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Production and Characterization of Magnetic and Luminescent $Gd_2O_2SO_4:Eu^{3+}$ Nanoparticles by Sol-gel Technique

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Abstract. Radiographic imaging which transforms y or x rays into visible light is one of the most important developments in the field of digital imaging systems in recent years. Materials consisting of rare earth elements have strong scintillation properties in digital radiology. $Gd_2O_2SO_4$ is studied due to having effective magnetic properties and excellent luminescence spectra. Additionally, $Gd_2O_2SO_4:Eu^{3+}$ has thermal stability and ideal red light emission performance with peak emission at around 618 nm when activated under X-ray or ultraviolet light. In this study, $Gd_2O_2SO_4:Eu^{3+}$ nanoparticles were synthesized by sol-gel method and were calcined at 950 ^oC in argon gas atmosphere. In the analysis of the produced nanoparticles XRD, SEM, XPS and optical luminescence devices were used to evaluate the results.

Keywords: Radiographic imaging; $Gd_2O_2SO_4$ nanoparticles; Sol-gel.
PACS: 78.55.Kz, 81.05.Bx, 81.20.Fz.

INTRODUCTION

Digital radiographic imaging is one of the most important developments in the field of imaging application in recent years [1, 3]. Studies on this subject are still continuing. Phosphors transforming y or x rays into visible light are used in radiographic imaging systems [4, 6]. Gadolinium oxysulfide (GOS) formed with the combination of gadolinium, oxygen and sulphide is one of the most efficient phosphorescence materials [7, 9]. $Gd_2O_2SO_4$ nanoparticles indicate very good luminescence behavior and unique magnetic properties at low temperature [10]. In studies on this material; $Gd_2O_2SO_4$: Eu^{3+} (GOS: Eu^{3+}) material was produced by hydrothermal synthesis method which had good thermal stability and showed red luminescence emission performance under X-rays [11-12]. Eu^{3+} dopant provides high packing density and low light scattering [13]. In another study, $(Gd_{1-x},Eu_x)_2O_2SO_4$ structure was synthesized by homogeneous precipitation method and the strong emission peak for GOS:Eu^{3+} was 618 nm under 270 nm light excitation [14]. Further, GOS structure has been studied in combination Gd_2O_2S and different synthesis methods [15]. $(Gd_{1-x}, Pr_x)_2O_2S$ nano-phosphors were synthesized using a novel co-precipitation method and GOS structure showed a green emission at 511 nm as the most prominent peak under 301 nm excitation. Also the optimal x value is 0.01 for the strongest luminescent emission under excitation light [16].

In this study, $Gd_2O_2SO_4:Eu^{3+}$ nanoparticles were produced by sol gel technique which was not previously applied before and the characteristic properties of this material were investigated via XRD, XPS, SEM and PL Spectra device.

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EXPERIMENTAL DETAILS

Europium doped Gadolinium oxysulfide ($Gd_2O_2SO_4:Eu^{3+}$ = GOS: Eu^{3+}) nanoparticles were synthesized using the sol-gel technique. Gadolinium (III) Acetate Hydrate ($Gd(CH_3CO_2).xH_2O$), Urea ($CO(NH_2)_2$), Ammonium sulfate ($(NH_4)_2SO_4$) and Europium (III) nitrate pentahydrate ($Eu(NO_3)_3 \cdot 5H_2O$) were the starting chemical materials. The amount of Eu dopant was %0.01 mole. Firstly, after the starting chemicals were weighed stoichiometrically, the solution was prepared in pure water and then the solution was stirred until it became transparent in an ultrasonic mixer at 80°C. The sol-gel is a unique method of producing various particles of gel, polymeric crystalline and glassy phases from liquid forms. If the starting point in the sol form is achieved and characterized carefully, the final gel form will be successful. At this point, various tests were applied for the characterization of the synthesized solution. A blur test of solution was implemented by standard VELP TB1 Model turbidity meter. The pH value was measured via the standard pH meter with the help of the Mettler Toledo Electrode. After the gel form was obtained the drying process was realized at 200°C. The dried gel was annealed at 950°C and GOS: Eu^{3+} nanoparticles were produced.

The detection of the valid phase of the GOS: Eu^{3+} particles annealed at 950°C were analyzed by Thermo Scientific Cu-Kα model X-ray diffraction (XRD). Furthermore, the surface composition of particles was analyzed via Thermo-Scientific Al-Kα model X-ray Photoelectron Spectroscopy (XPS). Microstructure of GOS: Eu^{3+} was obtained using Scanning Electron Microscope (SEM) and the excitation and emission peaks of GOS: Eu^{3+} particles were measured using the photoluminescence spectrum device.

RESULTS and DISCUSSION

Fig. 1 shows the XRD pattern of the $Gd_2O_2SO_4:Eu^{3+}$ particles annealed at 950°C. Shown in Fig. 1, it is clear that all of these peaks can be indexed to $Gd_2O_2SO_4$ according to JCPDS Card no.29-0613. When the produced particles annealed at 950 °C, well defined diffraction peaks appeared and the lattice parameters (a=4.04 Å, b= 4.17 Å, c= 12.97 Å) could be indexed to the orthorhombic phase of $Gd_2O_2SO_4$ matched with the standard of the literature values. It can be understood that doping of Eu^{3+} ions in the $Gd_2O_2SO_4$ host lattice has no influence on the crystal structure due to similar radius ($r_{(Gd3+)}$=0.0938 nm, $r_{(Eu3+)}$=0.095 nm) [17].

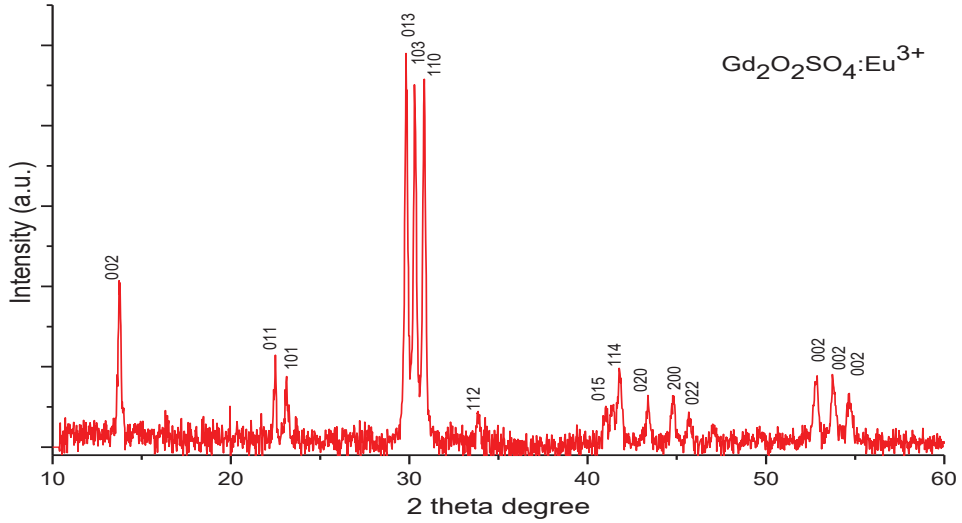


Figure 1. XRD pattern of the $Gd_2O_2SO_4:Eu^{3+}$ particles annealed at 950°C.

The surface composition of $Gd_2O_2SO_4:Eu^{3+}$ nanophosphor was further analyzed by XPS measurements. As demonstrated in Fig. 2, the elements of Gd, O, S, C and Eu can be detected. The carbon on the surface of $Gd_2O_2SO_4:Eu^{3+}$ nanophosphor is ascribed to the adsorbed CO_2 from the ambient environment and the adventitious hydrocarbon from the XPS instrument itself [18].

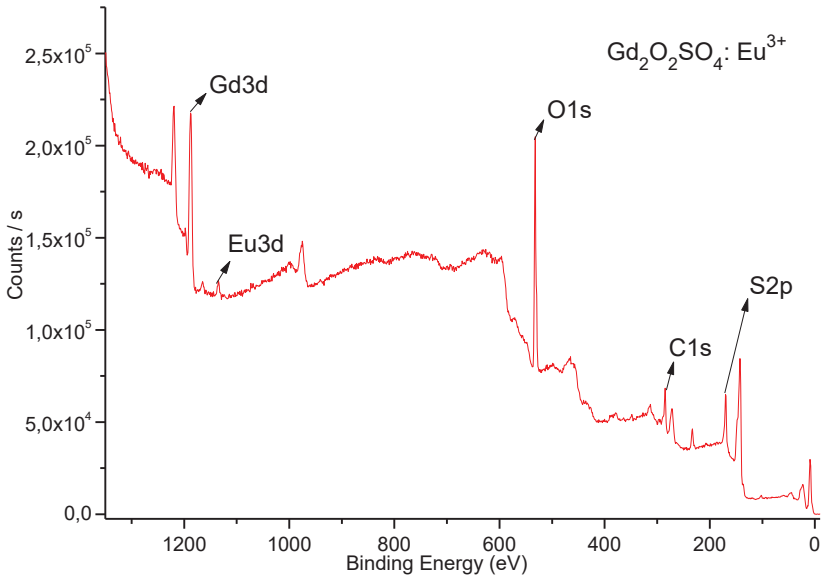


Figure 2. XPS pattern of the $Gd_2O_2SO_4:Eu^{3+}$ particles annealed at 950°C.

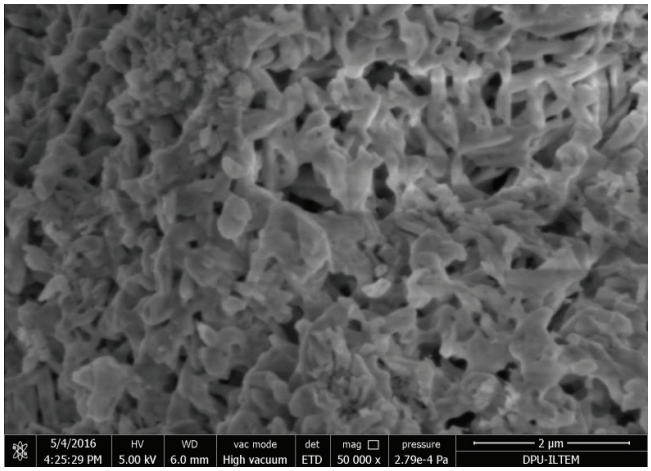


Figure 3. SEM image of GOS: Eu^{3+} nanoparticles (20x).

Fig. 3 indicates the SEM image of the Gd2O2SO4:Eu^{3+} nanoparticles. The particles are seen to be holding each other and be agglomerated when the structure is analyzed. It can be seen that the $Gd_2O_2SO_4$: Eu^{3+} nanoparticles are cactus needle-like in shape, dispersed and about 100–300nm in crystallite size, consistent with the result calculated from XRD patterns. The high magnification SEM image exhibits the formation of needle shaped particle with sharp headed tips.

Figs. 4a and b show the excitation and emission spectra of the $Gd_2O_2SO_4$:Eu^{3+} phosphor. The excitation spectra of $Gd_2O_2SO_4$:Eu^{3+} phosphor investigated at 612 nm emission were measured in the range of 340-440 nm as is shown in Fig. 4 a. The peaks near 365, 380, and 390 nm can be assigned to the higher energy level (f-f) transitions of Eu^{3+} [19].

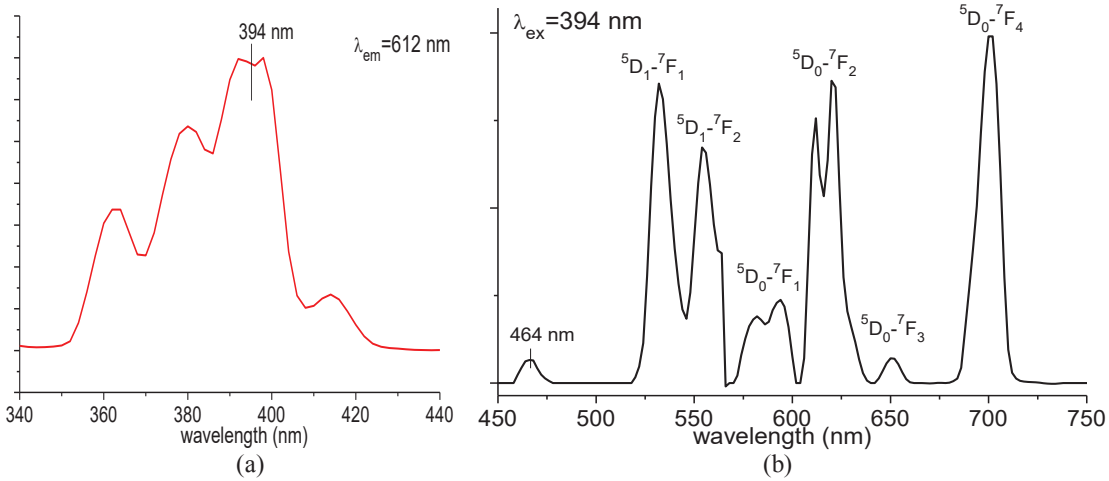


Figure 4. a) Excitation spectra of the GOS:Eu^{3+}; b) Emission spectra of the GOS:Eu^{3+} .

Fig.4.b. gives the emission spectrum of $Gd_2O_2SO_4$: Eu^{3+} excited at 320 nm consists of sharp lines ranging from 450 to 750 nm which are associated with the transitions from the excited level of $^5D_{0,1}$ to the levels of ^7F_J (J = 1, 2, 3and 4) of Eu^{3+}. Among several luminescence transitions of Eu^{3+}, the $^5D_0$-$^7F_1$ (~590 nm) emission transition is mainly magnetically allowed (a magnetic dipole transition). It is structure independent while $^5D_0$-$^7F_2$ (~620 nm) and $^5D_0$-$^7F_4$ (~700 nm) are hypersensitive forced and weak electric-dipole transitions, respectively, being allowed only at low symmetries with no inversion center.

These transitions are known to be hypersensitive to crystal-structure and chemical surroundings [20].

CONCLUSION

The $Gd_2O_2SO_4:Eu^{3+}$ phosphor particles with good scintillation characteristics were successfully produced by sol-gel method. XRD, XPS, SEM and luminescence characterization of the produced phosphor powders were completed and discussed with similar structures in the literature have been evaluated successfully. Luminescence spectra of $Gd_2O_2SO_4:Eu^{3+}$ phosphor particles under UV excitation show an emission at 612 nm as the most prominent peak. This study aims to develop GOS scintillators which are used in technological devices with regard to effect of the additive elements and the suitable production techniques.

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