Article

Evaluation of the antibacterial properties of copper-based mixed metal oxide nanocomposite

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Abstract: Copper-based nanocomposite has a wide variety of applications in various fields of science. The present study focuses on the preparation of copper oxide and zinc oxide nanocomposites by the chemical precipitation method. The prepared samples were analyzed with the help of various characterization techniques, such as XRD, SEM, UV/visible spectroscopy, and EDAX. Using the XRD pattern, the crystallite size determination is carried out, and the crystalline nature of the nanocomposite is confirmed. With SEM analysis, surface morphological studies were performed. EDAX analysis conforms to the formation of mixed metal oxide nanocomposites. The antimicrobial behavior of samples was evaluated for E. coli bacteria using the disc diffusion method.

Keywords: nanocomposite; metal oxide; antimicrobial

1. Introduction

In the field of material science, metal oxide nanocomposites have some peculiar qualities. The main reason for this quality is attributed to its surface-to-volume ratio. Metal oxide nanoparticles have a particular type of magnetic, electronic, and optical properties [1]. Metal matrix nanocomposites are very useful for different types of applications. Chemistry plays an important role in the preparation of different nanocomposite materials; compared with physical methods, chemical methods have some unique qualities. Nanocomposite made from copper oxide [2–4] has applications in various scientific fields.

2. Materials and methods

The copper-based nanocomposites were prepared by a controlled co-precipitation method. Samples were prepared using analytical-grade reagents. The preparation started by mixing a 0.1 M solution of copper nitrate, a 0.1 M solution of zinc nitrate, and 0.02 M citric acid. The mixed solution was taken into a conical flask. The solution was continuously stirred for half an hour. After that, a 0.5 M solution of sodium hydroxide was added to the above solution using a burette. Using a magnetic stirrer, the above solution stirred continuously for another three hours until a thick precipitate was obtained. The precipitate was filtered, washed, and dried; later, it was annealed at temperatures of 500 °C and 800 °C.

In the above preparation, citric acid is used as a capping agent to prevent the growth or agglomeration of the sample. For comparative purposes, individual nanoparticles of copper oxide and zinc oxide were also prepared and annealed at 500 °C to maintain a smaller crystalline size [5]. The obtained XRD patterns of

mixed metal oxide nanocomposite of copper oxide/zinc oxide annealed at 500 °C and 800 °C (CZ5 and CZ8) were shown in (**Figure 1**).
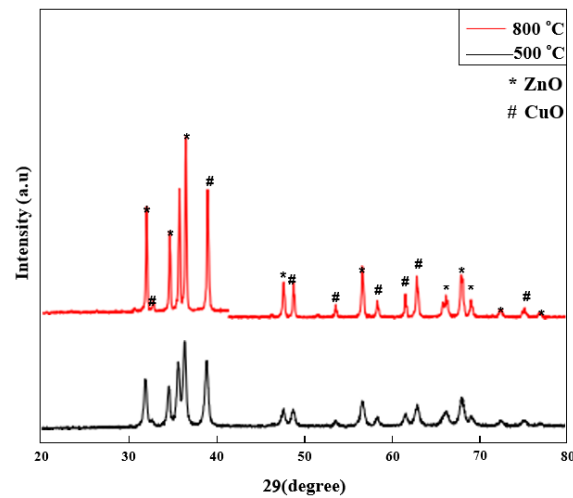

Figure 1. XRD pattern of CZ5 and CZ8.

3. Result and discussion

3.1. XRD pattern analysis

The nanocomposite nature of the prepared samples is verified with the help of XRD analysis. The average crystallite size of the prepared samples was determined by two different approaches, such as the Williamson-Hall plot and the Scherrer equation. The Scherrer formula is represented as $t = k\lambda/\beta_{hkl} \times \cos\theta_{hkl}$. Here, t is the crystallite size normal to the reflecting planes, and k is the shape factor, which lies between 0.95 and 1.15 depending on the shape of the grains ($k = 1$ for spherical crystallites). λ is the wavelength of X-ray used, and β_{hkl} is the measured full width at half maximum (FWHM), which was determined by a regular Psd-Vogit1 curve fitting, assuming the XRD peaks are symmetrical in shape. The curve fitting was done using Microl Origin version 8.5, where θ_{hkl} is the Bragg angle. The average crystallite size obtained for samples is represented in **Table 1**. The XRD pattern of nanocomposite shows peaks of both CuO and ZnO, which is a clear-cut representation of the formation of mixed metal oxide nanocomposite. The peaks of CuO were matched with ICCD card number 801916, and the peaks of ZnO were matched with card number 750576.

Table 1. Crystalline size of CZ5 and CZ8.

Sample	Crystallite size	
	Scherrer equation (nm)	Williamson-Hall (W-H) plot (nm)
CZ5	21	24
CZ8	35	41

3.2. UV-visible studies

The UV-visible studies of both CZ5 and CZ8 were carried out in the range of 210 nm to 800 nm with the help of a Jasco 650V UV/visible spectra photometer. The absorbance and reflectance spectra of both samples were recorded. The optical bandgaps of CZ5 and CZ8 were calculated using Tauc plots and Kubelka Munk plots. The optical bandgap obtained from the absorbance and reflectance spectra is shown in **Table 2**. **Figure 2** represents the UV/visible absorption and reflectance spectra of CZ5 and CZ8, and the corresponding Tauc plot and Kubelka Munk plot are represented in **Figure 3**.

Table 2. Optical bandgap of CZ5 and CZ8.

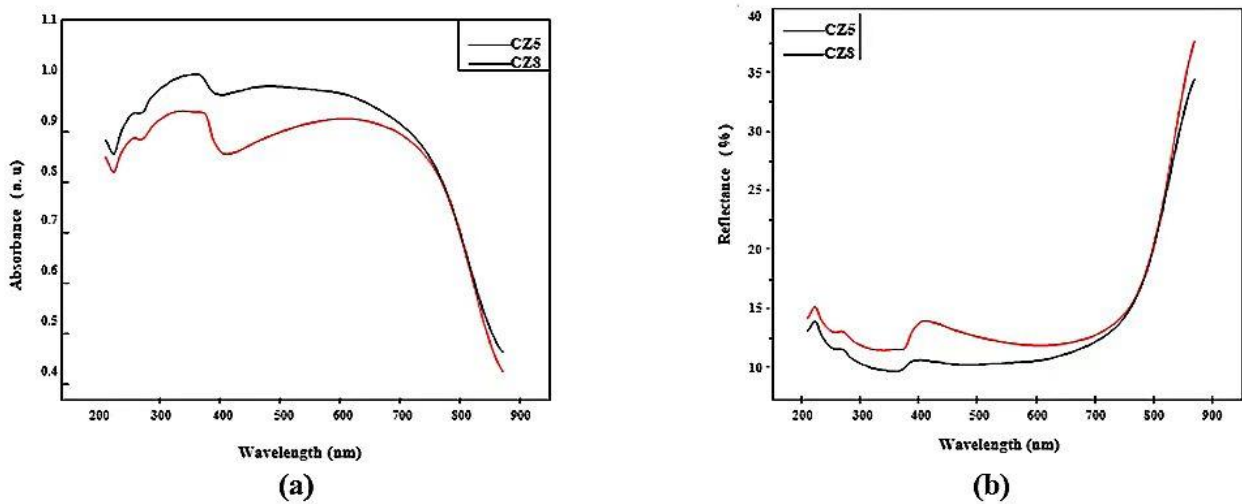| Sample	Optical bandgap	
	Tauc plot eV	**Kubleka Munk plot eV**
CZ5	1.36	1.23
CZ8	1.09	1.05

Figure 2. (a) UV/visible absorbance spectra of CZ5 and CZ8. **(b)** Reflectance spectra of CZ5 and CZ8.
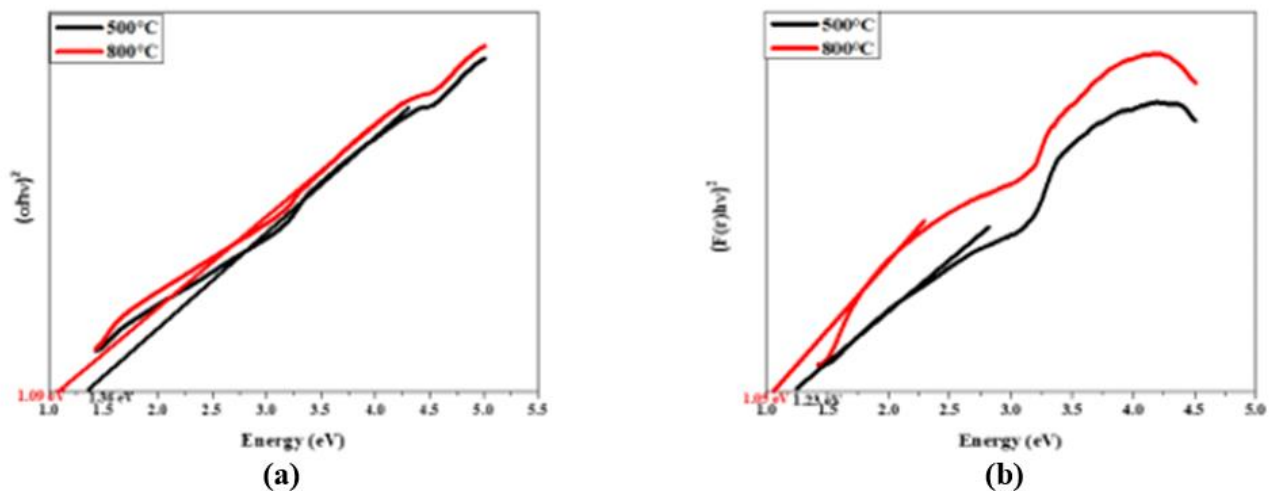

Figure 3. (a) Tauc plot of CZ5 and CZ8. **(b)** Kubelka Munk plots of CZ5 and CZ8.

3.3. SEM with EDAX analysis

SEM images show that the prepared particles are almost spherical in shape and have a slight level of agglomeration. The SEM images of CZ5 and CZ8 are represented in **Figure 4a,b**, respectively. The EDAX spectrum confirms the formation of nanocomposites. The prepared samples were almost homogeneous in nature. The presence of oxygen and corresponding metals, such as copper and zinc, conformed to the analysis. No impurities are found in the samples. The EDAX spectra of CZ5 are shown in **Figure 4c**. The percentage of various elements in the nanocomposite is represented in **Table 3**.
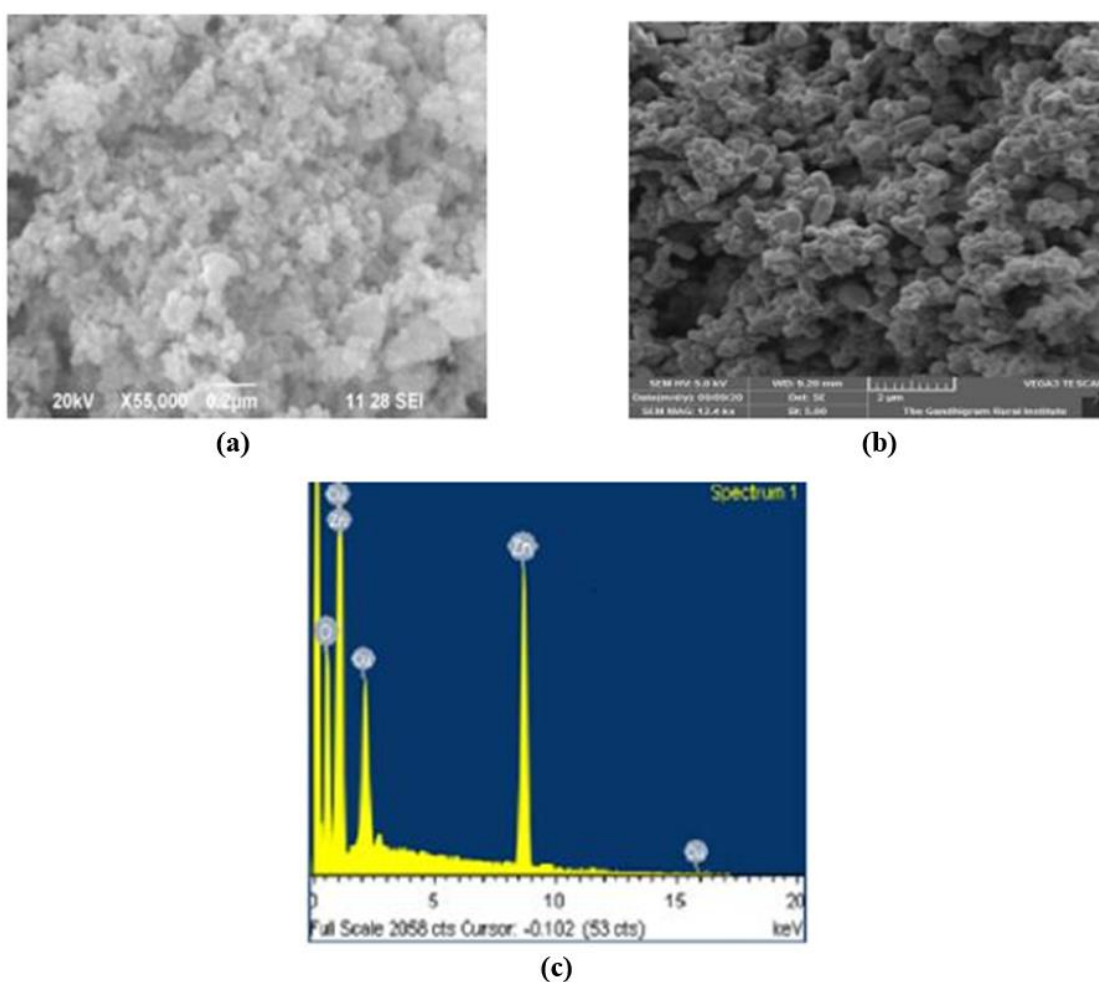

(a)

(b)

(c)

Figure 4. (a) SEM image of CZ5. **(b)** SEM image of CZ8. **(c)** EDAX image of CZ5.

Table 3. Chemical composition of the sample.

Sample	O		Cu		Zn	
	Mass %	Atom %	Mass %	Atom %	Mass %	Atom %
CZ5	26.96	59.81	34.38	19.20	38.66	20.99

3.4. Antimicrobial property evaluation analysis

Detailed studies of the antibacterial properties of C5, Z5, CZ5, and CZ8 were carried out in the bacterial strain E coli. Basically, the antimicrobial behavior of mixed metal oxide nanocomposites is mostly due to the enhanced surface area. Superior surface area enriches the contact with the bacterial cell with nanocomposite. Various researchers suggest different types of mechanisms to explain the antibacterial properties of mixed metal oxide nanocomposites. Most of the antimicrobial properties of nanocomposite materials are attributed to different steps, such as the production of highly reactive oxygen species and bacterial cell membrane disruption by the accretion of metal oxide nanoparticles. The release of metal ions inside the cytoplasmic region of bacteria will lead to the total collapse of the bacterial cell [6–8]. **Table 1** shows the zone area obtained for C5, Z5, CZ5, and CZ8, respectively. Selected bacteria were Gram-negative bacteria, which have an additional cell membrane for visitant antimicrobial action. Even with the presence of this cell wall-mixed metal oxide nanocomposite, it shows good bacterial inhibition. The studies reveal that mixed metal oxides provide better antimicrobial properties than individual metal oxides. As the size increases, the antimicrobial properties of the mixed metal oxide nanocomposite gradually decrease. The zones of inhibition produced by various samples are represented in **Figure 5** and **Table 4**.

Figure 5. Image of zone of inhibition.

Table 4. Antimicrobial zone of inhibition.

Sample	Zone of inhibition
C5	8 mm
Z5	6 mm
CZ5	12 mm
CZ8	10 mm

4. Conclusion

Transition metal oxide-based mixed metal oxide nanocomposites were prepared using the co-precipitation method. From the study, it is found that mixed metal oxide nanocomposite shows improved antimicrobial properties as compared with individual metal oxide nanoparticles. As the annealing temperature of the sample

increased, the crystalline size increased, and as a result, the antimicrobial properties decreased.

Conflict of interest: The authors declare no conflict of interest.

References

1. Zhang X, Wang G, Zhang W, et al. Seed-mediated growth method for epitaxial array of CuO nanowires on surface of Cu nanostructures and its application as a glucose sensor. Journal of Physical Chemistry 2008; 112(24): 8856–8862. doi: 10.1021/jp800694x

2. Wang H, Pan Q, Zhao J, et al. Fabrication of CuO film with network-like architectures through solution-immersion and their application in lithium ion batteries. Journal of Power Sources 2007; 167(1): 206–211. doi: 10.1016/j.jpowsour.2007.02.008

3. Wang F, Li H, Yuan Z, et al. A highly sensitive gas sensor based on CuO nanoparticles synthetized via a sol–gel method. Royal Society of Chemistry 2016; 6(83): 79343–79349. doi: 10.1039/C6RA13876D

4. Kida T, Oka T, Nagano M, et al. Synthesis and application of stable copper oxide nanoparticle suspensions for nanoparticulate film fabrication. Journal of the American Ceramic Society 2007; 90(1): 107–110. doi: 10.1111/j.1551-2916.2006.01402.x

5. Biju R, Ravikumar R, Thomas C, Indulal CR. Enhanced photocatalytic degradation of Metanil Yellow dye using polypyrrole-based copper oxide–zinc oxide nanocomposites under visible light. Journal of Nanoparticle Research 2022; 24(6): 117. doi: 10.1007/s11051-022-05495-3

6. Balouiri M, Sadiki M, Ibnsouda SK. Methods for in vitro evaluating antimicrobial activity: A review. Journal of Pharmaceutical Analysis 2016; 6(2): 71–79. doi: 10.1016/j.jpha.2015.11.005

7. Díez-Pascual AM. Antibacterial action of nanoparticle loaded nanocomposites based on graphene and its derivatives: A mini-review. International Journal of Molecular Sciences 2020; 21(10): 3563. doi: 10.3390/ijms21103563

8. Biju R, Ravikumar R, Thomas C, et al. Optimization and multifunctional applications of polypyrrole-modified copper oxide–zinc oxide nanocomposites. Arabian Journal for Science and Engineering 2023; 48(2): 919–937. doi: 10.1007/s13369-022-07199-1