RASĀYAN *J. Chem.*
Vol. 16 | No. 4 |2256-2260| October - December | 2023
ISSN: 0974-1496 | e-ISSN: 0976-0083 | CODEN: RJCABP
http://www.rasayanjournal.com
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SOL-GEL SYNTHESIZED COPPER OXIDE (CuO) NANOPARTICLES AND THEIR PHOTOCATALYSTS AND ANTIBACTERIAL APPLICATIONS.

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ABSTRACT

This study aims to report the synthesis of copper oxide nanoparticles using the Sol-Gel autocombustion technique. According to the diffraction data, a monoclinic crystal structural phase had been formed due to the crystallization process. Electronic microscopic images prove that the nanoparticles are growing in a spherical structure. Using Methylene blue as a pollutant, a study was conducted to determine whether the prepared copper oxide nanoparticles have a photocatalytic activity. A photocatalytic rate constant of 0.011 min^{-1} was obtained for the sample, indicating that the synthesized copper oxide nanoparticles have good photocatalytic activity. During the antimicrobial study, the CuO nanoparticles were found to have significant antimicrobial activity against the selected microorganisms, *S.aureus* and *E.coli*. Furthermore, *E. coli* bacteria have shown the highest sensitivity to the synthesized CuO nanoparticles.

Keywords: Sol-Gel Auto Combustion, Nanoparticles, XRD, SEM, Photodegradation, Antibacterial Activity.
RASĀYAN*J. Chem.*, Vol. 16, No.4, 2023

INTRODUCTION

The majority of people worldwide are presently suffering from health issues as a result of drinking untreated, contaminated water.[1] In this regard, developing drugs manufactured at the nanoscale has proven to be the most effective approach in treating many microbes. The surface area of nanomaterials is exceedingly large in terms of their volume. Due to the increased nanoparticles surface area, microbes can interact with the surface more readily, thus enhancing the antimicrobial properties.[2]As a result of nanomaterials' increased surface-to-volume ratio, they have a large number of active sites that are capable of trapping pollutants.[3]Upon photon exposure, these nanomaterials produce electron-hole pairs that break down the pollutant molecules. Metal oxide nanoparticles are one of the most versatile nanomaterials available. In addition to metal oxide nanomaterials, copper oxide (CuO) is significant. This compound has several uses, including antibacterial, antiviral, antiparasitic, antipyretic, and antiparasitic qualities and antioxidant capabilities.[4]The submitted work used Sol-Gel auto combustion synthesis to make CuO nanoparticles easily and affordably. The Sol-Gel auto-combination process extensively forms extremely porous catalytically active nanomaterials. A good diffusion method and optical absorption measurements were performed to test the prepared monoclinic CuO nanoparticles' antibacterial properties and photocatalytic activity. According to the findings, it was confirmed that the CuO nanoparticles possess significant antibacterial activity against *E. coli* and *S.aureus* microbes, as well as being more effective in reducing MB solution colour.

Synthesis Procedure

The Sol-Gel auto-combustion method was used to create CuO nanoparticles. A 0.2M concentrated copper solution was prepared by adding 4.863grams of copper nitrate salt(starting material) to 100 ml of methanol(solvent). Then, citric acid was added as organic fuel to the obtained copper solution. The complex solution was stirred for one hour at 60°C to get a gel form. The viscous gel was heated at 200^0C to ignite the auto-combustion process. As a result of the combustion, a metal oxide compound dust is formed. This product was crushed into a fine powder and heated at 500°C to create a fine nanopowder.

RESULTS AND DISCUSSION

Structural Configuration Analysis

Aside from phase identification, x-ray diffraction also provides information about structural parameters, including unit cells' dimensions (a,b, and c) and volume. As a confirmation of the presence of the CuO phase in the prepared nanoparticle, x-ray diffraction was taken using Cu-$K_{\alpha 1}$ radiation. A diffraction pattern of CuO nanoparticles is shown in Fig.-1at 20^0-70^0 angular Bragg's displacements.

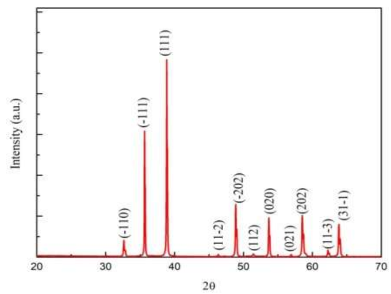

Fig.-1: X-ray Diffraction Spectra of CuO Nanoparticles

Based on the obtained XRD data, a monoclinic CuO crystal structure was confirmed, which corresponds well to the standard database (RRUFF ID: R120076). A PowderX program was used to analyze the diffraction data obtained. Lattice parameters obtained from this study are well in agreement with those previously reported.[5,6,7] According to Debye Scherrer's relation($D = \frac{k\lambda}{BCos\theta}$)[8], the lattice grain size measured along the most intense diffraction peak (111) is 57 nm. A summary of the crystal structural parameters obtained in the experiment is presented in Table-1.

Table-1: Structural Parameters of Prepared CuO Nanoparticles

Peak position of (111) 2θ	Plane spacing d (Å)	Peak width (B) FWHM	Grain size D nm	Lattice parameters
38.83	2.3173	0.1537	57	a = 4.66759 Å b = 3.41020 Å c = 5.11713 Å V = 80.31983Å^3

Particle Surface Morphology

An image of the surface morphology of the fabricated copper oxide nanoparticles can be seen in Fig.-2. It is evident from the electron microscopy image that nanoparticles are distributed homogeneously and that they are almost flakes in shape. Image J free version software was used to evaluate the flake-shaped particle thickness.[9] The histogram of particle thickness represents the average size of the particles. These observations show that the average synthesized CuO nanoparticle flake size is approximately 129nm.

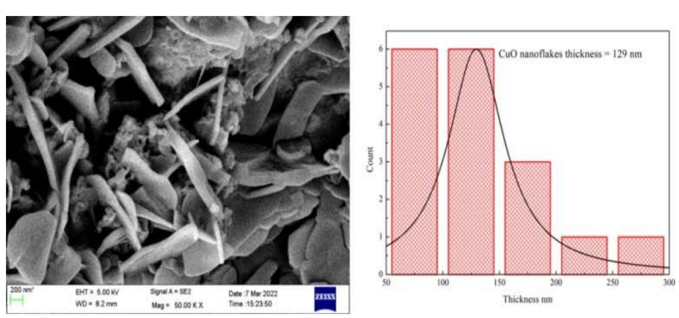

Fig.-2: Electronic Micrograph and Histogram of CuO Nanoparticles

The surface-to-volume ratio increases when reducing a substance to its nanoscale form from bulk form. As a result of these raised surfaces, chemical reactions on the surface of nanoparticles become much more reactive than when they were in bulk form. The prepared CuO nanoparticles' antibacterial and photodegradation properties might be explained by the growth of nanoflakes and increased surface area.This may be viewed as one of the advantages of nanoparticles' photocatalytic activity.

FTIR Analysis

Fourier transform infrared spectroscopy is a sophisticated analytical technique for identifying the chemical structure of a sample. Fig-3 depicts the I.R. spectrum of the synthesized CuO nanoparticles between 400 and 4000 cm^{-1}. A prominent absorption peak at 640 cm^{-1} demonstrates the formation of a monoclinic phase of CuO[10]. The absence of additional vibrational frequencies indicative of various phases, such as Cu_2O, in the fingerprint area indicates the purity of the synthesized CuO phase. A peak at wavenumber 3431cm^{-1} was observed, which may be attributed to H-O-H vibration.[11]

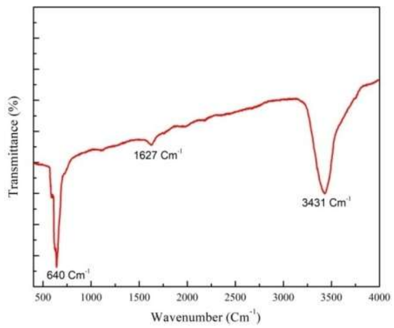

Fig.-3: FTIR Spectrum of CuO Nanoparticles

PhotodegradationActivity

The photocatalytic degradation efficiency of synthesized CuO nanoparticles was investigated using a blend of 50 mg of nano CuO powder and 4ppm concentrated methylene blue (MB) solution. The photodegradation activity of CuO nanoparticles is depicted in Fig.-4. After exposure to sunlight, the solution colour was degraded to almost transparent. A maximum dye degradation percentage of 78% was achieved within 140 minutes. CuO nanoparticles generate electron-hole pairs when interacting with photons, which can lead to photocatalytic activity in sunlight. These pairs react with oxygen and water molecules in MB solution, forming reactive oxygen species like $O_2^-$and OH•, which causes color deterioration.[12]

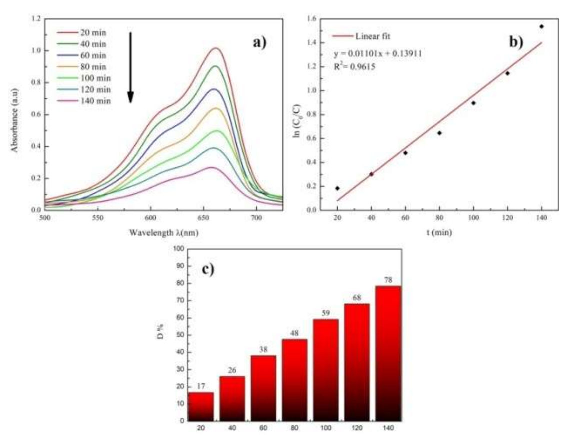

Fig.-4: Photodegradation Activity a) Color Degradation Rate b) Linear Fit-Rate Constant c) Color Degradation Percentage

The size and form of CuO nanoparticles might affect their photodegradation efficacy, among other things. It has previously been observed that nanoflake-shaped particles have exceptional photocatalytic capabilities.[13] The dye degradation of MB at 664 /cm[13] was calculated based on the initial and final absorbance of CuO nanoparticles.

$$D\% = \frac{C_0 - C_t}{C_0} \times 100$$

The Langmuir-Hinshelwood kinetics explain the photodegradation of MB solution by CuO nanoparticles.[14] The obtained linear fit parameters of the $\ln(C_0/C)$ Vs t curve indicates that the photocatalytic activity of CuO nanoparticles follows pseudo-first-order kinematics.[15]

AntibacterialActivity

The antibacterial activity of prepared copper oxide nanoparticles was assessed using a well diffusion method.[16] The antibacterial effect was measured in terms of the diameter of the inhibition zone around the exposed nanoparticles. Fig-5 illustrates the CuO nanoparticles' antibacterial activity. The antibacterial activity of CuO nanoparticles against *S. aureus* and *E. coli* bacteria increased with the increase in nanoparticle dose (20 μg/mL, 40 μg/mL, and 60 μg/mL), indicating a concentration-dependent effect. CuO nanoparticles were more effective against *E. coli* bacteria than against *S. aureus* bacteria.[17]Nanoparticles with an increased surface-to-volume ratio have active sites on their surface, which act as catalysts for forming Cu^{2+} and reactive oxygen ions when exposed to light. These ions have antimicrobial properties, reducing bacteria by damaging the cell wall. When bacterial microbes come into contact with the nanoparticle surface, these ions can break down the cell wall.

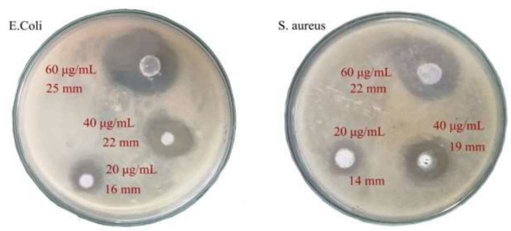

Fig.-5: Inhibition of Bacterial Growth by CuO Nanoparticles

CONCLUSION

CuO nanoparticles were successfully synthesized by Sol-Gel auto combustion. It was determined by XRD diffraction that CuO has grown in polycrystalline monoclinic structure, with a crystal lattice grain size of 57 nm. Furthermore, the obtained FTIR data also supported the monoclinic CuO phase. The electronic micrograph demonstrated that the nanoparticles are formed as nanoflakes with an average size of 129 nm. Furthermore, the nanoparticles exhibited greater antibacterial capabilities against *E. coli* bacteria than against *S.aureus*, which may be due to the thinner cell wall thickness of *E. coli* when compared to *S.aureus* cell wall thickness. Based on the outcomes, it was established that methylene blue organic dye is predominantly demolished by photocatalysis. It was discovered that 78% of the photodegradation occurred in 140 minutes.

ACKNOWLEDGMENTS

Authors are thankful to the authorities of their institutions.

CONFLICT OF INTERESTS

The authors confirm that they do not have any conflicts of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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[RJC-8663/2023]