Nanoscale Reports

RESEARCH ARTICLE

Eleyon Publishers

DOI:10.26524.nr.4.4

4(1) (2021) 19-24

OPEN ACCESS

Synthesis and Characterization studies of pure Mg and Cu Doped MgO Nanoparticles by Co-Precipitation Method

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Abstract

The pureMgO and Cu doped MgO nanoparticles were synthesized by Co - Precipitation method. The synthesized samples are characterized by X-ray diffraction, Scanning Electron Microscope, Fourier transform infrared spectrometer&UV-Vis spectrometer. The XRD studies of the sample confirmed the formation of cubic andorthorhombic structure.The particle size and lattice constants were analysed. The XRD patterns show that the average particle size is in the range of 6nm for MgOand Cu doped MgO Nanoparticles was found to increase as 8 nm respectively. The presence of functional groups of thepure& Cu doped Magnesium Oxide nanoparticles were confirmed by FTIR analysis.SEM results show both the presence of cubic and agglomeration of the smaller particles.A broad absorbance band from UV-Vis spectra is located at around 264 nm and 260 nm. This is the simple synthesis method and they are used in optical and gas sensor applications, telecommunication cables, conductor wires, connector wires and automotive switches.

Keywords: X-ray diffraction, Scanning Electron Microscope, FT-IR & UV-VIS, Co-Precipitation method.

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How to cite this article: P.Praveena, Synthesis and Characterization studies of pure Mg and Cu Doped MgO Nanoparticles by Co-Precipitation Method 4(1), 19-24 Retrieved from https://nanoscalereports.com/index.php/nr/article/view/67

Received: 4 March 2021 **Revised:** 7 April 2021 **Accepted:** 9 April 2021

I.INTRODUCTION

Magnesium oxide is an interesting basic oxide that has many applications in catalysis, adsorption and in the synthesis of refractory ceramics. It is a unique solid because of its highly ionic character, simple and crystal structure, and it can be prepared in widely variable particle sizes and shapes.It has been documented that the shape and size of nanocrystalline magnesium oxide particles endow them with high specific surface and reactivity, because of the high concentration of edge/corner sites and structural defects on their surface.[1,2] MgO is an important material which has many applications in catalysis, toxic waste remediation, paint, superconducting products and anti-bacterial activities. The compound MgO have boiling and melting points as 3600ºC and 2852ºC. [1,2] Pure CuO is one of the most typical p-type metal-oxide semiconductors and exhibits interesting antiferromagnetic ordering below its Néel temperature of 225 K.

The co-precipitation method is one of the most appropriate ways of synthesizing a nanopowder. Coprecipitation is the name given by analytical chemists to a phenomenon whereby the fractional precipitation of a specified ion in a solution results in the precipitation not only of the target ion but also of other ions existing side by side in the solution. The additional precipitation of unwanted ions is, of course, an impediment to the analytical process. Some of the most commonly substances used in coprecipitation operations are hydroxides, carbonates, sulphates and oxalates. [5]

2. Experimental details

2.1. Materials

Magnesium chloride,Copper chlorideand reagents included a KOH are used for the synthesis Pure and Cu doped MgO nanoparticles. All chemicals, double distilled water and reagents used were procured from Sigma-Aldrich (United States of America) and Merck (Germany) and were of analytical grade.

2.2. Sample Preparation

The pure magnesium Nanoparticles were synthesized by co-precipitation method. 0.6 mol of magnesium chloride was added in 100ml of distilled water and it was stirring for half an hour. Then, 0.4 mol of KOH was added to the precursor solution. The whole solution was continuously stirred for 3 hours at room temperature. Then, finally the resulting solution is kept at room temperature for three hours under constant stirring. A white precipitate is formed. Then the whole solution is washed with distilled water for several times then it is kept in oven at 100°C for 3 hours. Finally it was calcinated in a muffle furnace at 300°C for 2 hours and then cooled down to room temperature. Then obtained sample is grained finely using mortar. Finally pure magnesium oxide Nanoparticles were obtained.

The Cu doped MgOnanoparticle was synthesized by co-precipitation method. 0.6 mol of magnesium chloride was added to 100 ml of distilled water and it was stirred for half an hour. Then, 0.01 mol of copper oxide was added. 0.4 mol of KOH was added to the precursor solution. The whole solution was continuously stirred for 3 hours at room temperature. Then, finally the resulting solution is kept at room temperature for three hours under constant stirring. The bluish green precipitate is formed. Then the whole solution is washed with distilled water for several times then it is kept in oven at 100°C for 3 hours. Finally it was calcinated in a muffle furnace at 300°C for 2 hours and cooled down to room temperature. Then obtained

sample is grained finely using mortar. Finally Cu doped MgONanoparticles are obtained.

3. Results and Discussion

3.1 Powder X-ray diffraction (XRD) analysis

Fig 3.1 (a) and (b) show the XRD pattern of undoped and Cu doped MgO2 Nanoparticles calcinated at 300°C for 2 hrs respectively. All the spectra were recorded in the ranges from 10°C to 90°C 2θ. The XRD pattern of Fig. 1.9 (a) show prominent peaks observed at 2θ values of 30.2°, 34.9°, 50.3° and 59.82° indexed to the (111), (200), (220) and (221) planes respectively. These peaks belong to the Cubic structure of MgO Nanoparticles, which matches well with the standard XRD pattern (JCPDS card No: 76-1363). [6]

Interestingly, Fig. 3.1 (b) shows prominent peaks observed at 2θ values of 30.22°, 34.99°,50.39°, and 59.93° indexed to the (051), (101), (280), and (082) planes respectively. These peaks belong to the orthorhombic structure of Cu Nanoparticles, which matches well with the standard XRD pattern (JCPDS card No: 41-1364). [7]

The average diameter (D) of the pure MgO and Cu doped MgO Nanoparticles are estimated from the Scherer's formula using the FWHM for undoped <111> 100% peak at 30.2° 2θ value, for doped <051> 100% peaks at 30.22° 2θ value.

$$D=K\lambda/\eta\cos\theta$$

Where λ is the X-ray wavelength (1.5418Å), η is the full width of the peaks in radians at half-maximum intensity; θ is the Bragg's angle and K the Scherer's constant (0.9). The estimated average diameter of pure MgO Nanoparticles calcinated at 300°C is found to as 6 nm and respectively Cu doped MgO Nanoparticles was found to increase as 8 nm respectively. The reduction of FWHM is the clear indication of the increase of average diameter of the pure MgO and Cu doped MgOnanoparticles. [8]

3.2 Fourier Transform Infra-Red spectroscopy Analysis (FTIR)

Fourier transformation infrared spectroscopy (FTIR) was carried out in a KBr medium at wave number ranging from 4400 to 400 cm-1 with a resolution of 4 cm-1 to evaluate the purity and chemical composition of the prepared pure MgO, Cudoped MgO Nanoparticles. FTIR spectroscopy used for functional group identification and is based upon the simple fact that chemical substance shows selective identification.

Fig 3.2 (a) and (b) show the FTIR spectra of pure MgOand Cudoped MgONanoparticles calcinated at 300°C respectively. The strong absorbance bands appear at around 678.94 to 680.12 cm-1 was assigned to the Cu-O and Mg-O stretching modes which clearly confirm the formations of Cu doped MgO2 Nanoparticles. The absorbance band at 3386 to 3375 cm-1 is due to the presence

2.3 FLOW CHART

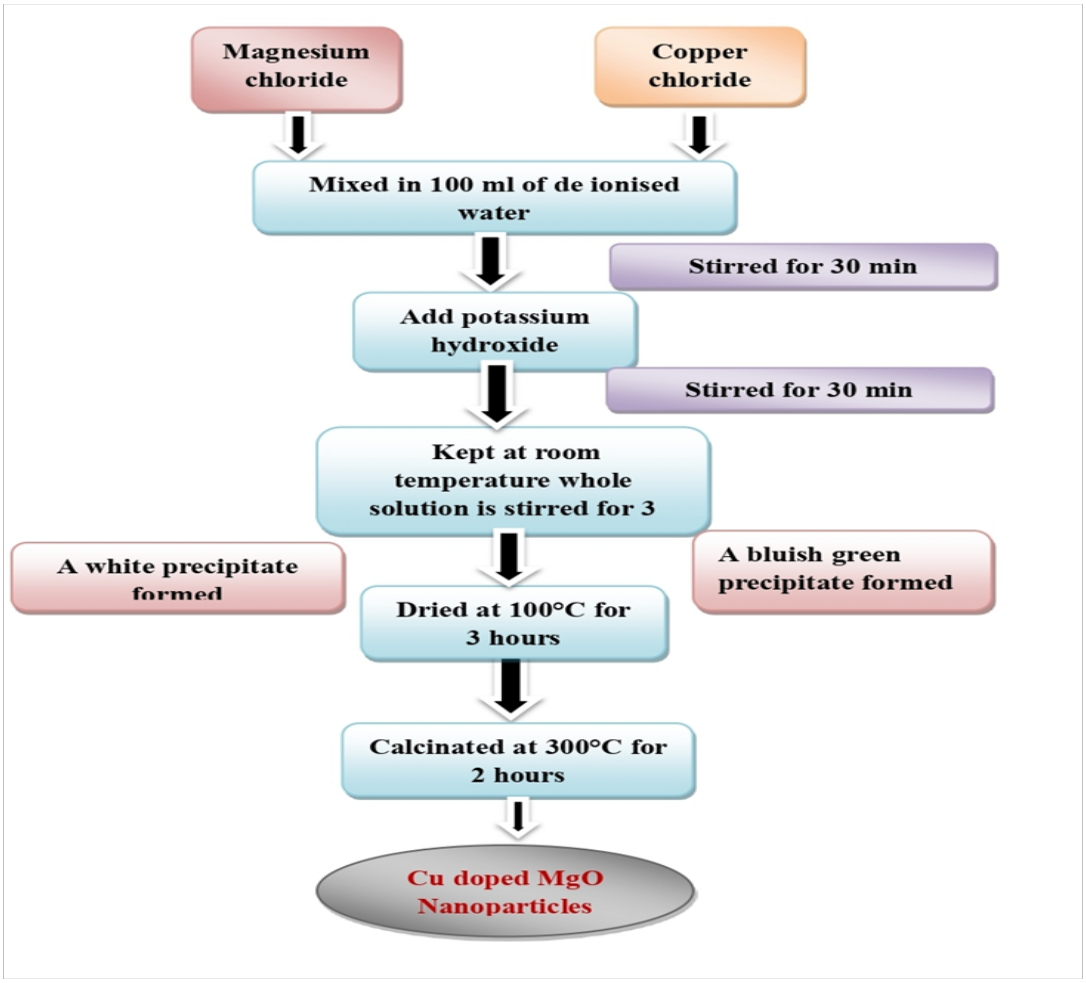

Fig 1.1Flow chart for the synthesis of Pure and Cu doped MgO Nanoparticles

of O-H stretching band. The band appeared at 2265 to 2268cm-1 is due to the presence of C=O stretching band. The band appeared at 1637to 1638.77 cm-1 is due to presence of C-H symmetric stretching of band. [9-15]

3.3 Scanning electron microscopy (SEM) analysis:

The surface morphology of pure MgOandCu doped MgO nanoparticle calcinated at 300°C were shown in Fig. 3.3 (a) shows the cubic shape of pure MgO Nanoparticles. Fig 3.3 (b) shows the agglomeration of Cu doped MgO Nanoparticles. These Nanoparticles are very dense and closely arranged together on the sample surfaces and very clear boundaries are also observed. [16,17,18,19]

3.4 UV visible spectroscopy analysis

UV-VIS spectrum of the undoped and Cu doped MgO nanoparticles calcinated at 300°C for 2hrs recorded in the wavelength ranges from 200 to 800 by using spectrophotometer at room temperature in order to analyse the absorption band of the undoped Cu doped MgO nanoparticles respectively, are shown in the figure.Fig 3.4 (a) shows the absorbance band of undoped MgOis located around 264 nm and Fig 3.4 (b) shows the absorbance band of Cu doped MgOnanoparticlesis around at 260 nm. The band gap of undoped and Cu doped MgOis found to be 7.5 eV and 7.6 eV. [20,21,22,23]

5. CONCLUSION

- Undoped MgO and Cu doped MgO Nanoparticles calcinated at 3000C were obtained using a “Co-precipitation method”.
- The XRD pattern of Pure MgO and Cu doped MgO Nanoparticles shows that they were in cubic and orthorhombic structure.
- From FTIR analysis, the presence of functional groups of the Cu doped magnesium nanoparticles was confirmed.
- The SEM images clearly suggest the formation of cubic shape and agglomeration of undoped and Cu doped MgO Nanoparticles.
- A broad absorbance band from UV-Vis spectra is located at around 264 nm and 260 nm. The band gap is calculated.

Data Availability

The data used to support the findings of this study are included within the article. More information could be obtained from the authors upon request.

Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

Acknowledgments
The authors would like to thank themanagement, principal and head of the department for their continuous support and encouragement for making this article successful as part of my research.

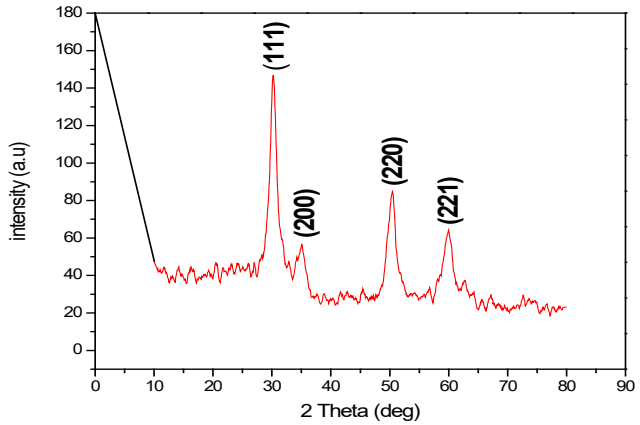

Fig.3.1 (a) XRD pattern for pure Magnesium oxide Nanoparticles

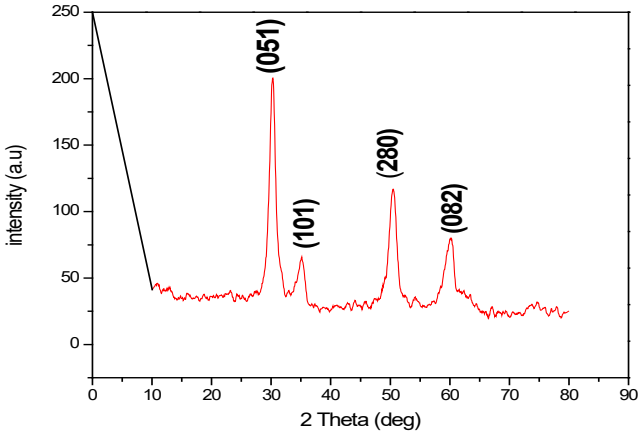

Fig 3.1 (b) XRD Pattern for Cu doped MgO Nanoparticles

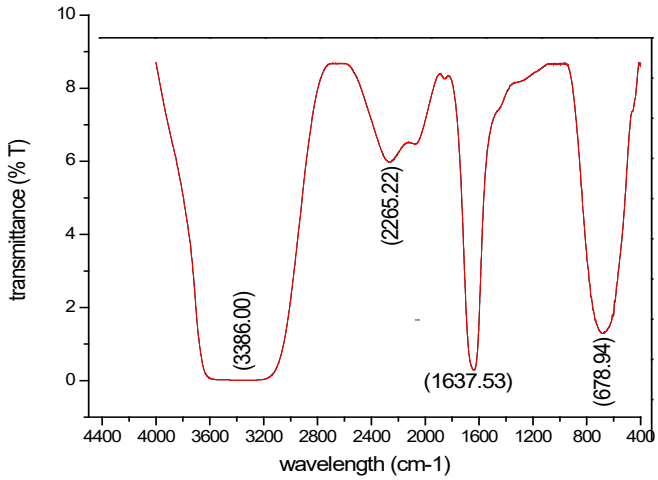

Fig.3.2 (a) FTIR undoped Magnesium oxide Nanoparticles

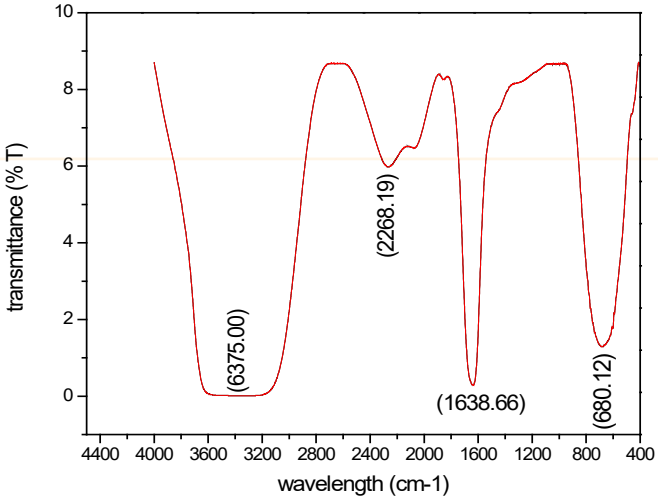

Fig.3.2 (b) FTIR Cu doped MgO Nanoparticle

Table 1 FTIR Vibrational band assignment for pure and doped MgO

Vibrational band	Absorbance band (cm-1)
C-H Symmetric stretching	2265 to 2268
O-H Stretching band	3386 to 3375
C=O Stretching band	1637 to 1638.66
Cu-O and Mg-O Stretching band	678.94 to 680.12

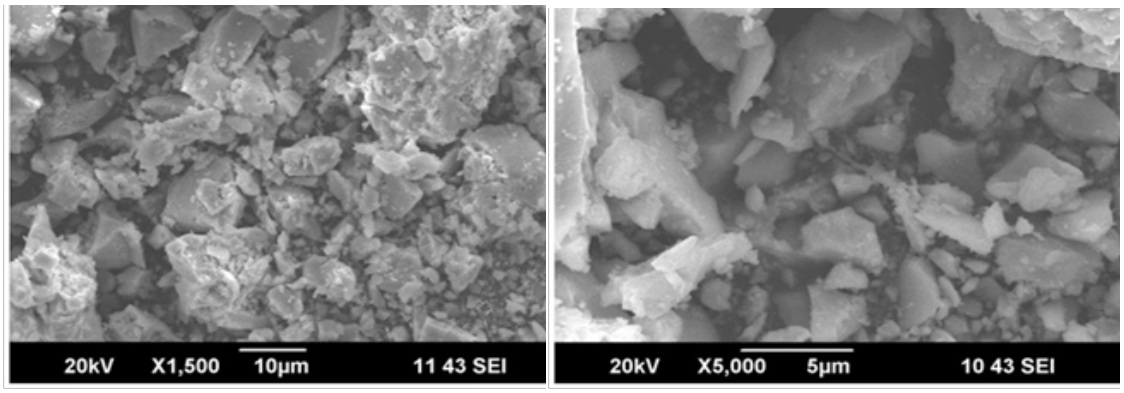

Fig 3.3 (a) SEM Image for pure Magnesium Nanoparticles

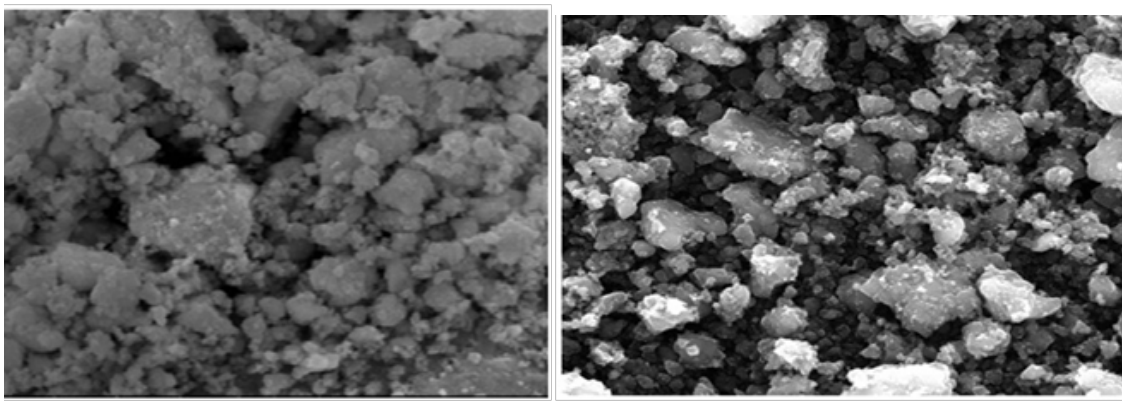

Fig 3.3 (b) SEM image for Cudoped MgO Nanoparticles

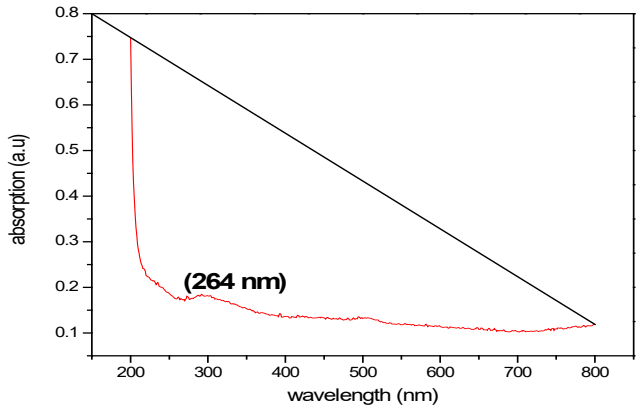

Fig. 3.4 (a) UV undoped Magnesium oxideNanoparticles

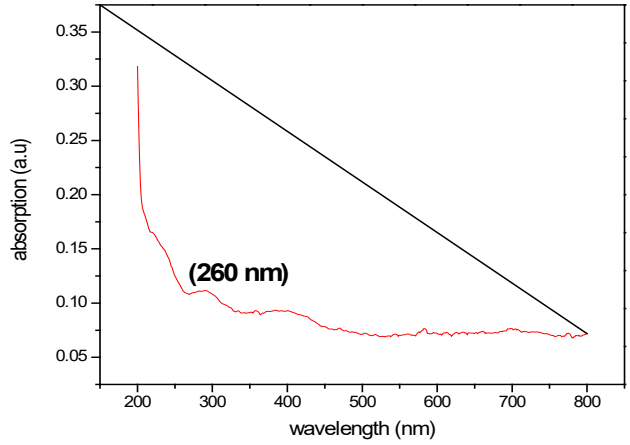

Fig. 3.4 (b) Cu doped MgONanoparticles

Acknowledgments

The authors would like to thank themanagement, principal and head of the department for their continuous support and encouragement for making this article successful as part of my research.

Funding

No funding was received to carry out this study.

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