Effect of Annealing on the Photoluminescence Intensity of Gehlenite:Eu Doped Phosphor Prepared in Different Gas Atmospheres

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Abstract. Europium doped gehlenite phosphor glassy powders were successfully prepared by spray pyrolysis followed by annealing under air, N_2 and N_2/H_2 treated gases. In this paper, it is of great attention and importance to find that the reductions of Eu^{3+} to Eu^{2+} ions can be realized. To identify the oxidations of europium ions, the X ray photo spectroscopy (XPS) was used. The photoluminescence (PL) properties intensively studied under different excitation in which the result shows that the europium doped phosphor gehlenite glassy powders emit a strong red light in air and N_2 treated gases; while under N_2/H_2 treated gas, blue light was observed.

Keywords: Gehlenite · Photoluminescence · Air · N_2 and N_2/H_2 · Eu^{2+}/Eu^{3+} · Spray pyrolysis

1 Introduction

It is known that aluminosilicates have excellent chemical and thermal stability, good corrosion resistance and low cost, which can be used in different fields of applications such as, in the areas of sensors, security labels, compact fluorescent lamps (CFLs), and field emission displays (EDs), white light emitting diodes (WLEDs) and excellent phosphor hosts [1–7]. Recently among, the aluminosilicate group amorphous gehlenite materials have superior photoluminescence properties when doped with rare earth metal ions [8].

Rare-earth ions doped gehlenite ($Ca_2Al_2SiO_7$) have been widely investigated for the last few decades [9]. For example, Er^{3+} and Nd^{3+} ions doped $Ca_2Al_2SiO_7$ have large absorption bands which enhance, for the fabrications of laser typed pumped materials. Zhang et al. have reported $Ca_2Al_2SiO_7$:Eu^{3+} as a potential red phosphor used in WLEDs [10]. Eu^{3+} is commonly, used in luminescence studies due to its intra

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transition electrons and the longlife times of the excited states. Most, red phosphors activated by Eu^{3+} ion were extensively reported, for example, $LiEu(PO_3)_4$ [11] and $Na_5Eu(WO_4)$ [12]. These phosphors can be effectively excited by the ultravio-let LED along with orange/red emission originating from the $^5D_0$ ^7F_J (J = 0, 1, 2, 3, 4) [13]. In addition, Eu^{2+} ions are more sensitive to the ligand field and luminescent color strongly depend on the host lattices in which the emission colors change, for example from blue to yellow (and even red). Its emission wavelength strongly depends on the nature of the host lattice due to the participations of d orbitals ($4f^65d^1 \rightarrow 4f^7$) [6]. In the previous report, Eu^{2+} ions are mainly obtained at eleveated temperature solid state reaction under specific atmospheres such as reducing atmosphere (N_2/H_2, H_2 and CO) [14, 15]. Haoyi et al. reported that Eu^{2+} ions doped $Ca_2Al_2SiO_7$ are prepared by solid state reaction method in which a blue-green emission were observed [16].

Several methods have been used for the synthesizes of phosphors materials. Among these methods: solid state reaction [17], sol-gel [1] and spray pyrolysis methods have been used. Solid- state reaction is a conventional method, but has some demerits such as, high heating temperature, low chemical uniformity, and large average particle size [18]. To improve these drawback, sol-gel methods were suggested, which resulted fine particle sizes and low preparation temperatures. However, they also have limitations such as, difficulties in powder morphology controlling and huge energy loss for the removal of residue during calcination process [19]. Thus, problems may reduce the enhancement of luminescent properties of phosphor particles. To overcome those limitation spray pyrolysis methods were used [20].

Particles prepared using spray pyrolysis method are chemical uniform in size and composition, spherical morphology and non-agglomeration, because of the microscale reactions that proceed inside a droplet [21, 22]. In additions, spray pyrolysis has many advantages such as its low annealing temperature, cost effective, high purity, fast process and continuous processes [23–25]. Shih et al. reported that, the gehlenite doped europium was prepared by spray pyrolysis methods were successfully prepared in which red emissions was obtained [8]. In this work we propose a single phosphor material to produce more than one color emissions in different atmospheric condition.

In this paper, a novel gehlenite: Eu glassy powders phosphors was systematically, studied under air, N_2 and N_2/H_2 treated gases. Additionally, for the determination of the atomic composition the oxidation state of europium ions was estimates, from peak area. We believe this work will greatly promote the development and application of the europium doped gehlenite phosphors.

2 Experimental Procedures

2.1 Preparation of Gehlenite: Eu Doped Glassy Powders

For the preparation of gehlenite: Eu doped glassy powder, aluminum nitrateenneahydrate (AlN), (AlN) (Al $(NO_3)_3.9H_2O$, 99%, Alfa Aesar), calcium nitrate tetrahydrate (CaN) (Ca $(NO_3)_2.4H_2O$, 99%, Alfa Aesar, Heysham, UK), tetraethylorthosilicate (TEOS) ($C_8H_{20}O_4Si$, 98%, Acros, Pittsburgh, PA) and europium oxide (Eu_2O_3, 99%, Alfa Aesar) were used. All chemicals were mixed and stirred at room temperature for

24 h with DI water. Then, the precursor, of the mixture was atomized by ultrasonic to form a droplet. The small droplets were supplied to the three different region temperatures, i.e., evaporation, calcination, and cooling at 250, 1000, and 350 °C, respectively. The phosphor particles produced were collected inside the metal cylinder wall. Finally, the collected powders prepared were treated at 800 °C for 1 h inside a tubular furnace under an air, N_2 and N_2/H_2 gases with the heating and cooling rate of 5 °C/min.

2.2 Characterization

The phase compositions of gehlenite: Eu phosphor particles prepared under air, N_2 and N_2/H_2 treated gases were examined, using XRD (D2 Phaser, Bruker, Karlsruhe, Germany), with Cu-Kα radiation, has used to obtain the XRD patterns with the range from 20° to 80° and with the scanning rate of 6°/ min. The surface morphology of gehlenite: Eu phosphors particles prepared under air, N_2 and N_2/H_2 treated gases were examined using scanning electron microscope (SEM, JSM-6500F, JEOL, Tokyo, Japan). Photo luminescent properties were determined using Xe-ramp as the excitation source. All the luminescence characterization of the phosphors was carried out at room temperature using a fluorescence spectrometer (FP-8500, JASCO, Tokyo, Japan). A Xenon lamp of 150 W was used as the excitation light source to record the spectra of gehlenite: Eu doped powders at a wavelength of 254 and 394 nm. The surface compositions of europium ions, oxidation state and the binding energies of the 3d core levels were determined by X-ray photoelectron spectroscopy (XPS, Perkin-Elmer PHI 5600, and Waltham, MA, USA). All measurements were carried out at the room temperature.

3 Results and Discussion

Figure 1 shows the phase composition of the gehlenite: Eu phosphor glassy powders treated in air, N_2 and N_2/H_2 gases. The XRD spectra showed that all powders have a broad band between 23° and 38°, indicating the absence of the crystalline phase for a glassy structure. In short, the gehlenite: Eu phosphor glassy powders treated in air, N_2 and N_2/H_2 gases treated exhibited the amorphous phase. Furthermore, the dopant didn't influence the structure of the prepared specimen.

The surface morphologies of the all powders were observed with FE-SEM, as shown in Fig. 2(a)–(c). As seen from Fig. 2, the image seems like similar, spherical and smooth shape in air, N_2 and N_2/H_2 treated gases, which was the morphology, does not influenced under thus gases treatment. Based on statistical measurement, the average particle sizes prepared phosphors are calculated from 300 particles were, 1.39 ± 0.52 μm, 1.32 ± 0.72 μm and 1.25 ± 0.5 μm in air, N_2 and N_2/H_2 treated gases respectively. In addition, the spherical and smooth, morphology which is considered to be of benefit in enhancing Photoluminescence properties [22].

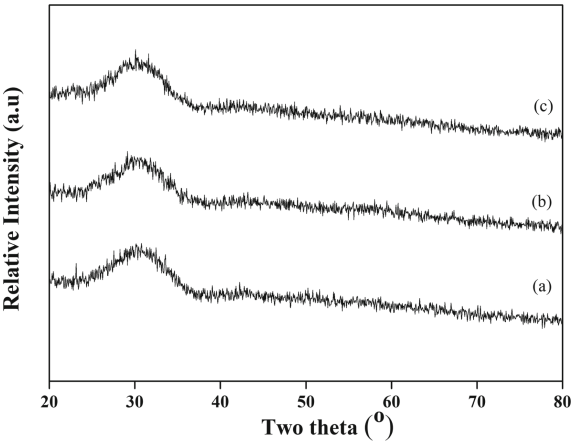

Fig. 1. XRD patterns of gehlenite: Eu phosphor glassy powders treated in (a) air, (b) N_2 and (c) N_2/H_2-gases treated.

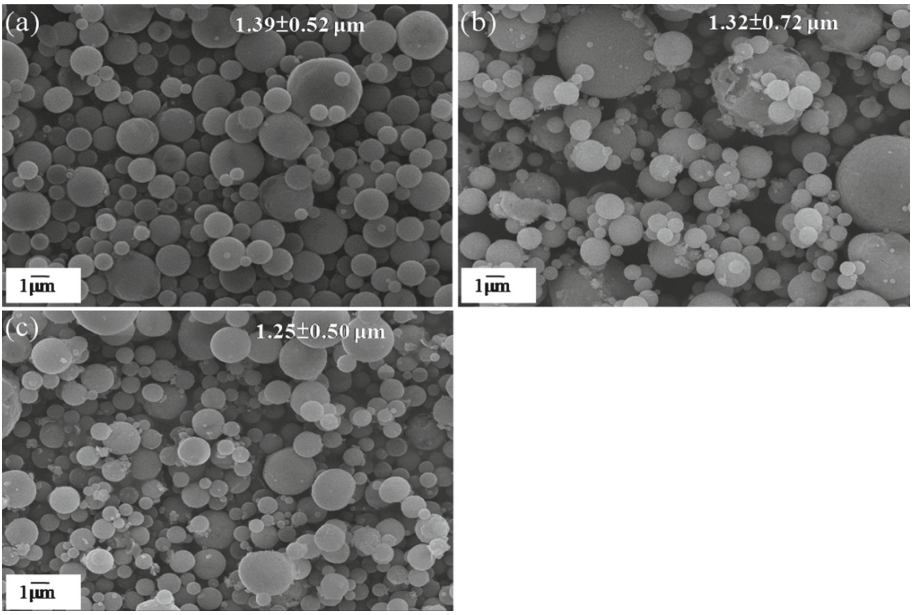

Fig. 2. SEM images of gehlenite: Eu phosphor glassy powders treated in (a) air, (b) N_2 and (c) N_2/H_2 gases treated.

To confirm the presence of Eu^{2+} and Eu^{3+} ions, all prepared powders were analysis by XPS techniques. X-ray photoelectron spectra (XPS), results were shown in Fig. 3 There were four photoelectron emission signals at the binding energy range between 1120 and 1180 eV in the XPS spectrum for gehlenite: Eu phosphor particles prepared under air, N_2 and N_2/H_2 treated gases in Fig. 3. From Fig. 3(a) two broad band peaking were observed at 1124.23 eV and 1133.79 eV, which is ascribed to Eu^{2+} $3d_{5/2}$ and Eu^{3+} $3d_{5/2}$, respectively. The other two band peaking were observed at 1153.94 eV and 1163.70 eV, which belongs to Eu^{2+} $3d_{3/2}$ and Eu^{3+} $3d_{3/2}$, respectively. In addition, in Fig. 3(b) there are four photoelectron emissions signals were observed at binding energy of 1124.44, 1133.82, 1154.09 and 1163.76 eV which attributed to Eu^{2+} $3d_{5/2}$, Eu^{3+} $3d_{5/2}$, Eu^{2+} $3d_{3/2}$ and Eu^{3+} $3d_{3/2}$ respectively. Lastly, in Fig. 3(c) four photoelectron emissions were observed at the binding energy of 1124.23, 1133.87, 1153.77 and 1163.55eV belongs to Eu^{2+} $3d_{5/2}$, Eu^{3+} $3d_{5/2}$, Eu^{2+} $3d_{3/2}$ and Eu^{3+} $3d_{3/2}$ respectively. The binding energy difference between the trivalent and divalent states is about $\sim$9.1eV, in which similar discussions were given for the Eu ion in $KBaPO_4$: Eu phosphor as well [26].

The emission spectra of Eu ions doped gehlenite glassy powder treated are shown in Fig. 4(a) (λ_{ex} = 254 nm) and (b) (λ_{ex} = 394 nm) under air, N_2 and N_2/H_2 gases treated.

The emission spectrum of the gehlenite: Eu phosphor glassy powder from Fig. 4(a) were measured by adjusting the excitation at 254 nm, which exhibits a broadband at 400 nm and it attributed to $4f^65d \rightarrow 4f^7$ transitions of Eu^{2+}. Up on 254 nm UV excitation, the gehlenite: Eu^{2+} phosphor shows a strong blue emission band with a peak between 360–470 nm, which is attributed to the 5d-4f allowed transition of Eu^{2+} ions. Furthermore, for transitions of Eu^{3+} ions there is the existence of narrower peaks between 550 and 750 nm due to the $^5D_0 \rightarrow {}^7F_J$ (J = 1–4) transitions. From Fig. 4(b), there are two main emission peaks at 613 nm and 592 nm which is assigned to the induced electric dipole (ED) transition $^5D_0 \rightarrow {}^7F_2$ and magnetic dipole (MD) transition $^5D_0 \rightarrow {}^7F_1$ of Eu^{3+}. The magnetic dipole transition appears due to both centrosymmetric and non centrosymmetric lattice centres, whereas the induced electric dipole transition appears when Eu^{3+} ions occupy non-centrosymmetric lattice centres. The relative intensity ratio of the $^5D_0 \rightarrow {}^7F_1$ and $^5D_0 \rightarrow {}^7F_2$ transitions strongly depends on the local symmetry of the Eu^{3+} ions [27].

As mentioned in Table 1 the atomic composition of sample prepared under air, N_2 and N_2/H_2 treated gases were calculated from the XPS analysis for both Eu^{2+} and Eu^{3+} ions. Atomic composition of Eu^{2+} in percentage under air, N_2 and N_2/H_2 treated was 56.33%, 50.65% and 61.67% respectively. Comparatively, N_2/H_2 treated Eu-doped gehlenite glassy powders were 61.67% (Eu^{2+}) in which the highest intensity of blue emitted color was obtained at excitation of 254 nm compare to air and N_2 treated gases. However, in air and N_2 treated Eu-doped gehlenite glassy powders were 56.33% and 50.65% respectively. The photoluminescence intensity recorded for air and N_2 treated gases at the excitation of 254 nm was low which suppressed their emissions due to

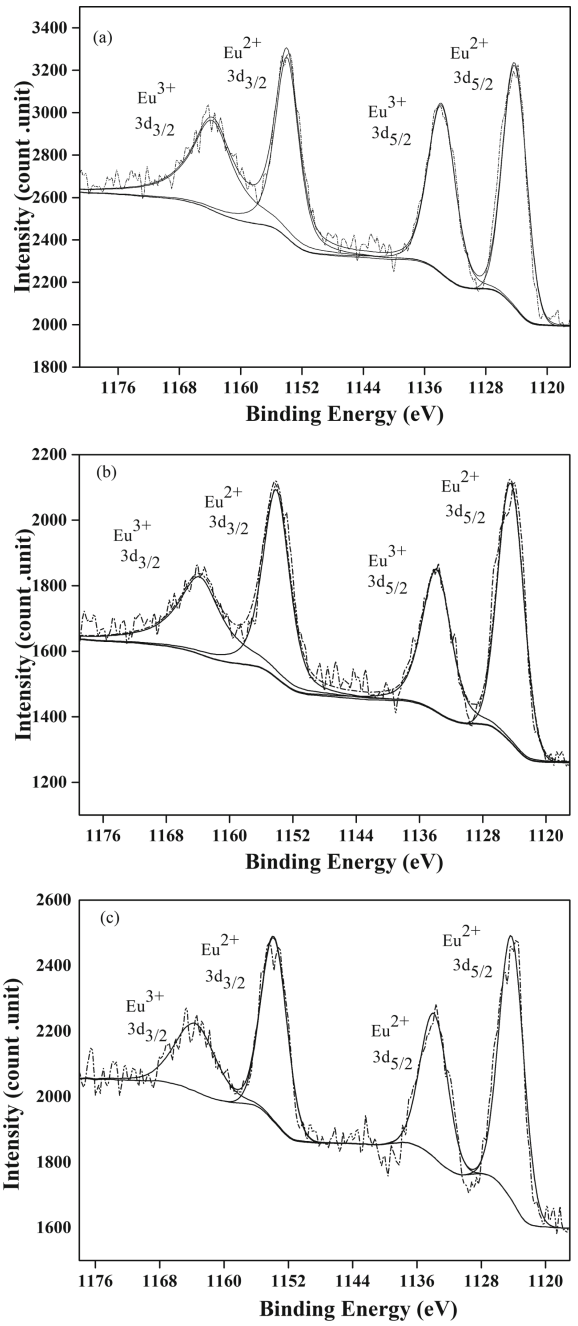

Fig. 3. XPS spectra of the Eu 3d peaks of gehlenite: Eu phosphor glassy powders treated in (a) air, (b) N_2 and (c) N_2/H_2-gases treated. Solid line represents the experimental spectrum (after background subtraction) and the dotted lines are the results with curve fitting.

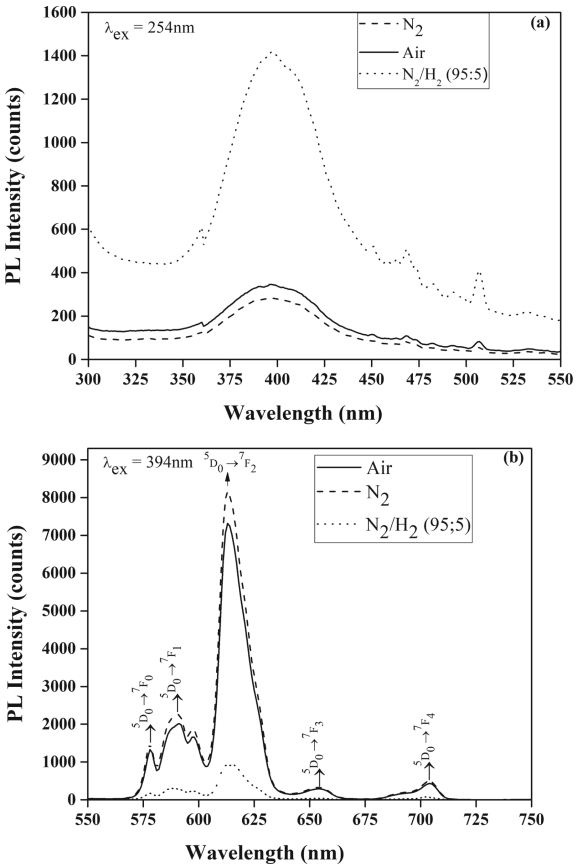

Fig. 4. PL spectra of gehlenite: Eu phosphor glassy powder (a) (λ_{ex} = 254 nm) and (b) (λ_{ex} = 394 nm) treated in air, N_2 and N_2/H_2 gases treated.

reduction of Eu^{3+} to Eu^{2+} ions, whereas at the excitations of 394 nm the highest intensity of red emission was obtained. From this analysis the N_2/H_2 treated were the appropriate for blue emissions in which it favored to Eu^{2+} ion but under air and N_2 treated shows better red emissions. Generally, two colors from a single phosphor material were successfully obtained under air, N_2 and N_2/H_2 treated gases.

Figure 5 shows the ratio of the integrated emission intensity caused by the ($^5D_0$ $^7F_2$) transition to the ($^5D_0 \rightarrow {}^7F_1$) transition of Eu^{3+} ions, i.e., ($^5D_0 \rightarrow {}^7F_2$) / ($^5D_0$ $^7F_1$). As we seen from Fig. 5 the intensity ratios of N_2 treated gases where higher than in air and N_2/H_2 treated gases. The values of intensity ratios are 3.64, 3.69 and 3.16 in air, N_2 and N_2/H_2 treated gases respectively.

Table 1. Compositions of Eu^{2+} and Eu^{3+} ions from XPS data of Eu-doped gehlenite glassy powders treated in air, N_2 and N_2/H_2 gases treated.

Atmospheric gases	Peak area						Atomic composition (%)	
	Eu^{3+} $3d_{5/2}$	Eu^{2+} $3d_{5/2}$	Eu^{3+} $3d_{3/2}$	Eu^{2+} $3d_{3/2}$	$\sum Eu^{3+} + Eu^{3+}$ $3d_{5/2}$ $3d_{3/2}$	$\sum Eu^{2+} + Eu^{2+}$ $3d_{5/2}$ $3d_{3/2}$	Eu^{2+}	Eu^{3+}
N_2	3944.4	4333.1	4263.9	4097.2	8208.3	8430.3	50.7	49.4
Air	2393.3	3256.9	2419.1	2952.4	4812.4	6209.3	56.3	47.6
N_2/H_2	1879.7	3097.9	1458.8	2271.4	3338.5	5369.3	61.6	38.3

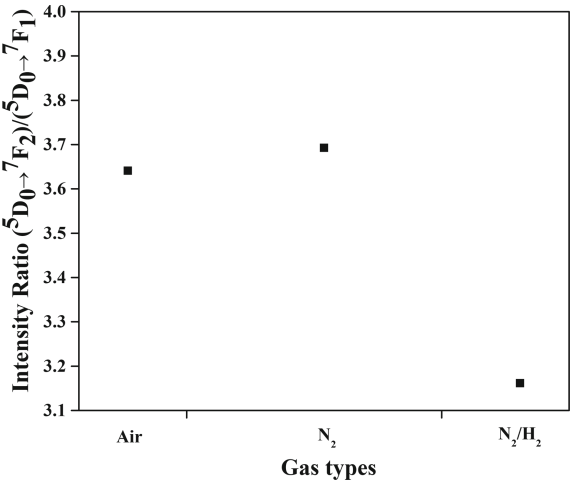

Fig. 5. PL Intensity Ratio ($^5D_0 \rightarrow {}^7F_2$) / ($^5D_0 \rightarrow {}^7F_1$) of gehlenite: Eu phosphor glassy powder treated in air, N_2 and N_2/H_2 gases treated.

The significance of ($^5D_0 \rightarrow {}^7F_2$) / ($^5D_0 \rightarrow {}^7F_1$) offers how far the Eu^{3+} ion deviates from the Centro symmetry, i.e., a measure of the site symmetry of Eu^{3+} ion. The intensity ratio of N_2 treated where greater, than air and N_2/H_2 treated. This larger in intensity ratio which results better color purity for red emissions [28]. The ratio was relatively the similar in air and N_2 treated, but at N_2/H_2 treated lower intensity ratio. Thus, the environment around the Eu^{3+} ions was changed under different atmospheric conditions. In other words, the asymmetric ratio increases with decreasing the degree of distortion from the inversion symmetry of the local environment of Eu^{3+} ions [27]. This effect can be explained by the preparation method in which the sample was conducted under air and N_2 treated less reduction takes place. However, sample conducted under the N_2/H_2 treated, europium is mainly in the form of divalent in which reduction were realized compare to the air and N_2 treated.

4 Conclusions

Photoluminescence properties of europium doped gehlenite have been measured at a different excitation wave length in which the prepared sample in air treated, N_2 treated and N_2/H_2 treated were successfully studied. The atomic composition of Eu^{3+} and Eu^{2+} ions were obtained from peak area with irrespective of binding energy. The photoluminescence properties of both Eu^{3+} and Eu^{2+} ions in phosphor glassy powder shows red and blue emission were obtained at different excitation wavelength respectively, which gives a crucial hint on developing new phosphors with different rational designs in the future. The XPS results revealed that the europium (Eu^{3+}) ion doped gehlenite phosphor with N_2 treated > air treated > N_2/H_2 treated shows better red emission intensity. Whereas, for blue emissions the effect of atmospheric gases i.e., N2 treated < air treated < N_2/H_2 treated was obtained for Eu^{2+} ion doped gehlenite phosphor. The intensity ratio (R) in N_2 treated was the highest values compare to air treated and N_2/H_2 treated.

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Conflicts of Interest: The authors declare no conflict of interest.

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