery, photonics, sensors, etc.[12] Microwave heating has been actively developed recently because of being simple to organize and economically efficient, and because of allowing easy control and variation of nanoparticles sizes and shapes. The basic drawback of this method is the broad nanoparticles size distributions. Limited information on the possible antimicrobial activity of copper and its oxide is available. Copper and its oxide are cheaper than silver, easily mixed with polymers and relatively stable in terms of both chemical and physical properties. Highly ionic nano particulate metal oxides, such as copper and its oxide may be particularly valuable antimicrobial agents as they can be prepared with extremely high surface areas and unusual crystal morphologies.[13] The aims of this study were to characterize copper and its oxide nanoparticles physically and chemically and to investigate this compound with respect to its potential antimicrobial applications.[14]

II. MATERIALS METHODS

All the chemicals, reagents used in the experiments were of analytical grade and were used as received without further purification. Succinic acid, Copper sulphate and NaOH were purchased from Aldrich and were used as received.

A. Synthesis of the Copper(II) maleate precursor

The $CuSO_4.5H_2O$ (2 mmol) was dissolved in 10 mL of distilled water to form a homogeneous solution. A stoichiometric amount of sodium hydroxide (NaOH) and maleic acid were dissolved in distilled water. The sodium maleate thus formed was drop wise added into the above solution under constant stirring.

The solution was stirred for about 30 min and a blue precipitate was obtained which was centrifuged and washed with ethanol several times. The product was dried. The Copper(II) maleate was characterized by various spectral techniques.

B. Synthesis of the Copper nanoparticles

Copper and its oxide nanoparticles were synthesized by a domestic microwave method in the presence of Copper(II) maleate. The Copper(II) maleate was placed in a domestic microwave oven . The reaction system was heat treated at 473K for 2 h. Copper and its oxide nanoparticles were obtained as brown powder.

III. RESULTS AND DISCUSSION

A.UV Visible Spectra

Copper nanoparticles typically absorbed around 650 nm. However the Copper nanoparticles synthesized here show an absorption peak around 653 nm (Figure.1). This peak can be assigned to the absorption of nanoparticles of copper. [15-18]

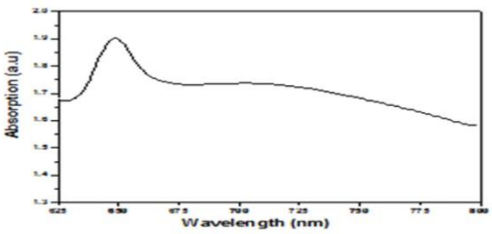

Fig. 1. The UV–Vis absorption spectrum of the copper and its oxide nanoparticles.

B. IR measurement

The IR spectrum of Copper(II) maleate precursor is shown in the Figure 2a. The IR spectrum of nanoparticles is shown in Figure 2b. A twin peak at 621 cm^{-1} & 680 cm^{-1} indicated the Cu-O Stretching vibration. The metal salt (Cu-O-C) Peak appeared at 1192 cm^{-1}. A Peak at 3541 cm^{-1} indicates OH stretching of the water in the precursor which disappeared in the nanoparticles. In Figure.2a, peak at 1637 cm^{-1} indicates metal carbonyl (C=O) group reduced [19-21].

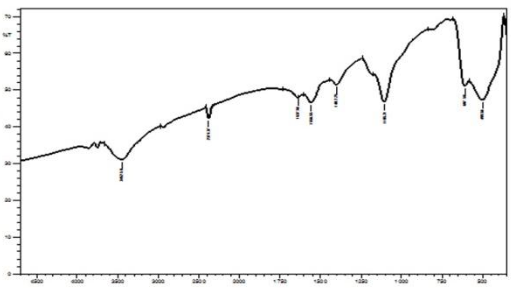

Fig. 2(a). FT-IR spectra of Copper(II) maleate Precursor.

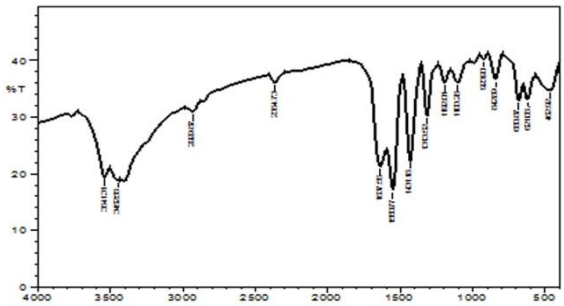

Fig. 2(b). FT-IR spectra of copper and its oxide nanoparticles.

C. XRD measurement

Powder diffraction analysis indicated that the product was copper and its oxides (Figure. 3). Particle size was predicted by using Debye Scherrer equation.

Average Crystalline Size

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

$\lambda = 1.5406 \times 10^{-10} m$

β = Full width at half maximum (radian) The Size of the nanoparticles is 45.07 nm.

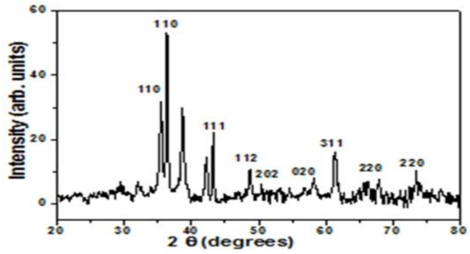

Fig. 3. XRD pattern of the copper and its oxide nanoparticles.

D. Cyclic Voltagramm (CV)

Figure. 4 shows the Cyclic Voltagramm (CV) of the copper nanoparticles. It was recorded in DMF with 0.1 M tetrabutylammonium perchlorate as supporting electrolyte in the potential range -2 to + 0.1 V, with a conventional three electrode system composed of a platinum auxiliary, glassy carbon working electrode and Calomel (Saturated KCl) as reference electrode. The reductive peaks correspond to Cu (II)/Cu (I) and Cu (I)/Cu (0).

$CuO \rightarrow Cu_2O \rightarrow Cu$.

The redox peak currents increase linearly with increase in the scan rate from 20 to 100 Mvs^{-1} (Figure 4). The oxidative peak corresponds to $Cu \rightarrow Cu_2O \rightarrow CuO$.

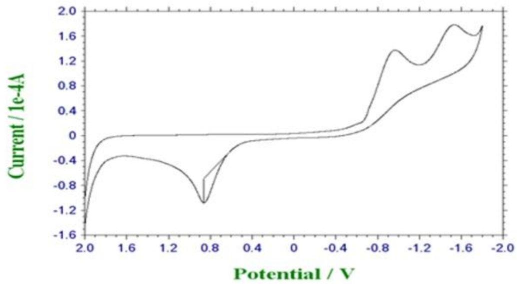

Fig. 4. C V spectrum of the copper and its oxide nanoparticles.

E. SEM

The morphology of the product was examined by SEM. Figure. 5 depict the SEM images of nanoparticles. It shows that the Copper and copper oxide nanoparticles are spherical in shape.[22]

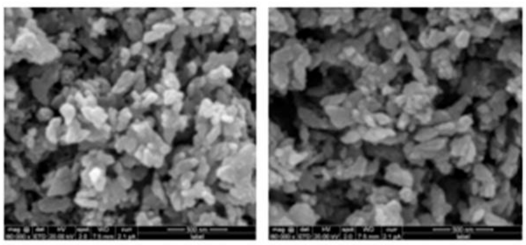

Fig. 5. SEM images of the copper and its oxide nanoparticles.

F. AFM

The Atomic force microscopic (AFM) photographs of the product are (cubic, fcc) given in Figure 6. The area roughness and RMS are in the range 160 to 487 nm which indicates its semiconducting nature. Copper and its oxide nanoparticles are spherical like and have loose solid morphologies.

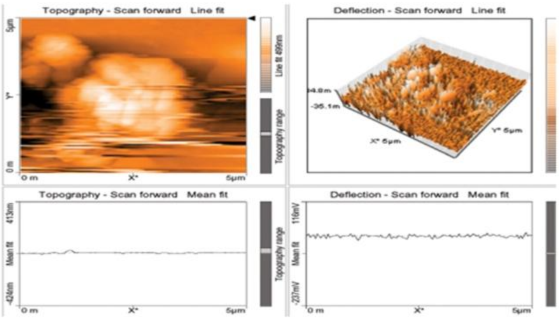

Fig. 6. The AFM images of copper and its oxide nanoparticles.

G. Antibacterial activity

All bacterial isolates were obtained from clinical samples (*Streptococcus pyogenes*, *Pseudomonas aeruginosa*, *Escherichia coli* and *Staphylococcus aureus*). The antimicrobial test was carried out primarily by the agar well diffusion method. Bacterial pathogenic inoculums were prepared from 18 h grown cultures (∼104−106cells/mL). Petridishes containing the bacterial inoculums on nutrient agar was used for the study. Using a cork borer (6 mm diameter), wells were made in the culture plates and 50 μ L of the synthesized nanoparticles was loaded. The plates were then incubated at 37° C for 24h. After incubation, the zone of inhibition (ZOI) was measured.[23]

Table 1. *In vitro* antimicrobial activity of some human pathogenic bacteria on nanoparticles by disc diffusion assay

S. No.	Test organism	Zone of inhibition (mm)		
		Nature of Bacteria	Copper (II) maleate Precursor	Copper nanoparticles [Microwave]
1.	Streptococcus pyogenes	Gram (+)	15mm	10mm
	Pseudomonas aeruginosa	Gram (-)	16mm	18mm
3.	Escherichia coli	Gram (-)	20mm	22mm
4.	Staphylococcus aureus	Gram (-)	20mm	21mm

IV. CONCLUSIONS

The green chemistry approach used in the present work for the synthesis of nanoparticles is simple, cost effective and the resultant nanoparticles are highly stable and reproducible. The overall antimicrobial activities of Copper(II) maleate are consistent It showed excellent activity against *Streptococcus pyogenes*, *Pseudomonas aeruginosa*, *Escherichia coli* and *Staphylococcus aureus*, with strong zone of inhibition. In the future, Copper and copper oxide nanoparticles could replace some antibiotic medicines used to combat human pathogenic microorganisms (bacteria), safe and cost effective in the Pharmaceutical industry. Copper and its oxide nanoparticles with a diameter of around 50 nm were obtained in the absence of any reducing agent.

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