

INTENSE GREEN EMISSION PROPERTIES OF RARE-EARTH-ELEMENT-DOPED CaZrO₃ PEROVSKITE PHOSPHOR

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ARTICLE INFO		ABSTRACT
Received:	28/10/2024	In this work, we report the optical properties of Ho ³⁺ doped CaZrO ₃ perovskite phosphor synthesized using a solution combustion method. The crystal structure, morphology, and optical properties of resulting phosphors were investigated by X-ray diffraction, field emission scanning electron microscope, and photoluminescence analysis. X-ray diffraction result confirms that the single-phase orthorhombic CaZrO ₃ was obtained. Under 975 nm excitation of the laser source, the phosphors emitted a strong green region at 523/542 nm and a weak red region at 660 nm, corresponding to the ⁵ F ₄ - ⁵ I ₈ / ⁵ S ₂ - ⁵ I ₈ and ⁵ F ₅ - ⁵ I ₈ transitions of Ho ³⁺ in the host lattice. Power laser excitation-dependent emission intensity indicated that the two-photon absorption produces a green and red upconversion emission. In addition, the obtained phosphor exhibits intense green upconversion emissions and high color purity (99.66%), making it suitable for optoelectronic devices and solid-state lighting applications.
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Upconversion emissions		

TÍNH CHẤT PHÁT XẠ MÀU XANH CỦA BỘT HUỖNH QUANG PEROVSKITE CaZrO₃ PHA TẠP NGUYÊN TỐ ĐẤT HIẾM

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THÔNG TIN BÀI BÁO	TÓM TẮT
Ngày nhận bài: 28/10/2024	Trong nghiên cứu này, chúng tôi trình bày về tính chất quang học của bột huỳnh quang perovskite CaZrO ₃ pha tạp ion Ho ³⁺ được tổng hợp bằng phương pháp đốt cháy dung dịch. Cấu trúc tinh thể, hình thái và tính chất quang của vật liệu thu được đã được nghiên cứu bằng phương pháp phân tích giản đồ nhiễu xạ tia X, ảnh hình thái bề mặt và phổ phát quang. Kết quả giản đồ nhiễu xạ tia X xác nhận rằng vật liệu CaZrO ₃ thuộc vật liệu có cấu trúc perovskite trực thoi pha đơn và không phụ thuộc vào nồng độ pha tạp Ho. Dưới sự kích thích 975 nm của nguồn laser, vật liệu phát ra vùng xanh lục mạnh ở 523/542 nm và vùng đỏ yếu ở 660 nm, tương ứng với các chuyển tiếp ⁵ F ₄ - ⁵ I ₈ / ⁵ S ₂ - ⁵ I ₈ và ⁵ F ₅ - ⁵ I ₈ của Ho ³⁺ trong mạng nền CaZrO ₃ . Cường độ phát xạ phụ thuộc vào sự kích thích laser công suất chỉ ra rằng sự hấp thụ hai photon tạo ra sự phát xạ chuyển đổi lên màu xanh lục và đỏ. Ngoài ra, bột huỳnh quang thu được thể hiện sự phát xạ chuyển đổi ngược màu xanh lục mạnh và độ tinh khiết màu cao (99,66%), cho thấy vật liệu phù hợp để chế tạo thiết bị quang điện tử và các ứng dụng chiếu sáng trạng thái rắn.
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1. Introduction

Rare earth ion-doped-upconversion (UC) materials are widely used for multifunction applications such as lasers, solar cells, optical temperature sensors, biomedical, and catalytic due to their excellent properties, including long-lifetime, high stock shift, thermal stability, and ease of synthesis [1] – [3]. The UC emission is a nonlinear optical phenomenon. The UC phosphor material can absorb photons at the near-infrared band (low energy) to emit at the visible band (high energy) through two or more photon absorption [4], [5]. The Ho^{3+} ion, typical rare-earth elements widely used activators for UC materials, have multiple emission regions, from green to red. Notably, the green/red intensity ratio of the Ho^{3+} doped UC phosphor could affect its application area. This value depends on many parameters, such as dopant content, host lattice, sensitivity ions, and condition synthesis [6], [7]. Singh et al. [8] synthesized intense green UC emission of nano $\text{Y}_2\text{O}_3:\text{Ho}^{3+}$ phosphors using the solution combustion method. They found that the phosphor with high green color purity could be used for green LEDs and display applications. Liu et al. [9] studied strong green UC emission of $\text{Ho}^{3+}/\text{Yb}^{3+}/\text{Nd}^{3+}$ co-doped BaGd_2O_4 phosphors synthesized using solid-state reaction. These results presented high green color purity upto 93% and temperature sensitivity of $3.2 \times 10^{-4} \text{ K}^{-1}$, suggesting that it is suitable for thermal sensor application. Meanwhile, Wei et al. [10] investigated UC emission and optical temperature sensing properties of $\text{Gd}_3\text{BWO}_9:\text{Yb}^{3+}, \text{Ho}^{3+}$ phosphor that indicated a high absolute sensitivity ($5.08 \% \text{ K}^{-1}$ at 300 K) of the obtained phosphor, which could be used for thermal sensor application.

Oxides have low phonon energy, thermal stability, wide-range transparency, and accessible synthesis, making them suitable for luminescent host [11] – [13]. CaZrO_3 perovskite host is a typical oxide widely used for optoelectronic applications and has an orthorhombic structure and space group $Pbnm$ [14], [15]. It has low phonon energy, resulting in reduced non-radiative transition, enhancing UC emission efficiency [16]. Van et al. [6] successfully synthesized intense green UC emission of $\text{Er}/\text{Yb}/\text{Mo}$ doped CaZrO_3 using a solid-state reaction method. These results indicate that the optical properties of the phosphor depend on Mo^{6+} doping content, and phosphors showing high green color purity and temperature sensitivity are suitable for optoelectronic devices. Khan et al. [17] studied tuning white light UC emission of $\text{Er}/\text{Tm}/\text{Yb}$ co-doped CaZrO_3 synthesized by solid-state reaction for display devices and laser field applications. Meanwhile, Maurya et al. [18] investigated the enhanced green UC emission of Mg^{2+} doped $\text{CaZrO}_3:\text{Ho}/\text{Yb}$ phosphor using a solid-state reaction. They found that the presence of Mg^{2+} resulted in decreased lattice parameters and increased crystallinity of the phosphor, which caused its enhanced green UC emission. However, the intense green UC emission of $\text{CaZrO}_3:\text{Ho}$ phosphor synthesized by the solution combustion method has yet to be well documented.

In this work, intense green UC emission $\text{CaZrO}_3:\text{Ho}$ phosphors were synthesized via a solution combustion method using urea as fuel. The structural, morphological, and optical properties of the phosphors were explored using X-ray diffraction (XRD), high-resolution scanning electron microscopy (FE-SEM), and photoluminescence (PL) measurement.

2. Experiments

$\text{CaZrO}_3:x\text{Ho}$ ($x = 1, 2, 3, 4,$ and $5 \% \text{ mol}$, labeled S1, S2, S3, S4, and S5, respectively) nanophosphors were synthesized by the solution combustion method. The prepared processed using raw materials with high purity such as $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ (99.90%), $\text{ZrOCl}_2 \cdot 4\text{H}_2\text{O}$ (99.90%), $\text{Ho}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ (99.99%), and $\text{CO}(\text{NH}_2)_2$ (99.9 %) as fuel. The synthesis process followed several steps. First, the Ca^{2+} (1M), Zr^{4+} (1M), and Ho^{3+} (0.5M) solutions were obtained by dissolving salt in de-ionized water. Then, the dopant ion (Ho^{3+}) was drop-by-drop added to the ($\text{Ca}^{2+}, \text{Zr}^{4+}$) solution under stirred conditions for 60 min. Next, 20% mol urea (compared to Ca^{2+}) was added to the mixed solution and stirred magnetically for 1 h at 80°C . The mixed solutions were in the reaction combustion at 400°C for 4 h in air. Finally, the powder was annealed at 1200°C for 2 h in air.

The structure of phosphors was carried out using an X-ray diffractometer (Bruker D8 Advance). Analyzed morphological property was determined using high-resolution scanning electron microscopy (JEM 1010, JEOL Technique, Japan). The luminescence properties of UC phosphor were characterized using a NANO LOG spectrometer (Horiba, USA) equipped with a 975 nm laser radiation source.

3. Results and Discussion

Figure 1 shows the XRD patterns in the 20–70° region of investigated phosphor samples S1, S2, S3, and S5. It can be seen that all diffraction peaks of all samples match well with the standard card PDF#00-061-0228 of single phase CaZrO_3 , which has an orthorhombic structure and space group $Pbmn$. These typical planes, such as (110), (112), (220), (312), and (224) are observed clearly in all samples. In addition, all diffraction peaks indicate high and sharp intensity, and the good agreement between the experimental data and the X-ray pattern suggests the high degree of crystallinity of the obtained phosphors. Notably, the diffraction peak attributed to the (112) plane shows the highest intensity, suggesting a preferred orientation of grains grown along with the crystallographic c -axis. The preferred orientation phenomenon can improve the UC emission efficiency of the material [19], [20]. Moreover, these impurity phases were not observed, confirming the successfully synthesized of the single phase-phosphors. The single phase with high crystallinity of the phosphors can improve its UC emission efficiency.

Furthermore, the grain size of the samples was calculated using the Scherrer equation [20]:

$$L = \frac{0.9\lambda}{\beta \cos(\theta)} \quad (1)$$

where L (nm) presents grain size, 0.9 is Scherrer constant, the wavelength of the X-ray sources is 0.15406 nm, β (radians) is full width at half maximum, and θ (radians) is peak position. The diffraction peak corresponding to the (112) plane was chosen to estimate the grain size of the phosphors. The grain size (L) of all samples is 66 nm (S1), 62 nm (S2), 58 nm (S3), and 52 nm (S5), indicating a decrease in L values as the increase of Ho^{3+} contents, which is due to different radius ionic between Ho^{3+} (0.91Å) and Ca^{2+} (1Å) ions.

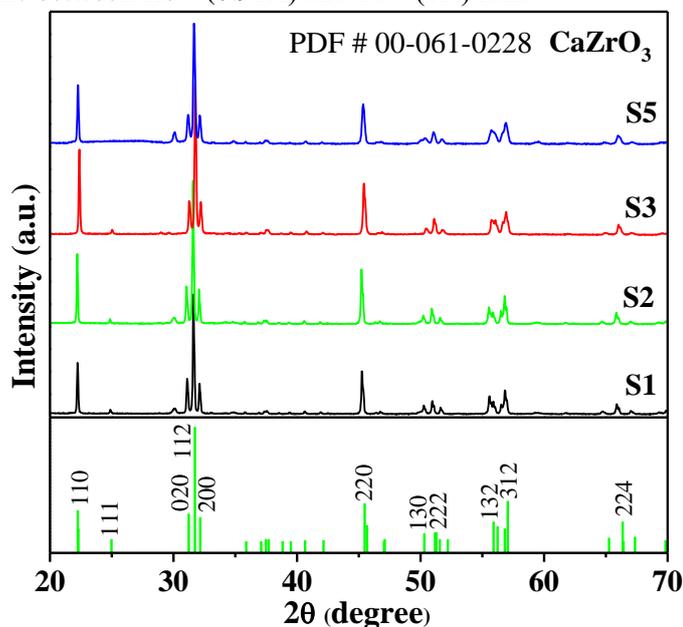


Figure 1. XRD patterns of S1, S2, S3, and S5 investigated phosphors

Figure 2a presents the SEM image of a typical S2 sample annealing at 1200 °C for 2 h in air. It is seen that the S2 sample showed grain clusters with a cubic-like morphology with an average

grain size of 462 nm (inset of Figure 2a). Meanwhile, all peaks observed in Figure 2b are attributed to the elements of the dopant ion (Ho^{3+}) and host lattice (Ca, Zr, O), suggesting the presence of Ho in the sample. Notably, other impure elements are not observed, indicating the high purity of the obtained phosphor, which agrees with XRD results.

The optical properties of investigated samples are measured using a 975 nm excitation wavelength from a laser source, as shown in Figure 3a. Under 975 nm excitation, the phosphors emitted intense green UC region (523/542 nm) corresponding to $^5\text{F}_4/^5\text{S}_2 - ^5\text{I}_8$ transitions and weak red region (660 nm) attributed to $^5\text{F}_5 - ^5\text{I}_8$ transition of Ho^{3+} ion. Its emission intensity depends on Ho^{3+} content (Figure 3b) and reaches the maximum value at 2% mol Ho^{3+} . When Ho^{3+} content is higher than 2% mol, the emission intensity decreases, which is caused by concentration quenching [21].

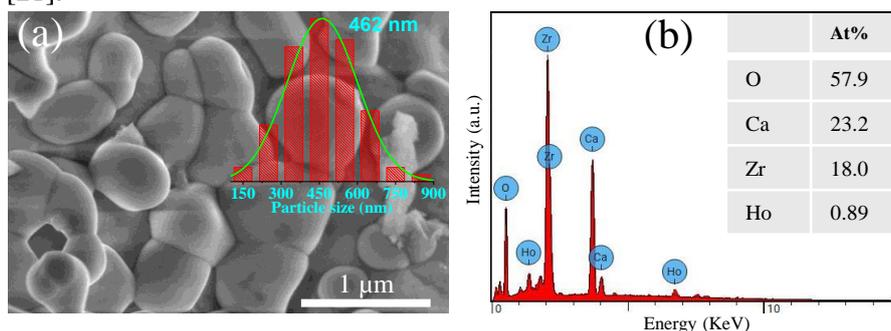


Figure 2. a) SEM image of typical samples S2 and b) EDS spectrum of S2 sample

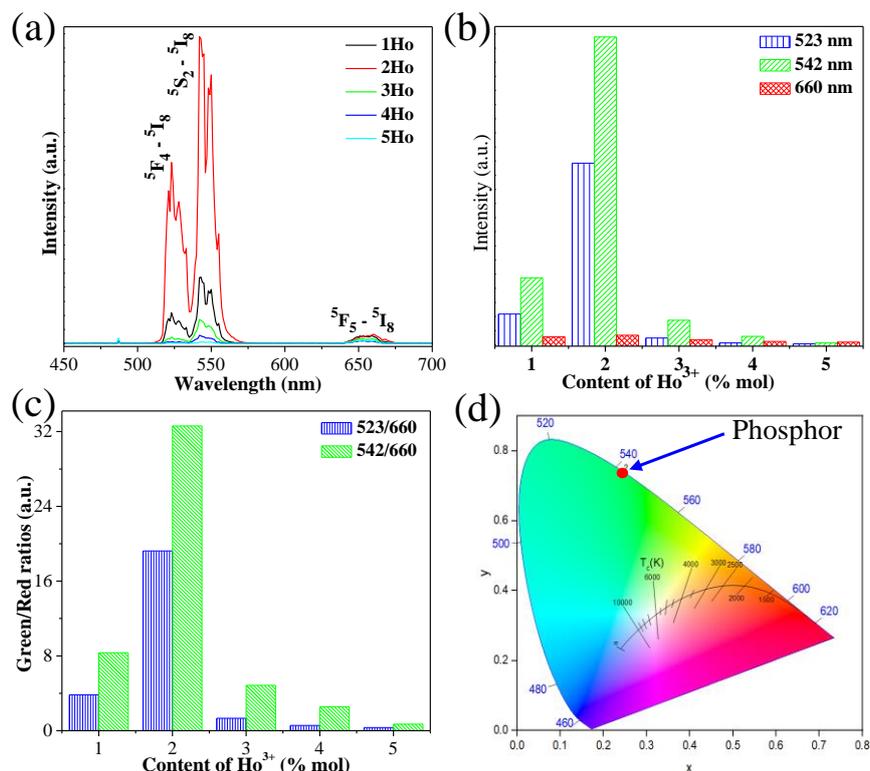


Figure 3. (a) UC emission spectra of all resulted samples; (b) emissions intensity-dependent on dopant content; (c) green/red ratios of all samples; (d) CIE coordinates of typical sample S2

The green/red intensity is estimated (Figure 3c) to show the highest value at 2% mol Ho^{3+} (542/660 nm ratio achieved 32 times), confirming the strong green UC emission of the obtained

phosphors. Furthermore, Commission International de l'Eclairage (CIE) [22] chromaticity coordinates of the resulting phosphor are calculated based on their emission spectra, as presented in Figure 3d. The calculated values are shown in Table 1. In this case, the CIE coordinate of S2 (0.242, 0.734) is close to the pure green color [22]. Notably, the chromaticity coordinates from Table 1 were used to estimate the color purity of the phosphors [23]:

$$\text{Color purity} = \sqrt{\frac{(x - x_i)^2 + (y - y_i)^2}{(x_d - x_i)^2 + (y_d - y_i)^2}} \times 100\% \quad (2)$$

where (x, y) are the color coordinates of samples, $(x_i = 0.3333, y_i = 0.3333)$ corresponding to the color coordinate of the white light source [6], and (x_d, y_d) presents the color coordinates of the dominant wavelength. Significantly, the highest color purity achieved for the S2 sample is 99.66%, confirming the pure green light emission. These results indicate that the S2 phosphor is suitable for solid lighting and optoelectronic devices.

Table 1. The CIE chromaticity coordinates and color purity of investigated samples

Samples	Chromaticity coordinates (x, y)	Color purity (%)
S1	(0.262, 0.771)	92.32
S2	(0.242, 0.734)	99.66
S3	(0.287, 0.681)	83.28
S4	(0.303, 0.633)	73.37
S5	(0.383, 0.497)	42.73

The excitation power (P)-dependent emission intensity (I) can be used to explain the intense green UC emission mechanism based-phosphor [24]. It can be written as $I \propto P^n$, where n is the number of photons absorbed for emitter UC. Figure 4a shows the relationship between I and P from 300 to 700 mW of the typical sample (S2). As shown in Figure 4a, the I value increases with the increase of the P values. In this case, the slope values (n) are 1.36/1.15 and 1.23 (obtained by fit linear between the $\log [I]$ and the $\log [P]$), corresponding to the ${}^5F_4/{}^5S_2 - {}^5I_8$ (green) and ${}^5F_5 - {}^5I_8$ (red) transitions, respectively. The results confirm the two-photon absorption process for the obtained sample's green/red emissions of (Figure 4b) [25], [26].

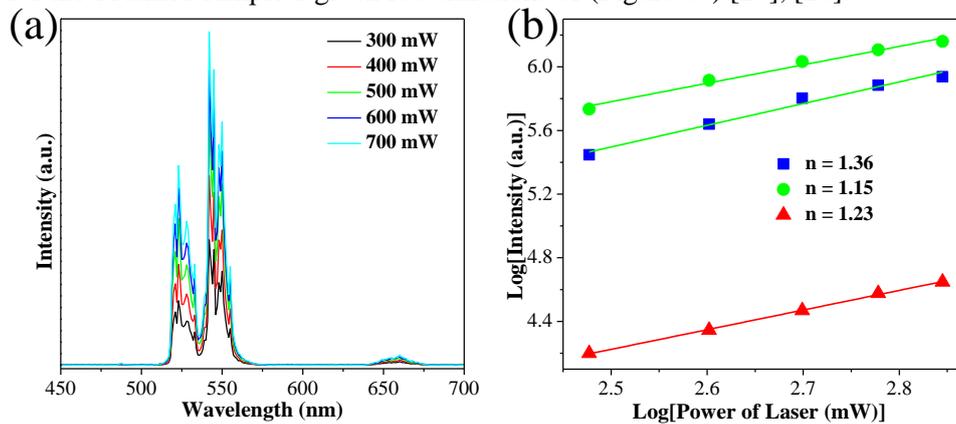


Figure 4. (a) Emission intensity-dependent the power of laser from 300 to 700 mW and (b) the log–log plots of green/red emission as a function of the power of the sample S2

4. Conclusion

Intense green UC emission of Ho^{3+} doped CaZrO_3 phosphors was successfully synthesized using the solution combustion method to explore its structure, morphology, and optical properties. The XRD measurement confirmed the formation of high crystallinity of the single-phase orthorhombic structure CaZrO_3 . Upon 975 nm excitation of the laser source, the phosphors emitted strong green UC at 523/542 nm corresponding to ${}^5F_4/{}^5S_2 - {}^5I_8$ transitions and weak red at

660 nm, attributing to ${}^5F_5 - {}^5I_8$ transition of Ho^{3+} on CaZrO_3 lattices. Its emission intensity depended on Ho^{3+} dopant content and reached a maximum value at 2% mol Ho^{3+} ; beyond that, it is decreased owing to concentration quenching. Significantly, the $\text{CaZrO}_3:2\text{Ho}$ phosphor shows intense green UC emission with high color purity (99.66%), indicating that it is suitable for solid-state lighting and optoelectronic applications.

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