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Preparation and Characterization of MgO Nanoparticles by Co-Precipitation Method

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Abstract

Nanocrystalline Metal oxides are interesting area for scientists due to their unique surface chemistry and high surface area. Among the metal oxide nanomaterials, magnesium oxide (MgO) is an exceptionally important material and has been extensively used in,fire retardation ,catalysis, refractory materials,toxic waste remediation, antibacterial materials, paints, and superconductor products owning to its unique photonic, optical, electronic ,magnetic, mechanical and chemical properties. In the present work MgO nanocrystals were synthesis by co-precipitation method in which ammonia is used as co-precipitating agent were subsequently calcinated at 600°C in air for 4hrs and 6hrs to transform into MgO. X-ray diffraction (XRD) pattern revealed that synthesized $Mg(OH)_2$ nanocrystals are polycrystalline in nature with hexagonal structure, and after annealing at 600 ^oC it transforms to MgO nanocrystals with cubic structure. Fourier transform infrared spectroscopy (FTIR) of the as-synthesized Mg(OH)2 showed the OH-antisymmetric stretching vibration at 3686cm-1, which on annealing disappears completely indicating the Mg(OH)2 to MgO phase transformation. Transmission electron microscopy (TEM) images of MgO nanocrystals exhibited flakes-like structure. Optical band gap energy of MgO nanocrystals was estimated using UV-Vis diffused reflectance spectroscopy (DRS), and found to be 5.92eV respectively.

Keywords: *Nano metaloxides, MgO, X-ray diffraction, FTIR, TEM, Optical properties.*

1. Introduction

In past decade, the unique properties of nanomaterials have created interest to the researchers to develop simpler and inexpensive techniques to synthesis nanostructures for technologically importance. Metal oxides nanomaterial with high surface area and porosity have attracted considerable interest for scientific research due to their potential application such as functional components for nanoelectronics, optoelectronics and sensing devices [1]. In particular, magnesium oxide (MgO) as a versatile oxide materials with assorted properties finds extensive applications in catalysis, ceramics, toxic waste remediation, or as an additive in refractory, paint and superconductor products [2, 3]. Also, owing to its very large band gap, excellent thermodynamical stability, low dielectric constant and refractive index, it has been used as a transition layer for growing various thin film materials [4, 5]. Generally, MgO nanostructures were synthesized by dehydration of Mg(OH)2 or by decomposition of various magnesium salts using co-precipitation method, thermal evaporation [6], flame spray pyrolysis [7], sol-gel techniques, combustion aerosol synthesis [8], chemical vapor deposition [9], hydrothermal [10], and surfactant methods [11]. etc,these preparation methods involve complex procedures, sophisticated apparatus/ equipments, rigorous experimental conditions, high-temperature annealing,. Hence, in the present work we explore a facile route to synthesis MgO nanoparticles under mild reaction conditions without using any surfactants or organic solvents. Systematic study of the structural, morphological and optical properties of the as-synthesized MgO nanoparticles were carried out using XRD, FTIR, TEM, and UV–VIS (DRS).

2. Experimental

Synthesis: All the chemicals were analytic grade (HIMEDIA, India)) and used without further purification 0.1M of $Mg(NO_3)$(HIMEDIA, India)) was prepared in 100ml of deionized water. Then NH_4OH solution was injected to the above solution at 100°C under constant stirring, and the resulting mixture was refluxed at the same temperature for 24hrs. After the reaction was complete, the resulting white precipitate was washed with deionized water and ethyl alchohel for several times to remove the by-products or impurities, and then dried in air at 100°C for 4hr. The as-synthesized material was calcinated at 600°C for 4hrs and 6 hrs in air to obtain MgO. The structural properties MgO nanoparticles were analyzed by XRD using a Bruker Axs D8 analyzed automated with CuK radiation = 1.5406Å)..FTIR was recorded on a Bio-Rad (Hercules, CA) FTS-165

spectrometer. The morphology of the nanoparticles was analysed using JSM-6360 JEOL TEM. The optical band gap of the nanoparticles was estimated from DRS using JASCO V- 670 double beam spectrophotometer.

3. Result and Discussion

3.1 XRD analysis

X-ray Powder Diffraction (XRD) studies were carried out to confirm the the structure (crystallinity) using X-ray diffractometer with Copper (kα) radiation (λ = 1.5418 Å) in the range of 10–80° in steps of 0.0170 at a scan speed 0.4°/min. The XRD pattern is shown in Fig.1.

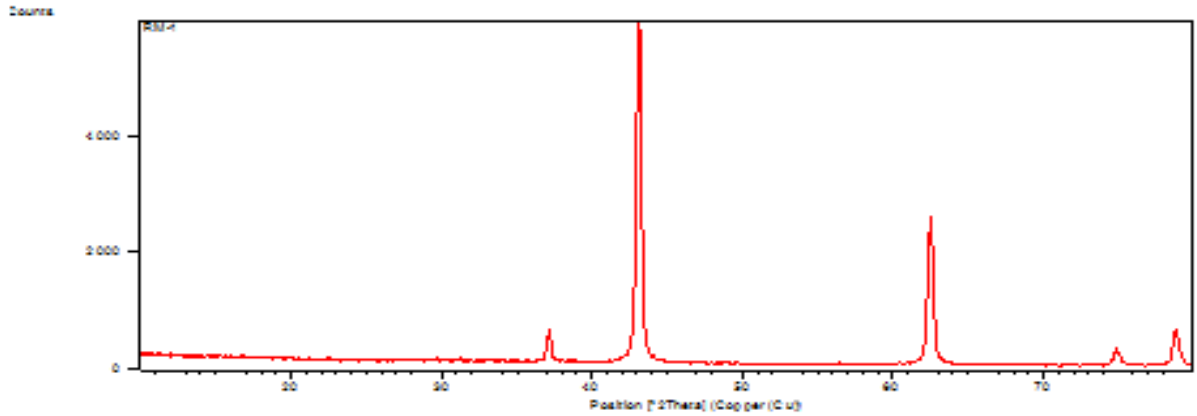


Fig1. XRD OF MgO nano particles at 600 ^{o}C (4HRS)

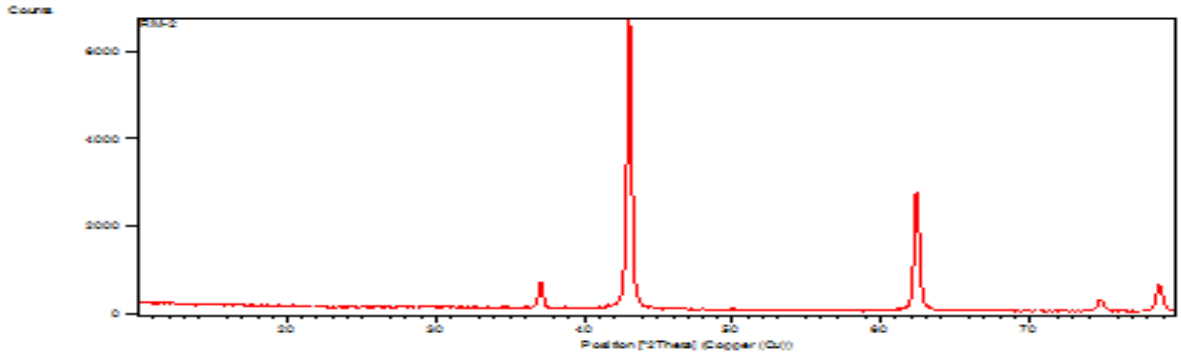


Fig2. XRD OF MgO nano particles at 600 ^{o}C (6HRS)

The spectrum reflect the good crystallinity for MgO nanoparticles.The highest peaks position (2θ)of MgO nanoparticles were at 43.1164 & 43.0396, change in peaks show that particle size increase with increase the sintering time. All the diffraction peaks can be indexed to the face-centered cubic structure of MgO (JCPDS card No: 78-0430). No characteristics peaks of Mg(OH)2 and other impurities was detected in the XRD pattern Considerably broadened lines in the XRD patterns are indicative of the presence of nano-size particles. The XRD patterns is used for obtaining the average particle size with the help of Debye - Scherrer's equation

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

Where D is the crystallite size, λ is the X-ray wavelength, β is the full width at half maximum of the diffraction peak, and θ is the Bragg diffraction angle of the diffraction peaks. The calculated average particle sizes for MgO nanosamples were 11 and 12 nm respectively. It shows that increase the heating time the size of the sample increase.

3.2 FTIR STUDY

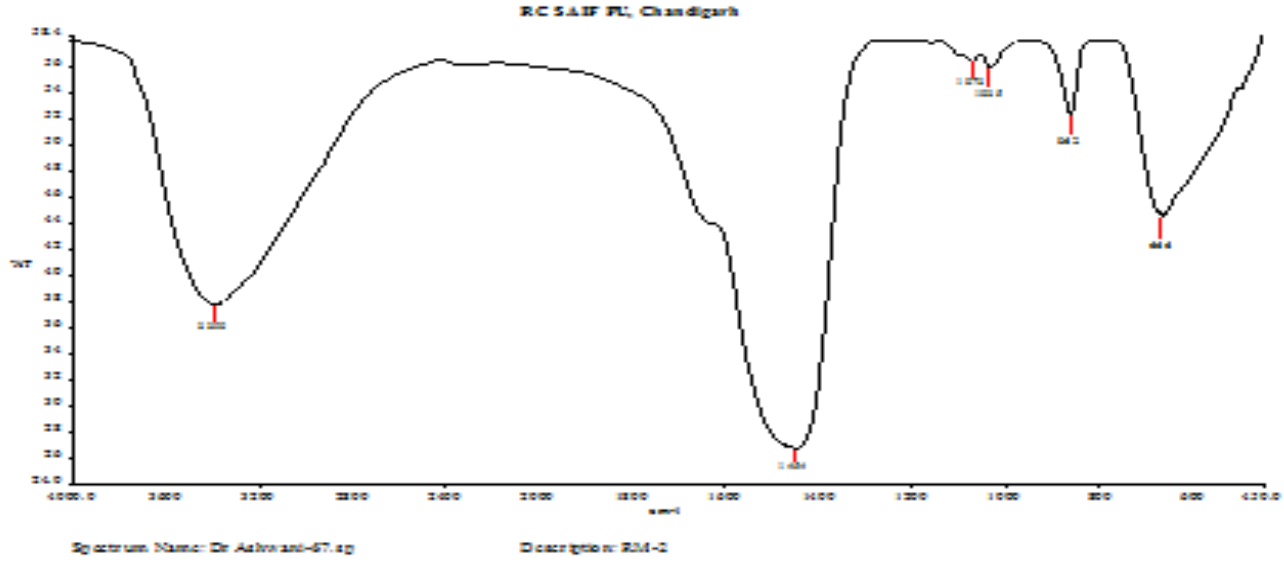
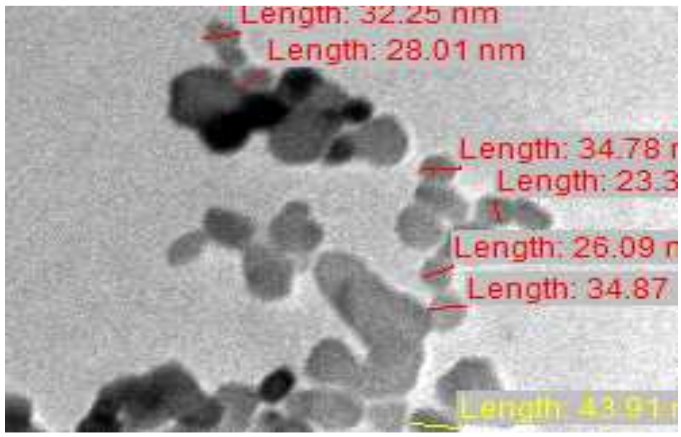
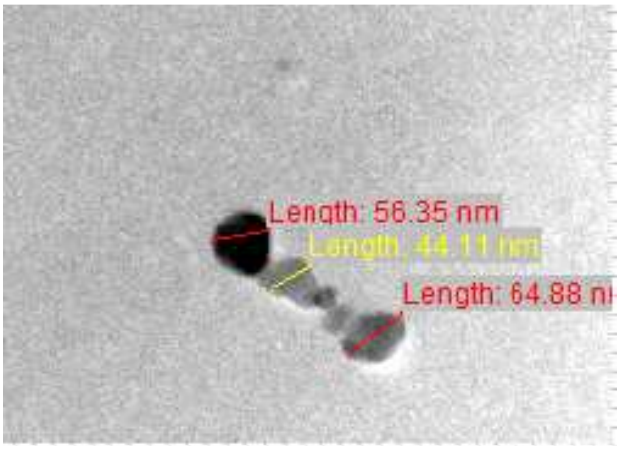


Fig. 3 FTIR Spectroscopy of MgO Nanoparticles

The sharp and intense peak at 3390cm-1 is attributed to the OH antisymmetric stretching vibration in the crystal structure of Mg(OH)2 due to water presence. The peak at 666cm^{-1} is assigned to the Mg–O stretching vibration [12]. The absorption peaks at 1454cm^{-1} and 1073,1035cm^{-1} corresponds to the bending vibration of OH bond and Mg–OH stretching vibrations respectively. The other absorption solder peaks at 1640cm−1 is due to the stretching and the bending vibration of water, respectively [13]. The FTIR spectrum of MgO nanocrystals is shown in Fig.3. Absence of 3686cm-1 sharp peak due to antisymmetric stretching vibration in the Mg(OH)2 crystal structure further confirms that calcinating at 600°C for 4hrs in aircompletely transforms hexagonal structure of $Mg(OH)_2$ to cubic MgO.

3.3 TEM Studies





The Transmission electron microscopy (TEM) measurement was carried out in AIIMS, Delhi instrument in order to analyze the structure and morphology of synthesized MgO nanoparticles. From The TEM images the particle sizes of the MgO nanoparticles were found to be in the range 28-64 nm, which is in quite accordance with the reported value. It is also clear that the synthesized MgO nanoparticles sample is very hygroscopic in nature.

4. Conclusion

MgO nanoparticles can be synthesized in nano-scale in the presence of ultrasonic waves .The MgO nanoparticles can be used as Bacteriocide , so used for water purification [20]. MgO-Al_2O_3 have high strength, so aluminium polycarboxylate is used as dental cement, and this method may be developed to synthesize other effectiveness dental cements[21] .We also see toward other property of MgO nanoparticles in future.

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