Structural Characterization of Combustion Synthesized Gd_2O_3 Nanopowder by Using Glycerin as Fuel

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***Abstract** -Combustion synthesis were accomplished to synthesize pure Gd_2O_3 phosphor by using glycerin as fuel. To investigate the luminescence behavior, excitation and emission spectra have been recorded. The prepared phosphor sample was characterized by using XRD, FTIR, SEM and TEM. From the XRD data, using the Scherer's formula the calculated average crystallite size of pure Gd_2O_3 phosphor is around 8nm. The TEM (Transmission Electron Microscope) and SEM (Scanning Electron Microscope) results agree with the XRD results.*

***Keywords:** Pure Gd_2O_3, Phosphors; XRD, FTIR, SEM & TEM; Corresponding author Email Id: - raunak.ruby@gmail.com.*

I. INTRODUCTIONS:-

Nowadays, Rare earth oxide luminescent materials have attracted great attention due to their size, shape and phase dependent luminescence properties, which make them suitable for fundamental and technological applications [1-4]. Among different families of crystalline materials, oxide crystals are of great interest due to their unique optical properties, such as long fluorescent lifetime, large Stokes shift, favourable physical and chemical properties as well as good photochemical stability. Several of the rare earth elements and their corresponding oxides are of exceedingly technical importance and are used in critical parts. Rare earth oxides are this kind of advanced materials and which are widely used as high performance luminescent devices, magnets, catalysts, and other functional materials such as electronic, magnetic, nuclear, optical, and catalytic devices [2, 5-6].

Gadolinium oxide (Gd_2O_3) is a good choice to investigate for luminescence properties due to its high refractive index (2.3), high optical transparency, good thermal and chemical stability, high dielectric constant, and low phonon energy among the family of oxide hosts [7-10]. Because of these favourable properties, it exhibits numerous applications such as three way catalysts for exhaust gas treatment from vehicles, oxide ion conductors in solid oxide fuel cells, oxygen gas sensors, electrode materials for sensors, optoelectronic devices, high definition televisions, biological imaging, high temperature superconducting materials, excellent ultraviolet light absorber, photo-catalyst, therapeutic effects in cancer treatment-enhancing the effect of radiation on cancerous cells while reducing damage to normal cells, luminescent inks, paints and dyes sunscreen cosmetics and luminescent materials[11-17].

In the presented work, combustion synthesis technique has been utilized to prepare nanopowders of gadolinium oxide by using glycerin as a fuel. The synthesis and characterization of Gadolinium oxide via different techniques have attracted considerable attention. The glycerin and metal nitrate get decomposed and give flammable gases such as NH_3, CO_2, and NO. When the solution reaches a point of spontaneous combustion, it begins burning and becomes a burning solid. The combustion continues until all the flammable substances have burned out and it turns out to be a loose substance with voids and pores formed by the escaping gases during the combustion reaction. The whole process takes only a few minutes to yield powder of oxide. The structural and optical characterizations of the synthesized powders were carried out using X-ray powder diffractometer. Scanning electron microscopy (SEM) was used to illustrate the formation of crystallites and TEM was used for particle size confirmation. Fourier Transform Infrared Spectroscopy (FTIR) spectrum of Gd_2O_3 nanopowder was obtained by using FTIR spectrophotometer (Model; MIR 8300TM) with KBr mixture in the pellet form.

Synthesis:- For synthesis of Gd_2O_3 phosphor an aqueous precursor solution containing 1 gm/ml $Gd(NO_3)_3.6H_2O$ and 2.5 gm/ml urea was used. When the solution was stirred for 1 h it was transformed into a transparent gel. This semisolid sample was heated at 600 ± 10°C in a muffle furnace. After few minutes spontaneous ignition occurred. Conversion of the sample to the anhydrous form was followed by a vigorous redox reaction which produced Gd_2O_3 phosphor with liberation of gaseous byproducts by libration of oxides of carbon and nitrogen. The heat of this redox reaction is sufficient for decomposition of the mixture. The resulting mixture was heated until a controlled explosion took place yielding a very fine powder [14]. There would be different mechanism of combustion reaction with different fuel-oxidizer combinations. When a mixture containing oxidizer and fuel with required stoichiometry is heated rapidly at or above the temperature of exothermic decomposition of fuel, it undergoes melting and dehydration initially. Later on this mixture foams due to the generation of gaseous decomposition products as intermediates and leads to enormous swelling.

The crystallinity as well as the particle size of the phosphor were monitored X-ray diffraction measurement. The X-ray powder diffraction data was collected by using Bruker D8 Advanced X-ray diffractometer using Cu Kα radiation. The X-rays were produced using a sealed tube and the wave length of X-ray was 0.154 nm. The X-rays were detected using a fast counting detector based on Silicon strip technology (Bruker Lynx Eye detector). Molecular structure was determined by FTIR analysis done by Nicolet Instruments Corporation USA MAGNA-550. The surface morphology of the prepared phosphor was determined by field emission scanning electron microscopy (FESEM) JSM-7600F. Energy dispersive X-ray analysis (EDX) was used for elemental analysis of the phosphor. Particle diameter and surface morphology of prepared phosphor were determined by Transmission Electron Microscopy (TEM) using Philips CM-200.

II. RESULTS & DISCUSSIONS:-

(I) **XRD Results:-** The powder X-ray diffraction (PXRD) was used to investigate the phase structures of the resulting powder. No crystalline impurity apart from rare earth oxides can be determined from the PXRD analysis. The crystallite size was calculated by the X-ray line broadening method using the Scherrer formula [18]:

$$D = \frac{k\lambda}{\beta \mathrm{Cos}\theta}$$

where λ is the wave length of radiation used (Cu Kα in this case), k is the constant, β is the full width at half maximum (FWHM) intensity of the diffraction peak for which the particle size is to be calculated, θ is the diffraction angle of the concerned diffraction peak and D is the crystallite dimension (or particle size). It can be seen that the diffraction pattern of the phosphor is consistent with the JCPDS data (43-1015), indicating that the sample of Gd_2O_3 phosphor is in the pure monoclinic phase [19]. The broadening of the diffraction peaks is due to the small crystallite size of the formed nanoparticles, which are less than 10 nm.

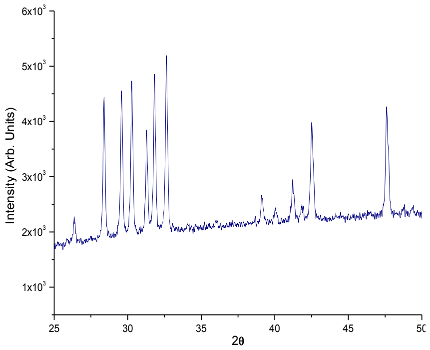

Figure 1. Powder X-Ray Diffraction pattern of Gd_2O_3 phosphor

(II) **FITR Results:-** FTIR spectra have been recorded for combustion synthesized Gd_2O_3 powders by using glycerin as a fuel as shown in Fig. 2. The broad peak cantered around 3400 cm^{-1} to 3500 cm^{-1} corresponds to the O-H symmetric stretching from hydroxyl group [4,20]. FTIR spectra revealed that the peak at 1534 to 1456 cm^{-1} is may be due to surface-adsorbed NO_3^- group, stretching vibration of NO [21]. The weak peak at 1600 cm^{-1} are correspond to the stretching mode of C=O in a carbonyl functional group for the prepared material [22]. The similar results have also been observed elsewhere [23]. Hydroxyl groups provide an effective pathway for the radiation less energy transfer of OH vibration and quench the emission intensity. The band at approximately 542 cm^{-1} is ascribed to the Gd–O vibration of Gd_2O_3 phosphor [4,14,17, 24].

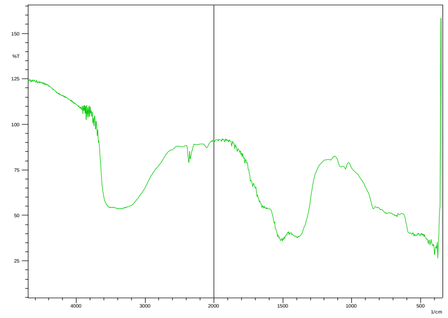

Figure 2. FTIR spectra of Gd_2O_3 phosphor

(III) Scanning Electron Microscope (SEM) results:-

The morphology analysis of Gd_2O_3 powders have been studied by SEM using backscattered electron detection mode. The black and white SEM micrograph of the prepared powder indicates that all the particles are looking like agglomerated in homogeneously in different shapes/sizes of the order of nano range

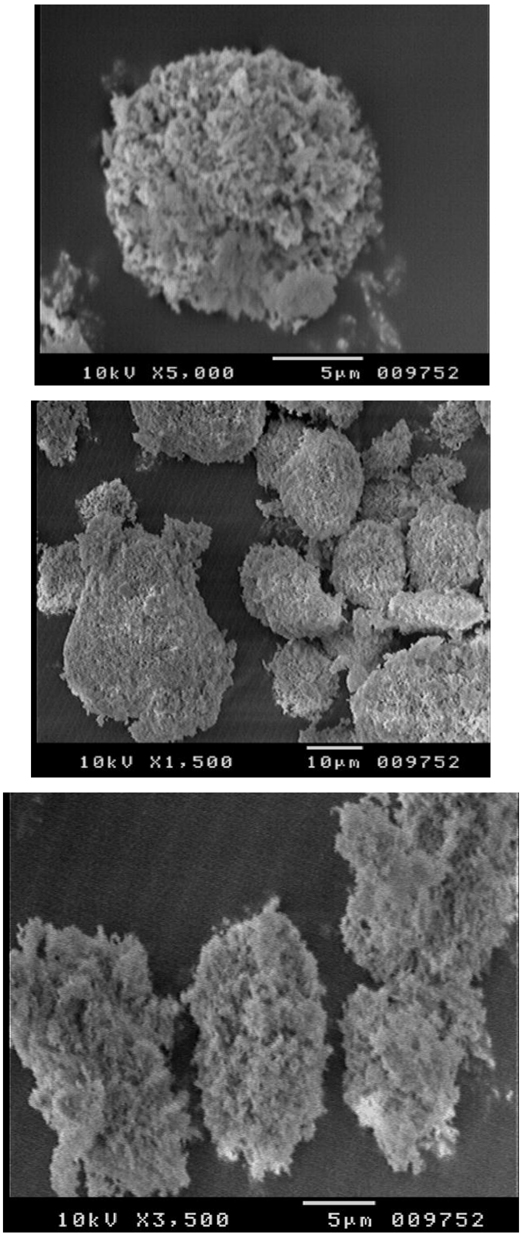

Figure 3. Scanning Electron Microscope image of Gd_2O_3 phosphor

Energy Dispersive X-ray analysis (EDX)

The EDX spectra represent elemental analysis of the prepared sample (Fig4). In the spectrum intense peak of Gd, and O are present which confirms the formation of Gd_2O_3 phosphor (fig 4). For EDX analysis, entire area of the black and white SEM micrographs was analyzed with EDX mapping and spectrum. The EDX mapping measurements were carried Gd_2O_3 powders to analyze the composition of the clustered particles.

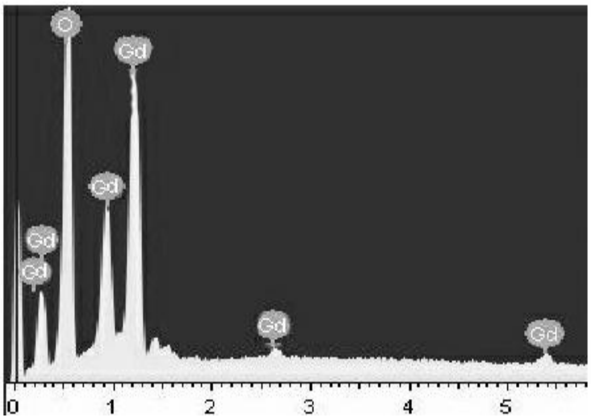

Figure 4. Energy dispersive X-ray analysis of Gd_2O_3 phosphor

Transmission electron microscopy (TEM) Results: - Transmission electron microscopy was used to study the morphology of combustion synthesized Gd_2O_3 phosphor using glycerine as fuel (Figure 5). TEM analysis shows the nanocrystalline behavior of the prepared phosphor with particle size in the range of 8nm to 12nm which is identical with XRD result.

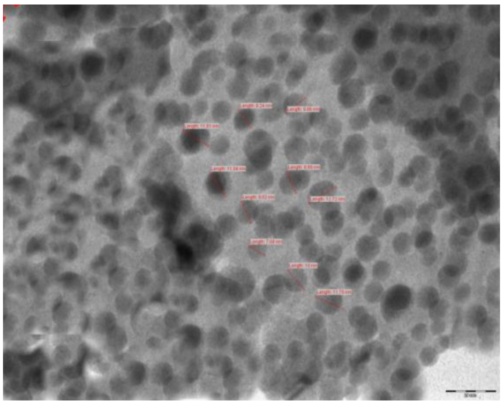

Figure 5. Transmission electron microscope image of Gd_2O_3 phosphor

III. CONCLUSION: -

From above studies it is concluded that, this method is suitable for large scale production and eco-friendly. The sample was characterized by XRD, FTIR, SEM, and TEM studies. From XRD pattern it is confirmed that prepared phosphor has monoclinic structure and particle size was determined by Debye- Scherer formula, which was found 8 to 12 nm range. The functional group analysis was determined by FTIR analysis which confirms the formation of Gd_2O_3 nano phosphors. Also confirmation found by the SEM and TEM micrographs which is good agreement with XRD pattern.

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