J. Mountain Res. P-ISSN: 0974-3030, E-ISSN: 2582-5011 DOI: https://doi.org/10.51220/jmr.v16i2.12
Vol. 16(2) Special Issue ICRAMS-2021, (2021), 83-88

Synthesis And Structural Characterization of Samarium Doped Silica Nanopowder

Neelam Rani[1] • Rachna Ahlawat[1] • Surender Ahlawat[2]

[1]*Department of Physics, Material Science Lab., Chaudhary Devi Lal University, Sirsa, Haryana (India).*
[2]*Department of Economics, Chaudhary Devi Lal University, Sirsa, Haryana (India).*

*Corresponding Author Email: neelam.rani.sardiwal@gmail.com

Received: 2.8.2021; Revised: 29.8.2021; Accepted: 25.9.2021

Abstract: In the current research work, we demonstrate the synthesis of Samarium doped silica nanopowder through wet-chemical technique. The phenomenon correlated to structural morphology is proposed and conferred. Nanocrystalline form of Sm-SiO_2 powder has been formulated via sol-gel process. Performance of this method is absolutely correct for massive production and it is also a profitable in the sense of money. The ready sample are treated thermally at temperature 900ºC which are characterized through different supportive instrumental techniques as like 'X-Ray Diffraction' (XRD), 'Fourier Transform Infrared Spectroscopy' (FTIR) and 'Scanning Electron Microscope' (SEM) with EDX etc. The cubic phase of prepared sample is confirmed by XRD with average crystalline size ~18 nm using well known Debye-Scherer's formula and lattice constant is calculated as 10.8 Å. The surface morphology and microparticles structure information are collected from the SEM study. Investigation proposed that different nanoscopic collaborations play a key role in defining the morphology and crystal phase of ready materials. Presently, activity in this area is concentrated on the synthesis of glowing material via advanced methods and inspecting standard utilization in the area of microchips, photonics, flashes, sensors, optical intensification and fluorescent detecting appliances.

Keywords: Rare earth (RE) ions, Sol-gel, Annealing, Doped silica

Introduction

At present, nanomaterials have got universal attraction because of their brilliant structural and optical changes appearing due to surface morphology, dimensionality and magnitude of them. Oxide's form of rare-earth have been largely examined owed to their inimitable and stimulating properties as like enhanced scintillating efficiency (Murlidaran et al., 2009; Neto et al., 2010). This type of nanomaterial having translucent or visible gel holding high rare-earth amount are excellent applicants for photosensitive cleaner as thin skims or monoliths. Despite of several research, the formulation of crystal-clear silica gels containing rare-earth (Sm) with their natural configuration of nanomaterial in powder form has not been depicted largely till now. Recently, curiosity in this field is noticed for the synthesis purpose of luminescent materials by using superior methods and exploring standard applications in the field of integrated circuit technology, photonics, flashes, sensors, optical magnification and luminous detecting tools (Prasad PN, 2004). Rare-Earth doped materials have been produced using several approaches together with plasma-enhanced CVD (chemical vapour deposition), flame hydrolysis and co-precipitation (Sloff et al., 2001; Kundu et al., 2012; Kumar et al., 2010). In this investigation, we have adopted research with solution in majority-based procedure like 'soft chemical technique' to generate a

monitored nanostructure material. Also, the technique to prepare rare earth doped silica nanopowder using a wet chemical process is 'Sol-gel'. Sol-gel approach is an effectual course of action to study gel nanomaterials which are formed at normal room temperature (below 110ºC). Sol-gel method resulting in silica networked materials incapacitated with ions of rare-earth have supported exciting structural and optical properties alike luminescence, coloration and energy transmission (Ahmed et al., 2007). Here in current research work, we have explored the properties of samarium doped silica material in powder form in demand to accomplish more data regarding the characteristics of rare-earth doped silica nanopowder for modern optoelectronic devices.

Experimental

Sample Preparation

The chemical materials used in the synthesis of rare earth doped silica were tetraethyl orthosilicate (TEOS, purity >98%), spectroscopic grade ethyl alcohal (C_2H_5OH 99.8%), analytical grade ($Sm(NO_3)_2.6H_2O$), double distilled water catalyzed by hydrochloric acid (HCl). The groundwork of the prepared sample was centered on the sol-gel process styled in Fig. 1. Afterwards, formed gel ready for aging process at ambient temperature for 1 week and ultimately desiccated stepwise from 80°C to 110°C for 5 days to yield as-groomed sample T1. The as-groomed powder was positioned in furnace at 900°C for 3 h called as T2.

Characterizations

The most synthesized samples are characterized using eminent experimental methods as like: XRD, FTIR and SEM. XPERT-PRO x-ray diffractometer functioned at 45kV and 40 mA through Cu Kα monochromatic rays having wavelength 1.5406 Å in distinct arrays to produce x-ray pattern.

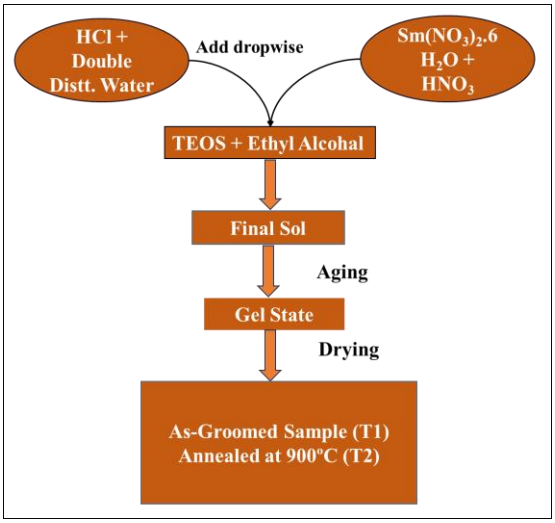

Fig. 1: Sol-gel method for formulation of samarium doped silica nanopowder.

As-groomed and annealed sample also go through FTIR spectra of 'Perkin Elmer 400 spectrophotometer' in IR region i.e., 400-4000 cm^{-1}. 'JEOL-JSM-6100' scanning electron microscope (SEM) joined by an energy dispersive x- ray spectrometer (EDX) used to study the surface morphology of ready sample.

Results & discussion

X-ray diffraction (XRD) analysis

The segment with erection of prepared nanopowder are determined by XRD. Crystalline phase was not

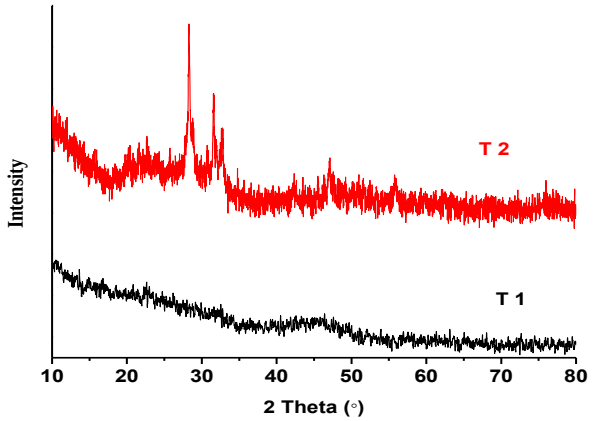

Fig. 2: XRD patterns of as-groomed (T1) and annealed sample (T2) at 900ºC.

observed in the Sm doped silica powder named as sample T1 i.e., as prepared sample. The XRD pattern of the sample T1 displays irregular peaks which strictly supports that the samples are in amorphous nature. The planes in this sample shown in Fig. 2 below are irregularly arranged so there is no long-range order in the distribution of planes in these samples. So, from studying the XRD spectrum it is clear now that the as-groomed sample is amorphous. The broad humps with no major diffraction peaks at 2θ ~ 22.62^0 and 45.40^0 might be ascribed to amorphous silica in T1 i.e., as-groomed sample linked to JCPDS card no. 29–0085 from collected works (Rani and Ahlawat, 2019). The hump near 2θ ~ 43.5^0, show that the activator ion (Sm^{3+}) can be arbitrarily scattered in the SiO_2 network (Chiad et al., 2011). The broad peaks were disappeared for sample T2 when sample annealed at 900℃ which give indication about the cumulate of these ions in the pore's structures of sol-gel materials. The strong peaks at 28.28^0 (222), 31.57^0 (400) with some minor peaks at 47.08^0 (440) and 55.79^0 (622) visibly specify the development of the pure cubic phase (JCPDS card no -42-1461) together with a slight volume fraction of monoclinic phase (Ghosh et al., 2010). The average nanocrystalline size of annealed sample T2 was estimated to be 18 nm using standard equation (1) given by Debye-Scherer's:

$$D = 0.9 \lambda / \beta \cos \theta \qquad (1)$$

here 'D' is mean or average nanocrystalline dimensions, 'λ' is wavelength of x-rays and 'β' denoted as full width at half maxima. Conferring to Scherer formula, the slim and sharp peak is obtained when the crystal size is larger. The lattice constraints for ready sample have been detected as, a = b = c = 10.8 Å and α = β = γ =90° confirmed that sample belongs to cubic system with $Ia\bar{3}$ space group (Antoinette et al., 2017). Thus, outcome of this type also be accredited to development of the crystal with thermal treatment because of disappearance of scums with accumulation of nanoparticles that are become bigger in size.

Fourier transform infrared (FTIR) analysis

FTIR spectrum of $Sm:SiO_2$ nanopowder of as-groomed and thermally treated sample have been revealed in Fig. 3 at normal temperature in scale of IR i.e., 400–4000 cm^{-1}. FTIR spectroscopy delivers the appreciated data and documentation of unlike chemical group/bonds existing in ready samples.

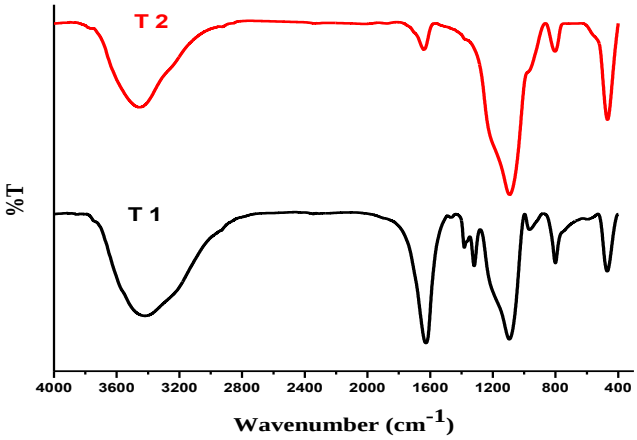

Fig. 3: FTIR Spectrum of as-groomed (T1) and annealed sample (T2) at 900ºC.

In the sample T1, two groups were seemed at about 3420-3454 cm^{-1} and 1627-1639 cm^{-1} owed to vibration of O-H bond in water molecules and expresses that the dehydrating process at room temperature does not completely catch the water molecules from the openings of silica gel network (Rani and Ahlawat, 2019). Therefore, the as-groomed sample needs heating at higher temperature to obtain dry gel. So, decrement of intensities in the annealed sample T2 (at 900ºC) showed that the water got removed from the gel. Some weak band at 1382, 1319 and 965 cm^{-1} related to nitrates/hydroxyl group is completely disappeared in sample T2 by effect of heating. The peak about 1100 cm^{-1} shows the presence of asymmetric stretching mode of Si-O-Si (Rani et al., 2020). The characteristics vibrational bands of silica were originated in FTIR spectra, where; the absorption bands at about 470 cm^{-1} and 800 cm^{-1}which may be due to bending and symmetric stretching vibrations of Si-O-Si group. Few researchers have also obtained Sm-O vibrational modes in the lowest wavenumber region (Xavier et al., 2013). Also, in annealed sample T2, the band appeared at 470 cm^{-1} became deeper may be because of overlapping of stretching vibrations of Si-O-Si group with Sm-O modes. The doped samarium ions, depending on its size and its high activation energy, cannot substitute the silicon atom and thus not form any ligands in the silica network (Mackenzie et al., 1984). FTIR measurements in the present work proved this suggestion.

Scanning electron microscope (SEM) analysis

The geomorphology of the as-groomed and annealed sample was inspected via scanning electron microscope (SEM) and the picture is shown in Fig. 4

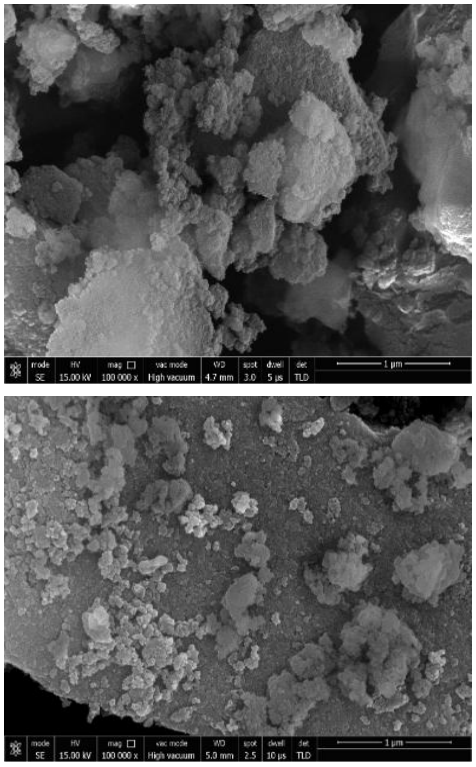

Fig. 4: SEM micrograph of as-groomed (T1) and annealed sample (T2) at 900ºC.

The particles are spherical in shape with better resolution.

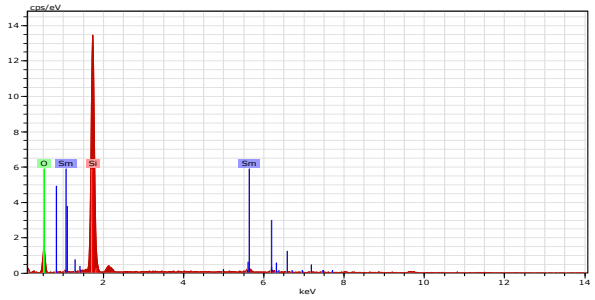

Fig. 5: EDX (Energy dispersive X-ray) spectrum of annealed sample T2.

The particles are agglomerated in the sample T1 indicate the amorphous nature of prepared sample. In sample T2 individual particle morphology can be seen due to annealing of sample.

Also, the EDX spectra of annealed sample shows the presence of samarium doped in silica powder with elemental composition of Sm having 0.67, Si having 43.90 and Oxygen having 55.43 at. % which is good agreement with other results.

Conclusions

Samarium doped in silica nanopowder were synthesized with the help of easy and simple sol-gel method. The prepared sample were obtained to be well crystalline and cubic in structure with slight volume of fraction monoclinic phase. Debye formulation used to compute the size of nanoparticles with average crystalline size of the ready sample was achieved to be ~ 18 nm. SEM was used for surface morphology simultaneously elemental composition with the help of joined EDX spectra.

References

Ahmed G, Koleva B, Gutzov S, Petkov I (2007) Experimental and theoretical spectral properties of ethyl 2-(7-hydroxy-2-oxo-2H-chromen-4-yl) acetate doped sol-gel materials: New materials with potential optical application, J. Incl. Phenom. Macro., 59: 167.

Antoinette MM, Israel S (2017) Synthesis and Characterization of Sm_2O_3 Nanoparticles using combustion method, Int. Res. Jr. of Engg. and Tech., 4: 1-5.

Chiad BT, Al-Maliki FJ, Twej WAA, Hameed MA (2011) Study the spectroscopy of samarium encapsulated in silica Xerogel matrices, Iraqi Jr. of Physics, 9: 49-54.

Ghosh P, Kundu S, Kar A, Ramanujachary KV, Lofland S, Patra A (2010) Synthesis and characterization of different shaped Sm_2O_3 nanocrystals, J. of Phys. D: Appl. Phys., 43: 405401-405408.

Kumar P, Sharma SK, Knobel M, Singh M (2010) Effect of La^{3+} doping on the electric, dielectric and magnetic properties of cobalt ferrite processed by co-precipitation technique, Journal of Alloys and Compounds, 508: 115-118.

Kundu S, Sutradhar N, Thangamathu R, Subramanian B, Panda AB, Jayachandran M (2012) Fabrication of catalytically active nanocrystalline samarium (Sm)-doped cerium oxide (CeO_2) thin films using electron beam evaporation, J. Nanopart. Res., 14: 1-16.

Mackenzie JD and Ulrich DR (1984), John Wiley and Sons, New York, Ch. 43: 589-601.

Murlidaran MN, Rasmitha CA and Ratheesh R (2009) Photoluminescence and FTIR studies of pure and rare earth doped silica xerogels and aerogels, J. of Porous Materials, 16: 635-640.

Neto MC, Sliva GH, Carmo AP, Pinheiro AS, Dantas NO, Bell MJV and Anjos V (2010) Optical properties of oxide glasses with semiconductor nanoparticles co-doped with rare earth ions, Chem. Phys. A, 101: 188.

Prasad PN, Nanophotonics (2004) New york: John Wiley & Sons Publications, pp. 1-418.

Rani N, Ahlawat R (2019) Role of Ceria Nanocrystals on Morphology and Luminescence of Eu^{3+} doped SiO_2 nanopowder, Jr. of Luminescence, 208: 135-144.

Rani N, Ahlawat R (2019) Tailoring the Structural and Optical Parameters of Eu^{3+}:CeO_2-SiO_2 Nanopowder Via Thermal Treatment, Silicon, 11:2521-2529.

Rani N, Ahlawat R, Goswami B (2020) Annealing effect on bandgap energy and photocatalytic properties of CeO_2–SiO_2 nanocomposite prepared by sol-gel technique, Material Chem. and Phys., 241: 122401.

Sloff LH, Dood MJA de, Blaaderen AV and Polman A (2001 Effects of heat treatment and concentration on the luminescence properties of erbium-doped silica sol-gel films, J. of Non-Crystalline Solids, 296: 158-164.

Xavier S, Thankachan S, Jacob BP, Mohammed EM (2013) Effect of Samarium Substitution on the Structural and Magnetic Properties of Nanocrystalline Cobalt Ferrite, Journal of Nanoscience, 524380: 1-7.