Study on luminescent properties of Tb$^{3+}$ and Sm$^{3+}$ co-doped CaSiO$_3$ phosphors for white light emitting diodes

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Abstract

A series of CaSiO$_3$:x%Tb$^{3+}$, y%Sm$^{3+}$ compounds was synthesized by the conventional solid state reaction. X-ray diffraction, scanning electron microscopy, and energy dispersive x-ray spectroscopy investigations were conducted to determine crystal structure. The photoluminescence (PL) excitation spectrum of Tb$^{3+}$ and Sm$^{3+}$ doped compounds consists of a series of peaks corresponding to electronic transitions of Tb$^{3+}$ and Sm$^{3+}$ ions. The wavelength of 375 nm was optimal to excite the luminescence of Tb$^{3+}$/Sm$^{3+}$ co-doped compounds. The PL emission spectrum consists of a set of intense lines at wavelengths of 415 nm, 436 nm, 457 nm, 488 nm, 543 nm, 586 nm, and 622 nm, corresponding to Tb$^{3+}$ electronic transitions of $^5D_3 \rightarrow ^7F_4$, $^5D_3 \rightarrow ^7F_6$, $^5D_4 \rightarrow ^7F_5$, $^5D_4 \rightarrow ^7F_6$, respectively. The PL emission spectrum of the Sm$^{3+}$ doped compound has a series of three peaks located at wavelengths of 565 nm, 602 nm, and 648 nm, corresponding to the electronic transitions of Sm$^{3+}$ from $^4G_5/2$ to $^6H_{5/2}$, $^6H_{7/2}$, and $^6H_{9/2}$, respectively. The energy level diagram was proposed. The lifetime measurements were conducted for all samples and energy transfer process was also examined by this way. The Commission Internationale de l’Eclairage chromaticity coordinates, correlated color temperature, and color rendering index of phosphors were determined and the results indicate that the obtained materials are optimal candidate for white LED applications.

1. Introduction

White light-emitting diodes (WLEDs) have been extensively investigated due to great characteristics such as environment-friendly, low energy consumption, high brightness, and long lifetime [1–4]. Rare earth elements are traditionally used as activators for various hosts to create efficient active materials for phosphors in visible and near infrared range of the wavelength. Alkaline earth based phosphates [5–8], oxides [9–11], thiogallates [12, 13], and fluorides [14–17] are the best hosts for lanthanide ions with the aim to create efficient active media for WLEDs. There are two methods to produce WLEDs. The first one is to assemble blue, green, and red LEDs. The second one, which is cheaper, is to utilize a LED chip with phosphors. Commercial WLEDs are made of a GaN-based blue LED chip combined with a yellow phosphor YAG:Ce$^{3+}$. However, a low luminous efficiency, resulting from the independent degradation rate between LED chips and phosphors, and poor color rendering index (CRI) due to the lack of a red light component are still issue. Park et al produced a high luminous efficiency WLED by synthesizing a Eu$^{3+}$ doped-Sr$_2$SiO$_4$ yellow phosphor and combining it with a GaN blue LED chip. However, its CRI is lower than that of the industrially available GaN-based YAG:Ce$^{3+}$ [3]. Huang et al...
The x-ray diffraction

The phosphors CaSiO₃:Eu²⁺, Sm³⁺ activated Sr₆Mg₃Y(PO₄)₂ and Sr₆Mg₃La(PO₄)₂ yellow-emitting phosphors and obtained high CRI s of 95.375 and 91.75, respectively [18]. However, the luminous efficiency of these WLEDs was not reported.

A method for enhancing the CRI is to co-dope or tri-dope transition-metal or rare-earth activated ions in host materials. Many researches have focused on glass host materials. Lakshminarayana et al co-doped Tb⁵⁺ and Dy³⁺ in oxyfluoride germinate glasses for white light emission [19]. Yu et al tri-doped Tb⁵⁺, Sm³⁺, and Eu³⁺ in phosphate glasses and produced a white-light-emitting material close to the standard equal energy white-light illumination [20].

Samarium ions are important activator with 4f⁵ electronic configuration which is widely used in various hosts [21–28]. The trivalent Sm³⁺ ions have a strong luminescence in the reddish-orange range of wavelength due to \( ^{5}G_{7/2} \rightarrow ^{7}H_{1}(I = 5/2, 7/2, 9/2) \) transitions. Simultaneously, the trivalent Tb⁵⁺ ions in different hosts are known as green emitters due to \( ^{3}D_{4} \rightarrow ^{7}F_{j}(i = 0, 1, \ldots, 6) \) transitions [29–34]. Moreover, Tb⁵⁺ ions are also good sensitizer for Sm³⁺ ions, which helps to increase reddish-orange luminescence. In Tb⁵⁺/Sm³⁺ co-doped systems, terbium plays the role as sensitizer, and samarium is an activator. The energy transfer from Tb⁵⁺ to Sm³⁺ occurs nonradiatively by quenching (Tb⁵⁺) and increasing (Sm³⁺) luminescence. Luminescent properties of various hosts singly doped with Sm³⁺ or Tb⁵⁺ are well reported in literatures [21–34] while only a few published works are devoted to Tb⁵⁺/Sm³⁺ co-doped host matrices [17, 35, 36].

Moreover, CaSiO₃ has been one of promising host luminescent materials not only due to the simple fabrication process but also its outstanding physical properties such as chemical and thermal stability, low thermal expansion, and chemical inertness [37]. Notably, in comparison with Ca₂SiO₄ and Ca₃SiO₅, CaSiO₃ is more stable. It is known that the Ca₃SiO₅ is stable in the range of 1523 and 2323 K but not in the low temperature range. At temperatures below ~1523 K, Ca₃SiO₅ is converted to Ca₂SiO₄ and CaO [38]. This high temperature range is not suitable for WLEDs if we look at the application point of view. Therefore, in this work we have synthesized and systematically investigated the structural and luminescent properties of phosphors CaSiO₃ co-doped with Tb⁵⁺ and Sm³⁺ activated ions for the application of WLEDs.

### 2. Experimental details

The phosphors CaSiO₃:x%Tb⁵⁺, y%Sm³⁺ were synthesized by the conventional solid state reaction using high-pure powdered precursors of CaCO₃, SiO₂, Tb₂O₃, and Sm₂O₃ purchased from Sigma-Aldrich as raw materials. These precursors with stoichiometric quantities were mixed and ground by using an agate mortar and pestle in several hours. The obtained mixtures were then calcined at 1200 °C for 2 h. The synthesized phosphors are listed in table 1.

The crystal structure of CaSiO₃:x%Tb⁵⁺, y%Sm³⁺ phosphors was investigated by an x-ray diffractometer D8 Advance Eco (Bruker) equipped with a Cu-Kα radiation source (\( \lambda = 1.54056 \) Å). The surface morphology and chemical compositions of the phosphors were analyzed using a field-emission scanning electron JSM-7600F Schottky microscope equipped with energy dispersive x-ray. Luminescent properties of the phosphors were studied by photoluminescence (PL) emission, PL excitation (PLE), and lifetime measurements using the fluorescence spectrophotometer Fluorolog 3 FL3-22 (Horiba).

### 3. Results and discussion

The x-ray diffraction (XRD) pattern of the synthesized phosphors was shown in figure 1(a). For angle calibration, before carrying out XRD measurement, a small amount of Si powder was mixed with the samples. All the diffraction peaks are well fit with the reference data PDF 01-080-9543 of pseudowollastonite (CaSiO₃). This result clearly reveals that all the phosphors are single phase and have monoclinic structure, belonging to the space group of C2/c. Moreover, it can be seen in figure 1 (b) that the position of the structural diffraction peak (008) of all these doped phosphors tends to shift towards larger angle with increasing the doping concentration.
This result clearly indicated that ions Tb\(^{3+}\) and Sm\(^{3+}\) were incorporated into the CaSiO\(_3\) host lattice. In addition, the average crystal size \(d\) of phosphors was also estimated based on the full width at half-maximum (FWHM) of the peak (008) using Scherrer’s equation \(d = 0.9\lambda/\beta\cos\theta\), where \(\lambda = 1.54056 \text{ Å}\) is the wavelength of x-ray, \(\beta\) and \(\theta\) are its FWHM and angle position, respectively. It has been established that the average crystal size ranges from 100 to 170 nm.

Figures 2(a) and (b) show scanning electron microscopy (SEM) images of undoped CaSiO\(_3\) and CaSiO\(_3\) doped with 1.0\% Tb\(^{3+}\) and 1.0\% Sm\(^{3+}\). The SEM images show non-uniform and irregular shapes of particles in the phosphors. The SEM results along with the estimated crystal size indicated that all synthesized phosphors were polycrystalline bulk. Furthermore, the SEM images of both undoped CaSiO\(_3\) and doped CaSiO\(_3\) are identical, meaning that doping Tb\(^{3+}\) and Sm\(^{3+}\) did not change the morphology of CaSiO\(_3\) particles. Figures 2(c) and (d) show the energy dispersive x-ray spectroscopy (EDS) analysis result of undoped CaSiO\(_3\) and CaSiO\(_3\) doped with 1.0\% Tb\(^{3+}\) and 1.0\% Sm\(^{3+}\). From the EDS analysis, elements of Ca, Si, and O were detected in both undoped and doped CaSiO\(_3\) with the ratio of Ca and Si elements close to 1:1, which clearly indicated the formation of CaSiO\(_3\) in the synthesized materials. In addition, peaks of Tb and Sm elements were also detected in the doped CaSiO\(_3\). The EDS analysis result indicated the purity of synthesized materials.

The PLE spectra of Tb\(^{3+}\) and Sm\(^{3+}\) in CaSiO\(_3\) are shown in figure 3. The PLE spectrum of Tb\(^{3+}\) monitored at the emission wavelength of 543 nm consists of a series of peaks observed at wavelengths of 318 nm, 340 nm, 352 nm, 369 nm, 377 nm, and 485 nm, corresponding to electronic transitions from \(^7F_6\) to \(^5D_1\), \(^7F_{6}\), \(^5D_2\), \(^7F_{10}\), \(^5D_3\), and \(^5D_4\), respectively \([40]\). Whereas, that of Sm\(^{3+}\) monitored at the emission wavelength of 602 nm is composed of a series of peaks observed at wavelengths of 345 nm, 362 nm, 375 nm, 404 nm, 417 nm, and 464 nm, corresponding to electronic transitions from \(^6H_{5/2}\) to \(^4I_{15/2}\), \(^4D_{3/2}\), \(^4P_{7/2}\), \(^4F_{7/2}\), \(^4P_{5/2}\), and \(^4I_{13/2}\), respectively.

Figure 1. (a) X-ray diffraction (XRD) pattern and (b) enlarged view of XRD region containing (008) peak of CaSiO\(_3\)\(x\%\)Tb\(^{3+}\), \(y\%\) Sm\(^{3+}\).
From the PLE spectra, we can see that the wavelength of 375 nm is optimal to excite the luminescence of both Tb$^{3+}$ and Sm$^{3+}$ in CaSiO$_3$ host materials.

Figure 2. Scanning electron microscopy images and corresponding energy dispersive x-ray spectroscopy analysis of undoped CaSiO$_3$ ((a) and (c)) and CaSiO$_3$:1.0%Tb$^{3+}$, 1.0%Sm$^{3+}$ (b) and (d)).

Figure 3. Photoluminescence excitation spectra of Tb$^{3+}$ and Sm$^{3+}$ in CaSiO$_3$.

[41]. From the PLE spectra, we can see that the wavelength of 375 nm is optimal to excite the luminescence of both Tb$^{3+}$ and Sm$^{3+}$ in CaSiO$_3$ host materials.

Figure 4 shows the PL emission spectra of the phosphors under the excitation of 375-nm-wavelength. For the Tb$^{3+}$ singly doped CaSiO$_3$ phosphor (S1), the PL emission spectrum displays a series of peaks located at wavelengths of 415 nm, 436 nm, 457 nm, 488 nm, 543 nm, 586 nm, and 622 nm, corresponding to Tb$^{3+}$.
electronic transitions of \( ^5D_3 \rightarrow ^7F_5, ^5D_3 \rightarrow ^7F_6, ^5D_4 \rightarrow ^7F_3, ^5D_4 \rightarrow ^7F_4, ^5D_4 \rightarrow ^7F_5, ^5D_2 \rightarrow ^7F_4, \) and \( ^5D_4 \rightarrow ^7F_3 \), respectively [40, 42]. The PL spectrum of the phosphor S2, singly doped with Sm\(^{3+}\), has a series of three peaks located at wavelengths of 565 nm, 602 nm, and 648 nm, corresponding to the electronic transitions of Sm\(^{3+}\) from \( ^4G_{5/2} \) to \( ^6H_{5/2}, ^6H_{7/2}, \) and \( ^6H_{9/2}, \) respectively [41]. As we can see in figure 4, when the concentration of Sm\(^{3+}\) increases, the emission intensity of Sm\(^{3+}\) increases while that of Tb\(^{3+}\) decreases. This result indicated the energy transfer from Tb\(^{3+}\) to Sm\(^{3+}\).

The probable processes of energy transfer under 375-nm UV excitation wavelength in the CaSiO\(_3\):x\%Tb\(^{3+}\), y\%Sm\(^{3+}\) phosphors are given in figure 5. For Tb\(^{3+}\) doped phosphor, the PLE spectrum consists of a set of intense lines formed by intra-4f transitions originating from the \( ^7F_6 \) ground state to \( ^5D_4 \) and \( ^5D_3 \) excited states of Tb\(^{3+}\) ions. Two non-radiative transitions of \( ^5D_3 \rightarrow ^5D_4 \) of Tb\(^{3+}\) ion and \( ^4P_{7/2} \rightarrow ^4G_{5/2} \) of Sm\(^{3+}\) ion are shown in the energy diagram. Because the energy level \( ^7D_4 \) of Tb\(^{3+}\) ion is just above the energy level \( ^4F_{3/2} \) of Sm\(^{3+}\) ion, the energy can be transferred from the level \( ^7D_4 \) of Tb\(^{3+}\) ion to the level \( ^4F_{3/2} \) of Sm\(^{3+}\) ion, followed by the non-radiation relaxation process to the level \( ^4G_{5/2} \) of Sm\(^{3+}\) ion for emission. There are several bands of Sm\(^{3+}\) are
present in the PLE spectrum (figure 3) at 350–500 nm range and the transitions for each bands are mentioned above. The peaks with strongest intensity are located at 404 nm which corresponds to $^6H_5/2 \rightarrow ^{4}F_7/2$ transition of Sm$^{3+}$ ion. Therefore, this energy is usually used for excitation. But in this study we investigated Tb$^{3+}$/Sm$^{3+}$ co-doped system and 375 nm is best wavelength (which corresponds to $^6H_5/2 \rightarrow ^{6}P_{7/2}$ transition of Sm$^{3+}$) for excitation. Our experimental results indicated that energy transfer occurs between Tb$^{3+}$ and Sm$^{3+}$ ions. Spectral overlapping between the Sm$^{3+}$ absorption and the Tb$^{3+}$ emission spectra shows that the energy transfer occurs from Tb$^{3+}$ to Sm$^{3+}$ [43]. It’s known that the absorption band around 477 nm of Sm$^{3+}$ ion overlap with the emission band of Tb$^{3+}$ around 488 nm [35]. Thus, the overlap region refers that Tb$^{3+}$ act as a sensitizer for Sm$^{3+}$.

The energy transfer was confirmed by results of lifetime measurements of the phosphors as shown in figures 6(a) and (b) and table 2. As we can see from figure 6(a) and table 2, the lifetime of Tb$^{3+}$ ion at the emission wavelength of 543 nm decreases with the increase of Sm$^{3+}$ ion. On the contrary, as we can see in figure 6(b) and table 2, the lifetime of Sm$^{3+}$ ion at the emission wavelength of 602 nm increases with the presence of Tb$^{3+}$ ion. These results clearly confirm the energy transfer from Tb$^{3+}$ to Sm$^{3+}$. The energy transfer efficiency was estimated using the equation [44]:

$$\epsilon = 1 - \frac{\tau_{T}}{\tau_{T}}$$

where $\tau_{T}$ and $\tau_{TS}$ are the lifetime of the Tb$^{3+}$ in the absence and presence of Sm$^{3+}$, respectively. The result is shown in table 2.

The Commission Internationale de L’Eclairage (CIE) chromaticity coordinates of synthesized phosphors excited at 375 nm are given in figure 7. As shown in figure 7, the pure Tb$^{3+}$ doped CaSiO$_3$ phosphor (S1) emits blue light. CIE coordinates of phosphor S1 are 0.1994 and 0.2256. The pure Sm$^{3+}$ doped phosphor (S2) emits reddish-orange light and its CIE coordinates are 0.3137 and 0.3367. The obtained results showed that Tm$^{3+}$/Sm$^{3+}$ co-doped phosphors emit white light when excited at 375 nm. CIE coordinates of Tm$^{3+}$ and Sm$^{3+}$ co-doped samples S3, S4, S5, and S6 are (0.3137; 0.3367), (0.3339; 0.3431), (0.3662; 0.3442), and (0.3623; 0.3446), respectively. From the CIE result, the correlated color temperature (CCT) of phosphors was calculated using McCamy’s equation [45, 46]:

Table 2. List of luminescent parameters of the synthesized phosphors.

| Phosphor | Lifetime $\tau$ (ns) | Energy transfer efficiency $\epsilon$ (%) | CIE (x, y) | Correlated color temperature (K) | CRI |
|----------|----------------------|------------------------------------------|------------|----------------------------------|-----|
| S1       | 747                  | —                                        | 0.1994, 0.2256 | 84000                           | N/A |
| S2       | —                    | 343                                      | 0.3084, 0.3804 | 1900                            | 45  |
| S3       | 720                  | 361                                      | 0.3137, 0.3367 | 6400                            | 72  |
| S4       | 712                  | 367                                      | 0.3339, 0.3431 | 5440                            | 79  |
| S5       | 623                  | 16.3                                     | 0.3662, 0.3442 | 4210                            | 74  |
| S6       | 851                  | —                                        | 0.3623, 0.3446 | 4340                            | 80  |

Figure 6. Decay characteristic of (a) Tb$^{3+}$ and (b) Sm$^{3+}$ in CaSiO$_3$. 

Figure 7. CIE chromaticity coordinates of synthesized phosphors.
$CCT = -449n^3 + 3525n^2 - 6823.3n + 5520.33$  \hspace{1cm} (2)

where $n = (x - 0.3320)/(y - 0.1858)$. The color rendering index (CRI) of the phosphors was also determined from the measured PL spectrum of the phosphors employing the ColorCalculator software developed by OSRAM SYLVANIA Inc. [47]. The CCT and CRI for the phosphors are given in table 2. The results showed that the CRI of phosphors significantly increases in Tm$^{3+}$/Sm$^{3+}$ co-doped phosphors S3, S4, S5 and S6. The highest value of CRI is 80 for phosphor S6 with CCT of 4340 K. As we can see from table 2, almost phosphors have cool colors. Especially, the CCT of the phosphor S4 belongs to the Daylight of color temperature. These results indicated that the phosphor CaSiO$_3$ doped with 1.0% Tb$^{3+}$ and 1.0% Sm$^{3+}$ (S4) is an optimal candidate for WLED applications.

Although the PL results clearly showed that the synthesized phosphors have good PL properties, it is possible to further improve the PL properties of these phosphors. It is known that when doped in CaSiO$_3$, ions Tb$^{3+}$ and Sm$^{3+}$ replace Ca$^{2+}$ ions. Due to the difference in valence state of these ions, Ca$^{2+}$ vacancies are created in the host material in order to keep charge balance. At the Ca$^{2+}$ vacancy, a net negative environment will allow to trap holes and, therefore, the luminescence intensity is reduced. In order to eliminate such defects in the host material and improve the luminescence intensity, ions Na$^+$ or Li$^+$ can be doped in the Tb$^{3+}$/Sm$^{3+}$ co-doped CaSiO$_3$ phosphors to make the charge balance in the host materials. As a result, the luminescent efficiency of phosphors can be improved [48, 49]. Another way for improving the PL properties of the phosphors is to improve their particle morphology through other phosphor synthesis methods such as combustion [50] or citrate gel methods [51].

4. Conclusion

The structural and photoluminescence properties of phosphors CaSiO$_3$:x%Tb$^{3+}$, y%Sm$^{3+}$ were studied at room temperature. X-ray analysis shows that all the phosphors are single phase and have monoclinic structure, belonging to the space group of C2/c. The PL emission spectrum consists of a set of intense lines formed by intra-4f transitions of Tb$^{3+}$ and electronic transitions of Sm$^{3+}$ ions from $^4G_{5/2}$ to $^6H_J$ ($J = 5/2, 7/2, 9/2$). The energy level scheme is proposed based on the obtained results and energy transfer process is examined. The emission intensity of Sm$^{3+}$ increases while that of Tb$^{3+}$ decreases with respect to the Sm$^{3+}$ concentration. This result indicated the energy transfer from Tb$^{3+}$ to Sm$^{3+}$. Because the energy level $^3D_4$ of Tb$^{3+}$ is just above the energy level $^4F_{3/2}$ of Sm$^{3+}$, the energy can be transferred from the level $^3D_4$ of Tb$^{3+}$ to the level $^4F_{3/2}$ of Sm$^{3+}$, followed by a non-radiation relaxation process to the level $^4G_{5/2}$ of Sm$^{3+}$ for emission. This energy transfer was confirmed by results of lifetime measurements of the phosphors. The lifetime of ion Tb$^{3+}$ at the emission wavelength of 543 nm decreases with the increase of Sm$^{3+}$ ion. On the contrary, the lifetime of Sm$^{3+}$ ion at the emission wavelength of 602 nm increases with the presence of ion Tb$^{3+}$. Correlated color temperature and color rendering index of phosphors were determined from their PL results. It was determined that almost phosphors...
have cool colors. Especially, the CCT of the phosphor CaSiO$_3$ doped with 1.0% Tb$^{3+}$ and 1.0% Sm$^{3+}$ belongs to the Daylight of color temperature. The results show that the obtained phosphor is an optimal candidate for white-LED applications.

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