Materials Research Express

PAPER

Luminescent property of La(OH)₃: Eu³⁺ nanorod and its decomposed compounds of LaOOH and La₂O₃

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Keywords: La(OH)₃: Eu³⁺ nanorod, luminescent property, decomposition, LaOOH, La₂O₃

Abstract

In this work, aiming to evaluate and compare the luminescent property of Eu³⁺ doped La(OH)₃ nanorods and its decomposed compounds of LaOOH and La₂O₃, Eu³⁺ nanorods were synthesized and the decomposition behaviors in air and hydrogen were revealed. Then the luminescent property of 0%–8% Eu³⁺ doped La(OH)₃ nanorods and the 300 °C–800 °C calcined La(OH)₃: Eu³⁺- 4% were studied. Results show that LaOOH is intermediate product during the degradation of La(OH)₃ in air or H2, which can be formed at a temperature range in 372.3 °C–592.9 °C in air. Intensity of emission spectra of La(OH)₃: Eu³⁺ nanorods increases with the rising of Eu³⁺ dopant concentration, and the emission property of Eu³⁺ doped LaOOH and La₂O₃ is varied and strengthened with that of La(OH)₃: Eu³⁺. Luminescent matrixes of LaOOH and La₂O₃ show similar emission spectrum and intensity, and the transitions of the D₀ → 7F₁ and D₀ → 7F₂ of Eu³⁺ each bifurcate into two peaks for both europium (III) doped La₂O₃ and LaOOH. The revealed results suggest that LaOOH is a relative thermal-stable compound and should be an appropriate matrix as similar as La₂O₃ for luminescent applications.

1. Introduction

Rare earth elements attract many research interests for luminescent applications, due to their unique 4f5d electronic structure and electron transitions between the f-for f-d [1], and the rare earth atoms have variety of electronic energy levels which offer abundant possible transition channels and resulting colorful emission lights [2]. Lanthanum compounds such as La₂O₃, LaOCl [3], La(OH)₃ [4, 5], etc are normally employed as matrix of phosphors. In which, La(OH)₃ owns a hexagonal phase and P63/m space group, it has a relatively stable phase and is easily produced by hydroxylation of lanthanum [5]. Hence, lanthanum hydroxide have been widely studied as light-emitting materials [6], catalysts [7], adsorbents [8, 9], up-conversion materials and bioluminescent matrixes [10–12] based on its excellent electrical, optical and magnetic properties [13].

On the other hand, europium (III) has a special electronic structure with the unfilled 4f level, showing a large atomic magnetic moment (3.4 μB), meanwhile, Eu³⁺ has the strongest emission peak located at around 611 nm which is one primary color light source [14–16]. The 4f orbital electron of La³⁺ is completely empty and has an optically inert closed shell, which is suitable to be used as the substrate for luminescent materials. The unpaired electrons in the Eu³⁺ electron shell can successively fill the 4f orbital and generate luminescence through orbital transition, which is suitable to be the activator ion of luminescent centrals [16, 17]. Eu³⁺ doped La(OH)₃ phosphors have been synthesized [4, 5, 18–21], and based on its luminescent properties studies, the Eu³⁺ doped
La(OH)$_3$ was suggested to be a potential luminescent materials for the applications e.g. luminescent bioimaging probe [4, 18].

In literatures, La(OH): Eu$^{3+}$ nanobelts [20], nanoparticles [21–23] or nanoflowers [24] have been synthesized and their luminescent property have been investigated. As it is known that luminescent property of phosphor is somewhat affected by the shape of the matrixes [25], while the luminescent properties of Eu$^{3+}$ doped La(OH)$_3$ nanorods have not been well studied yet. Moreover, it has been reported that La(OH)$_3$ will decompose into LaOOH with the rising of temperature, while LaOOH is a relative thermal stable compound and an intermediate product during the degradation of La(OH)$_3$ [3, 26]. Comparative study of the luminescent properties of La(OH)$_3$: Eu$^{3+}$ and La$_2$O$_3$: Eu$^{3+}$ had been reported [5, 24], but the emission and excitation characterization of LaOOH: Eu$^{3+}$ has not been well revealed before.

Hence, in this work, we synthesized 0–8 mol. % Eu$^{3+}$ doped La(OH)$_3$ nanorods, and investigated the dehydration behaviors of La(OH)$_3$: Eu$^{3+}$-4%, then decomposed the La(OH)$_3$: Eu$^{3+}$-4% under different temperature in air or H$_2$ at 300°C–800°C and studied the luminescent property of Eu$^{3+}$ doped La(OH)$_3$ nanorods and its degradation products of LaOOH and La$_2$O$_3$.

2. Experimental

2.1. Materials

La$_2$O$_3$ (99.99%), Eu$_2$O$_3$ (99.99%) and KOH were purchased from Aladdin Chemistry Co. Ltd (Shanghai, China), and nitric acid (65%–68%) was ordered from Jiangchuan Chemistry Co. Ltd (Chongqing, China), all chemicals were used without any further purification. H$_2$ and Ar gas with the purity of 99.999% were ordered from Shen-Jian Co., Guiyang, China.

2.2. Methods

The sample synthetic and treated process in this work is shown in figure 1. In a typical synthetic route, a certain amount of La$_2$O$_3$ was mixed with 0–8 mol.% of Eu$_2$O$_3$ and then dissolved into 16 ml nitric acid. 60 ml of 5 mol·l$^{-1}$ KOH solution was rapidly poured into the prepared solution for stirring around 15 min. The mixture was transferred into a 100 ml Teflon-lined stainless steel autoclave and reacted at 180°C for 12 h, then the white precipitate was filtered and washed for several times and followed by drying the powder at 60°C for 24 h. The obtained white powders were henceforth named as La(OH)$_3$: Eu$^{3+}$-x% (x = 0, 2, 4, 6, and 8, means the mole ratio of Eu$^{3+}$).

Then the La(OH)$_3$: Eu$^{3+}$-4% sample was chosen to be decomposed in a tube furnace in air at 300°C–800°C or in H$_2$ at 800°C for 2 h. During the calcination of sample in air, the flanges at both sides of the tube furnace were opened, while before a hydrogen calcining with the H$_2$ flow rate of 400 ml·min$^{-1}$, air in tube furnace was repeatedly expelled by alternately flowing argon and vacuuming for several times. The roasted samples were then named as La(OH)$_3$-T-A, where T means the temperature of 300, 600 or 800°C, and A represents the atmosphere of air or H$_2$.

2.3. Analysis

The synthesized and roasted samples were subjected to several analyses. X-ray diffraction (XRD) pattern was recorded by X - PertPowder with Cu Kα radiation (λ = 1.5418 Å) from 5.00° to 90.00° by a step of 0.02°/s. SEM and TEM images were taken by SIGMA-X-Max 20 (ZEISS, German) and Tecnai G2 F20 (FEI, USA) respectively. The TG-DTA data was collected by TGA/SDTA 851e (Mettler, Switzerland) in a 50 ml min$^{-1}$ air/hydrogen flow.
from room temperature to 900 °C. The excitation and emission spectra were recorded by Edinburgh Instruments FLS980 at room temperature.

3. Results and discussion

3.1. XRD patterns

The XRD patterns of synthesized La(OH)$_3$:Eu$^{3+}$-x % are presented in figure 2, it can be found that all diffraction patterns are similar with PDF #36-1481 of La(OH)$_3$, no diffraction peaks of Eu(OH)$_3$ can be observed. Meanwhile, as shown in the enlarged patterns (figure 2(b)), the peak of (101) located around 28° is shifted to a larger 2θ with the increasing concentration of dopant Eu$^{3+}$, which should be due to the replacement of La$^{3+}$ by Eu$^{3+}$ in La(OH)$_3$ lattice. Eu$^{3+}$ has a radius of 0.95 Å (CN = 6) which is smaller than that of La$^{3+}$ (r = 1.03 Å, CN = 6) [27], the La(OH)$_3$ lattice trends to shrink after doping Eu$^{3+}$ in crystal. Hence, the XRD patterns indicate that the Eu$^{3+}$ has been successfully doped in to La(OH)$_3$ lattice.

3.2. Micrograph

SEM images are shown in figure 3, it can be seen that the La(OH)$_3$:Eu$^{3+}$-x % has a well rod-shaped with the length of 73–107 nm and the diameter of 15–22 nm. With the increasing of the dopant Eu$^{3+}$, the shape of La(OH)$_3$ nanorod becomes more regular and the average length of nanorod is incremental. EDS mapping of the La(OH)$_3$:Eu$^{3+}$−4% shows that Eu$^{3+}$ has been homogenously doped into the La(OH)$_3$ nanorods.

TEM and HRTEM images of La(OH)$_3$:Eu$^{3+}$−4% are shown in figure 4, it also proves that the synthesized La(OH)$_3$ has a rod shape, where the lattice spaces are measured as 0.332 and 0.217 nm which correspond to the (101) and (210) facet of La(OH)$_3$, respectively.

3.3. Decomposition behaviors of La(OH)$_3$:Eu$^{3+}$

The TG and DSC curves of La(OH)$_3$:Eu$^{3+}$−4% in air or H$_2$ are presented in figure 5, the weightlessness before 264 °C should be the dehydration of free water, then the La(OH)$_3$ nanorods start to be decomposed at a higher temperature. Based on the literatures [3, 28], the dehydration of La(OH)$_3$ includes two steps:

\[
\text{La(OH)}_3 \rightarrow \text{LaOOH} + \text{H}_2\text{O} \quad (1)
\]

\[
2\text{LaOOH} \rightarrow \text{La}_2\text{O}_3 + \text{H}_2\text{O} \quad (2)
\]

Above 264 °C, the intermediate product of LaOOH is formed, when the temperature raised to 372.3 °C the weight of tested sample keeps as a similar value which means the LaOOH can be stably existed in the temperature range of 372.3 °C–592.9 °C in air and 372.3 °C–555.7 °C in hydrogen respectively. The result further proves that the LaOOH is thermal-stable at relative high temperature below 550 °C, and which suggests that LaOOH can be further used in the low temperature applications. The decomposition of LaOOH ends at 695.2 or 644.3 °C in air/H$_2$ atmosphere and forms La$_2$O$_3$, where the mass of sample continually decreases in hydrogen is due to the reduction of Eu$^{3+}$ to Eu$^{2+}$ by H$_2$. The slightly weightlessness of La$_2$O$_3$:Eu$^{3+}$ in air above 695.2 °C may be due to the part reduction of Eu$^{3+}$ to Eu$^{2+}$, because it was reported that Eu$^{3+}$ will be reduced to Eu$^{2+}$ at a high temperature even in air atmosphere [29, 30].
Based on the TG results, La(OH)$_3$: Eu$^{3+}$4% was roasted at 300, 600 and 800 °C in air and 800 °C in hydrogen respectively. XRD patterns of the calcined samples are shown in figure 6.

The La(OH)$_3$:300-Air has same diffraction patterns as La(OH)$_3$, which means most of La(OH)$_3$ have not been decomposed for 2 h in air at 300 °C. La(OH)$_3$:600-Air is determined as LaOOH which is in agreement with the TG data and further proves the relative thermal-stability of LaOOH. The phase turns to La$_2$O$_3$ when the calcination temperature elevated to 800 °C in air atmosphere. In addition, the phase of La(OH)$_3$:800-H$_2$ is also found as La$_2$O$_3$, but the peaks located at around 29.1° (002) is much higher than that of La(OH)$_3$:800-Air, which may due to the tuning of the predominant growth facet of La$_2$O$_3$ caused by the change of atmosphere from air to hydrogen.

**Figure 3.** SEM images of La(OH)$_3$: Eu$^{3+}$, (a)–(e) is the micrograph of La(OH)$_3$: Eu$^{3+}$-x % respectively, and (f) is the EDS mapping of La(OH)$_3$: Eu$^{3+}$–4%.

**Figure 4.** TEM and HRTEM images of La(OH)$_3$: Eu$^{3+}$–4%.


3.4. Luminescent properties

The excitation and emission spectra of as-prepared La(OH)$_3$: Eu$^{3+}$-x % nanorods are shown in figures 7(a) and (b). It can be seen from figure 7(a) that the excitation intensity increases with the rising of Eu$^{3+}$ dopants, where the excitation peaks located at 362, 376, 395 and 415 nm are corresponding to the $^7F_0 \rightarrow ^5D_4$, $^7F_0 \rightarrow ^5G_{2,3}$, $^7F_0 \rightarrow ^7L_{6,5}$, and $^7F_0 \rightarrow ^3D_3$ characteristic f-f transitions of Eu$^{3+}$ within its 4f$^6$ configuration respectively [31–33], and the summarized energy level diagram is given in figure 7(c).

In the emission spectrum, the emission peaks are located at 592 and 616 nm which results by the transition of $^5D_0 \rightarrow ^7F_1$ and $^5D_0 \rightarrow ^7F_2$ [34, 35], which shows the red luminescent color. It is interesting to found that the emission intensity of La(OH)$_3$: Eu$^{3+}$-x % is continually enhanced with the elevation of Eu$^{3+}$ dopant.

Figure 5. TG and DSC curves of La(OH)$_3$: Eu$^{3+}$—4% in air or hydrogen.

Figure 6. XRD patterns of decomposition products.
concentration, and no concentration quenching effect has been observed even the Eu$^{3+}$ dopant concentration elevated to 8%, which suggests the La(OH)$_3$ nanorod is an appropriate matrix material for Eu$^{3+}$ based phosphor.

Comparing with the as-synthesized La(OH)$_3$: Eu$^{3+}$−x% (x = 3%), the excitation and emission spectra of roasted samples are displayed in figure 8, where significant difference can be observed from that of un-calcined La(OH)$_3$:

Figure 7. Excitation spectra of La(OH)$_3$: Eu$^{3+}$−x% (λ$_{em}$ = 616 nm) (a), emission spectra of synthesized La(OH)$_3$: Eu$^{3+}$−x% (λ$_{em}$ = 395 nm) (b), schematic energy level diagram of La(OH)$_3$: Eu$^{3+}$−x% (c)
Eu$^{3+}$−4%. The samples roasted in air have similar excitation peaks located at 363, 382, 395, 416, 466, and 535 nm, which corresponds to the $7F_0 \rightarrow 5D_{4,3}$, $7F_0 \rightarrow 5G_{2,1}$, $7F_0 \rightarrow 5L_{6,5}$, $7F_0 \rightarrow 5D_{3,2}$, and $7F_0 \rightarrow 5D_1$ of Eu$^{3+}$, respectively.

The La(OH)$_3$-600-Air is determined as LaOOH: Eu$^{3+}$−4% shows the strongest excitation spectrum. La(OH)$_3$-800-H$_2$ just has two un-conspicuous peaks located at 395 and 466 nm can be found in figure 8(a). In the emission spectra as shown in figure 8(b), La(OH)$_3$-300-Air has similar emission spectrum with the un-roasted sample but varied from that of La(OH)$_3$-600-Air or La(OH)$_3$-800-Air/H$_2$. A small difference is found as

Figure 8. Excitation spectra of roasted La(OH)$_3$:Eu$^{3+}$−4% ($\lambda_{em}$ = 616 nm) (a), emission spectra of synthesized roasted La(OH)$_3$: Eu$^{3+}$−4% ($\lambda_{ex}$ = 395 nm) (b), schematic energy level diagram of roasted La(OH)$_3$:Eu$^{3+}$−4% (c).
a new peak located at 580 nm, while a similar emission peak can be observed in that of La(OH)$_3$–600–Air or La(OH)$_3$–800–Air/H$_2$. From XRD pattern as shown in figure 6, no peaks of LaOOH can be found while TG results show that LaOOH should be formed at 300 °C. The additional emission peak found in La(OH)$_3$–300–Air indicates that part of LaOOH may be generated in La(OH)$_3$–300–Air but which cannot be detected by XRD analysis. Emission spectrum of La(OH)$_3$–300–Air located at 615 nm corresponds to the ($^7$D$_0$ → $^7$F$_2$) and 591 nm ($^7$D$_0$ → $^7$F$_1$) of Eu$^{3+}$. The emission spectra of La$_2$O$_3$ and LaOOH are similar, but it can be observed that the strongest peak is located at 625–627 nm or 613 nm for La$_2$O$_3$ and LaOOH respectively. Moreover, emission peak which located at 613–627 nm and 586–596 nm is the transition of the $^7$D$_0$ → $^7$F$_1$ and $^7$D$_0$ → $^7$F$_2$ of Eu$^{3+}$, each of them bifurcate into two peaks for both of europium (III) doped La$_2$O$_3$ and LaOOH. Đorđević and coauthors [5] also found that the red line of Eu$^{3+}$ ($^7$D$_0$ → $^7$F$_{2}$) exhibited double centered at 613 nm and 626 nm for La$_2$O$_3$ and as a single line at 616 nm for La(OH)$_3$ sample, which is in agreement with the results shown in figure 8. The bifurcation of the emission peaks are mainly related to the lattice site of the Eu$^{3+}$ in the host crystal [3, 34, 36], where the La(OH)$_3$ and La$_2$O$_3$ have a hexagonal crystal with the space group of P63/m and P-3m1 respectively, and LaOOH is reported owns a monoclinic crystal space group was P2$_1$/m (No.11) [28]. The emission spectra show major emission peaks of red color, and the LaOOH: Eu$^{3+}$ has similar and good emission property as the La$_2$O$_3$: Eu$^{3+}$.

Besides the described emission peaks above, the La(OH)$_3$–800–Air/H$_2$ samples own additional the emission peaks located at 511 nm, 539 nm and 554 nm which cannot be observed in other samples should be represented to the emission characterizations of Eu$^{2+}$. Eu$^{2+}$ was observed to be reduced to Eu$^{3+}$ in air atmosphere [29, 30], and from the TG results it can be found the Eu$^{3+}$ is reduced at the high temperature e.g. 800 °C. For the emission properties of Eu$^{3+}$, the emission peak located at 511 nm, 539 nm and 554 nm is corresponding to the transition of 4f$^5$5d$^4$–4f$^6$ of Eu$^{2+}$ [37, 38]. On the other hand, the La(OH)$_3$–800–H$_2$ owns a lower emission intensity than that of La(OH)$_3$–800–Air, which suggests that more reduced Eu$^{2+}$ ions may adverse to the luminescent property of Eu$^{3+}$ doped La$_2$O$_3$.

4. Conclusions

In summary, homogeneously Eu$^{3+}$ doped La(OH)$_3$ nanorods were synthesized by a facile hydrothermal method, and obtained LaOOH and La$_2$O$_3$ by decomposing La(OH)$_3$ at 600 or 800 °C, and found LaOOH is a relative thermal-stable compounds. The luminescent property of La(OH)$_3$: Eu$^{3+}$ nanorod is enhancing with the rising of Eu$^{3+}$ concentration from 0–8 mol. % and no concentration quenching effect has been observed. LaOOH: Eu$^{3+}$ shows the best excitation spectrum compared with La(OH)$_3$: Eu$^{3+}$ and La$_2$O$_3$: Eu$^{3+}$, and the transitions of the $^7$D$_0$ → $^7$F$_1$ and $^7$D$_0$ → $^7$F$_2$ of Eu$^{3+}$ each bifurcate into two peaks for both europium (III) doped La$_2$O$_3$ and LaOOH. Similar emission spectrum of LaOOH: Eu$^{3+}$ and La$_2$O$_3$: Eu$^{3+}$ suggests that the LaOOH: Eu$^{3+}$ is a potential luminescent matrix which may be used for similar luminescent applications as La$_2$O$_3$: Eu$^{3+}$.

Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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