Article

Investigations on Europium doped Magnesium Oxide Nano Particles: Synthesized by Polyol mediated Technique

K.Sujatha, D.Vijay, Ch.Ashok, G.Nirmala Kumari, K.Subramanyam, S.Sravani, S.Rajyalakshmi and K.Ramachandra Rao

Crystal Growth & Nanoscience Research Center, Department of Physics, Government College (A), Rajahmundry-533105, Andhra Pradesh, INDIA



MgO: Eu³⁺ (1 mol %) nanoparticles were synthesized through eco-friendly, inexpensive and simple low temperature chemical route Polyol using Ethylene glycol as solvent. The synthesized powders were characterized by Powder XRD, SEM and UV-Vis Spectroscopy. Powder XRD data revealed the formation of cubic system. From SEM and UV-Vis Spectroscopy, the influence of Eu³⁺ on the structure morphology, UV absorption and optical band gap of MgO: Eu³⁺ nanoparticles (NPs) were investigated. Powder XRD confirms the crystallinity of the sample and the average crystallite size calculated from Debye-Scherer equation was about 23.88 nm and the particle size calculated from Absorption spectra is around 9-10 nm. The same was confirmed by SEM. From UV-Vis spectrum it is evident that the doped MgO has a reduced band gap of 5.47 eV than undoped MgO (5.58 eV). The dopant concentration, effective crystallite size, textural properties and decreased band gap findings open up new possibilities for the exploration of Eu-doped MgO NPs to be used in the development of display devices.

Received on 8th Febraury 2021

Revised on 3rd May 2021 and 6th August 2021

Accepted on 12th August 2021

1.Introduction

Nanotechnology is the science of the small. It is the use and manipulation of matter at a tiny scale (1nm – 100nm). At this size, atoms and molecules work differently, and provide a variety of surprising and interesting uses. Nanoparticles exhibit completely new or improved properties with larger particles of the bulk materials and these novel properties are derived due to the variation in specific characteristics such as size and morphology of the particles [1]. Nanomaterials have found to exhibit interesting applications in various fields like catalysts, color display, flat panel displays, solar energy converters, optical amplifiers, electroluminescent devices, photodiodes, biodetectors, lamp industries, radiation dosimetry, host for solid-state lasers, solid

electrolytes, chemical sensors, magnetic refrigeration materials and thermal barrier coatings [2-8]. MgO is a well-known photocatalyst with unique chemical, mechanical, optical and electrical properties; inexpensiveness and nontoxicity were identified as the main reason for the acceptability of MgO materials.

The luminescence properties of rare earth doped MgO NPs depends on various factors such as phase purity, crystallite size, surface morphology, nature of dopants and the synthesis route [9,10]. Several physical and chemical procedures were used for the synthesis of large quantities of metal NPs in relatively short period of time. Approaches such as co-precipitation method and hydrothermal method [11,12] were widely used.

However, among all the polyol method is one of the more widely recognized methods due to its several advantages like soft chemistry, easy to handle and requiring no special or expensive equipment.[13]. In the present study, we have synthesized Eu doped MgO nanoparticles by simple Polyol method and the work was focussed to estimate the structural, optical and luminescence properties of the synthesized material.

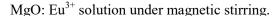
2. EXPERIMENTAL PROCEDURE:

Eu-doped MgO Nano particles were synthesized by a simple and low cost Polyol method using Magnesium nitrate, Europium nitrate (SDFINE –CHEM LIMITED) as starting materials. Ethylene glycol was used as a solvent and stabilizing ligand. First, an accurate weighed amount of Magnesium nitrate and Europium nitrate are added in 10 ml double distilled water and stirred well for 20 minutes.

After that 20 ml of ethylene glycol was added to the solution. Solution was shaken and kept under stirring. A thermometer was used to measure the temperature. When the temperature was raised to 100° C, around 2 gm. of urea was added and temperature was raised further to 120° C and maintained at this temperature for 2 hours.

The precipitate obtained after 2 h of reaction was cooled, centrifuged, washed twice with methanol, and twice with acetone. The precipitate was dried overnight under ambient conditions. The real-time synthesis process is shown in fig-1(a) & (b).







b) MgO: Eu³⁺ nanoparticles obtained at 120°C.

Flowchart for the preparation of Eu-doped MgO nanoparticles

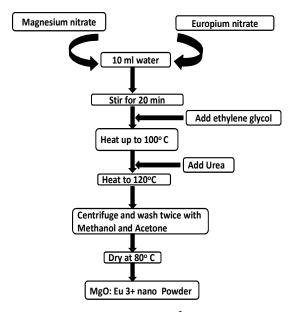


Fig-1: Polyol synthesis of MgO: Eu³⁺ nanoparticles

3. RESULTS AND DISCUSSION:

3.1 Powder X-Ray Diffraction Analysis

The powder x-ray diffraction pattern was recorded for better understanding of structure and crystallite size of the synthesized sample (MgO: Eu³⁺ 1 mol %). X-ray diffraction was recorded for the sample by means of a slowly moving radiation detector in the range of 10°-90 where monochromatic wavelength of 1.5405 Å (Cu) was used.MgO: Eu³⁺ phase was obtained at 700°C. The figure 3.1 shows the powder x-ray diffraction pattern of MgO: Eu³⁺ (1 mol %) nanoparticles calcinated at 700°C.

3.1 XRD Analysis

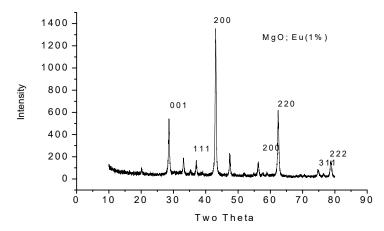


Figure 3.1: X-ray diffraction pattern of the Eu³⁺ doped MgO nanoparticles.

The XRD patterns of the Mgo Nano material with 1% Eu⁺³doping concentration are presented in Fig.3.1. The broadening of the diffraction peaks of all the MgO: Eu⁺³can be observed. For this sample the diffraction peaks correspond to the lattice planes are (111),(200),(220),(311) and (222) matching the cubic structure of MgO (JCPDS 4-829) with the lattice parameters a =4.2011 A^o. X-ray diffraction was recorded for the sample by means of a slowly moving radiation detector in the range of 10°-80°. No diffraction peaks from europium impurities are detected. The average size of the nanoparticle were calculated using diffraction maxima from the half-width of diffraction peaks using the Debye Scherrer formula.

$$\mathbf{D} = \frac{0.9\lambda}{\beta \cos \theta}$$

 $\mathbf{D} = \frac{0.9 \lambda}{\beta Cos\theta}$ Where λ = wavelength (1.7902A°), Θ is the bragg diffraction angle of the XRD peak, β is the measured broadening of the diffraction line peak at an angle 20, at half its maximum intensity (FWHM) in radian. The average size of our nano particle is found nearly 23.88nm.

3.2 Scanning Electron Microscopy (SEM):

The surface morphology of pure MgO and MgO: Eu³⁺ (1 mol %) nanoparticles studied through SEM analysis were shown in Fig-:3.2(a) & (b). The MgO: Eu³⁺ nanoparticles showed non-uniform size with nearly spherical shape. The crystallite size estimated from XRD and SEM measurements were in good agreement.

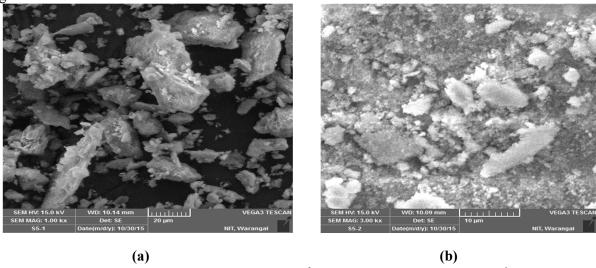


Figure 3.2: SEM micrographs of the pure (a) and Eu³⁺ doped MgO (b) nanoparticles at 900⁰C.

3.3 UV-Vis Spectroscopy & Optical Band gap calculation:

The UV-Vis absorbance of the sample was measured using a LABINDIA UV 3092 UV-Vis Spectrophotometer. The absorbance spectra of MgO: Eu³⁺ (1 mol%) nanoparticles were recorded by distributing the particles uniformly in solvent Ethelyne glycol in the wavelength range of 190-900 nm to estimate the optical energy band gap (Fig.3.3). The Fig.3.3.(a) shows strong and prominent absorption spectrum band with maximum around 230 nm which can arise due to transition between valence band to conduction band [14]. The smaller sized crystallites were found to have high surface to volume ratio which results in increase of defects distribution on the surface of nanomaterials and exhibit strong absorption bands [15]. In MgO: Eu³⁺, the particles size was in nanometer which results in high surface to volume ratio as a result, there is an increase in defects distribution on the surface of the nanomaterials.

The energy gap was determined by plotting Absorption coefficient and band gap from which the direct band gap was obtained in the range of 5.47–5.58 eV. This suggests that decrease in the band gap of Eu-doped MgO nanoparticles is due to the incorporation of Eu³⁺ ion into MgO matrix which alters the electronic structure leading to the appearance of intermediate energy level [16].Eu doped MgO nanoparticles can absorb light in a wider range of wavelength extending into to visible region and thereby use more luminous energy that results in higher quantum efficiency leading to higher photocatalytic activity. Doping with Eu³⁺ affects the CB shifted toward lower energy and a systematic decrease in valence band – CB occurs which leads to decrease in Fermi level and hence a reduction in the optical bandgap [17]. This would help the host laattice to exhibit high luminescence property. The decrease in optical band gap 5.47eV is observed when 1 % of Eu³⁺ is doped into MgO at 900°C temperature.

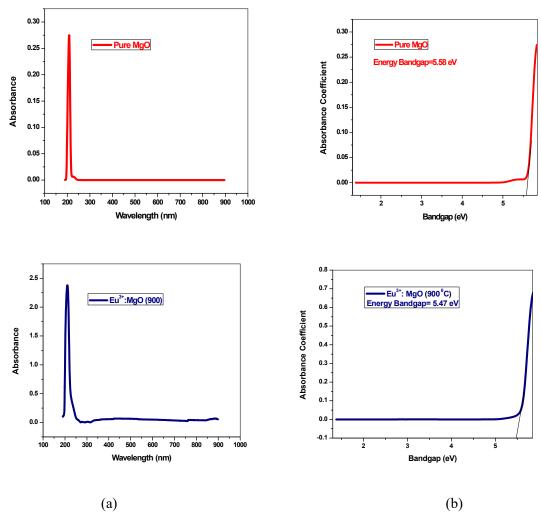


Figure 3.3 (a & b): UV-Vis Absorbance spectrums and Optical Band gaps of pure MgO and Eu³⁺ doped MgO nanoparticles annealed at 900°C.

4. CONCLUSION

Eu doped Magnesium oxide nanoparticles were synthesized by simple and inexpensive Polyol technique using Magnesium nitrate, Europium nitrate and ethylene glycol. When the synthesis temperature was 150- 170 °C in the polyol process, we obtained Eu-doped MgO nanoparticles not only with a small size but also with good absorption in the Ultraviolet and Visible regions. The powder XRD and SEM analysis confirms the crystallinity of the synthesized sample with an average crystallite size of 23.88 nm and average particle size of 10 nm for the doping concentration of 1 mol %. From UV-Vis spectrum it is evident that the doped MgO has a reduced band gap of 5.47 eV than undoped MgO (5.58 eV). The dopant concentration, effective crystallite size, textural properties and decreased band gap findings open up new possibilities for the exploration of Eu-doped MgO nanoparticles to be used in the development of display and optoelectronic devices and luminescent materials.

References

- 1. P. Ravindra Singh, K. Vineet Shukla, S. Raghvendra, K. Yadav, Prashant Sharma, Prashant K. Singh, Avinash, C. Pandey, Biological approach of zinc oxide nanoparticles formation and its characterization, Adv. Mater. Lett 2 (4) (2011) 313–317.
- 2. D. Norris, A. Efros, S. Erwin, Science 319 (2008) 1776–1779.
- 3. R. Si, Y.W. Zhang, L.P. You, C.H. Yan, Angew. Chim. Int. Ed. 44 (2005) 3256–3260.
- 4. L. Curkovic, D. Ljubas, S. Segota, I. Bacic, J. Alloys Compd. 604 (2014) 309–316.
- 5. Y.S. Vidya, K.S. Anantharaju, H. Nagabhushana, S.C. Sharma, H.P. Nagaswarupa, S.C. Prashantha, C. Shivakumara, Danithkumar, Spectrochim. Acta Part A Mol.Biomol. Spectrosc. 135 (2015) 241–251.
- 6. M.N. Huang, Y.Y. Ma, X.Y. Huang, S. Ye, Q.Y. Zhang, Spectrochim. Acta Part A Mol. Biomol. Spectrosc. 115 (2013) 767–771.
- 7. Y.S. Vidya, K.S. Anantharaju, H. Nagabhushana, S.C. Sharma, J. Alloys Compd.619 (2015) 760-770.
- 8. M. Chandrashekar, H. Nagabhushana, S.C. Sharma, Y.S. Vidya, K.S. Anantharaju, B. Daruka, S.C. Prasad, D. Prashantha, P. Kavyashree, Sadananda Maiya, Mater.Res. Express 2 (2015) 045402.
- 9. Y. Huang, S. Ou, G. Xu, M. Fang, X.Z. Li, Appl. Surf. Sci. 254 (2008) 2013–2016.
- 10. P. Mishra, R.S. Yadav, A.C. Pandey, Ultrason. Sonochem. 17 (2010) 560–565.
- 11. K. Sato, F. Sag, K. Nagaoka, Y. Takita, Int. J. Hydrogen Energy 35 (2010) 5393–5399.
- 12. H.R. Mahmoud, S.A. El-Molla, M. Saif, Powder Technol. 249 (2013) 225–233.
- 13. Noriya Izu, Kazuhiko Shimada, Takafumi Akamatsu, Toshio Itoh, Woosuck Shin, Kentaro Shiraishi, Taketoshi Usui, Ceramics International 40 (2014) 8775-8781.
- 14. R. Harikrishna, B.M. Nagabhushana, H. Nagabhushana, R.P.S. Chakradhar, R.Sivaramakrishna, C. Shivakumara, T. Thomas, J. Alloys Compd. 585 (2014) 129–137.
- 15. S. Das, C.Y. Yang, C.h. Lu, J. Am. Ceram. Soc. 96 (2013) 1602–1609.
- 16. V. Stengl, S. Bakardjieva, N. Murafa, Mater. Chem. Phys. 114 (2009) 217–226.
- 17. M.C. Wang, H.J. Lin, T.S. Yang, J. Alloys Compd. 473 (2009) 394–400.