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Preparation and Characterization of Cu Doped MgO Nanoparticles using Co-precipitation Method

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Abstract: The objective of the research was mainly focused on the synthesis of Cu dopped MgO nanoparticle at room temperature. The segment composition of the Synthesized Cu dopped MgO nanoparticles were confirmed from powder X-ray diffractometer (XRD). In the present work we focused on the synthesis of MgO and Cu doped MgO nanoparticles using Co-precipitation method. From the XRD analysis, the crystalline size of MgO and Cu doped MgO nanoparticles are calculated by Debye Scherrer's formula and found to be 25.15nm 13.37nm respectively.
Keywords: Synthesis, XRD, UV-Vis, Cu doped MgO.

I. INTRODUCTION

In recent years, synthesis of nano structured oxide materials have been attracted considerable attention, Oxide semiconductor nanostructures have been widely investigated in current years because of their radiant properties [Zieliński, P. A., (1993) Travitzky, N., (2003) and Martínez E., (2003)]. Nanotechnology is a formation of handling and the usage of resources having a size range of 1-100 nm, and they are known as Nanoparticle. scientists concentrated their focus on nanoparticles due to their different magnetic, chemical sensors, spintronics, optoelectronic(1-6) and physicochemical properties that are regulated by their size and shape distribution (Shah M, et al 2015).Nanoparticles are used in different applications like catalysts, chemical sensors, electronic components, imaging for medical diagnostic and medical diagnostic protocols, pharmaceutical products, drug delivery (various nanoparticle platforms especially liposomes, polymeric nanoparticles, inorganic nanoparticles and dendrimers have acquired significant attention) because of their novel properties like physical, chemical and optoelectronic properties (Bhattacharya R and Mukherjee P 2008) .MgO is an important material, which used in many applications like catalysis, toxic waste remediation, paint, superconducting products(7), anti-bacterial activities(8) against food borne pathogens (Tony Jin and Yiping He 2011 ; K. Mageshwari, 2013; MohdSufriMastuli, 2012).. The experimental conditions used in the preparation of these resources play animportant role in the particle size of the product [Yang Y., et al (2004), Xu B.Q., et al (2001)].MgO is typical wide band gap semiconductor; it possesses unique optical, electronic, magnetic, thermal, mechanical and chemical properties due to its characteristic structures [K. Ramanujam, et al (2014)]. In this present paper, MgO and Cu doped MgO nanoparticles are prepared by co-precipitation method. The samples were synthesized under standard laboratory conditions in clean room and analyzed using such as X-ray diffraction (XRD)

II. EXPERIMENTAL PROCEDURE

In this present study the Synthesis of Cu doped MgO Nanoparticles is done by using Co- precipitation method and the Characterization Techniques are discussed.

A. *Synthesis of MgO Nanoparticles*

To prepare MgO nanoparticles, 100mL of 0.4 M NaOH solution is added drop-wise into a solution containing 100mL of 0.6M Magnesium Chloride solution under constant stirring at room temperature, double distilled water is used to prepare solutions. Then the resulting solution is kept at 30o C(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 100oC in an oven for 3 hours. The obtained samples are calcinated in at 300oC for 2 hours to get MgO nano particles. In a typical procedure for the preparation of MgO nanoparticles, 0.1M of Magnesium chloride (MgCl2) was dissolved in 100 ml de-ionised water and 0.2M of aqueous NaOH solution was added drop wise into the above solution under constant stirring. The precipitate is centrifuged by using ultra centrifuge, then it was dried and make it powder. A magnetic stirrer or magnetic mixer is a laboratory device that employs a rotating magnetic field to cause a stir bar immersed in a liquid to spin very quickly, thus stirring it ,the rotating magnetic field may be created either by a rotating magnet or a set of stationary electromagnets, placed beneath the vessel with the liquid. Since glass does not affect a magnetic field appreciably and most chemical reactions take place in glass vessels because magnetic stir bars work well in glass vessels.

B. Synthesis of Cu doped MgO Nanoparticles

To prepare Cu doped MgO nanoparticles, 100 mL of (0.4M) NaOH 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 waskept at room temperature for three hours under constant stirring. Obtained bluish green precipitate is washed several times with distilled water and dried at 100C in an oven for 3 hours. Finally the precalcinated in at 300^o C for 2 hours to get Cu doped MgO nanoparticles.

III. RESULTS AND DISCUSSIONS

A. Powder X-ray Diffraction

X-ray diffraction (XRD) is an important technique in the field of materials Characterization to obtain structural information on an atomic scale from both crystalline and non-crystalline materials. XRD is a non-destructive and can be successfully applied to determine crystal structures of metals and alloys, minerals, inorganic compounds, polymers and organic materials as well as to derive such information as crystallite size, lattice strain, surface and interface roughness, chemical composition and crystal orientation. Because the wavelength of X-rays is comparable to the size of atoms, they are ideally suited for probing the structural arrangement of atoms and molecules in a wide range of materials.

The energetic X-rays can penetrate deep in to materials and provide information about the bulk structure. Diffraction is governed by the Bragg's formula, 2dsinθ=nλ, here 'd'is inter-planar spacing, θ is angle of diffraction, λ is wavelength of the incident beam and n is order of diffraction,in a typical setup,a collimated beam of X-rays is incident on the sample. The intensity of diffracted X-ray is measured as a function of diffracted angle 2θ. The intensity of diffracted beams provided information about the atomic array.The sharpness and shape of reflection are related to the perfection of crystal. As mentioned earlier, X-ray diffraction method can yield information on particle size. Smaller the particle size, broader is the diffraction peak.Nano particles cause appreciable broadening of diffraction lines. The breadth B of diffraction line is related to the particle size 'D' via the Debye Scherrer's formula(9).

$$D=0.9\lambda / \beta \cos\theta \qquad (1)$$

Where D is crystallite size,
λ is wavelength of X-ray radiation (1.5406Å),
θ is Bragg diffraction angle and
β is full width at half maxima (FWHM) intensity.

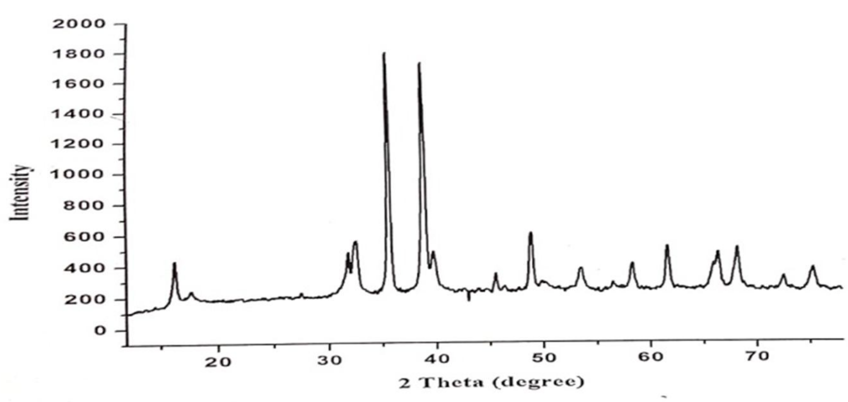

Fig .1 XRD Spectra of MgO nanoparticles

The prepared nano particles are of crystalline nature. The strong diffraction peak considered to determine crystallite size which yields and an average value of 25.15nm for MgO nanopowder.The XRD patterns of as-synthesized and calcined MgO nanoparticles using different precursors are shown in figure.XRD peaks confirm that the formation of MgO from each precursors was in monoclinic phase the characteristic peaks located at 2θ=35.63.

Table 1: Average Crystallite size of MgOnano particles.

Sample code	Position(2θ)	FWHM(β)	Crystalline size(D)
MgO	35.63	0.3281	25.15

B. XRD Spectra of Cu doped MgO Nanoparticles

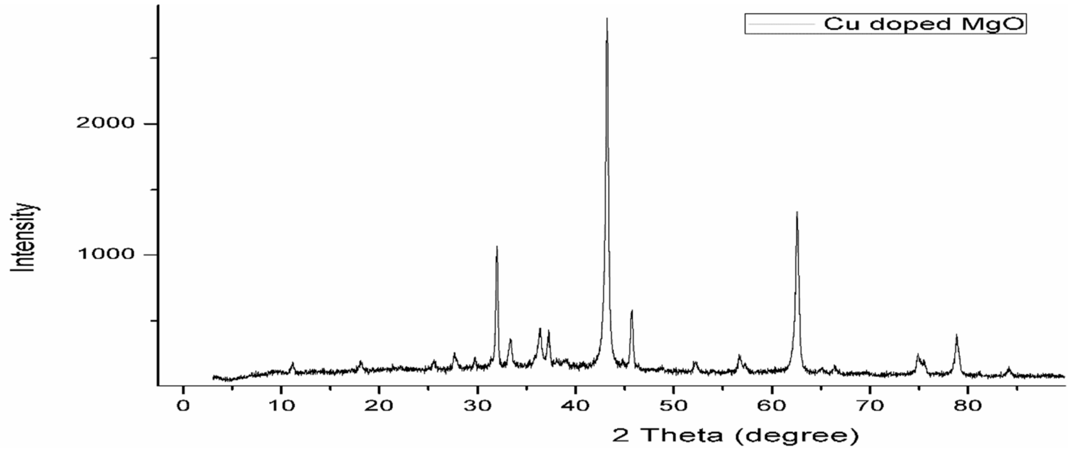

Fig.2 XRD spectra of Cu doped MgO nanoparticles

For Cu doped MgO nano powder the strong diffraction peak considered to determine crystallite size which yields an average value of 13.37nm for Cu doped MgO nanopowder.The XRD patterns of a synthesized and calcined Cu doped MgO nanoparticles using different precursors are shown in fig.XRD peaks confirm that the formation of Cu doped MgO from each precursors was in monoclinic phase.The characteristic peaks located at 2θ=43.29.

The obtained value of dislocation density (δ)[(10)] of Cu doped MgO is greater than pure MgO nanoparticles.

Table 2 :Average crystallite size of MgO and Cu doped MgOnanoparticles

Sample Code	Position(2θ)	FWHM(β)	Crystallite size(D)nm	DensityDislocation $\delta=1/D^2(\times10^{15})$
MgO	35.63	0.3281	25.15	1.5809
Cu doped MgO	43.29	0.6683	13.37	5.5941

C. UV-Vis Absorption Spectroscopy

The UV-Vis absorption spectroscopy shows the strong confinement effect of the prepared sample which suggest the formation of nanoparticles.

The optic absorption spectrum (Essic J, Mather R 1993) was used to study the optical properties of the synthesized Cu doped MgOnanoparticles, from this the band gap and the type of electronic transitions were determined.

Essic J, Mather R Characterization of a bulk semiconductors band gap via near-absorption edge optical transmission experiment.Am J Phys 1993; 61:646-9

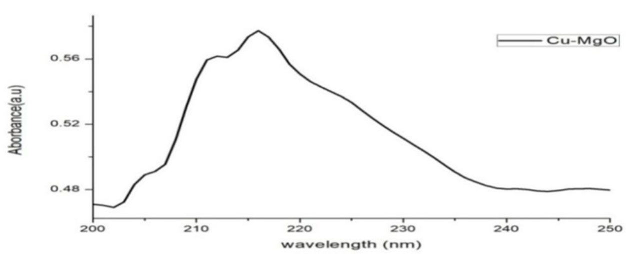

Fig.3 UV-Vis spectra of Cu doped MgO nanoparticles

The optical properties of the MgO and Cu doped MgO Nano Particles remain calculated in part by means of the UV–Visible absorption spectra in the wavelength range between the 100–900 nm. In the obtained spectra there is a strong absorption peak appeared at around 280nm.

IV. SUMMARY

The nano particles are prepared by co-precipitation technique. The nano particles are calcined at 400^0C for 4 hours.The XRD, and UV-Vis studies are made to determine the structural and optical properties of Cu doped MgO Nano Particles.

V. CONCLUSION

Cu doped MgO nanoparticles are synthesized by chemical co-precipitation method. It is confirmed by XRD analysis. From this study the size of the particle (D), dislocation density (δ) is determined. The UV -Vis spectra of Cu doped MgO nanoparticles shows sharp absorption edge observed around 280nm.

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