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Antimicrobial Activities of Copper Oxide Nanoparticles using Ginger (*Zingiber Officinale* Rosc.) Rhizome Extracts and Spectroscopic Analysis

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***Abstract* - A novel copper oxide nanoparticles was synthesized using ginger (*Zingiber officinale* Rosc.) rhizome extract from copper complex containing Schiff base ligand (metal precursor) via calcination approach and applied for antibacterial activities against gram negative species. The structure of the nanoparticles has been examined by XRD, SEM, TEM and FT-IR spectroscopic techniques. X-ray diffraction reveals the size of nanoparticles. Antibacterial potential of the prepared nanoparticles were carried out by using gram negative E.Coli bacteria and shows good antibacterial potential.**

Keywords: Synthesis, Spectroscopy, Schiff base ligand, Copper complex, copper oxide nanoparticles and antibacterial activities.

I. INTRODUCTION

Nanotechnology is an interdisciplinary area of science which has been growing and developing rapidly across the world forming Nano revolution by producing Nano products and nanoparticles that can have novel and size related physicochemical properties differing significantly from larger matter. [1]. Nano particles have become important materials in modern technologies compared with their bulk analogues not only as a result of their excellent structural features but also as a result of their unusual attributes[2]. Nanostructure metal oxides have been attended in the field of nanotechnology both from a fundamental and industrial point of view. Metal oxides play a very important role in many areas of chemistry; physics and material science. The metal elements are able to form a large diversity of oxides [3]. Transition metal oxides and metals have been researched extensively due to their interesting catalytic, electronic and magnetic properties. Nanometer sized metal oxides and metals find wide applications in data storage devices, catalysis, drug delivery and biomedical imaging [4-5]. Presently several methods, viz. hydrothermal, thermal decomposition, sol–gel, solvo thermal so no chemical etc. are available to prepare nanoparticles.

However, thermal decomposition of transition metal is one of the simplest and less costly techniques for preparing Nano sized transition metal oxides [7]. Copper oxide is an important transition metal oxide with many practical applications, such as it is the basis of several high-Tc superconductors and materials with giant magneto resistance, and is also used as catalysts, pigment, p-type semiconductor, gas sensors, solar cells, magnetic storage media and cathode materials [8]. Zinc oxide is also a well-known semiconductor material with a wide band gap (3.37 eV) and a large exciton binding energy (60 meV) at room temperature. CuO nanoparticles have been extensively studied over the past decades because of their fascinating electrical, mechanical, optical, and piezoelectric properties. CuO nanoparticles have a wide range of applications such as gas sensors, dye-sensitized solar cells, ultra violet photo detectors, UV lasers, photocatalysts, piezoelectric transducers, and for biomedical applications [9]. Because of the practical reasons mentioned above, the synthesis of nano structured CuO has also attracted considerable attention. In this paper we described the synthesis and characterization of CuO nanoparticles by solid state thermal decomposition of metal complex containing Schiff base ligand.

II. EXPERIMENTAL

2.1 Synthesis of Schiff base ligand

1.07 ML (10.00mmol) of salicylaldehyde was refluxed with 1.10g (10.00mmol) of 4-Bromoaniline in ethanol for 2 hours to obtain and orange solution. The solution was reduced under suction to form and orange precipitate. The precipitate was filtered under suction, washed with ethanol and recrystallized from ethanol. It was dried over silica gel in desiccator [11].

2.2 Synthesis of Copper (II) complex

A copper acetate monohydrate $Cu(OAc)_2.H_2O$, (0.123 g, 0.617 mmol) was completely dissolved in ethanol (10 mL) and ethanolic solution of the synthesized ligand (0.30 g, 1.23

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mmol) was added drop-wise with vigorous stirring. A green precipitated of the product (86.9%) was form which was suction filtered, washed with ethanol and dried in a desiccator [12].

2.3 Preparation of CuO nanoparticles

To prepare CuO nanoparticles the prepared Schiff base – copper complex were transferred in to a porcelain crucible and placed in a muffle furnace, heated to700^{0}C for 3 hrs. To remove any impurities, the final products are washed with ethanol and dried at room temperature for 3 days.

2.4 Preparation of gingerLeaf Extract

The fresh leaves of ginger (*Zingiber Officinale* Rosc.) were obtained from a Digil Word, Mubi North Local Government Area of Adamawa State. The collected fresh leaves were washed thoroughly with deionized water to remove dirt particles if adsorbed on the surface of the leaves. The washed leaves were then air-dried at room temperature. The cleaned and dried leaves were ground into fine powder using an electrical blender followed by sieving using a 20 mesh sieve. In order to prepare a flavonoid-rich extract, 2 g of the dried leaf powder were extracted with 200 mL deionized water in a Soxhlet apparatus for 6 hours. The aqueous leaf extract were then stored at 5°C for further use [13]

2.5 Optimization of Copper oxide NPs

The optimization of CuO NPs was carried out at different volumes of leaf extract (1, 2, 3, 4 mL) at pH 8 and room temperature. The pH of the reaction was varied by adding 0.1 NaOH to achieve the pH of the solution 8, 9, 10 and 11, accordingly. The reaction temperature was optimized by conducting the experiment at three different temperatures, namely, at room temperature, 60°C and 80°C. Finally, the reaction time was recorded up to the saturation time. The entire optimization process of biosynthesis CuO NPs was monitored by UV-Vis spectroscopy. After completion of the optimization experiment, the colloidal solution was centrifuged at 14800 rpm for 20 min. The obtained precipitate was washed with distilled water, and dried and kept in a desiccator for further use [13].

III. RESULTS AND DISCUSSIONS

3.1 Antibacterial studies

Disc diffusion method was carried out by using gram negative bacteria- *E.Coli* to evaluate the antibacterial potential of the sample. Bacterial culture of *Escherchia-coli (E. Coli)* was swabbed on freshly prepared nutrient agar which was poured on sterile petridishes. After solidification, the prepared polymer samples at different concentrations (25, 35and 45 µg/mL) for Schiff base ligand, copper(II) complex and copper oxide containing ginger respectively were loaded in discs and the plates were incubated at 37^{0}C for 24 hours. After incubation, the diameter of inhibitory zones formed around each well/disc was measured in mm and compared with positive control (Tetracycline).

Table 1: Antibacterial activity of copper complex containing ginger

Samples	25 µg/ml	35 µg/ml	45 µg/ml
Schiff base ligand	+	+	++
Copper (II) oxide	++	++	+++
Copper (II) oxidecontaining ginger	+++	+++	+++

Key to symbols
Highly active = +++ (inhibition zone>10
Moderately active = ++ (inhibition zone 7-10)
Slightly active = ++ (inhibition zone 4-6)

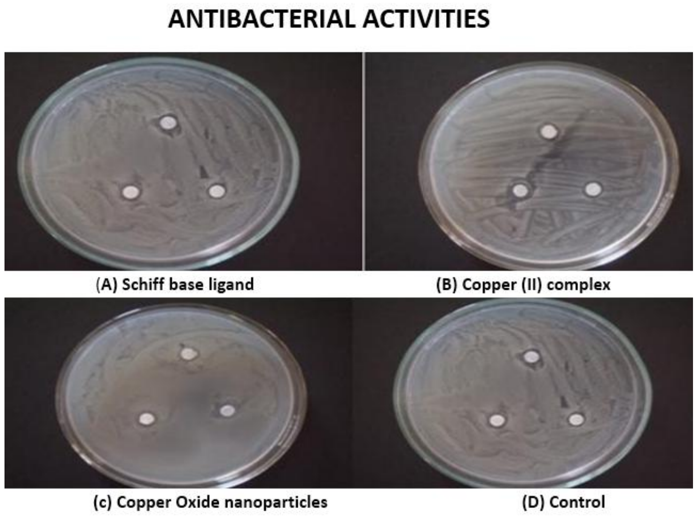

Figure 1: Antibacterial activities of the three samples including control

3.2. Morphology of the copper complex containing ginger

Agglomeration and large grains formation were observed in the SEM images (Figure 2 & 3) and the image also revealed that few spherical shaped particles are also present. Some particles are quite separated from each other and agglomeration formations are due to the oxidation of metal oxide. The TEM results (Figure 4) depicted that synthesized compounds were FCC in accordance with XRD results. The TEM results also indicate the formation of copper oxide nanoparticles. XRD revealed the monoclinic single phase structure [15].

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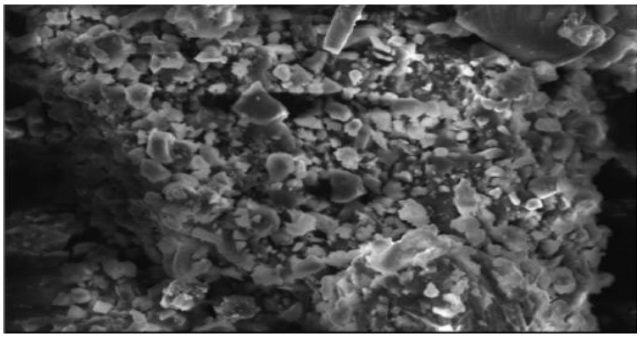

Figure 2: SEM image of Schiff base ligand at magnification of 5µm

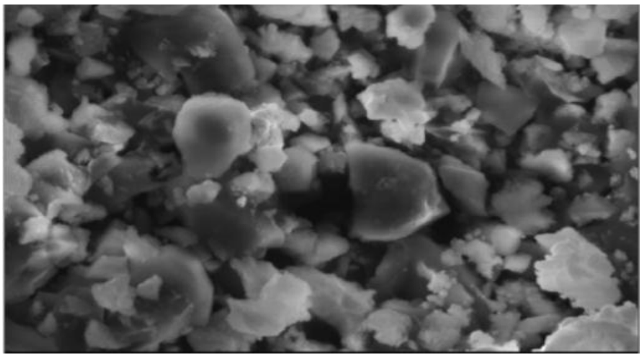

Figure 3: SEM image of copper oxide containing ginger at magnification of 5µm

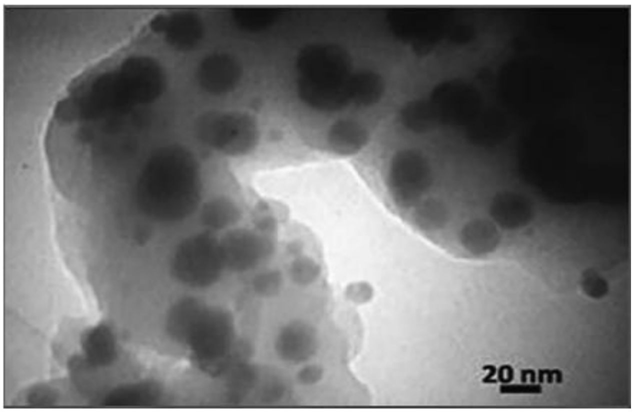

Figure 4: TEM image of copper oxide containing ginger at magnification of 20µm

3.3 XRD analysis

The purity and crystallinity of the synthesized copper oxide nanoparticles containing ginger were examined using XRD pattern. All the diffraction peaks are absolutely matched with the standard spectrum JCPDS number 96-901-6058 [16], in the case of figure 8, JCPDS number 96-900-4180 in the case of Figure 6.Diffraction peaks corresponding to the impurity were not found in the XRD patterns, confirming the high purity of the o products. The average crystallite size is calculated by X-ray Diffraction, line broadening using the Scherrer formula.

D=Kα/B Cosθ

- D=Crystalline size
- K=0.9 scherrer constant
- α=wavelength of X rays (1.5406 A° unit)
- θ =diffraction angle of the peak
- B=full width half maximum of the peak

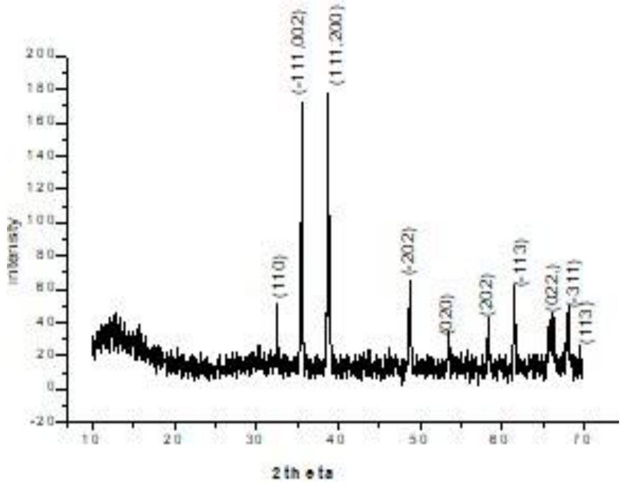

Figure 5: XRD pattern of Schiff base ligand

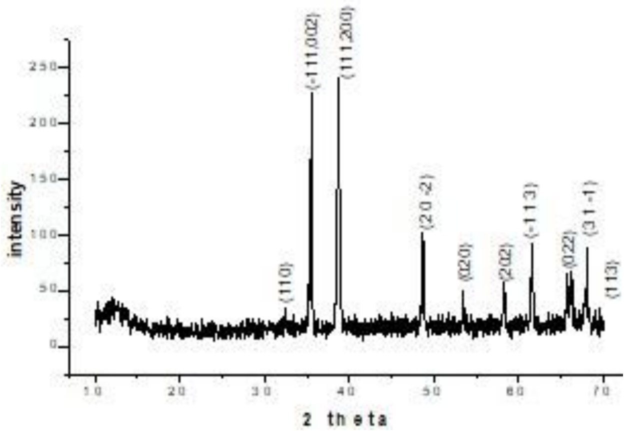

Figure 6: XRD pattern of copper (II) complex

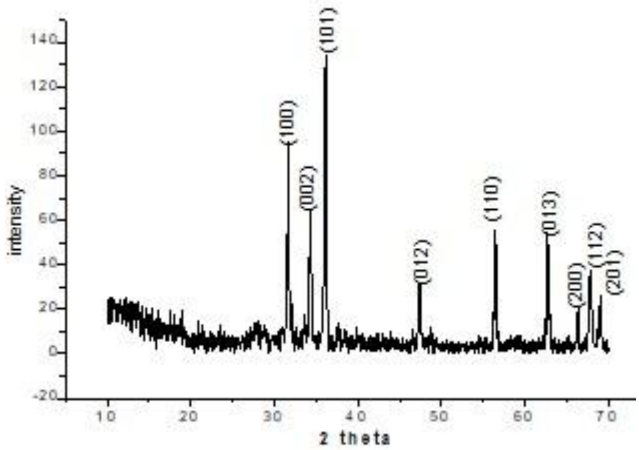

Figure 7: XRD spectrum of the copper oxide containing ginger

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3.4 FT-IR spectra analysis of the copper oxide nanoparticles

The infrared spectra of Schiff base ligand and copper oxide exhibit a sharp band at 1632cm^{-1} and 1635cm^{-1} respectively due to azomethine linkage. In the complex, this band appears at a frequency lower than that of the free ligand. This clearly indicates the involvement of nitrogen atom in coordination due to a reduction in the electron density in the azomethine linkage. The appearance of 3433 cm^{-1} frequency is due to OH functional group in the compounds. The appearance of frequency at 2921 cm^{-1}is due to C-H functional groups [17].

IV. CONCLUSIONS

A copper oxide nanoparticles was successfully synthesized using ginger leaf extract via hydrothermal calcinations approach. The compounds were applied for the antibacterial activities against gram negative bacteria. The compounds were characterized for its physicochemical, optical, and structural properties by XRD, SEM, TEM and FT-IR spectroscopic techniques. X-ray diffraction reveals the size of nanoparticles. Antibacterial potential of the prepared compounds were carried out by using gram negative bacteria (E.Coli and shows good inhibition potential.

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