Synthesis and Characterization of Gd doped Y$_2$O$_3$ Phosphor Material

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ABSTRACT

In particular, the nanostructures phosphor materials are of great interest as they offer brighter cathodoluminescence and much improved screen packing. Inorganic nanoparticles manifest unique size and shape (surface to volume ratio) dependent properties, which in some extent depend on their crystallinity, defect contents, and preparation techniques. In the present work we have synthesized phosphor material by homogenous coprecipitation method (HCP) using Y$_2$O$_3$ as host material and inner transition metal, Gd as dopant. The synthesized phosphor material has been analyzed by XRD, SEM, and FTIR for their morphology and by PL spectrophotometer for their luminescence property. Using obtained data the effect of concentration of dopant on morphology and luminescence property of phosphor material have been studied.

Keywords: phosphor, homogeneous coprecipitation method, Y$_2$O$_3$:Gd$^{3+}$, photoluminescence.

INTRODUCTION

In literature large numbers of reports are found on inner transition metal doped Y$_2$O$_3$ nanostructures with different morphologies. These materials have been synthesized by different methods such as gas phase condensation technique, sol-gel route, homogeneous coprecipitation, spray pyrolysis, and hydrothermal method by various research groups.
Y$_2$O$_3$ phosphors doped with rare earth (RE) ions are considered as promising candidate in the field of cathodoluminescent display, catalysis and luminophors due to their high chemical durability and thermal stability [1,2]. In some reports HCP method was described to synthesize Y$_2$O$_3$: Eu red phosphors [3]. We observed few reports which describes synthesis of Y$_2$O$_3$ phosphors codoped with rare earth ions like Eu$^{3+}$, Gd$^{3+}$ and Tb$^{3+}$ for their color tunable properties [4,5]. Over last few years, Y$_2$O$_3$:Ln$^{3+}$ has been recognized as one of the best commercial color phosphors for plasma display panels (PDP) and Hg free fluorescent lamps. Synthesis of Eu$^{3+}$ doped Y$_2$O$_3$ and effect of different surfactant on morphology of phosphors was reported [6]. Study on novel preparation method and luminescent properties of Eu$^{3+}$ doped YBO$_3$ phosphors was reported [7]. Synthesis and characterization of Eu$^{3+}$ doped Y$_2$O$_3$(red phosphor) and Tb$^{3+}$doped Y$_2$O$_3$(green phosphor) by hydrothermal method was reported [8]. Sensitization effect of Yb$^{3+}$ in upconversion luminescence of Eu$^{3+}$ codoped Y$_2$O$_3$ phosphor was reported [9]. Photoluminescence studies of Eu$^{3+}$ doped Y$_2$O$_3$ phosphor was reported [10].

The present work describes the homogenous co precipitation method (HCP) for synthesis of inner transition metal ion, Gd$^{3+}$ doped Y$_2$O$_3$ phosphor material in aqueous medium. The formed precipitate after sintering and calcinations gives nanostructure phosphor material in powder form. The nanostructure, Y$_2$O$_3$:Gd$^{3+}$ is obtained by changing, the concentration of dopant ion, time for precipitation and calcinations, with suitable capping agent. Thus, formed phosphors can be tested towards their homogenous crystallinity, high distribution and good luminescence intensity with required color- tuneable properties.

The characterization of phosphor material towards its crystalline phase can be done by X-ray analysis. The morphology and crystal size of samples can be determined by SEM, technique. The presence of desired constituents/ groups in as-synthesized product is determined by FTIR spectra. The photoluminescence characterization will be done by PL spectra at room temperature. Thus, formed phosphors shows wide ranged applications in various fields.

METHODOLOGY

The phosphor Y$_2$O$_3$:Gd$^{3+}$ are prepared by homogenous coprecipitation method (HCP) in aqueous medium offers comparatively low temperature route, higher crystal controllability. The starting materials, Yttrium Nitrate hexahydrate [Y(NO$_3$)$_3$.6H$_2$O], Sigma-Aldrich (99.9%) and Gadolinium Nitrate hexahydrate [Gd (NO$_3$)$_3$.6H$_2$O] Alfa-Aesar (99.9%) were purchased. Other chemicals, oxalic acid (AR grade) used, as precipitant and CTAB (Cetyl Trimethyl Ammonium Bromide) as a capping agent/surfactant were purchased from Loba Chemie Co. Ltd. The aqueous solution of mixed metal nitrate precursor was prepared in 25 ml DI water by taking [Y(NO$_3$)$_3$.6H$_2$O] and [Gd (NO$_3$)$_3$.6H$_2$O] in specific mole ratio represented as [1-x. Y (NO$_3$)$_3$.6H$_2$O + x. Gd (NO$_3$)$_3$.6H$_2$O]. Thus, prepared solution was kept on magnetic stirrer for one hour at 40°C. Afterwards, the aqueous solution of oxalic acid (precipitant) was added drop wise (4 drops per min.) in above prepared aqueous solution of mixed metal nitrate precursor in presence of 1-2 ml CTAB (capping agent) for 2 hours, with constant stirring on magnetic stirrer keeping temperature at about 40°C and pH about 6.5 to 7.0. After complete addition of oxalic acid, thus formed precipitate was removed by filtration, dried under IR lamp. After step wise sintering and calcinations at about 800°C, for 4 hour we get stochiometric white powdered phosphor material. For synthesis of Y$_2$O$_3$: Gd $^{3+}$ phosphor material CTAB is used as capping agent/surfactant which control the particle size, avoids agglomeration effectively by forming reverse micelle. It reduces the oxygen bridge bonds between the particles.

The characterization of phosphor material was done by X-ray analysis, SEM, FTIR techniques. The photoluminescence characterization was done by PL spectra at room temperature. The step wise chemical reactions are represented in stepwise manner as follows:

\[
2 \text{Y(NO}_3)_3.6\text{H}_2\text{O + 2 Gd(NO}_3)_3.6\text{H}_2\text{O + 3 H}_2\text{C}_2\text{O}_4.2\text{H}_2\text{O} \quad \text{Precipitation as Oxalate}
\]

\[
\downarrow \quad \text{In presence of CTAB (capping agent)}
\]

\[
(Y \text{Gd})_2(\text{C}_2\text{O}_4)_3.12\text{H}_2\text{O}+6\text{HNO}_3+2\text{H}_2\text{O} \quad \text{(I)}
\]

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\[(\text{Y Gd})_2(\text{C}_2\text{O}_4)_3\cdot 12\text{H}_2\text{O} \rightarrow (\text{Y Gd})_2(\text{C}_2\text{O}_4)_3 + 12\text{H}_2\text{O}\] 
\[\text{(2)}\]

\[(\text{Y Gd})_2(\text{C}_2\text{O}_4)_3 \rightarrow (\text{Y Gd})_2(\text{CO}_3)_3 + 3\text{CO}\] 
\[\text{---- (3)}\]

\[(\text{Y Gd})_2(\text{CO}_3)_3 \rightarrow (\text{Y Gd})_2\text{O}_2\text{CO}_3 + 2\text{CO}_2 \] 
\[\text{--------(4)}\]

\[(\text{Y Gd})_2\text{O}_2\text{CO}_3 \rightarrow \text{Y}_2\text{O}_3 : \text{Gd}^{3+} + \text{CO}_2 \] 
\[\text{-----------(5)}\]

RESULTS AND DISCUSSION

XRD analysis:
The crystalline phase formation in products, \((\text{Y}_{1-x}\text{Gd}_x)_2\text{O}_3\) prepared by HCP method was confirmed by X-ray diffraction pattern shown in Fig.1. It agrees with the standard JCPD data. The peaks at 20.16, 29.42, 33.14, 36.24, 40.17, 43.23, 48.34 and 57.47 corresponding to (211), (222), (400), (411), (332), (134), (440) and (622) respectively, show exact matching with JCPD data. The space group of \(\text{Y}_2\text{O}_3\) is \((\text{I a}3)\). Also the XRD pattern confirms single crystalline phase of product having cubic structure with comparable cell parameters (\(a/\text{nm}\)) as that of \(\text{Y}_2\text{O}_3\). It indicates the substitution of \(\text{Gd}^{3+}\) in cation sites in host matrix of \(\text{Y}_2\text{O}_3\) internally.

Fig.1: XRD patterns of \((\text{Y}_{1-x}\text{Gd}_x)_2\text{O}_3\) prepared by HCP method with variable concentration of dopant (\(x= 0.05\) to 0.5 %)

SEM analysis:
The SEM image of \(\text{Y}_2\text{O}_3 : \text{Gd}^{3+}\) (\(x= 0.2\) %) crystal is shown in Fig.2. As can be seen, \(\text{Y}_2\text{O}_3: \text{Gd}^{3+}\) particles synthesized shows uniform shape and agglomerates of little spheres.

Fig.2: SEM image of \(\text{Y}_2\text{O}_3 : \text{Gd}^{3+}\) (\(x= 0.2\) %) phosphor prepared by HCP method.

FTIR spectra analysis:
The FTIR spectra of \(\text{Y}_2\text{O}_3 : \text{Gd}^{3+}\) (\(x= 0.2\) %) crystal is shown in Fig.3. It shows different peaks from 4000 - 600 cm\(^{-1}\) confirms presence of all constituents in prepared phosphor.

Fig.3: FTIR spectra of \(\text{Y}_2\text{O}_3 : \text{Gd}^{3+}\) (\(x= 0.2\) %) phosphor prepared by HCP method.

Luminescence properties:
The emission spectra of \(\text{Y}_2\text{O}_3 : \text{Gd}^{3+}\) (\(x= 0.2\) %) phosphor prepared by HCP method is shown in Fig.4. This spectrum is taken at room temperature using 220 nm Xenon lamps. It shows sharp emission line corresponding to \(5\text{D}_0 \rightarrow 7\text{F}_2\) at 610 nm for \(\text{Gd}^{3+}\) dopant ion. The other emission lines corresponding to \(5\text{D}_0 \rightarrow 7\text{F}_J\) (\(J= 0, 1, 3, 4\)) are also observed at 580, 590, 630, 660 nm are also observed. This observation confirms greater luminescence property of phosphor due to doping of \(\text{Gd}^{3+}\) of said concentration.
CONCLUSION

The cubic crystalline phosphor $\text{Y}_2\text{O}_3 : \text{Gd}^{3+}$ was successfully prepared by HCP method. The results of XRD, SEM and FTIR investigation shows doping of Gd $^{3+}$ in host matrix of $\text{Y}_2\text{O}_3$As compared with other methods phosphor prepared by HCP method shows comparable luminescent properties with less concentration of dopant ion. It is an efficient way to save the cost of the phosphor while luminescent properties are retained. Thus prepared $\text{Y}_2\text{O}_3 : \text{Gd}^{3+}$ color emitting phosphor used for display devices and lamp manufacturing.

Conflicts of interest: The authors stated that no conflicts of interest.

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