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Synthesis, structural, vibrational, thermal studies of Mg doped ZnO nano particles using chemical precipitation method

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Abstract
In the present work ZnO and Mg doped ZnO nano particles have been synthesized by Chemical co precipitation method and their structural, vibrational, thermal properties have been studied. The average particle size of the prepared pure ZnO and Mg doped ZnO samples have been found to be 51nm and 44nm using Debye-Scherr's formula. FTIR spectra analysis shows that the peak at 462.92cm^{-1}corresponding to the characteristic peaks of ZnO nano particle has been found to be shifted to 472cm^{-1} in the Mg doped ZnO nano particles. It indicates the presence of Mg in the ZnO nano particles. Doping of Mg to ZnO nano particles changes the thermal stability region of pure ZnO nano particles from 177^{0}C -288^{0}C to 268^{0}C -287^{0}C.

Keywords: Nano particle synthesis; XRD; FTIR; TG

1. Introduction
In current years, researchers have focused more on the synthesis of ZnO nano particles due to its novel applications in advanced technologies. Metal oxides are very significant technological materials to be used in electronic and photonic devices. The zinc oxide (ZnO) is a very suitable candidate for insulation applications due to its low heat capacity and high melting point. Recently, it was reported that ZnO has a good bactericidal show in aqueous environments due to the formation of super-oxide [1]. Various methods such as vapor phase growth, vapor liquid solid process, soft chemical method, electrophoresis deposition, sol gel process, homogeneous precipitation, chemical co precipitation etc have been used to synthesis ZnO nano particles**.** Mg doped ZnO nano particles has been prepared by using $ZnCl_2$, $MgCl_2$ and NaOH. The nano particles are subjected to analytical techniques such as XRD, FTIR, and TGA analysis.

2. Experimental procedure
2.1 Chemicals used
Zinc Chloride, Magnesium Chloride and Sodium hydroxide are purchased from Merck specialties private limited chemicals (Mumbai, India). All the chemicals were of analytical grade.

2.2. Synthesis of ZnO nano particles
Zinc Chloride, Sodium hydroxide (Merck, AR grade) are dissolved in 100 ml distilled water separately by using magnetic stirrer for half an hour. Then the sodium hydroxide solution is added drop wise to zinc chloride solution at room temperature and stirred well. Precipitate was formed. Then it is filtered out separately and repeatedly washed with double distilled water to remove unnecessary impurities.

2.3. Synthesis of Mg doped ZnO nano particles
Zinc Chloride, Magnesium Chloride, Sodium hydroxide (Merck, AR grade) are dissolved in 100 ml distilled water separately by using magnetic stirrer for half an hour. Solutions of Zinc Chloride and Magnesium Chloride are mixed together and stirred for one and half hours. Then the sodium hydroxide solution is added drop wise to this mixture at room temperature and stirred well. After 30 minutes milky white color precipitate of Mg doped zinc oxide is formed. Collected the white precipitate after filtration and drying

2.4 Characterization Technique
X-ray diffraction Patterns of the products are recorded at room temperature on Philips X'PERT-PRO diffractometer using CuKα radiation in the range of $2\theta=10^0$-80^0.Vibrational analysis was taken by using IR spectrum has been taken in the range 400-4000cm^{-1} at room temperature using SHIMADZU IR Affinity -1 Spectrometer. UV analysis of the prepared samples has been recorded by using UV Visible spectrometer SHIMDZU MODEL UV 2450 in the frequency range 200nm to 900nm. Thermal properties measured by TGA 4000 Perkin Elmer with a heating rate of 10^{0}C/min in the atmosphere of Nitrogen from the temperature of 35^{0}C to 800^{0}C.

3 Results and Discussion

3.1 Particle Size Analysis

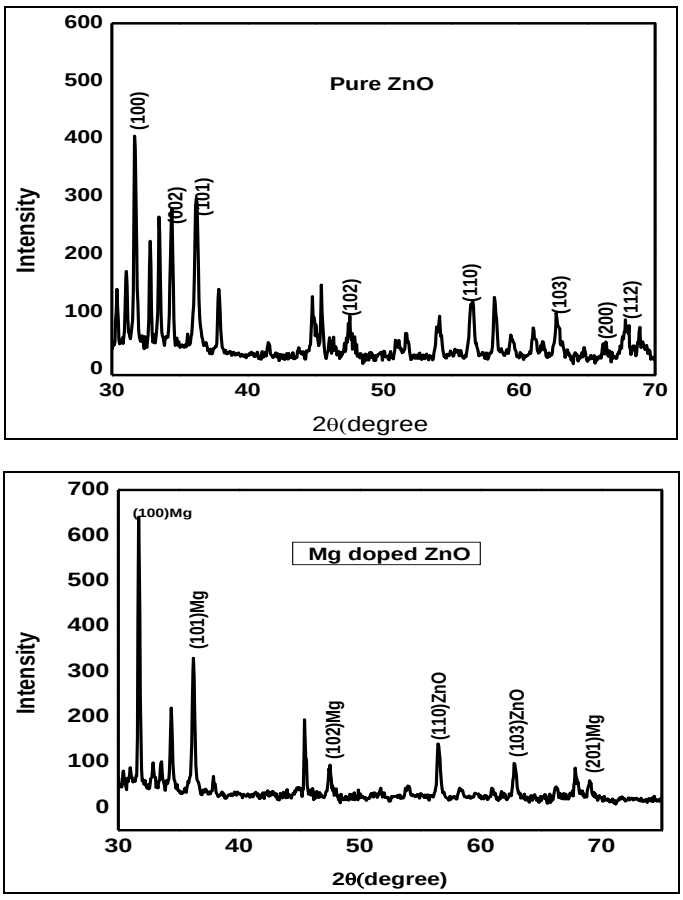


Fig1: XRD Pattern of Mg doped ZnO

XRD analysis is used to investigate the crystal structures of the samples. The X-Ray diffraction patterns of pure ZnO and Mg doped ZnO nano particles are shown in Fig.1.The diffractions peaks at 2θ = 31.770, 34.422, 36.253, 47.539, 56.603, 62.864, 66.380 and 67.897 are corresponding to lattice planes (002), (101), (102), (110), (103), (200) and (112) respectively of ZnO nano particles. Using unit cell software the lattice parameters of the pure ZnO nano particles are calculated. These found to be in close agreement with JCPDS ICDD file for hexagonal ZnO [36-1451] [2]. This revealed that the resultant nano particles are pure ZnO with a hexagonal structure [3]. The diffraction peaks at 34.40, 36.620, 47.815 and 57.386 could be assigned to the diffractions from (101), (102), (110), (103) and (201) planes of magnesium respectively according to the data base in JCPDS No. 89-5003. The particle size D of the samples have been determined using the Scherrer formula

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

Where λ is the wavelength of CuK_α radiation, β is half of its maximum intensity (FWHM), and θ is the Bragg's angle.

The calculated particle size of pure ZnO is 51 nm. It is same as the value reported by the author Amutha *et al.* 2016 [4]. The particle size of the Mg doped ZnO is 44nm. It has been observed that the there is decrease in the particle size of ZnO nano particles. This may be due to the influence of Mg ions**.**

Dislocation density (δ**)** is defined as the number of dislocation lines per unit volume of the crystal. The dislocation density is used to determine the amount of defects present in the prepared samples (table1). The dislocation density of pure ZnO is in close agreement to the value reported in the Literature. The dislocation density of Mg doped Zno nano particles is less than the pure ZnO nano particles.

Table 1: The Particle size, bond length, dislocation density and Lattice parameters of Mg doped ZnO nano particles.

Samples	Particle size (nm)	Bond Length (A^0)	Dislocation density 10^{14} (Lines\m^2)	Lattice parameters (A^0)	
				a =b	c
Pure ZnO	51	0.5078	5.1698	3.252	3.209
Mg doped ZnO	44	0.6542	4.9304	3.213	5.275

The Zn-O bond length L is calculated using the formula

$$L = \sqrt{\left[\frac{a^2}{3} + \left(\frac{1}{2} - u\right)^2 c^2\right]} \quad (3)$$

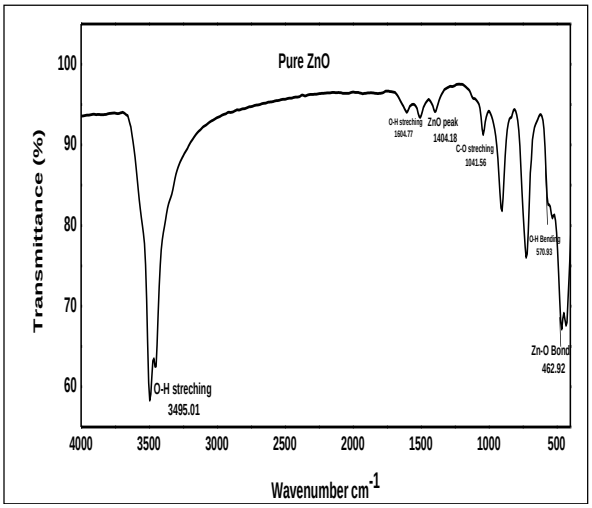
Where

$u = \frac{a^2}{3c^2} + 0.25$ for Hexagonal structure

The bond length of Mg doped ZnO increases from 0.5078 A^0 to 0.6542A^0 (table1)

3.2. Ftir Analysis

Fig 2 shows the FT-IR spectra of pure ZnO and Mg-ZnO nano particles recorded in the range of 500 to 4000 cm^{-1}. The peaks at 462.92cm^{-1}, 904cm^{-1} and 1404cm^{-1} are the characteristic absorption peaks of Zn–O bond [5]. The peaks at 578.93cm^{-1}, 1604$cm^{-1}$and 3459cm^{-1} are corresponding to bending and stretching vibration of hydroxyl (O-H) group from NaOH [6].These bands get shifted in the Mg doped ZnO as shown in table 2. It confirms the doping of Mg ion in the ZnO nano particles.



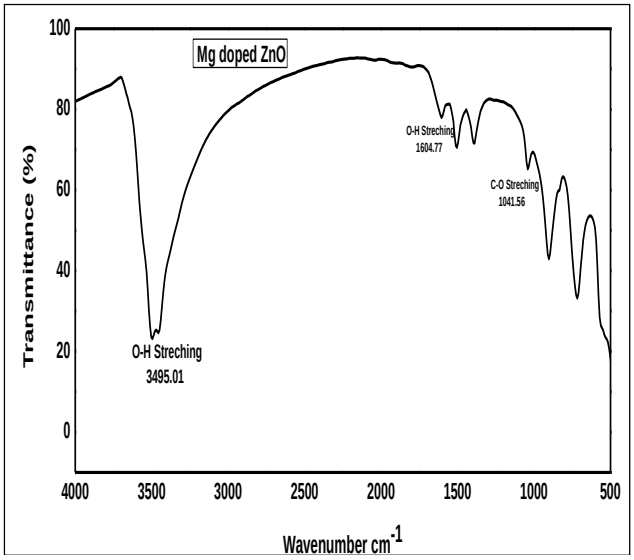


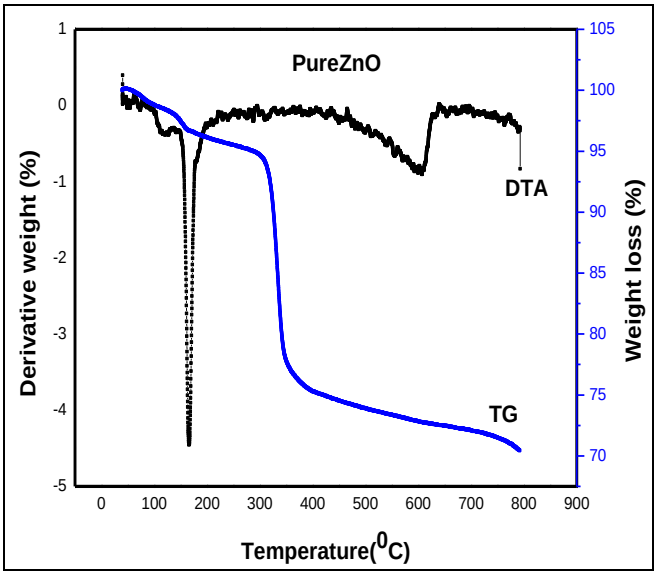
Fig 2: FTIR Spectra Mg doped ZnO nano particles

Table 2: Vibrational peaks & Assignment of the samples

Vibrational of the samples (cm^{-1})		Assignment
Pure ZnO	Mg-ZnO	
462.92	472.63	Zn-O(b)
570.93	578.93	O-H(b)
904	895	Zn-O
1404	1404.15	ZnO
1604.77	1604.77	O-H(S)
3456.43	3467.43	O-H(s)

3.3. TGA and DTA analysis

TG/DTA thermo gram of pure ZnO and Mg doped ZnO are shown in fig 3. In Fig 3 the first transition occurs in the temperature range 37-65^{0}C and 37-92^{0}C with weight loss of 2 and 2.5% in the TG thermo gram of pure ZnO and Mg doped ZnO nano particles respectively. These weight losses are accompanied by 50^{0}C and 67^{0}C in DTA curves of pure ZnO and Mg doped ZnO nano particles respectively. This transition is attributed to evaporation of water molecules. The second transition occurs in the temperature range 100-125^{0}C and 123-193^{0}C with weight loss of 2 and 19% in the TG thermo gram of pure ZnO and Mg doped ZnO nano particles respectively. These weight losses are accompanied by 115^{0}C and 192^{0}C in DTA curves of pure ZnO and Mg doped ZnO nano particles respectively. The third transition occurs in the temperature range 146-179^{0}C and 272-448^{0}C with weight loss of 18 and 2.7% in the TG thermo gram of pure ZnO and Mg doped ZnO nano particles respectively. These weight losses are accompanied by 172^{0}C and 246^{0}C, 417^{0}C in DTA curves of pure ZnO and Mg doped ZnO nano particles respectively. This transition is attributed to evaporation of water molecules. The fourth transition occurs in the temperature range 576-617^{0}C and 542-653^{0}C with weight loss of 2 and 21% in the TG thermo gram of pure ZnO and Mg doped ZnO nano particles respectively. In the temperature range 100ºC to 400ºC the weight loss caused by evaporation of inorganic materials. These weight losses are accompanied by 624^{0}C and 433^{0}C in DTA curves of pure ZnO and Mg doped ZnO nano particles respectively. The thermal event at 465°C that occurs after the transformation of the hydroxide to the oxide is associated with the phase transformation of amorphous ZnO into its cubic ordered phase [7].



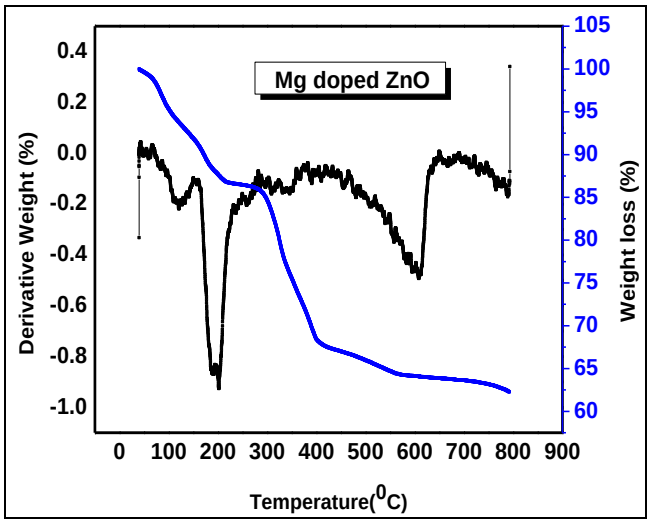


Fig 3: TG spectrum of Mg-ZnO nano particles

Table 3: Weight loss % and the DTA peaks for pure ZnO (R1) and Mg doped ZnO

Samples	Region	TG		DTA peaks (^{0}C)
		Temperature (ºC)	Weight Loss (%)	
Pure ZnO	I	37-65	2	50
	II	100-125	2	115
	III	146-179	18	166
	IV	576-617	2	604
Mg doped ZnO	I	37-92	2.4	65
	II	123-193	19	192
	III	276-448	2.7	246, 417
	IV	542-653	21	690

4. Conclusion

In these work nano particles of pure ZnO and Mg doped ZnO nano particles were synthesized by Chemical Precipitation method. The X-ray diffraction analysis of the nano particles confirms crystalline nature of the powder. FTIR analysis reveals the possible bonding present in the nano particles and confirms the stretching and bending mode in different

wavelength. TG analysis suggests the Mg doped Zno nano particle pure has less weight loss than pure Zno. The lesser weight loss in the sample the better it is. Thus this nano particle has more thermal stability than Mg doped ZnO nano particles.

5. Acknowledgement
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