Synthesis, Characterization And Antibacterial Assessment Of Cuo Nanoparticles

Chandrama Kalita, Sanjib Karmakar

Abstract: The sol-gel method has been implemented to synthesize CuO nanoparticles at 100ºC. X-ray Diffraction (XRD) pattern has been employed to establish the monoclinic phase structure of prepared CuO nanoparticles. Crystal size (using Debye-Scherrer's relation) of the prepared nanoparticles have been calculated from XRD data. Transmission Electron Microscope (TEM) has been utilized to confirm the nanometric size of prepared CuO nanoparticles. The band gap of the prepared CuO nanoparticles has been estimated from UV-visible absorption spectra. The chemical composition of prepared CuO nanoparticles has been inspected by Fourier Transform Infra Red (FTIR) spectroscopy. Surface morphology of prepared CuO nanoparticles has been analyzed by using Field Emission Scanning Electron Microscope (FESEM). CuO nanoparticles have shown adequate bactericidal effect against gram-positive bacteria L. Monocytogenes and gram-negative bacteria Escherichia Coli. The antibacterial activity of CuO nanoparticles has been investigated against these two bacterial strains.

Index Terms: CuO nanoparticles, sol-gel method, crystal size, band gap energy, bacterial strains, antibacterial activity, zone of inhibition

1. INTRODUCTION

Metal oxide nanoparticles are getting a lot of consideration because of broad consideration for their potential applications in optoelectronics, nanodevices, nanoelectronics, water purification, photocatalysis and data storage. Metal oxide nanoparticles go astray from bulk materials because of their peculiar electronic, optical and compound characteristics. CuO nanoparticles are the simplest compound of the copper compound family [1]. Copper oxide (CuO) nanoparticles are widely utilized in various applications for example as gas sensor [2], [3], photocatalyst [4], solar cells [5], humidity sensor [6]. CuO nanoparticles have a brilliant adsorbing limit. At a specific pH, it can adsorb component and at certain pH, desorption happens. Because of this property, CuO nanoparticles are by and large broadly utilized as harmful component remover, for example, arsenic(III), arsenic(V), fluoride from polluted water [7]. Distinctive medical issues of human ascent because of dif-ferent micro-organisms live in our condition and our body itself. Presently, microorganisms make obstruction against traditional antitoxins. So, the quest for new anti-infection material has turned out to be basic. CuO nanoparticles show very great antibacterial action against various microorganisms [8]. In this investigation, we incorporated CuO nanoparticles utilizing sol-gel strategy and we portrayed prepared nanoparticles using various instruments. Antibacterial behavior of the prepared nanoparticles was tried utilizing various microbes.

2 DETAILS OF EXPERIMENTAL PROCEDURE

2.1 Materials

The material $CuCl_2.2H_2O$ (Copper Chloride Dihydrate) and-NaOH (sodium hydroxide) pellets with 99% purity were bought from Merck specialities private limited The glacial acetic acid was bought from NICE CHEMICALS (P) LTD. Mueller–Hinton agar, Mueller–Hinton broth, Dimethyl sulfoxide (DMSO) and sterile disks were purchased from Hi-media India Pvt Ltd.

2.2 Preparation of CuO nanoparticles

An 0.8 M aqueous solution of $CuCl_2.2H_2O$ was made by dissolving 6.82 gm in 50mL DI water in a beaker [9]. 4mL glacial acetic acid was added dropwise to the above solution with constant stirring. Thereafter, the prepared solution was warmth to 100ºC. pH 7.5 was maintained by dropwise addition of 8M NaOH aqueous solution. A black precipitate was formed and the precipitate was filtered with the assistance of a Whatman filter paper. The precipitate was washed thoroughly with distilled water. Then it was air dried and converted into powder using mortar. The powders were utilized for characterization and antibacterial behavior assessment.

2.3 Characterization Techniques

Diffraction pattern of the prepared CuO nanoparticles was analyzed by XRD instrument (Phillips Expert Pro XRD). For investigating absorption spectra of the prepared nanoparticles, Perkin Elmer (Lambda 950) instrument was used. Field Emission Scanning Electron Microscope (FESEM) Model Sigma 300(ZEISS) was used to inspect the morphology of prepared nanoparticles. Identification of elements present was done by using Energy Dispersive X-ray Spectroscopy (EDS) Model Element. FTIR spectra of prepared nanoparticles was investigated by using Perkin Elmer, spectrum 65. Transmission Electron Microscope (TEM) analysis of prepared nanoparticles was done by using JEOL, Model: JEM 2100.

2.4 Investigation of Antibacterial Activity

The investigation of antibacterial behavior of prepared CuO nanoparticles was assessed by employing two pathogenic bacterial strains such as Escherichia Coli (ATCC 27853), a gram negative bacteria and Listeria Monocytogenes, a gram positive bacteria (PTCC 1298). Kirby-Bauer test or disk diffusion test was employed to inspect the antibacterial activity test of prepared CuO nanoparticles [10], [11]. The required amount of Muller Hinton Agar powder was mixed with distilled water in accordance with the instructions given on the container. To dissolve the powder properly into water, the mixture was heated for some time. The mixture solution was autoclave to it make sterile. The sterile solution was poured into sterile petri

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plates and let them dry. After that, the above mentioned bacterial strains were swabbed homogeneously with the help of an inoculation loop on each plates. After the solidification of agar, sterile disks were placed on it. 1mg CuO nanoparticles was dispersed in 100 µl DMSO to make a solution. 10 µl of this solution was poured on the sterile disks. Keeping it undisturbed till absorption of the CuO nanoparticles solution by sterile disks was taken place. After that, the plates were properly sealed and placed inside an incubator at 37ºC for 24 hrs. With the assistance of a ruler, the zone of inhibition was measured around the disks [12].

3 RESULTS AND DISCUSSIONS

3.1 XRD Analysis

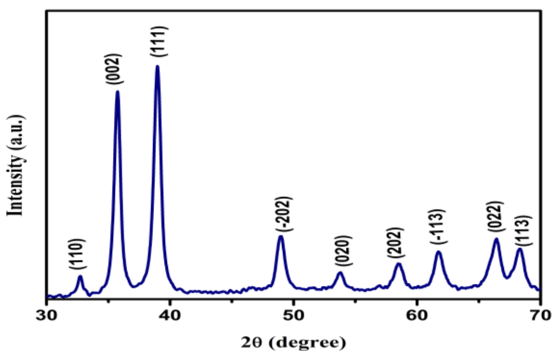

***Fig. 1**. XRD Diffraction pattern of prepared CuO nanoparticles*

X-Ray Diffraction pattern is employed for establishing the structure and phase of prepared CuO nanoparticles. Fig.1 demonstrates the XRD diffraction pattern of prepared CuO nanoparticles. The diffraction pattern revels clear peaks centred at diffraction angles (2θº) as 32.75º, 35.73º, 38.96º, 48.97º, 53.71º, 58.49º, 61.77º, 66.32º, 68.29º corresponds to (110), (002), (111), (-202), (020), (202), (-113), (022) and (113) lattice planes respectively. All the peaks in the diffraction pattern show the prepared CuO nanoparticles are of monocline structure. The peaks are matched with the standard data of ICDD card no 89-5895.The XRD pattern confirmed the crystalline nature of prepared CuO nanoparticles. The Debye-Scherrer's formula as given in equation (1) [13]

$$D = \frac{K\lambda}{\beta \cos \theta} \qquad (1)$$

Where K is a shape factor and its value is equal to 0.9, **β** is the Full width half maximum(FWHM), λ is the wavelength of X-ray radiation (value=1.54056Å), θ is the Bragg's angle of diffraction. The average crystal size of prepared nanoparticles was evaluated as 9.08 nm.

3.2 TEM Analysis

The TEM image of this synthesized CuO nanoparticles is shown in Fig. 2. The size of nanoparticles determined from TEM image is approximately 9.69 nm (diameter), which is in good accordance with that estimated value by Scherrer's equation from the XRD pattern.

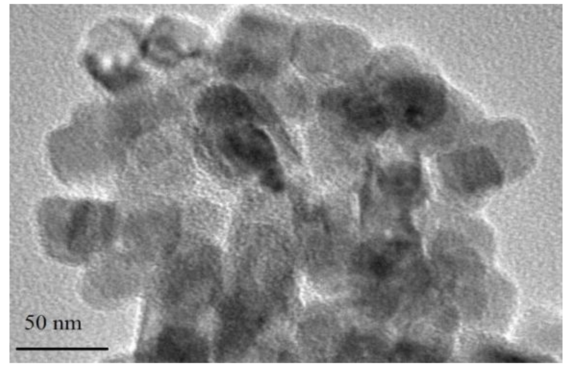

***Fig. 2.** TEM image of prepared CuO nanoparticles*

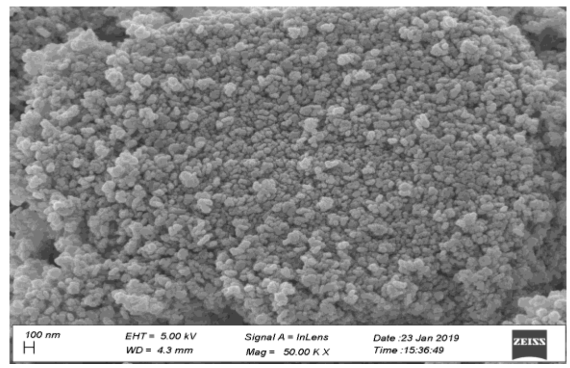

***Fig. 3.** FESEM image of prepared CuO nanoparticles*

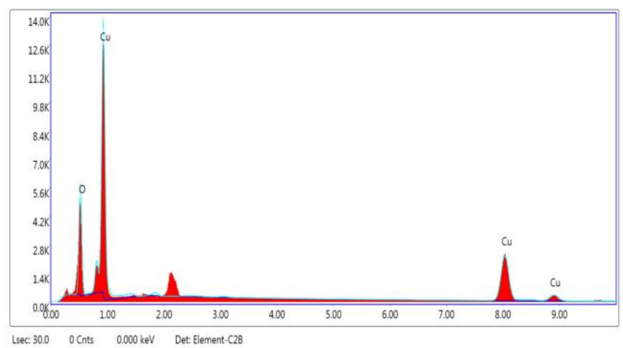

***Fig. 4.** EDS spectra of prepared CuO nanoparticles*

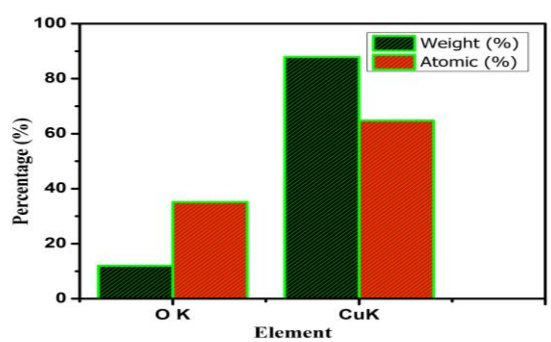

***Fig. 5.** Elemental Analysis of prepared CuO nanoparticles*

The EDS analysis as shown in Fig.4, proved that the prepared nanoparticles were free from impurity. The elements existed in the CuO nanoparticles were Cu (copper) and O (oxygen). The percentage composition of the elements present was shown in the Fig.5.

3.4 Optical Analysis

The optical properties of the prepared CuO nanoparticles were investigated with the help of optical absorbance spectra. Fig.6. displays the UV-vis absorbance spectra of prepared nanoparticles. The information of optical absorbance and band gap energy of a material predicts the field in which it can be used. A peak was observed at wavelength 367 nm in the absorption spectra.

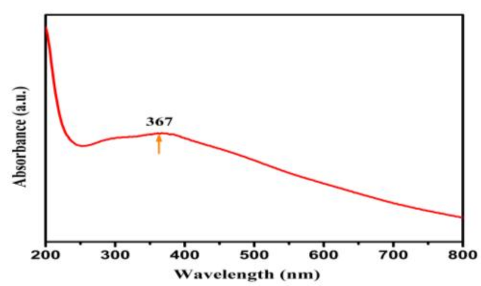

Fig. 6. *UV-Visible absorption spectra of prepared CuO nanoparticles.*

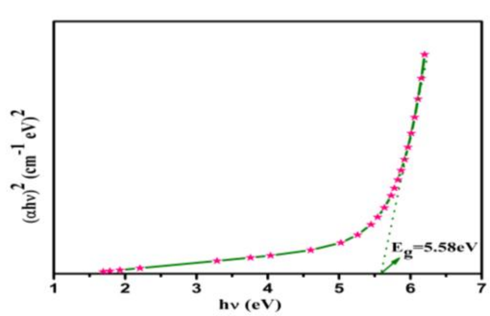

***Fig. 7**. $(\alpha h\nu)^2$ vs $h\nu$ spectra of prepared CuO nanoparticles*

The band gap energy of prepared nanoparticles was estimated by using Tauc Equation [14]

$$(\alpha h\nu)^{1/n} = A\left(h\nu - E_g\right) \qquad (2)$$

Where α is the absorption co-efficient, $h\nu$ is the incident photon energy, A is a constant. For materials, having direct band gap, such as CuO, the value of n is 1/2. The band gap energy has been calculated by plotting graph $(\alpha h\nu)^2$ versus $h\nu$ and then extrapolating the linear portion of plot as shown in Fig.7. The band gap energy of CuO nanoparticles was estimated as 5.58 eV.

3.5 FTIR Analysis

The FTIR spectra of prepared CuO nanoparticles were inspected by using the KBr pellets approach. Fig.4 displays the FTIR spectra of prepared CuO nanoparticles were recorded in the range of 4000–400 cm^{-1}. Formation of highly pure CuO nanoparticles was affirmed by bands at around 609, 512 cm^{-1} of FTIR spectra. These peaks were occurred due to the vibrations of Cu(II)-O bonds. The distinguishable peaks of CuO

TABLE 1

RESULT OF ANTIBACTERIAL TESTING OF PREPARED CUO NANOPARTICLES

Testing Bacteria	Zone of Inhibition (mm)
E. Coli	16
L. Monocytogenes	--

were found in between 984 cm^{-1} to 426cm^{-1}. A wide absorption

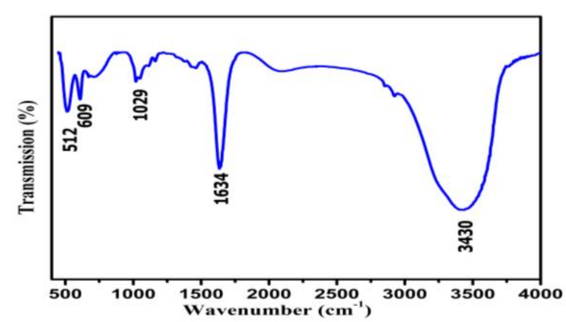

***Fig. 8**. FTIR spectra of prepared CuO nanoparticles*

band at around 3430 cm^{-1} was noticed which occurred because of absorption of moistures by the prepared nanoparticles. The peak at 1029 cm^{-1} was designated to -OH bending vibrations. The band located at around 1634 cm^{-1} was raised due to the formation of C=C bonds. The allocation of peaks in accordance with the values found in the literature [15], [16], [17], [18], [19], [20].

3.6 Antibacterial Assessment

Antibacterial activity of the prepared CuO nanoparticles was inspected by utilizing strains of E. Coli and L. Monocytogenes. A clear growth of inhibition by the CuO nanoparticles was observed as displayed in Fig.9. DMSO was utilized as a dispersing reagent to the nanoparticles (control) which has no antibacterial action towards any bacterial strains. The antibacterial action of nanoparticles can be described based on the size of the bacterial cell and its membrane pore size. In general, the size of bacterial cells is about a few microns. The size of pores on the bacterial membrane fall in the nanometer range. The CuO nanoparticles which were in the nanometer scale might have a size less than the size of membrane pore. Because of this, nanoparticles can readily penetrate through the cell membrane and prevent growth [21]. It was evident that CuO nanoparticles were not efficient towards L. Monocytogenes. The zone of inhibition against the two bacterial strains is given in Table 1.

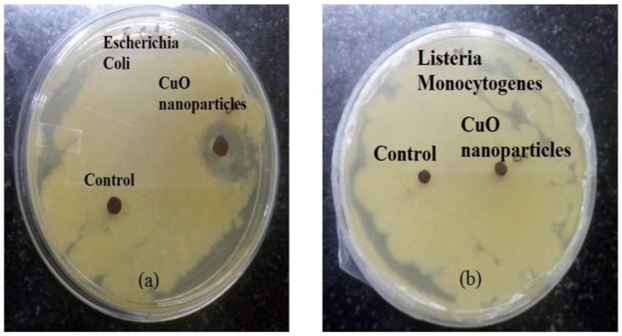

***Fig. 9**. Zone of inhibition in bacterial plates of (a) E. Coli and (b)*

4 CONCLUSION

The sol-gel method was successfully implemented to synthesized CuO nanoparticles. The single-phase monoclinic structure of prepared nanoparticles was affirmed with the assistance of X-ray Diffraction spectra. Owing to quantum confinement effect of nanoparticles, a blue shift was distinct in the band gap energy of prepared nanoparticles. Agglomeration of CuO nanoparticles were strongly apparent from the FESEM image. The EDS and FTIR spectra confirmed that the prepared nanoparticles were of pure form. The antibacterial activity of prepared nanoparticles against bacterial strains were evident from the experiments. It showed prominent antibacterial activity towards strain E. Coli. In case of L. Monocytogenes strain, the nanoparticles were not effective. Due to this antibacterial property, the CuO nanoparticles can be used in making safety equipment, cloths etc. utilized in hospitals. It can also be used in treatment of polluted water.

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