**Synthesis, structural and optical characterization of gadolinium mixed yttrium oxide nanomaterials**

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**ABSTRACT**

 Gadolinium mixed Yttrium oxide (Y2O3: Gd) nanomaterials have been synthesized by co-precipitation technique. The synthesized materials have been annealed at 500°C, 750°C and 1000°C using a muffle furnace at normal atmosphere. It is observed that the crystalline nature of the nano powders was increased due to annealing process which is evidenced by the observed diffraction planes in the powder diffraction patterns. In order to identify the oxide vibrations in response to the IR frequency, the FTIR spectra have been recorded and the different modes of vibration of rare earth oxides have been observed. The elemental analysis has been carried out using XPS and energy dispersive spectral analysis using a scanning electron microscope. The weight ratio and atomic ratio of the elements present in the sample has been identified and discussed. The morphology of the annealed samples has been studied through high resolution SEM images and the crystallite size was found by using TEM images. The photo luminescence spectral analysis indicated that the synthesized materials have emission in the blue region. However, it is also observed that the annealing temperature of the samples has influenced significantly on the structure, morphology and photoluminescence property.

**Keywords:** Rare earth; X-ray diffraction; HRSEM; TEM; PL Spectrum

1. **INTRODUCTION**

Nanomaterials are being the focused interest of researchers because of their impending applications in various fields of materials science and for the technological development in recent decades. Metal oxides, in particular rare earth oxides and their derivatives have very good electrical, semiconducting and insulating properties which have potential applications in various fields. Especially, rare earth oxides were identified and reported as potential materials for extraordinary luminescent property[1–3]. It has been observed that the doping of other rare earth elements in yttrium oxide has influenced to a greater extent on the enhancement of photoluminescence properties. The emission peak of europium doped yttrium oxide was found to be shifted to the blue region due to the change of crystal field resulting from great surface tension of its nanoscale [4]. Eu doped yttrium oxide nanostructure has been investigated for optical properties with respect to size and it is found that method of preparation has not affected the optical property of Y2O3: Eu [5]. However, the incorporation of lithium nano powder in Eu: Y2O3 prepared by co-precipitation method had enhanced the intensity of luminescence [6]. In view of enhancing optical property, europium incorporated Y2O3 has been synthesized and reported by several authors [7,8]. Nanocrystalline Y2O3/Pr3+ have been synthesized by sol-gel process and the photoluminescence property has been reported [9]. Incorporation of Tb3+ in yttrium oxide has also been investigated and found that the optical properties have been tuned for the enhancement [10,11]. Recently, Nd3+ mixed yttrium oxide has been synthesized and its structural, morphological and photoluminescence property have been reported [12,13]. In recent years, the yttrium oxide nanomaterials are being subjected to analyze the properties of different kind of charged surfactants, as photo active metal oxides, super capacitor applications and in antimicrobial applications [14,15]. Microstructure, cation distribution and magnetic susceptibility with respect to annealing temperature and size of (Y0.9Gd0.1)2O3 have been investigated and reported [16]. Photoluminescence and thermo luminescence properties have been reported for gadolinium doped yttrium oxide phosphor prepared by solid state reaction method [17]. Gama ray induced thermo luminescence studies have been reported in gadolinium doped yttrium oxide prepared by solid state reaction [18]. Along with structural and morphological investigation, thermo luminescence glow curve has been obtained for gadolinium doped yttrium oxide nanophosphor and the parameters such as activation energy and trap depth, order of kinetics and frequency have been obtained [19].

 However, limited works have been reported on gadolinium mixed yttrium oxide nanomaterials on their structural and photoluminescence characteristics. Hence, it is aimed to prepare the gadolinium mixed yttrium oxide nonmaterial and to study its structural, morphological and photoluminescence properties. Various nanostructures of rare earth yttrium oxides with different morphologies have been synthesized by methods like sol-gel [9,20], thermal decomposition [21], solvo-thermal [22] co-precipitation [23], auto-ignition [24], etc., and their properties were investigated. Among these methods, co-precipitation method has been widely used as it is simple and effective method for the preparation of rare earth oxides[23]. Hence, in this work, Y2O3:Gd has been prepared by co precipitation technique. The Y2O3:Gd has been prepared by replacing yttrium precursor with ten molar weight percent of gadolinium precursor. The synthesized materials were subjected to structural, morphological and photoluminescence characterization and discussed in this paper.

1. **SYNTHESIS**

 Bulk nano particles of gadolinium mixed Y2O3 have been synthesized by co-precipitation method. In a typical synthesis of gadolinium mixed Y2O3 nanomaterials, aqueous yttrium nitrate hexahydrate solution was prepared by dissolving yttrium nitrate hexahydrate (0.09 M) in Millipore water (resistance ~18MΩ) with ten weight percentage (0.01M) of Gadolinium trichloride hexahydrate. This aqueous solution was stirred well and the required amount of ammonia solution was added drop wise. After constant stirring at room temperature for 3 h, the white precipitate was collected and dried overnight at 70°C to remove the solvent. The dried particles were washed with ethanol and water several times in order to remove the ionic impurities. The possible chemical reaction for the formation of Y2O3:Gd nanomaterials can be expressed as,

$(0.09M)Y\left(NO\_{3}\right)\_{3} 6H\_{2}O+(0.01M)Gd\left(Cl\right)\_{3} 6H\_{2}O \rightarrow Y^{3+}+Gd^{3+}+3Cl\_{3}^{-}+6H\_{2}O$ (1)

$Y^{3+}+Gd^{3+}+3NH\_{4}OH \rightarrow Y(1-x)Gdx(OH)\_{3}+3NH\_{4}^{+}$ (2)

 $2Y(1-x)Gdx(OH)\_{3}→Y\_{2}O\_{3}:Gd+3H\_{2}O$ (3)

Y(NO3)3.6H2O and GdCl3.6H2O decomposes to form Y3+ and Gd3+ along with their byproducts as shown in Equation (1). The OH- in the base reactant reacts with the acid reactant of Y3+ in the solution, resulting in homogeneous precipitation. As per Equation (2) Y (OH)3 is formed through the hydrolysis of Y3+ followed by the oxidation of O2 from air to form Y2O3 (Equation (3)). In order to improve the crystalline nature of the as-prepared sample, it was annealed at 500°C, 750oC and 1000°C for 3 h in a muffle furnace.

1. **CHARACTERIZATION TECHNIQUES**

XRD analysis was carried out in reflection mode with Cu Kα radiation (λ=1.5406 Å) using a X'Pert Pro diffractometer with the scanning rate of 1°/min in the 2θ range from 20° to 80°. FTIR spectra of as prepared Y2O3:Gd nanomaterials annealed at 1000°C were recorded by NICOLET Infrared spectrophotometer. X-ray photoelectron spectra have been obtained using Shimadsu ESCA 3400 electron spectrophotometer. Surface morphology and elemental analysis have been studied by High resolution scanning electron microscope (HRSEM) attached with energy dispersive analyzer of X-rays (EDAX) system. Transmission electron microscope (TEM) JEOL JEM-200 CX model at 200 kV has been used to obtain TEM images and selected-area electron diffraction (SAED) pattern. The photoluminescence was studied using a Jobin Yvon Fluorolog-3-TAU steady-state/lifetime spectrofluorometer in the range of 200–900 nm for the as-prepared samples and the samples annealed at 500°C, 750°C and 1000°C.

1. **RESULTS AND DISCUSSION**
2. **Powder XRD Analysis**

 Powder XRD patterns of Gadolinium mixed Y2O3 as-prepared nanomaterials and annealed at 750°C and 1000°C temperatures are compared in Figure 1. The XRD patterns of as-prepared samples show amorphous nature with some humps around 30° and 45°, which is the indication of Y2O3 compound formation in the mixed Yttrium oxide as well. The amorphous nature observed in the as-prepared sample may be due to the hydroxide compound of Yttrium. When the samples are annealed at higher temperatures, the hydroxides have been transformed into oxide, which is confirmed by the powder XRD patterns obtained for the annealed samples. The XRD patterns of the annealed (at 750°C and 1000°C) samples, confirm the cubic phase of Y2O3 nanomaterials (JCPDS #83-0927), with lattice constant a = 10.6108 Å. The calculated lattice constant is reflected in almost all the lattice planes of the XRD patterns of Y2O3:Gd nanomaterials with only meager variations. Hence, It is inferred from the XRD patterns of Y2O3:Gd nanomaterials that there is no significant variation in the crystal structure due to annealing. However, a meager peak shift towards lower diffraction angle due to annealing at higher temperature has been observed in the pattern as shown in the Figure 1(B) which may due to lattice reorientation during the crystallization process at higher temperature. Also, the annealing at higher temperature has influenced to increase the crystallite size as expected. The dislocation density and stacking fault also have been calculated and found that these values have been decreased as the annealing temperature has been increased as shown in the table 1.

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**Figure 1: Powder XRD patterns of (A) as-prepared and annealed Y2O3:Gd** **nanomaterials at 750°C and 1000°C temperatures in air (B) Comparison of the peak for 222 diffraction planes.**

**Table 1 Comparison of Powder diffraction data of Y2O3:Gd** **nano particles annealed at 750°C and 1000°C**

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **hkl** | **Pos. [°2Th.]** | **FWHM [°2Th.]** | **Crystallite Size [nm]** | **Dislocation Density (x 1015 lines/m2)** | **Sacking fault (Å)** |
| **750°C** | **1000°C** | **750°C** | **1000°C** | **750°C** | **1000°C** | **750°C** | **1000°C** | **750°C** | **1500°C** |
| 211 | 20.62 | 20.48 | 0.36 | 0.41 | 22.44 | 19.76 | 1.986 | 2.562 | 0.002523 | 0.002885 |
| 222 | 29.26 | 29.13 | 0.44 | 0.41 | 18.67 | 19.83 | 2.869 | 2.542 | 0.002149 | 0.002031 |
| 400 | 33.92 | 33.75 | 0.48 | 0.45 | 17.31 | 18.45 | 3.337 | 2.938 | 0.002006 | 0.001892 |
| 440 | 48.62 | 48.49 | 0.50 | 0.44 | 17.44 | 19.82 | 3.287 | 2.545 | 0.001411 | 0.001245 |
| 622 | 57.74 | 57.60 | 0.60 | 0.48 | 15.12 | 19.20 | 4.371 | 2.764 | 0.001387 | 0.001106 |

1. **FTIR Spectral Analysis**

 FTIR spectra of asprepared and annealed samples of Y2O3:Gd nanomaterials were recorded by NICOLET Infrared spectrophotometer as shown in Figure 4.8. FTIR spectra of Y2O3:Gd nanomaterials contains O-H stretching vibration at 3490 cm-1. The weak intense peak around 1650 cm-1 is due to hydroxyl group in the water is also absorbed by the sample from the atmosphere. The intense peak around 1530 cm-1, 1400 cm-1 and 629 cm-1 are due to the stretching vibrations of rare earth element with oxygen. The shift in the peaks due to annealing effect has been observed and tabulated in Table 2.

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**Figure 2: FTIR spectra of as-prepared and annealed samples of Y2O3:Gd**

**Table 2. Wave numbers of important vibrations observed and their assignments**

|  |  |  |
| --- | --- | --- |
| **Sl. No.** | **Wavenumbers** | **Assignment** |
| **As-Prepared** | **Annealed at 1000°C** |
| **Y2O3** | **Gd:Y2O3** | **Y2O3** | **Gd:Y2O3** |
| 1 | 3490 | 3490 | 3440 | 3440 | O-H stretching |
| 2 | 1530 | 1530 | 1540 | 1530 | C-O asymmetric stretching |
| 3 | 1400 | 1400 | 1400 | 1410 | C-O asymmetric stretching |
| 4 | 1080 | 1080 | 1030 | 953 | C-O asymmetric stretching |
| 5 | 629 | 629 | 565 | 571 | Y-O stretching |

1. **X-Ray Photoelectron Spectral Analysis**

The XPS wide spectra obtained for pure Y2O3 and Y2O3:Gd nanomaterials annealed at 750°C are shown in Figure 3.It is observed that the spectra the show the signals for Y 3d, C 1s, and O 1s electrons at their respective binding energies. Figure 4 (a) shows the core level X-ray photo electron spectra of Y 3d electrons present in pure Y2O3 and the same for Y2O3:Gd is shown in Figure 4(b). In the case of pure Y2O3, Y 3d core level spectrum has a single high intense signal of electrons with the binding energy of 158.6 eV. It is observed that, the signal has two peaks related to Y 3d5/2 and Y 3d3/2 and their binding energies are 157.8 eV and 159.6 eV, respectively. The difference between the binding energies of these two electrons is nearly 1.8 eV. In the case of Y2O3:Gd, the binding energy of the single peak observed has no variation. However, the deconvoluted spectrum shows two signals related to Y 3d5/2 and Y 3d3/2 electrons and the binding energies related to this have been observed to be slightly increased 0.4 eV and 0.6 eV respectively. This ensures that the incorporation of doping elements are in favor since it is not significantly weakened the binding nature of Y 3d electrons.

The core level spectra of 1s electron of other prominent element (Oxygen) present in the pure and Y2O3:Gd samples has given its signal at the binding energy of 530.6 eV (Figs. 4 c&d) and the similar phenomenon of unaltered binding energy due to the dopants has been observed. However, it is observed that the single peak in both cases contains two signals on deconvolution. In the case of Pure Y2O3, the binding energies of the oxygen signals are 530.4 eV and 532.1 eV. The Oxygen having greater biding energy is lattice oxygen and the smaller one is for the oxygen diffused to the surface due to annealing process. The binding energy of lattice oxygen in Y2O3:Gd has only a meager change of 0.1 eV. The binding energies of Y 3d and O1s electron in both cases ensure that Gd has been incorporated into the lattice perfectly.

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**Figure 3: XPS spectra of as-prepared (a) pure Y2O3 and (b) Y2O3:Gd** **shows the signal for Y 3d, C1s and O1s electrons**



**Figure 4: Core level XPS spectra of (a&b) Yttrium 3d electrons from Pure and Y2O3:Gd, (c&d) Oxygen 1s electron obtained from Pure and Y2O3:Gd**

The core level spectra of 3d and 4d electrons of dopant element (gadolinium) present in the Y2O3:Gd samples have given their signals at their respective binding energies of 1241.6 eV and 150 eV, respectively as shown in the Figure. 5 (a&b). On deconvolution of Gd 3d electron signal, two different peaks related to Gd 3d5/2 and Gd 3d3/2 have been observed at 1236.3 eV and 1241.2 eV, respectively. On the other hand, Gd 4d electron gave its signal as doublet related to Gd 4d5/2 and Gd 4d3/2 have been observed at 142 eV and 150 eV, respectively with nearly the binding energy separation of 8 eV. These observed signals of 3d and 4d electrons of gadolinium are the signature of its presence in the Y2O3:Gd.



**Figure 5: Core level XPS spectra of Gadolinium (a) 3d electron and (b) 4d electron signals obtained from Y2O3:Gd**

1. **EDS and Morphological Analysis**

 The Y2O3:Gd.was subjected to elemental analysis using a scanning electron microscope attached with energy dispersive analyzer of X-rays (EDAX) system. The SEM images with energy dispersive spectrum of Y2O3:Gd are shown in the Figure 6(a-c). The elemental compositions of the samples are tabulated in the inset of Figure 6c. It is observed that 5.95 weight percentages is the actual incorporation of gadolinium in the Yttrium oxide nanaostructures. Surface morphology was studied by High resolution scanning electron microscopic (HRSEM) images. HRSEM images of as prepared Gadolinium mixed Y2O3 nanomaterials in two different magnifications are shown in Figure 6 (a&b). It is observed from the images that the morphology of the mixed samples is irregular in shape and no significant difference. However, the crystalline nature is high in the Gadolinium mixed samples, which ensures the agglomeration/aggregation of the powder samples to form a highly crystalline structure. TEM images with low and high magnification of Y2O3:Gd samples are shown in Figure 6 (d&e). It ensures that the particle sizes are less than 50 nm. The SAED pattern ensures that the particles are of high crystalline order as the bright dots are observed in the rings as shown in Figure 6f.



**Figure 6: HRSEM images and Energy dispersive spectrum (a-c) and TEM images of Y2O3:Gd** **sample annealed at 750°C at different magnification along with SAED Pattern(d-f). The inset of ‘c’ shows the elemental composition.**

1. **Photoluminescence Spectral Analysis**

 The photoluminescence was studied using a JobinYvon Fluorolog-3-TAU steady-state/lifetime spectrofluorometer in the range of 200–900 nm. The PL spectra of pure Y2O3 nanomaterials and Gadolinium mixed Y2O3 nanomaterials for the excitation wavelength of 350 nm are shown in Figure 7. The violet/blue light emission peak at 434 nm for Gadolinium mixed Y2O3 nanomaterials could be explained by the charge transfer from 4f band to the valence band of Y2O3 [25]. The emission peak for the annealed samples appears nearly at the same wavelength that of the as-prepared sample with a significant variation in the intensity. However, new peaks at 532 and 572 nm have been observed with increased intensity. This infers that the host emission at 434 has been suppressed and the defect level emission has been increased due to the annealing process.

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**Figure 7: PL spectra of Y2O3:Gd** **for 350 nm light excitation in the region between (A) 300-700 and (B) 520 - 600.**

1. **CONCLUSION**

 In conclusion, Gadolinium mixed Yttrium oxide nano particles were synthesized by co-precipitation technique. It is found that the as-prepared nano powders possess amorphous nature as it contains no significant diffraction planes in the corresponding powder X-ray diffraction patterns. The synthesized materials have been annealed at 750°C and 1000° C using a muffle furnace at normal atmosphere. It is observed that the crystalline nature of the as-prepared nano powders was increased due to annealing process which is evidenced by the observed diffraction planes in the powder diffraction patterns. The powder diffraction patterns of as-prepared and annealed samples of Y2O3:Gd were compared and found that the annealing process has enhanced the crystalline nature. The different modes of vibration of rare earth oxides have been observed and identified by FTIR spectra. The elemental analysis has been carried out using XPS and energy dispersive spectral analysis using a scanning electron microscope. The weight ratio and atomic ratio of the elements present in the sample has been identified and discussed. The morphology of the annealed samples has been studied through high resolution SEM images and the crystallite size was found by using TEM images. The photo luminescence spectral analysis indicated that the pure as well as Y2O3:Gd nanomaterials have emission in the blue region. However, it is also observed that the inclusion of gadolinium has significantly influenced and enhanced the emission in the blue region.

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