

Synthesis and Characterization of MgO Nanoparticles by Using Sol-Gel Method

D. V. SONAWANE¹, M. D. SANGALE², D. N. GAIKWAD¹,
S. S. GAIKWAD¹, A.A. KALE² and S. R. KUCHEKAR³

¹p. G. Department Of Chemistry, A. A. College, Manchar, Tal- Ambegaon, Dist- Pune (Ms), India.

²R. B. N. B. College, Shrirampur, Dist. A'nagar, (Ms), India.

³Pravara Womens College of Home Science, Pravaranagar, Tal-Rahata, Dist. A'nagar, (Ms), India.
Corresponding author email: drdvsonawane@rediffmail.com

Abstract

Nanoparticles of mgo is formed by successive sol-gel method. Nanoparticles are obtained by dissolving solid mgso₄ aqueous medium with 0.1M solution of acetic acid. The crystals of mgo were obtained by addition of 1 M naoh. The precipitate is dried at 60⁰ C temperature for 12 hours to get the final growth of mgo nanoparticles. The structural morphology, optical property of particles were studied by X-ray diffraction (XRD), Scanning electron microscop (SEM), UV spectrophotometer and furrier transformer infra-red spectroscopy (FTIR). The study of surface morphology reveals that mgo nanoparticles shows nanorods, niddle and spindles like shapes. The obtained nanoparticles of mgo shows optical band gap of 5.17 ev.

Keywords: MgO nanoparticles, Characterization: SEM, XRD, UV and FTIR.

Introduction

Nanoscience is the study of phenomena on a nanometre scale. Atoms are a few tenths of a nanometre in diameter and molecules are typically a few nanometres in size. The smallest structures humans have made have dimensions of a few nanometres and the smallest structures we will ever make will have the dimensions of a few nanometres. This is because as soon as a few atoms are placed next to each other, the resulting structure is a few nanometres in size ^[1]. The smallest transistors, memory elements, light sources, motors, sensors, lasers, and pumps are all just a few nanometres in size. Nanoscience is the study of phenomena and manipulation of materials at atomic, molecular and macromolecular scales, where properties differ significantly from those at a larger scale. Besides the technological relevance of nanoscience, there is an enormous hype associated with it. Fantastic claims have been made about faster computers, cheap production of goods, and medical breakthroughs. Nanotechnology is expected to appear in products such as tennis rackets, self-cleaning cars, paint, food, cosmetics, and thermal underwear. The European Union is has identified nanotechnology as an important research area ^[2]. The goal of this study is to introduce the concepts of nanoscience so that the issues can be understood and a constructive contribution to the debates can be made. Nanoscience is a science that describes manipulation of chemical and biological architectures with dimensions in the range from 1 to 100 nanometers. Nanoscience is about developing new chemical and biological nanostructures, uncovering and understanding their characteristics, and ultimately about learning how to organize and join these new nanostructures into larger and more complex functional architectures ^[3]. It is integrated with nanotechnology because both of them are almost same in use. Nanoscience building blocks ranges from 100 to millions of atoms in a single block. There are different methods are discovered for synthesis

for nonmaterial's viz: (1) Physical method such as high Energy Ball Milling, Melt Mixing, Physical Vapor Deposition With Consolidation, Ionized Cluster Beam Deposition, Laser Vaporization, Laser Pyrolysis, Sputter Deposition:-DC & RF Sputtering, Chemical Vapour Deposition, Electric arc Deposition. (2) Chemical methods such as Colloids And Colloids in Solution, Langmuir-Blodgett (L-B) Methods, Micro emulsions, Sol-Gel Methods, Hydrothermal Methods. (3) Biological method as Synthesis using microorganisms, Synthesis using plant extracts, Synthesis using DNA [4-5].

In the present study, a simple procedure is described for the synthesis of mgo nanoparticles via sol-gel method. Nanomaterials have attracted interest for their novel optical properties, which differ remarkably from bulk materials. The reduction in the particle size in the case of semiconductors results in the increase in the band gap which results in the shift of the light absorption towards in the high energy region. The aim of the present work is to study the microstructure and optical band gap of the synthesized nanoparticle.

Experimental Procedure (MgO):

Weight accurately 2.46 gm $MgSO_4$ on butter paper using balance. Transfer this substance to a beaker & add 100 ml distilled water to it and dissolve the substance completely to get 0.1M $MgSO_4$ solution. Stir the solution with magnetic niddle for 5 minutes. By using measuring cylinder take 0.6 ml acetic acid in a test tube. Transfer this into 100 ml distill water to get 0.1M solution of acetic acid .Stir the solution with magnetic niddle for 5 min. Weight accurately 8 gm NaOH substance on butter paper using balance & transfer into other beaker & add 200 ml distill water & proceed as above. Take out 100 ml $MgSO_4$ + 100 ml acetic acid in a beaker to get homogenous solution with continuous stirring. Fill burette with 1 M NaOH solution & add from burette NaOH solution drop by drop till the colorless solution changes white. This shows that precipitated is formed. This precipitated obtained was washed several time with distill water & filter this solution by filter paper. Collect the precipitated in Petri dish. Dry the precipitated kept at 60^0 for 12 hours to get the final product of MgO nanoparticles. The resultant product further used for characterization [6-7].

Results and Discussions:

Structural characterization (XRD):

The peak of this graph (XRD of MgO powder) is obtained at:

$2\theta = 37.16^0$ therefore, $\theta = 18.58^0$ and intensity = 4841a.u Since $d = (n \lambda) / 2 \sin \theta$ & $n = 1$, $\lambda = 1.54A^0$, we get; $d = (1 * 1.54) / 2 * 0.3186 = 2.4168A^0$ and grain size = $D = K \lambda / \beta \cos \theta$ Where $\beta =$ full width half maxima $(\theta_1 - \theta_2) = 0.006639$ rad, $k =$ constant = 0.9 for spherical particle, $\lambda = 1.54A^0$, $\theta =$

Corresponding angles for peaks, $\cos \theta = 0.9438$

$D = 0.9 * 1.54 / 0.006639 \text{rad} * 0.9438 = 221.197A^0 = 22.1197 \text{nm}$.

The peak of this graph (XRD of MgO powder) is obtained at: $2\theta = 49.99^0$. Therefore, $\theta = 24.99^0$ and Intensity = 2891a.u, Since $d = (n \lambda) / 2 \sin \theta$ & $n = 1$, $\lambda = 1.54A^0$, we get; $d = (1 * 1.54) / 2 * 0.4224 = 1.8229A^0$ and grain size = $D = K \lambda / \beta \cos \theta$

Where $\beta =$ full width half maxima $(\theta_1 - \theta_2) = 0.01328 / 868$ rad, $k =$ Constant = 0.9 for spherical Particle, $\lambda = 1.54A^0$, $\theta =$ Corresponding angles for peaks, $\cos \theta = 0.9063$

$D = 0.9 * 1.54 / 0.01328 / 868 * 0.9063 = 115.0825A^0 = 11.50825 \text{nm}$.

The peak of this graph (XRD of MgO powder) is obtained at: $2\theta = 57.85^0$. Therefore, $\theta = 28.925^0$

and Intensity = 3273a.u, Since $d = (n \lambda) / 2 \sin \theta$ & $n = 1$, $\lambda = 1.54 \text{ \AA}$, we get; $d = (1 * 1.54) / 2 * 0.48366$
 $= 1.5922 \text{ \AA}$ and grain size = $D = K \lambda / \beta \cos \theta$

Where β = full width half maxima $(\theta_1 - \theta_2) = 0.013438 \text{ rad}$, $k = \text{Constant} = 0.9$ for spherical particle,
 $\lambda = 1.54 \text{ \AA}$, θ = Corresponding angles for peaks, $\cos \theta = 0.8752$

$D = 0.9 * 1.54 / 0.013438 * 0.8752 = 221.197 \text{ \AA} = 11.7845 \text{ nm}$.

The greatest peak of this graph (XRD of MgO powder) is obtained at: $2\theta = 61.33^\circ$ therefore,
 $\theta = 30.665^\circ$ and intensity = 1640a.u, Since $d = (n \lambda) / 2 \sin \theta$ & $n = 1$, $\lambda = 1.54 \text{ \AA}$, we get; d
 $= (1 * 1.54) / 2 * 0.5100 = 1.5098 \text{ \AA}$ and grain size = $D = K \lambda / \beta \cos \theta$

Where β = full width half maxima $(\theta_1 - \theta_2) = 8.9606 * 10^{-3} \text{ rad}$, $k = \text{Constant} = 0.9$ for spherical particle,
 $\lambda = 1.54 \text{ \AA}$, θ = Corresponding angles for peaks, $\cos \theta = 0.8601$, $D = 0.9 * 1.54 / 8.9606 * 10^{-3} * 0.8601$
 $= 179.8347 \text{ \AA} = 17.98347 \text{ nm}$.

XRD pattern of MgO nanoparticles and characteristics properties are shown in Fig. 1 and Table 1.

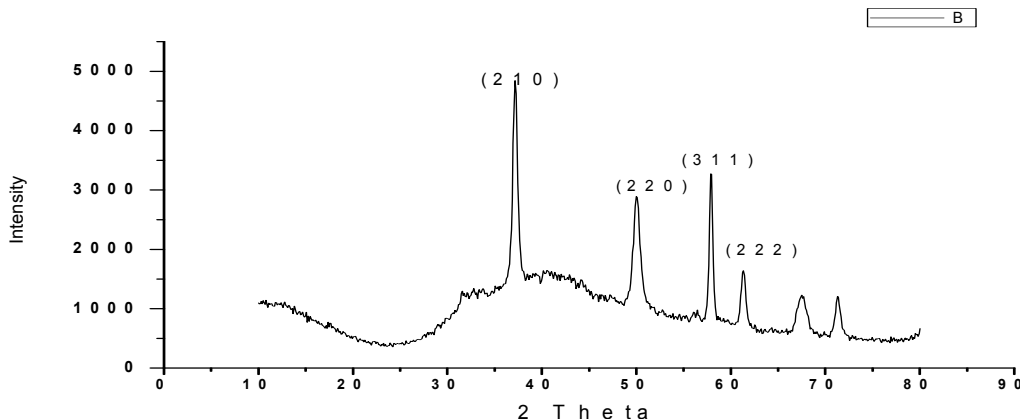


Fig. 1. XRD pattern of MgO nanoparticles

Table1. Characteristics of Mgo Nanoparticles

| 2θ (degree) | θ (degree) | sinθ | $d = \lambda / 2 \sin \theta$ | I(intensity) | $I/I_0 * 100\%$ | hkl | D nm |
|----------------|---------------|---------|-------------------------------|--------------|-----------------|-------|---------|
| 37.16 | 18.58 | 0.3186 | 2.4168 | 4841 | 100 | (210) | 22.1197 |
| 49.99 | 24.99 | 0.4224 | 1.8229 | 2891 | 59.71 | (220) | 11.5082 |
| 57.85 | 28.925 | 0.48366 | 1.5922 | 3273 | 67.60 | (311) | 11.7845 |
| 61.33 | 30.665 | 0.5100 | 1.5098 | 1640 | 33.87 | (222) | 17.9834 |

Optical Characterization:

UV absorption Spectra of MgO particles are shown in fig. 2. The UV-Visible spectra were taken from MgO powder and according to the graph slope is at $\lambda = 240 \text{ nm}$ and by the following formula we get band gap energy: $E = hc / \lambda$, since $c = 3 * 10^8 \text{ m/s}$, $h = 6.625 * 10^{-34} \text{ J.S}$ & $1 \text{ eV} = 1.6 * 10^{-19} \text{ J}$, Therefore $E = 3 * 10^8 * 6.625 * 10^{-34} / 240 \text{ nm}$ or $E = 5.17 \text{ eV}$ [8]

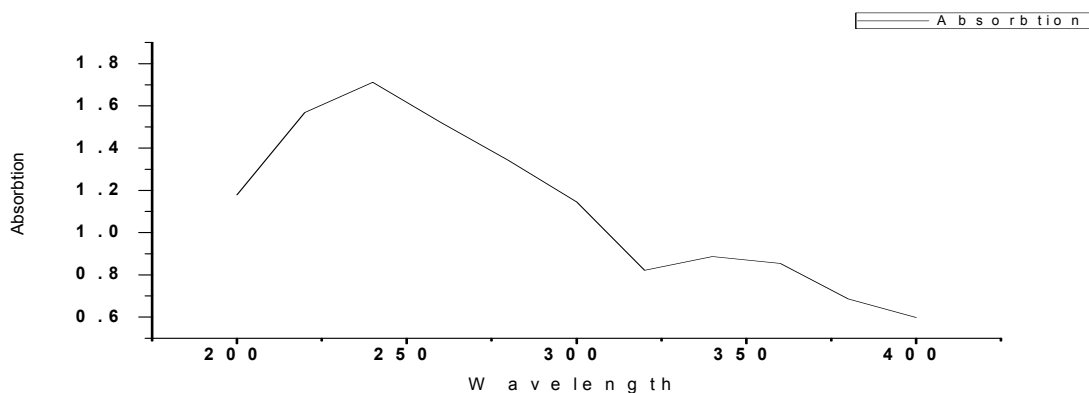


Fig. 2. UV absorption pattern of MgO nanoparticles

FTIR analysis:

FTIR Spectra of MgO particles are shown in fig. 3. Peaks at 3664 cm^{-1} , 3448 cm^{-1} corresponding to the O-H stretching mode of hydroxyl groups were present on the surface due to moisture. Peak at 1672 cm^{-1} was attributed to the bending vibration of water molecule. The major peaks at 449 cm^{-1} , 511 cm^{-1} , 671 cm^{-1} which confirmed the presence of Mg-O vibration [9].

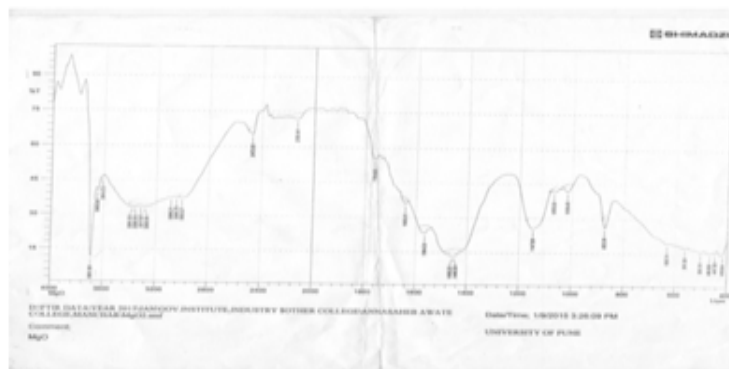


Fig. 3. FTIR spectrum of MgO nanoparticles

Scanning Electron Micrograph:

The surface morphology of prepared MgO nanoparticles was revealed through the SEM image shown in Fig. 4. It shows homogeneous distribution of spherical particles of the prepared MgO nanoparticles [10].

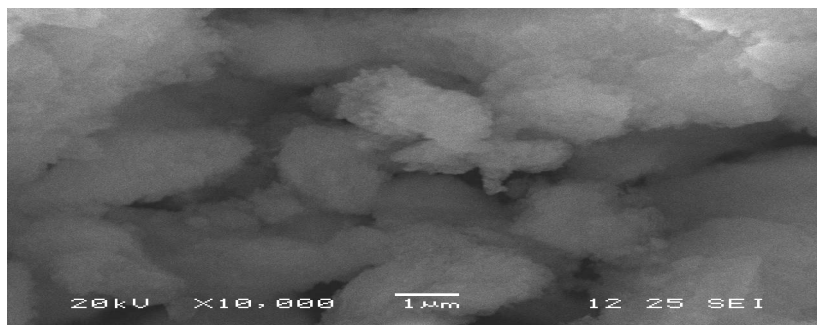


Fig. 4. SEM image of MgO nanoparticles

Conclusion:

Nanomaterials are important because of their extremely useful properties. They are exceptionally strong, hard and ductile at high temperatures. They resist wear, erosion and corrosion and are chemically very active. In most cases, nanomaterials outperform their conventional counterparts because of their superior chemical, physical and mechanical properties and outstanding formability. It is grain size and the order of 10^{-9} m (1nm). Some of the benefits like getting unique materials such as aerogels, zeolites, ordered porous solids by organic-inorganic hybridization are unique to sol-gel process. It is also possible to synthesize nanoparticles, nanorods, nanotubes etc. using sol-gel technique. The idea behind sol-gel synthesis is to “dissolve” the compound in a liquid in order to bring it back as a solid in a controlled manner. Multi component compounds may be prepared with a controlled stoichiometry by mixing sols of different compounds. The sol-gel method prevents the problems with co-precipitation.

References

- [1] Chikdu D, Pal P, Gujar A, Deshmukh R, and Kate S., “Green Synthesis and Characterization of Silver Nanoparticles by Using Aloe Barbadensis and its Antibacterial Activity”, *Journal of Global Biosciences*, Vol. 4, no.7, pp. 2713-2719, 2015.
- [2] W. Ueda, M. Sadakane, and H. Ogihara, “Nano-structuring of complex metal oxides for catalytic oxidation,” *Catalysis Today*, vol. 132, no. 1–4, pp. 2–8, 2008.
- [3] T. Gessner, K. Gottfried, R. Hoffmann et al., “Metal oxide gas sensor for high temperature application,” *Microsystem Technologies*, vol. 6, no. 5, pp. 169–174, 2000.
- [4] J. H. Kim, E. K. Kim, C. H. Lee, M. S. Song, Y.-H. Kim, and J. Kim, “Electrical properties of metal-oxide semiconductor nano-particle device,” *Physica E*, vol. 26, no. 1–4, pp. 432–435, 2005.
- [5] P. D. Pria, “Evolution and new application of the alumina ceramics in joint replacement,” *European Journal of Orthopaedic Surgery and Traumatology*, vol. 17, no. 3, pp. 253–256, 2007.
- [6] H. Farsi and F. Gobal, “Theoretical analysis of the performance of a model supercapacitor consisting of metal oxide nanoparticles,” *Journal of Solid State Electrochemistry*, vol. 11, no. 8, pp. 1085–1092, 2007.
- [7] A. C. Dillon, A. H. Mahan, R. Deshpande, P. A. Parilla, K. M. Jones, and S.-H. Lee, “Metal oxide nano-particles for improved electrochromic and lithium-ion battery technologies,” *Thin Solid Films*, vol. 516, no. 5, pp. 794–797, 2008.
- [8] L. D. Hart, *Alumina Chemicals: Science and Technology Handbook*, American Ceramic Society, Columbus, Ohio, USA, 1990.
- [9] A. Laachachi, M. Ferriol, M. Cochez, J.-M. Lopez Cuesta, and D. Ruch, “A comparison of the role of boehmite (AlOOH) and alumina (Al₂O₃) in the thermal stability and flammability of poly (methyl methacrylate),” *Polymer Degradation and Stability*, vol. 94, no. 9, pp. 1373–1378, 2009.
- [10] I. Lukic, J. Krstić, D. Jovanović, and D. Skala, “Alumina/silica supported K₂CO₃ as a catalyst for biodiesel synthesis from sunflower oil,” *Bioresource Technology*, vol. 100, no. 20, pp. 4690–4696, 2009.