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Solution Combustion Mediated Synthesis and Characterization Of Magnesium Oxide Nanoparticles

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Abstract - *The pursuit for novelty, unfolding the problems and elevating the industrial yield with energy efficient and cost-effective materials has unlocked the channels of nanotechnology. Among the diverse range of nanoparticles, magnesium oxide nanoparticles (MgO) is precedent due to its phenomenal physical and chemical properties. The objective of this study is to explore the possibility of synthesizing nano crystalline magnesium oxide particles. The prevalent accustomed method for synthesis of magnesium oxide is the decomposition of variant magnesium salts or magnesium hydroxide. Nano crystalline Magnesium oxide particles were propitiously synthesized by solution combustion technique employing magnesium nitrate as an oxidizer and glycine as fuel. It is an inexpensive method to produce nanosized magnesium oxide powders with controllable structure and morphology. The materials obtained by combustion method were grounded and annealed at various temperatures to improve the crystallinity and phase purity. The obtained MgO nanoparticles were characterized by powder X-ray diffraction analysis (XRD), Fourier transform infrared (FTIR) spectroscopy, FESEM and UV-Visible absorption spectroscopy. The obtained results validate that the combustion technique using Glycine as fuel can produce the materials with high crystallinity and this approach provides economically viable route for large scale synthesis of MgO nanoparticles.*

Key Words: Magnesium oxide, Combustion synthesis, Optical property, FESEM, nanoparticles.

1. INTRODUCTION

In the past decennium the remarkable properties of nanomaterials has contrived a concernment among the researchers to devise a facile and inexpensive techniques for synthesis of nanostructures which have scientific value [1-2]. There are various kinds of techniques which are used to synthesize the MgO nano-particles by using different methods such as pulse laser deposition (PLD) [3], Laser ablation [4], sol-gel method [5-6], flame spray pyrolysis [7], chemical vapour deposition [8], solution combustion synthesis [9] etc. Out of above all the synthesis technique, solution combustion method is reported for large scale production of MgO nanoparticles.

In the present work, a simple route of synthesis of MgO nanoparticles has been used under mild reaction conditions using an oxidizer and fuel. Systematic study of the structural, morphological and optical properties of the calcined MgO nanoparticles was then carried out by using XRD, FTIR, SEM and UV–VIS spectroscopic techniques.

1.1 Experimental Techniques: Synthesis of MgO Nanoparticles

Chemicals in the present investigation were analytical grade and used without any further purification. Nanocrystallites Magnesium oxide powder was set up with low-temperature solution combustion method at 300 degrees using magnesium nitrate [$Mg(NO_3)_2.6H_2O$] as an oxidizer and glycine [NH_2CH_2COOH] as a fuel [10-11].The compounds are dissolved and placed on preheated mantle maintained at 300 ± 10^0 C. At the point of spontaneous combustion, the solution starts off flaming and lets off heat. The whole solution vaporizes instinctively and becomes a fiery solid. The as-synthesized samples were calcined at different temperatures for different time duration in air to remove the nitrates and to verify its phase evolution

2. RESULTS AND DISCUSSION

2.1. XRD ANALYSIS

The typical XRD pattern of the MgO nanoparticles annealed at 400 ^{0}C and at 800^{0}C are shown in (Figure - 1) and (Figure - 2).

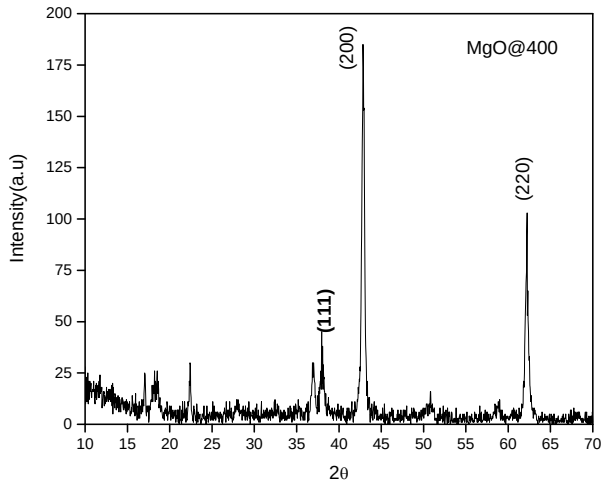

Figure -1: XRD Spectra of MgO annealed at 400^{0}C

The peak positions of sample exhibit the cubic phase of MgO which was confirmed from the standard data. Further, no other impurity peak was observed in the XRD pattern showing the single phase sample formation. Using Scherrer equation, D = 0.9 λ / β cosθ where D is the crystallite size, λ is the wavelength of X-ray beam, β is the full width at half maximum of the most intense peak, and 2θ is the Bragg diffraction angle of the most intense peak, the average crystal size was determined. Lattice parameters were calculated and reported as a = 4.2149Å, b =4.2149Å, c =4.2149Å which matches with the standard lattice parameters determined by ICDD card no. 80-1916.The crystal size of the synthesized MgO was determined and it was found to be 26.66nm.

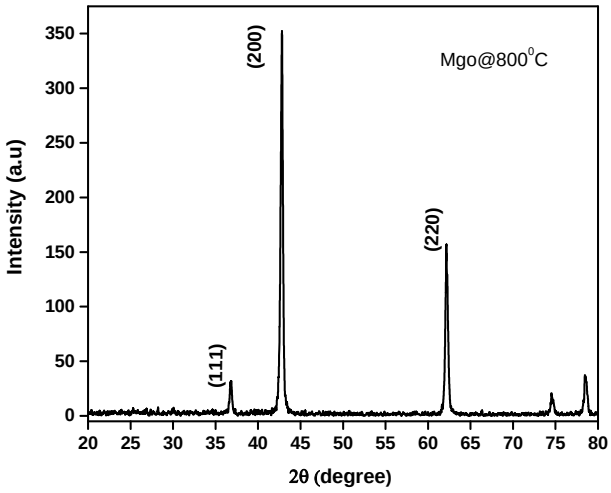

Figure -2: XRD Spectra of MgO annealed at 800^{0}C

2.2 FTIR ANALYSIS

FTIR Spectra of the MgO nanoparticles calcined at 400^{0}C for 2 hrs show presence of the IR peaks band at around 3445 cm^{-1}, 1568 cm^{-1}, 1420 cm^{-1}, 1017 cm^{-1} and 528 cm^{-1}.These peaks are attributed to stretching mode of -OH group, adsorption of CO_2 and different Mg-O-Mg vibration mode of MgO nanoparticles respectively.

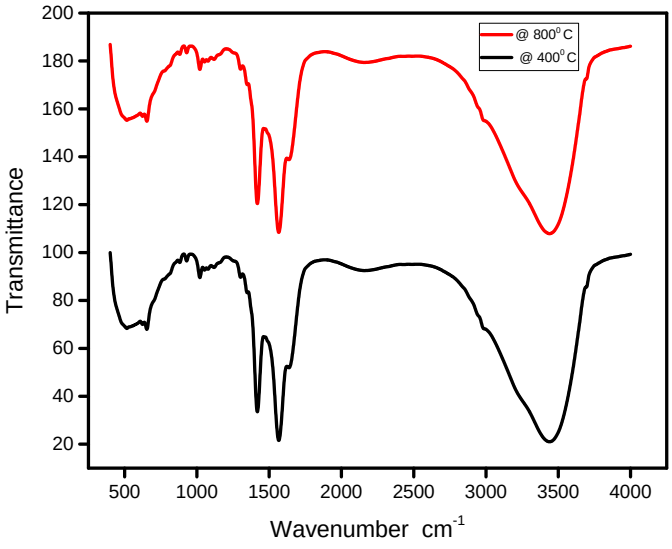

Figure -3: FTIR spectra for MgO annealed at 400^{0}C and 800^{0}C

MgO nanoparticles calcined at 800^{0}C for 1 hr show presence of the IR peaks band at around 3431 cm^{-1} , 1570 cm^{-1} ,1419 cm^{-1} , 1027 cm^{-1} and 621 cm^{-1}.The transmittance of the all calcined samples decreases with decrease in the duration of calcinations temperatures It might be due to the decrease of the condensation of the oxygen during calcination process.

2.3 SEM ANALYSIS

The Scanning Electron Microscopy (SEM) studies reveal the morphology of calcined nanoparticles. (Figure - 4) and (Figure - 5)

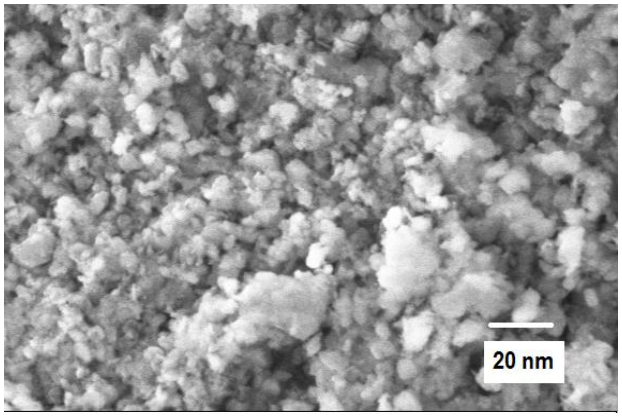

Figure -4: FE-SEM images of MgO annealed at 400^{0}C

The combustion derived product show highly porous, foamy and fluffy in nature. It has been observed that all the synthesized nanomaterials are agglomerated in nature and spherical in shape.

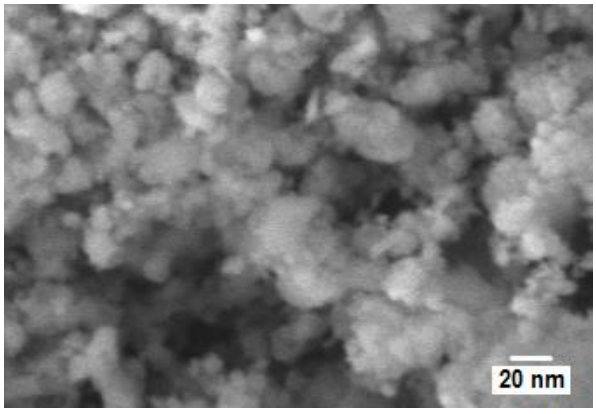

Figure -5: FE-SEM images of MgO annealed at 800^{0}C

2.4 UV – VISIBLE SECTROMETER ANALYSIS

For the UV–Vis absorption measurement, the calcined MgO samples are ultrasonically dispersed in absolute ethanol. The recorded graph between absorbance versus wavelength for MgO nanostructures calcined at fixed temperature (800 ^{0}C) for 1 hr is shown in (Figure - 6)

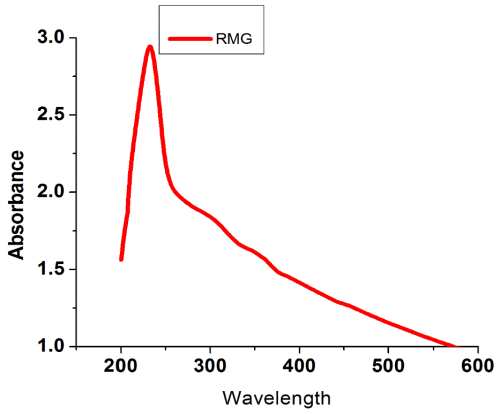

Figure -6: UV- Vis Spectra of MgO @800^{0}C

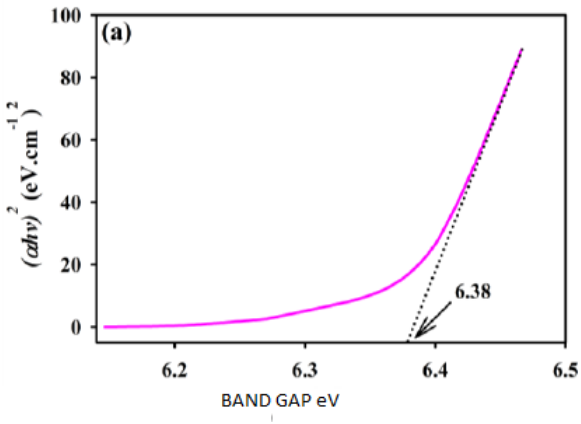

Figure -7: Band Gap of MgO nanoparticles

Optical band gap for all samples was calculated using well known Tauc relation given by $\alpha h\nu = A(h\nu - Eg)^n$ respectively where α absorption coefficient, A is the absorbance, t is the path length of wave which is equal to the thickness of the cuvette, A is the proportionality constant, Eg is the band gap, hν is the photon energy and n=1/2 and 2 for direct and indirect band gap semiconductors respectively. From the Tauc plot (Figure - 7), the band gap of pure MgO is found to be 6.38 eV.

3. CONCLUSIONS

MgO nanopowder was successfully prepared by solution combustion method. The powder XRD confirms the cubic phase of MgO. Porous nature of MgO product was confirmed from SEM micrographs. The band gap of the MgO is determined as 6.38 eV. Solution Combustion technique can produce the materials with high crystallinity and this approach provides economically viable route for large scale synthesis of MgO nanoparticles.

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