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SYNTHESIS AND OPTICAL CHARACTERIZATION OF MgO NANOPARTICLES USING CHEMICAL PRECIPITATION METHOD

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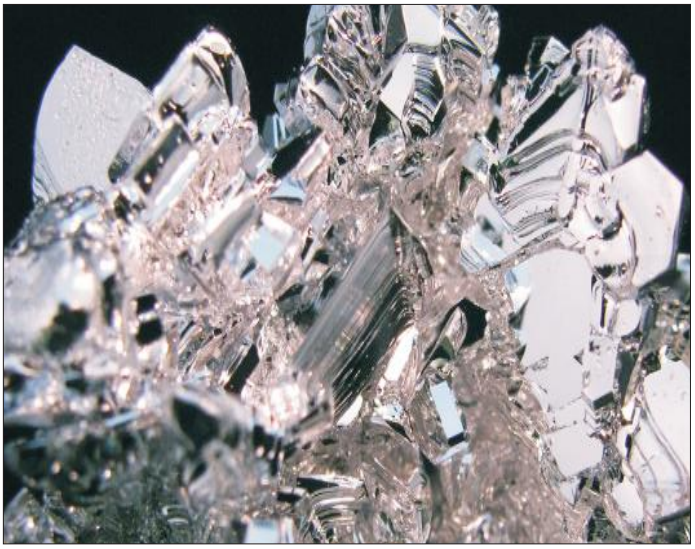


ABSTRACT

MgO nano particles have unique optical, thermal and structural properties. In the present work we focus on the Synthesis of MgO nano particles using $MgCl_2$ and NaOH with distilled water as solvent by chemical precipitation method. From the XRD analysis, the particle size of MgO nanoparticle has been calculated as 23.14 nm by using Debye Scherrer's formula. The presence of functional group in the samples has been analyzed using FT-IR analysis. From the UV analysis the optical band gap of MgO nano particle ranging from 5.59eV. The thermal behavior has been examined using TGA.

KEYWORDS

Nanoparticle, XRD, FTIR, UV, TG.

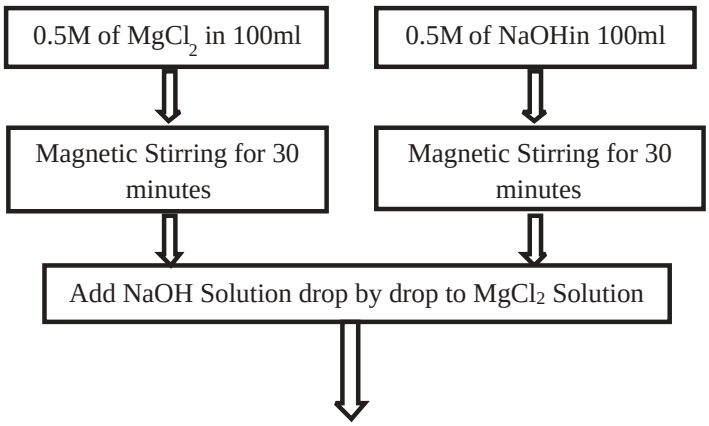
INTRODUCTION

Nanoscience and technology have emerged over the past decade as the forefront of science and technologies. Nanotechnology plays an important role in the industrial revolution. It has been reported nanoparticles can be prepared from aqueous solution by different ways such as hydrothermal, precipitation, Sol-Gel and microemulsion routes. Metal oxides such as TiO_2, ZnO, MgO and CaO are of particular interest as they are not only stable under harsh process conditions but also generally regarded as safe materials to human beings and animals. Magnesium oxide is an interesting basic oxide that has many applications in catalysis, adsorption and in the synthesis of refractory ceramics[1,2].It is a unique solid of high ionic character, simple stoichiometry and crystal structure and also it can be prepared widely with variable particle sizes and shapes. The present study was carried out with the main objective of evolving a simple method for the synthesis of MgO nanoparticles. MgO nanoparticles are characterized by XRD, FTIR UV and TG analysis.

EXPERIMENTAL PROCEDURES

Magnesium Chloride and Sodium hydroxide (Merck, AR grade)has been dissolved in 100 ml distilled water separately. Stir them separately for half an hour using magnetic stirrer for constant stirring. Solution of Magnesium Chloride is mixed together by using the magnetic stirrer for one and half hours. Then the sodium hydroxide solution is added to the mixture of Magnesium Chloride drop by drop with burette at room temperature and stirred well. After 30 minutes milky white color precipitate of magnesium oxide is formed. Collected white precipitate after filtration and drying.

Flowchart of MgO nanoparticles



CHARACTERIZATION TECHNIQUES

The average crystalline size and structure has been determined byX-ray diffraction patterns of the products are recorded at room temperature on Philips X'PERT – PRO diffractometer using Cu Kα 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 – 4000 cm^{-1} at room temperature using SHIMADZU IR Affinity – 1 Spectrometer. UV analysis of the prepared samples has been recorded by using UV Visible spectrometer SHIMADZU MODEL UV 2450 in the frequency range 200nm to 900nm.Thermal properties measured by TGA 4000 Perkin Elmer with a heating rate of 100C/min in the atmosphere of Nitrogen from the temperature of 35°C to 800°C.

RESULTS AND DISCUSSION

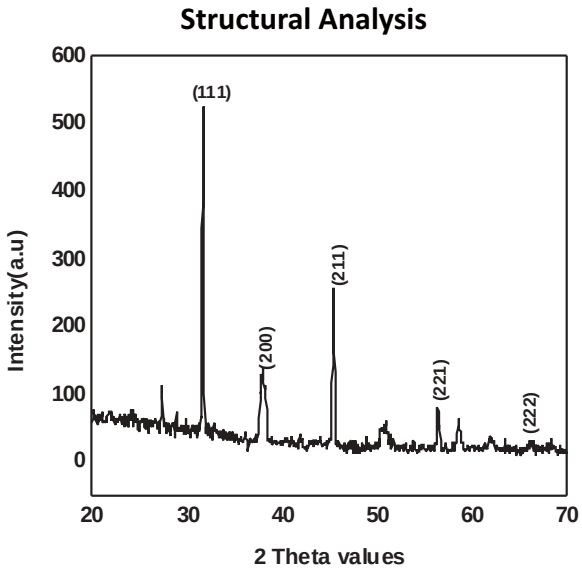


Figure 1: XRD Pattern of MgO nanoparticles

Figure 1shows the XRD pattern of five major diffraction peaks at 32.00, 37.12, 45.89, 57.05, 66.92 and 70.05 corresponding to lattice planes (111), (200), (211), (221) and (222) respectively of the pure MgO nanoparticle (JCPDS-76-1363). The diffraction pattern and interplanar spacing closely matched those in the standard diffraction pattern of MgO. The average particle size (D) of the nanoparticles is determined by using the Debye - Scherrer equation

$$D = 0.9\lambda/\beta \cos\theta$$

Where, θ is the wavelength of radiation used, β is the Full Width Half Maximum andθ is the Bragg's angle. The average particle size has been found to be 23.14nm for MgO nanoparticles[1].Lattice parameter for MgO nanoparticles are calculated by using the software of Cell Calculation as a = b = c = 0.4872 nm and space group Pa3mc (JCPDS card No-76-1363).The dislocation density (d), defined as the length of dislocation lines per unit volume, is given by the equation: $\mathbf{d = 1/ D^2}$.Dislocation density **(d)** of MgO has been detected as 1.867X10^15 (lines/m^2)[2]. The lattice strain (e) has been determined by using the tangent formulae = β / (4 $tan\vartheta$) Lattice Strain of the samples has been calculated using the above formula. Lattice Strain (e) of MgO has been detected as (3.648 x 10^{-3}).

FTIR ANALYSIS

The presence of functional groups in the samples has been analyzed using FT-IR analysis.Figure 2 shows the spectra of as MgO powder sample. The absorption band at 1637 cm^{-1} indicates the bending mode of vibration in water (HOH) and the broad and shallow band observed in the band at 3417 cm^{-1} shows the stretching mode of vibration in hydroxyl group (O-H)[3]. The peak at 3708 cm^{-1} is occurringdue to the brucite phase of $Mg(OH)_2$. The band at 1438cm-1is attributed to the Mg-O stretching vibration [4].

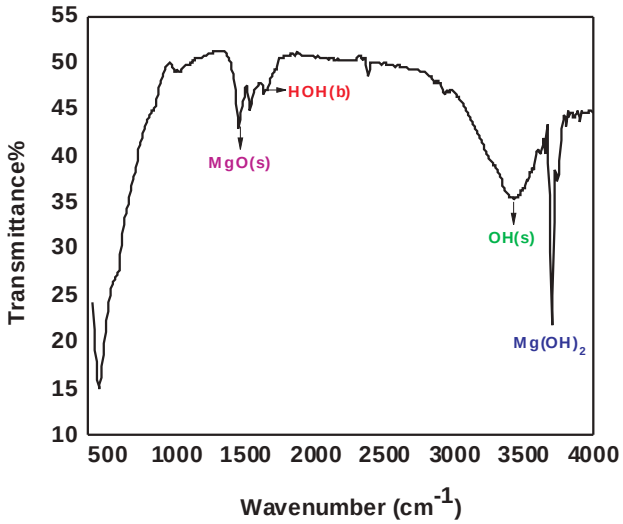


Figure 3: UV Spectrum of MgO nanoparticles

The band gap (E_g) of MgO is calculated by using the formula:

$$E_g = hc/\lambda$$

Where h is the planks constant, c is the velocity of light and θ is the wavelength of the band. The maximum absorption band of MgO nanoparticles have been found at 222 nm respectively[5]. This can be attributed to the electronic excitations of 4-coordinated surface anions at the edges. We observe that the band gap of doped MgO (5.59eV) is shifted slightly towards longer wavelength region.

TG ANALYSIS

TGA can be used to evaluate the thermal stability of a material. In a desired temperature range, if

species is thermally stable, there will be slight mass changes. Figure 4shows TG thermo gram of the sample MgO.

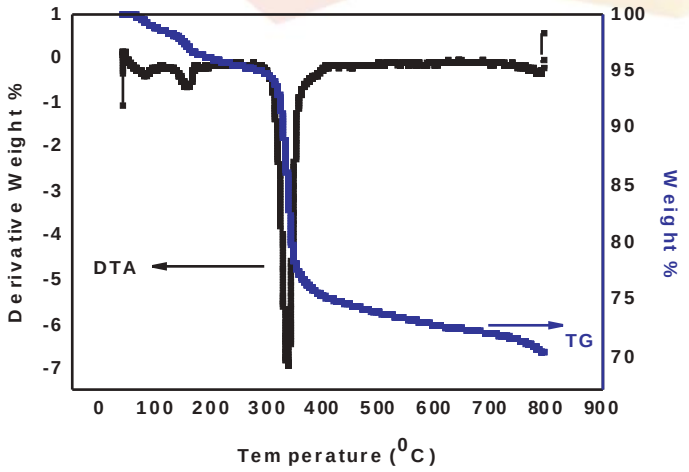


Figure 4: TG Spectrum of MgO nanoparticles

In Figure 4the first transition occurs in the temperature range 39-81^{0}Cwith weight loss of 2.5% in the TG thermo gram. It is accompanied by the endothermic peak at 770C in DTA curve. These transitions are attributed to the evaporation of water molecule[6]. The second transition occurs in the temperature range 138-160^{0}C with weight loss of 3.5% in the TG thermo gram. It is accompanied by the endothermic peak at 155^{0}C in DTA curve. The second degradation is due to the decomposition of $Mg(OH)_2$. The third transition occurs in the temperature range 300-347^{0}C with weight loss of 17% in the TG thermo gram. It is accompanied by the endothermic peak at 336^{0}C in DTA curve. The third degradation is due to the evaporation of inorganic materials. Weight loss percentage and the DTA peaks are observed in the Table 1. In a stable region (174-284^{0}C) there is no DTA peak appears in the MgO sample and the weight loss of 1.0%, these are shown in Table 1.

Table 1: Weight loss % and the DTA peaks for MgO nanoparticles

Sample	TG			DTA peaks (^{0}C)
	Region	Temperature Range (^{o}C)	Weight Loss %	
MgO	I. II. III.	39-81 138-160 300-347	2.5 3.5 17	77 155 336
	Stable	174-284	1.0	-

CONCLUSION

MgO nanoparticles have been successfully synthesized using chemical precipitation method. From XRD analysis average crystallite size of the MgO sample is 23.14 nm. From the FTIR studies we get the result of stretching and bending mode in different wavelength. The band at 1438 cm^{-1} is attributed to the Mg-O stretching vibration. The energy band gap of the prepared samples is 5.59 eV determined by using UV-visible studies. Thermal Stability of the prepared sample is analyzed by TG-DTA curves. These above results showed that as prepared MgO particles were in the nano range.

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