



INVESTIGATION ON PURE AND COPPER DOPED MGO NANOPARTICLE USING CO-PRECIPITATION METHOD

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ABSTRACT Using the co-precipitation method, pure and Cu-doped MgO were created. Magnesium chloride and copper chloride were used as precursors for the synthesis. The produced nanoparticles; structural, morphological, optical, and vibrational properties were assessed using XRD, SEM with EDAX, UV, PL, and FTIR. The monoclinic structure of the cubic crystal phase for both pure and Cu-doped MgO is confirmed by XRD. The produced nanoparticles were discovered to have diameters of 17 and 16 nm, respectively. The Cu, O, and Mg elements in the prepared sample are revealed by EDAX, while the cluster and porous agglomeration are visible in the SEM image. UV reveals that the produced samples band gap was 4.3 eV and 4.7 eV according to the Tauc relation. The emission peak detected at 424 and 465 nm is associated with PL band edge emission. The significant absorption bands seen at 603 cm⁻¹ and 1056 cm⁻¹ in the FTIR spectrum reveal that the magnesium metals have no shoulder peaks and that the bending vibration modes of Mg-O-C are responsible for their appearance.

KEYWORDS : MgO nanoparticles, Cu-doped MgO, Co-precipitation method, XRD and SEM-EDAX characterization

INTRODUCTION

The unique properties of nanomaterials have led to interest in the past decade in the development of simpler and more affordable ways of synthesizing nanoscale structures for industrial importance. Due to their radiant qualities, oxide semiconductor nanostructures have been extensively studied in recent years, and the fabrication of nanostructured oxide materials gained significant attention in recent years [1]. Because of their unique physical, chemical, and optoelectronic characteristics [2], nanoparticles are used in a variety of applications, including catalysts [3], chemical detectors [4], semiconductors [5], optical visualization for medical screening and medicinal products [6], and the delivery of drugs. Of these, liposomes [7], polymer nanoparticles, nanoparticles of inorganic material, and dendrimers [8] have drawn the most attention.

In the current work, magnesium oxide (MgO) is a significant substance that finds usage in a wide range of applications, including hazardous waste cleanup, catalysts, superconducting products, and antibacterial capabilities against pathogens that are transmitted through food [9].

The co-precipitation technique is used to synthesis MgO and Cu-doped MgO nanoparticles. One of the best ways to synthesis nanoparticles without agglomeration in the yield is by co-precipitation, which also makes size control easier [10]. Using XRD, SEM with EDAX, UV, PL, and FTIR, the Synthesized nanoparticles structural, morphological, optical, and vibrational properties were identified.

2 EXPERIMENTAL METHOD

2.1 FOR PURE MgO

To prepare MgO nanoparticles, 100mL of 0.4 M KOH solution is added drop-wise into a solution containing 100mL of 0.6 M Magnesium Chloride solution under constant stirring. Then the resulting solution is kept at room temperature for three hours under constant stirring. A white precipitate is formed. It is washed several times with distilled water and this precipitate dried at 100°C in an oven for 3 hours. The obtained samples are calcinated in at 300°C for 2 hours to get MgO nano particles.

2.2 FOR CU DOPED MGO

To prepare Cu doped MgO nanoparticles, 100 mL of (0.4M) KOH is added drop-wise into a mixture solution of 100 mL of (0.6 M) Magnesium Chloride and 100 mL of (0.01M) Copper Chloride under constant stirring. Then the resulting solution was kept at room temperature for three hours under constant stirring. Obtained bluish green precipitate is washed several times with distilled water and dried at 100°C in an oven for 3 hours. Finally, the pre-calcinated in at 300°C for 2 hours to get Cu doped MgO nano particles.

3.RESULT AND DISCUSSION

3.1 STRUCTURAL ANALYSIS

The crystalline structure and phase of the nanomaterial were characterized by X-ray diffraction analysis. The average size has been estimated by measuring the full-half at the half-maximum of the most

intense diffraction peak using Debye-Scherrer equation

$$D = K\lambda / \beta \cos\theta \quad (1)$$

Where λ is a wavelength (1.5418Å⁰), β is the full width of the peak in radians at half maximum intensity, θ is the Bragg's angle and K is the Scherrer's constant. The average diameter of pure and Cu doped MgO nanoparticles is found to be 17nm and 16 nm. The XRD pattern for pure and Cu doped MgO are in the Cubic crystal phase. The peak possesses Monoclinic structure of Cu doped MgO nanoparticles.

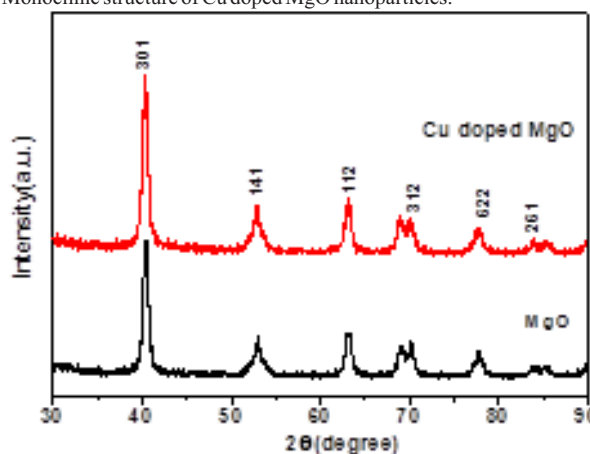


Figure 1 XRD pattern of pure and Cu doped MgO

Source:

The XRD pattern for Pure shows the prominent peak observed at 2θ values of 40.47, 53.06, 63.26 are attributed to (301), (141), (112) planes respectively. The XRD pattern for pure and Cu doped MgO are in the Cubic crystal phase. The peaks belong to the cubic structure MgO nanoparticles, the lattice plane is in the form of Face-centred cubic lattice (JCPDS card No :79-0612)[11] The XRD pattern for Cu doped MgO shows the prominent peaks observed at 2θ values 40.47, 53.06, 63.26, 70.38, 77.90 and 84.06 are attributed to (301), (141), (112), (312), (622) and (261) planes respectively. The peak possesses Monoclinic structure of Cu doped MgO nanoparticles (JCPDS card No:41-1381 [12,13])

3.2 OPTICAL ANALYSIS

UV-VIS spectrum of undoped and Cu doped MgO nanoparticles recorded in the wavelength range from 200 to 800 nm using spectrophotometer at room temperature in order to analyse the absorption band of the undoped and Cu doped MgO nanoparticle, are shown in the fig 3. the sample are in far UV region and absorbance maxima increase with Cu doping compared with undoped MgO and shifted to lower wavelength due to smaller particle size.

The optical band gap energy (E_g) was estimated from the Tauc' relation, The optical band gap energy was calculated by extrapolation

of the linear part of plot between $(\alpha h\nu)$ and $h\nu$. Pure MgO band gap energy 4.3 eV Cu doped MgO band gap energy 4.7 eV as shown in Fig 3(b)&(d). On the Cu doping in MgO the band gap energy increased systematically from 4.3 to 4.7 eV. This band gap narrowing is expected to find important application in photocatalysis. [14,15]

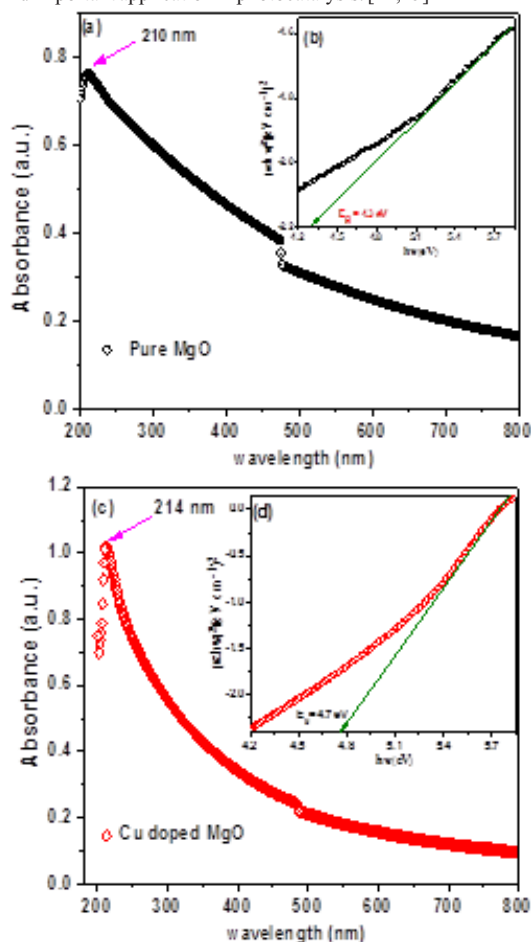


Figure 2 (a) & (c) UV absorption spectra of pure and Cu doped MgO NPs and (b) & (d) plot of $(\alpha h\nu)^2$ versus $h\nu$

3.3 SEM WITH EDAX

SEM is one of the promising techniques for the topography study of the sample and it gives important function regarding the growth mechanisms, shapes and size of the particle. The SEM image shows the formation of cluster. The SEM image shown in fig (b) different magnification the SEM results reveal that the annealed Cu doped MgO NMs are seemingly porous and highly agglomerated [16,17]. The elemental analysis of pure and Cu doped MgO nanoparticles were analysed by (EDAX). The EDAX of pure MgO shows prominent peaks of Mg and O as shown in Fig 4(c).

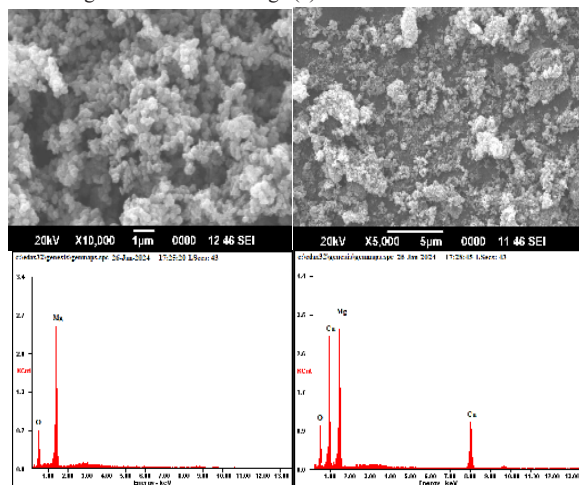


Figure 3 SEM WITH EDAX (a) Pure MgO (b) copper doped MgO

The weight contribution is 21.85 and 78.15 for Oxide and Magnesium respectively. This indicates the purity of Magnesium oxide as there is no impurity presence. The Cu doped MgO spectrum shows prominent peaks of Cu, Mg, O, as shown Fig 4(d). The weight contribution is 06.41, 65.53, 28.06 percentages for oxygen, Magnesium, copper respectively. It confirms that the adsorption of copper on MgO surface.

3.4 FTIR SPECTRUM

The FTIR spectrum of the MgO Powder Fig (5) exhibited characteristics bands at 3426 and 1636, which are attributed to the O-H stretching and vibration of water molecules. The band at 1056 cm⁻¹ Asymmetric C-O stretching vibration of C-O linkage. The bands that appear at low frequencies of 603 cm correspond to stretching vibration of Mg-O bending. In order to determine the molecular nature of the Cu doped MgO in Fig (5) FTIR spectra depend on the crystal structure of the material. [13,14] In all two-sample peak 3426 cm correspond to the vibration mode from the moisture absorbed on the sample and stretching vibration of the O-H bond and indicates the existence of hydroxyl groups and the surface of the sample. [18]

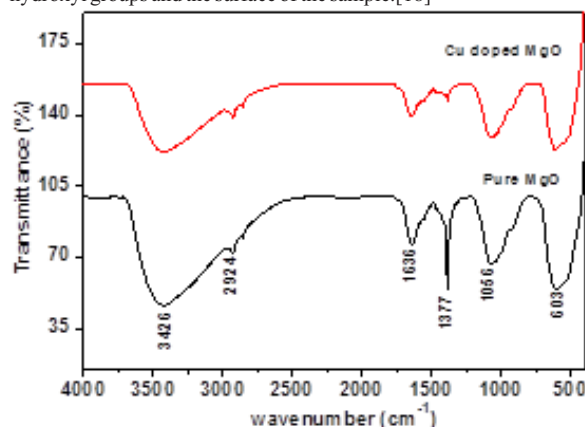


Figure 4 FTIR spectrum for pure MgO and Cu doped MgO

Table 1 : FT-IR Spectrum for pure MgO and Cu doped MgO

S.No	Compound	Peaks	Chemical Bonding	Functional group
1	Pure & Cu Mgo	3426	O-H stretching	hydroxyl groups
		2924	C-H stretching	Alkyl groups
		1636	O-H stretching	Water Molecule
		1377	Vibration stretching	Carbonate Species
		1056	Asymmetric C-O stretching	C-O linkage
		603	Vibration stretching	Mg-O bending

3.5 PHOTO LUMINESCENCE

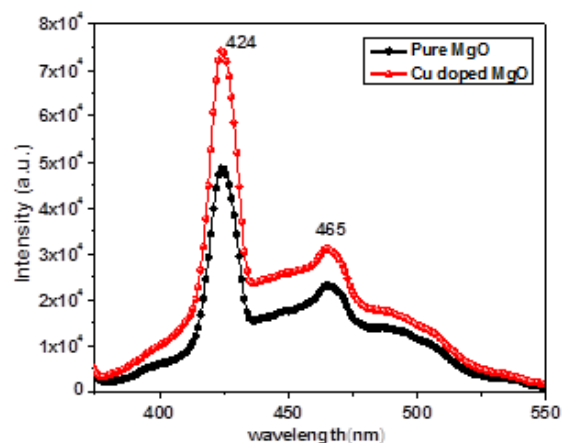


Figure 5 Photoluminescence spectrum for pure MgO and Cu doped MgO

The study of Photoluminescence (PL) properties of semiconducting

nanoparticles can generally emit two forms of light Band edge emission and surface defect emission, which occur in the region of wavelength below and above 500 nm, respectively. The PL spectra of MgO generally consist of two emission band on the UV Region near (370nm-400nm) and a broad band in visible region (400-700nm) of the spectrum.[19] The position of the peaks provides information about the depth and types of defects in the material in the emission spectra the peaks. The peaks were centre between (424nm) to and which are observed from UV region. whereas bands from visible region (465nm) are observed. Hence, PL spectrum shows that it belongs to band edge emission which below 500nm wavelength. [17,19]

4 CONCLUSION

The co-precipitation method was used to synthesize the pure and Cu doped MgO nanoparticles. XRD confirms that the existence of cubic crystal phase and the average diameter of pure and Cu doped MgO was around 17nm and 16nm. The doping of Cu with MgO enhances the antibacterial activity. The structural characterization was showed cluster and porous agglomeration with the structure for pure and Cu doped MgO nanoparticles respectively. further, it confirms that no impurities were found in EDAX analysis. Our study confirms that the lower band gap of UV suggested that the sample can be used for photocatalysis. Also, the PL shows that the corresponding emission peaks arise due to the oxygen vacancies which reflect the photocatalytic activities. FTIR confirms that the strong absorption peak and bending vibration modes of Mg-O. In the future, the characterization will also be extended towards the gas sensing application.

REFERENCES

1. Rani, V., Radha, K. P., & Ananthajothi, D. (2017). Structural analysis of Cu doped MgO nanoparticles using Co-precipitation Method. *INTERNATIONAL JOURNAL OF ENGINEERING DEVELOPMENT AND RESEARCH*, 5(4), 657-659.
2. Bhattacharya, R., & Mukherjee, P. (2008). Biological properties of "naked" metal nanoparticles. *Advanced drug delivery reviews*, 60(11), 1289-1306.
3. Wahab, R., Ansari, S. G., Dar, M. A., Kim, Y. S., & Shin, H. S. (2007, October). Synthesis of magnesium oxide nanoparticles by sol-gel process. In *Materials Science Forum* (Vol. 558, pp. 983-986). Trans Tech Publications Ltd.
4. Rajendran, V., Deepa, B., & Mekala, R. (2018). Studies on structural, morphological, optical and antibacterial activity of Pure and Cu-doped MgO nanoparticles synthesized by co-precipitation method. *Materials Today: Proceedings*, 5(2), 8796-8803.
5. Kaviyarasu, K., Manikandan, E., Kennedy, J., & Maaza, M. (2015). A comparative study on the morphological features of highly ordered MgO: AgO nanocube arrays prepared via a hydrothermal method. *RSC advances*, 5(100), 82421-82428.
6. Balamurugan, S., Ashna, L., & Parthiban, P. (2014). Synthesis of nanocrystalline MgO particles by combustion followed by annealing method using hexamine as a fuel. *Journal of Nanotechnology*, 2014(1), 841803.
7. Kulkarni, J., Ravishankar, R., Nagabhushana, H., Anantharaju, K. S., Basavaraj, R. B., Sangeeta, M., ... & Renuka, L. (2017). Structural, optical and photocatalytic properties of MgO/CuO nanocomposite prepared by a solution combustion method. *Materials Today: Proceedings*, 4(11), 11756-11763.
8. Subash, M., Chandrasekar, M., Panimalar, S., Inmozhi, C., Parasuraman, K., Utharkumar, R., & Kaviyarasu, K. (2023). Pseudo-first kinetics model of copper doping on the structural, magnetic, and photocatalytic activity of magnesium oxide nanoparticles for energy application. *Biomass Conversion and Biorefinery*, 13(4), 3427-3437.
9. Hadia, N. M. A., & Mohamed, H. A. H. (2015). Characteristics and optical properties of MgO nanowires synthesized by solvothermal method. *Materials Science in Semiconductor Processing*, 29, 238-244.
10. Sundarajan, S., Chandrasekaran, A. R., & Ramakrishna, S. (2010). An update on nanomaterials-based textiles for protection and decontamination. *Journal of the American Ceramic Society*, 93(12), 3955-3975.
11. Montero, J. M., Brown, D. R., Gai, P. L., Lee, A. F., & Wilson, K. (2010). In situ studies of structure-reactivity relations in biodiesel synthesis over nanocrystalline MgO. *Chemical Engineering Journal*, 161(3), 332-339.
12. Verziu, M., Cojocaru, B., Hu, J., Richards, R., Ciuculescu, C., Filip, P., & Parvulescu, V. I. (2008). Sunflower and rapeseed oil transesterification to biodiesel over different nanocrystalline MgO catalysts. *Green Chemistry*, 10(4), 373-381.
13. Glaspell, G., Hassan, Elzathary, A., Fuoco, L., Radwan, N. R., & El-Shall, M. S. (2006). Nanocatalysis on tailored shape supports: Au and Pd nanoparticles supported on MgO nanocubes and ZnO nanobelts. *The Journal of Physical Chemistry B*, 110(43), 21387-21393.
14. Rakmak, N., Wiyaratn, W., Bunyakan, C., & Chungsiriporn, J. (2010). Synthesis of Fe/MgO nano-crystal catalysts by sol-gel method for hydrogen sulfide removal. *Chemical Engineering Journal*, 162(1), 84-90.
15. Yu, X. Y., Luo, T., Jia, Y., Zhang, Y. X., Liu, J. H., & Huang, X. J. (2011). Porous hierarchically micro-/nanostructured MgO: morphology control and their excellent performance in As (III) and As (V) removal. *The Journal of Physical chemistry c*, 115(45), 22242-22250.
16. Kandiban, M., Vigneshwaran, P., & Potheher, I. V. (2015, January). Synthesis and characterization of MgO nanoparticles for photocatalytic applications. In *department of physics, Bharathidasan institute of technology (BIT) Campus, Anna University, Tiruchirappalli, Tamilnadu, India, conference paper* (Vol. 3, pp. 941-947).
17. Hu, J., Song, Z., Chen, L., Yang, H., Li, J., & Richards, R. (2010). Adsorption properties of MgO (111) nanoplates for the dye pollutants from wastewater. *Journal of Chemical & Engineering Data*, 55(9), 3742-3748.
18. Moussavi, G., & Mahmoudi, M. (2009). Removal of azo and anthraquinone reactive dyes from industrial wastewaters using MgO nanoparticles. *Journal of hazardous materials*, 168(2-3), 806-812.
19. Pashchanka, M., Hoffmann, R. C., & Schneider, J. J. (2010). Controlled synthesis and characterisation of MgO nanoparticles, thin films and polycrystalline nanorods derived from a Mg (ii) single source precursor. *Journal of Materials Chemistry*, 20(5), 957-963.