International Journal of Innovative Technology and Exploring Engineering (IJITEE)
ISSN: 2278-3075 (Online), Volume-9 Issue-4, February 2020

# Surfactant Free Synthesis of Magnesium Oxide Nanotubes by Simple Chemical Co-Precipitation Method

**Monika Tandon, Pratima Chauhan**

***Abstract*: A simple cost effective preparation of Magnesium oxide nanoparticles in nanotube morphology is reported using Chemical co-precipitation method. As prepared magnesium oxide nanoparticles were characterized using UV-visible spectroscopy, X-ray Diffraction, Field Emission Scanning Electron Microscope and Energy dispersive X-ray spectroscopy. As prepared magnesium oxide nanoparticles were found in nanotube morphology whose inner and outer diameter were 31 nm and 78 nm. The band gap of as prepared nanotubes were found to be 5.37eV with maximum absorbance at 200 nm.***

***Keywords*: Chemical co-precipitation method, magnesium oxide nanoparticles, nanotube morphology.**

## I. INTRODUCTION

In recent years, Magnesium oxide nanoparticle is of great interest among wide band gap metal oxides for the researchers. Because of their peculiar properties, magnesium oxide nanoparticles are being used in many applications like solar cells [1]-[3], sensors [4]-[6], catalysts [7]-[9], antibacterial or antipathogenic applications [10]-[12] and environmental remediation applications [13]-[14] etc. The performance of nanoparticles in all these applications depends mainly on their size, morphology and structure [15]-[16]. Nanostructured magnesium oxide can be synthesized in different morphologies like spherical [10], nanorod [17], nanoflake [9], hierarchical nanostructure etc [6]. There are many methods to synthesize magnesium oxide nanoparticles like hydrothermal method [14], chemical method [17], sol-gel method [18], combustion method [13] and electrospinning method [15] etc. Magnesium oxide nanoparticles have also been synthesized by Green route using neem leaves [19]. For synthesis of Magnesium oxide nanoparticles, Magnesium nitrate ($Mg(NO_3)_2$ [5], [7], Magnesium acetate ($Mg(CH3COO)_2$) [8] Magnesium sulfate heptahydrated ($MgSO_4.7H_2O$) [20] and Magnesium chloride ($MgCl_2.6H_2O$) [6], [9] etc are being used as precursors.

**Revised Manuscript Received on February 28, 2020.**
***** Correspondence Author
**Monika Tandon***, Department of Physics, University of Allahabad, Allahabad, India. Email: monika.tandon31@gmail.com
**Pratima Chauhan**, Department of Physics, University of Allahabad, Allahabad, India. Email: mangu167@yahoo.co.in



In the present work we have synthesized magnesium oxide nanotubes by Chemical Co-precipitation method and studied their optical, structural and morphological properties. Chemical Co-precipitation method is easy and cost-effective method for synthesis.

## II. EXPERIMENTAL DETAILS

### A. Materials/Chemicals Used

Magnesium acetate tetrahydrate Pure ($(CH_3COO)_2Mg.4H_2O$) (Merck), Sodium Hydroxide (NaOH) (Qualigens), Ethanol (Merck) and Distilled water (Merck) were used with no further purification

### B. Synthesis of Magnesium Oxide Nanoparticles

Magnesium oxide nanotubes were synthesized by simple Chemical Co-precipitation method. Two separate homogeneous aqueous solution of Magnesium acetate (0.2 M, 50 mL) and Sodium hydroxide (0.4 M, 50 mL) were prepared at constant stirring.
Prepared aqueous solution of sodium hydroxide was mixed drop wise with aqueous solution of magnesium acetate at constant stirring. The colour of the solution turned to milky white from transparent. The mixture was stirred at constant stirring for 3 hours. Then the solution was kept for precipitation and the precipitate was filtered after washing it several times by distilled water and ethanol. The whole reaction was performed at room temperature.

### C. Characterizations

For studying the different properties of synthesized sample following characterization techniques were used: The crystallographic information was obtained with the powder XRD method on a Proto A-XRD Bench top Diffractometer with fixed Cu-Kα (0.154 nm). The morphology and surface properties were investigated using Nova Nano SEM 450. UV-Vis absorption spectra were recorded on the Unicam 5625 UV-Visible spectrometer in the range 195 nm - 325 nm.

## III. RESEULT AND DISCUSSION

### A. UV-Visible characterization of as synthesized material

For studying the optical properties and band gap of synthesized nanoparticles UV-Vis characteristic has been taken. Fig. 1 shows the UV-Vis spectra of synthesized sample.

It shows intense absorbance peak exist at 200 nm while a broad absorbance peak exist at 270 nm. Fig. 2 shows the tauc plot of synthesized sample. The band gap of synthesized nanomaterial is 5.73 eV. The band gap of as-synthesized material is determined by the equation:

$$\alpha h\nu = A\,(h\nu - E_g)^n$$

Where α is the absorption coefficient, $E_g$ is the absorption band gap, A is constant, n depends on the type of transition, h is plank constant and ν is the frequency. As Magnesium oxide has direct allowed transitions, we have chosen n = 1/2. The band gap of the material is calculated from the $(\alpha h\nu)^2$ vs hν plot (Tauc plot).

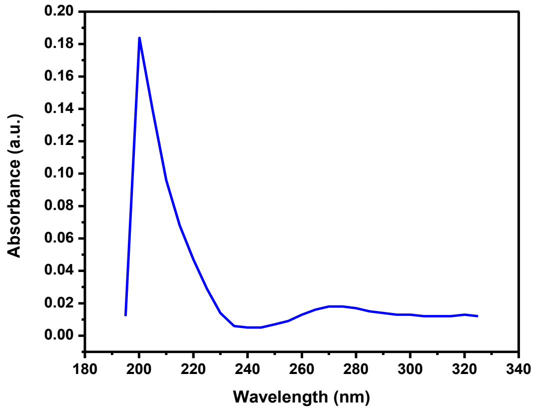


**Fig 1: UV-Visible spectra of synthesized Magnesium oxide nanomaterial**

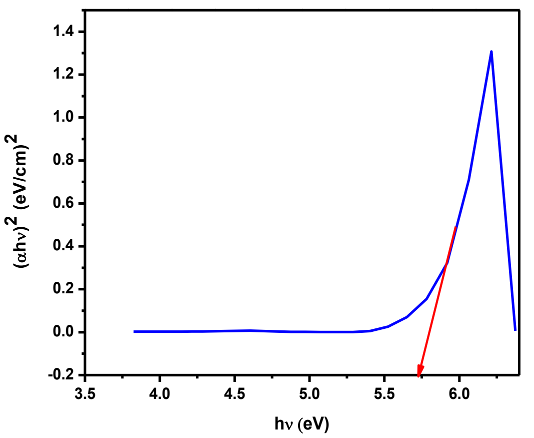


**Fig. 2: Tauc plot of synthesized magnesium oxide nanomaterial**

## B. Structural analysis of synthesized nanomaterial

Fig. 3 shows the XRD pattern of synthesized magnesium oxide nanoparticles. The XRD peaks assigned with '*' and '#' symbol correspond to the diffraction peaks obtained for Rhombohedral structure of $MgO_4$ (JCPDS file no. 27-0759) and Hexagonal phase of $Mg(OH)_2$ (JCPDS file no. 75-1527) respectively. The average crystallite size has been calculated for $MgO_4$ using the Scherrer formula [D= kλ/βcosθ]. In this formula, k is the constant (=0.91), λ is the wavelength of X-ray radiation used, β is the full width at half maximum (FWHM) intensity of diffracted peak and θ is the Bragg diffraction angle. The calculated average crystallite sizes for $MgO_4$ nanoparticles are 34.04 nm.

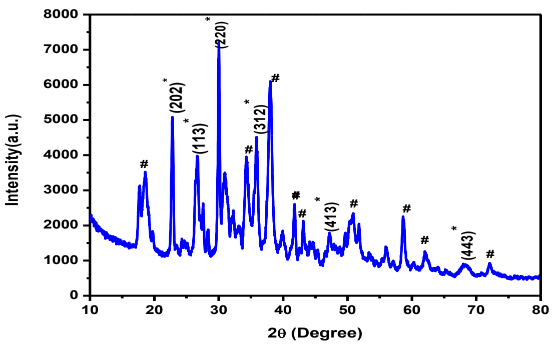


**Fig. 3: XRD pattern of synthesized nanomaterial**

## C. Morphological studies of synthesized nanomaterial

The FESEM images of synthesized nanomaterial are shown in Fig. 4 at different magnifications. It can be clearly seen that Magnesium oxide nanoparticles possessed nanotubes morphology with agglomeration. The diameter of outer wall of nanotubes is approximately ≈ 78 nm and inner diameter is approximately ≈ 31 nm. The diameters of nanotubes were analyzed using Image J software.

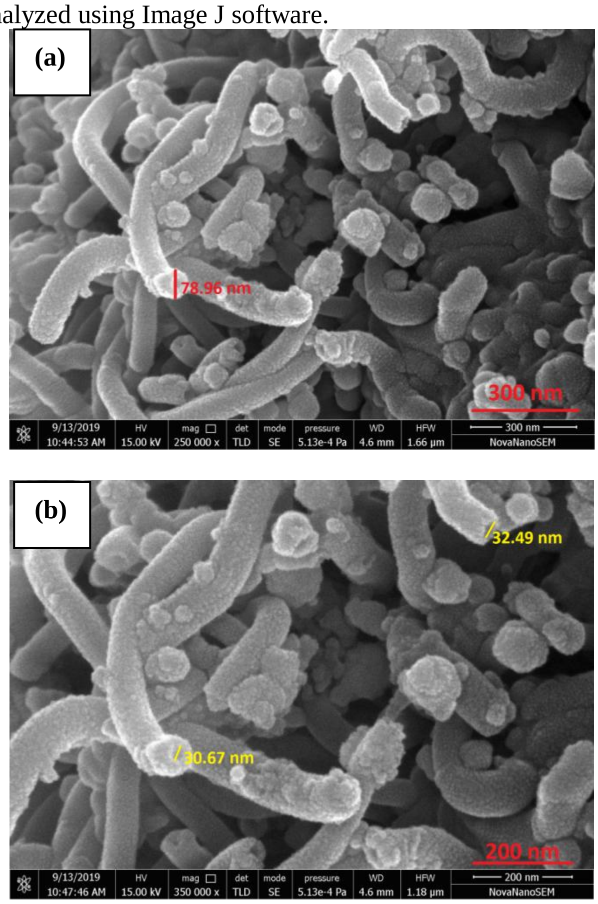


**Fig. 4: FESEM images of synthesized magnesium oxide nanotubes at (a) 300 nm magnification and (b) 200 nm magnification.**

## D. Chemical composition analysis of synthesized nanomaterial

The energy dispersive X-ray spectrum of synthesized nanomaterial is shown in Fig. 5 which shows only Mg and O peak. The other peaks correspond to C and Cu which were used for holding the sample.

This confirms the high purity of magnesium oxide nanomaterial synthesis.

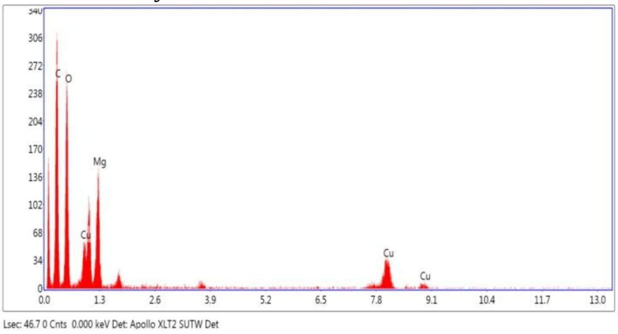


**Fig. 5: EDX spectrum of synthesized magnesium oxide nanomaterial**

## IV. CONCLUSION

From our present work, it is easy to conclude that Magnesium oxide with nanotube morphology can easily be synthesized using Chemical Co-precipitation method. With this method, we can easily synthesize magnesium oxide in nanotube morphology without using any surfactant or capping agent. The synthesized magnesium oxide nanotubes have shown wide band gap (5.73 eV) and the can be used in different applications like solar cells, catalysts and sensors etc.

## ACKNOWLEDGEMENT

We are thankful to U.G.C. for the financial support and DST for providing FIST grant to Physics Department, University of Allahabad, CIF (IIT-BHU) for providing FESEM and EDX facilities and CIR (Motilal Nehru Institute of Technology, Allahabad) for providing XRD facility. Authors are thankful to CSIR for financial support.

## REFERENCES

1. H. S. Jung, J. K. Lee and M. Nastasi, "Preparation of Nanoporous MgO-Coated TiO2 Nanoparticles and Their Application to the Electrode of Dye-sensitized Solar Cells." Langmuir, 21, 2005, pp. 10332-10335.
2. J. Ma, G. Yang, M. Qin, X. Zheng, H. Lei, C. Chen, Z. Chen, Y. Guo, H. Han, X. Zhao and G. Fang, "MgO Nanoparticle Modified Anode for Highly Efficient $SnO_2$-Based Planar Perovskite Solar Cells." Advanced Science News, 4, 2017, pp. 1700031-1700040.
3. C. Photiphitak, P. Rakkwamsuk, P. Muthitamongkol and C. Thanachayanont, "Performance Enhancement of Dye-sensitized Solar Cells by MgO Coating on TiO2 Electrodes." International Journal of Materials and Metallurgical Engineering, 6, 2012, pp. 411-415.
4. S. K. Shukla, G. K. Parashar, A. P. Mishra, P. Mishra, B. C. Yadav, R. K. Shukla, L. M. Bali and G. C. Dubey, "Nano-like magnesium oxide films and its significance in optical fiber humidity sensor." Sensors and Actuators B: Chemical, 98, 2004, pp. 5-11.
5. H. Li, M. Li, G. Qiu, C. Li, C. Qu and B. Yang, "Synthesis and characterization MgO nanocrystals for biosensing applications." Journal of Alloys and Compounds, 632, 2015, pp. 639-644.
6. K. Ahmad and S. M. Mobin, "High surface area 3D-MgO flowers as the modifier for the working electrode for efficient detection of 4-chlorophenol." Nanoscale Adv., 1, 2019, pp. 719-727.
7. N. Sutradhar, A. Sinhamahapatra, S. K. Pahari, P. Pal, H. C. Bajaj, I. Mukhopadhyay and A. B. Panda, "Controlled Synthesis of Different Morphologies of MgO and their use as solid base catalysts." J. Phys. Chem. C, 115, 2011, pp. 12308-12316.
8. A. B. Patil and B. M. Bhanage, "Novel and green approach for the nanocrystalline magnesium oxide synthesis and its catalytic performance in Claisen-Schmidt condensation." Catalysis Communication, 36, 2013. pp. 79-83.
9. Y. Zhang, M. Ma, X. Zhang, B. Wang and R. Liu, "Synthesis, characterization, and catalytic property of nanosized MgO flakes with different shapes." Journal of Alloys and Compounds, 590, 2014, pp. 373-379.
10. M. R. Bindhu, M. Umadevi, M. K. Micheal, M. V. Arasu and N. A. Al-Dhabi, "Structural, morphological and optical properties of MgO nanoparticles for antibacterial applications." Materials Letters, 166, 2016, pp. 19-22.
11. P. Bhattacharya, S. Swain, L. Giri and S. Neogi, "Fabrication of magnesium oxide nanoparticles by solvent alteration and their bactericidal applications." Journal of Materials Chemistry B, 7, 2019, pp. 4141-4152.
12. Y. He, S. Ingudam, S. Reed, A. Gehring, T. P. Strobaugh Jr. and P. Irwin, "Study on the mechanism of antibacterial action of magnesium oxide nanoparticles against foodborne pathogens." Journal of Nanobiotechnology, 14, 2016, pp. 1-9.
13. B. Nagappa and G. T. Chandrappa, "Mesoporous nanocrystalline magnesium oxide for environmental remediation." Mesoporous Materials, 106, 2007, 212-218.
14. T. H. Y. Duong, et al., "Synthesis of Magnesium oxide nanoplates and their application in nitrogen dioxide and sulfur dioxide adsorption." Hindawi Journal of Chemistry, vol. 2019, 2019, 9 pages.
15. R. Murugan, K. Ramamoorthy, S. Sundarrajan and S. Ramakrishna, "Magnesium oxide nanotubes: synthesis, characterization and application as efficient recyclable catalyst for pyrazolyl 1,4-dihydropyridine derivatives." Tetrahedron, 68, 2012, pp. 7196-7201.
16. M. Giannouli and F. Spiliopoulou, "Effects of the morphology of nanostructured ZnO films on the efficiency of dye-sensitized solar cells." Renewable Energy, 41, 2012, pp. 115-122.
17. Y. Zheng, X. Zhang, X. Wang, Q. Wang, Z. Bai and Z. Zhang, "Morphological and surface structural evolutions of MgO particles from paralleograms to rods." CrystEngComm, 18, 2016, pp. 2612-2616.
18. M. Sharma, D. Gandhi and M. Sharma, "Synthesis of nanostructured magnesium oxide by sol gel method and its characterization." International Journal of Pharmaceutical sciences and research, 9, 2018, pp. 1576-1581.
19. S. K. Moorthy, C. H. Ashok, K. V. Rao, C. Viswanathan, "Synthesis and Characterization of MgO nanoparticles by neem leaves through green method." Materials Today: Proceedings, 2, 2015, pp. 4360-4368.
20. A. Pilarska, M. Wysokowski, E. Markiewicz and T. Jesionowski, "Synthesis of magnesium hydroxide and its calcinates by a precipitation method with the use of magnesium sulfate and poly (ethylene glycols)." Powder Technology, 235, 2013, pp. 148-157.

## AUTHORS PROFILE

**Ms. Monika Tandon** is a D.Phil. student at Department of Physics, University of Allahabad under the supervision of Prof. Pratima. She has three publications on her own credit.

**Dr. Pratima** is Professor in Department of Physics, University of Allahabad. Her research field is applications of nanomaterials in Solar Cells, Control of Plant diseases, Pollution Control, Targeted Drug Delivery Systems and Gas sensors.