



Original Article

Effect of Fuel Contents on Upconversion Emission Properties of Rare-Earth Ions Doped Hydroxyapatite/Tri-Calcium Composite Phosphors

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Abstract: In this work, we report the effect of fuel content on the green upconversion emission of Er/Yb/Li tri-doped hydroxyapatite/ β -tricalcium phosphate (HA/ β -TCP) composite phosphor prepared by the solution combustion method using urea as the fuel. X-ray diffraction confirms the formation of mixed phases, including the main HA phase with a hexagonal structure and the secondary β -TCP phase; their percentage composition depends on the fuel content. Scanning electron microscopy indicates spherical particles with average sizes ranging from 118.7 nm to 306.6 nm. Under 975 nm excitation, the phosphors exhibit intense green upconversion (UC) emission (560 nm), which reaches its maximum at the optimal fuel content of 30 mol%. This phenomenon is due to the high fuel content, which enhances crystallinity and contributes to the improved emission-intensity of the phosphors. In summary, the phosphor exhibits intense green UC emission with a uniform particle size, making it suitable for biomedical applications.

Keywords: Green upconversion emission; HA/ β -TCP:Er/Yb/Li nanocomposite phosphor; Solution combustion method.

1. Introduction

Upconversion (UC) phosphor materials activated by rare-earth (RE) ions are widely applied in many fields, including solid-state lasers, bioimaging, and photovoltaic devices; these effects

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applications are contributed to their advantages of long lifetimes, narrow-band emission, and excellent physicochemical properties [1, 2]. Among them, RE ion-doped hydroxyapatite (HA) phosphor materials have received widespread attention for biomedical applications, such as bioimaging, bone substitutes, and drug delivery because of their excellent biocompatibility and bioactivity [3, 4]. Notably, the physicochemical properties of RE-doped HA, including morphology, particle size, degree of crystallinity, and optical properties, strongly affect its potential applications [3-5]. Therefore, many approaches have been applied to synthesize HA:RE phosphor materials to control their physicochemical properties, such as co-precipitation, sol-gel, the hydrothermal method, and solution combustion [4, 6]. Previous work indicated that synthesizing HA:RE nanophosphors with high crystallinity and high emission efficiency via low-temperature treatment is challenging. The solution combustion method has been widely applied to prepare RE-doped phosphors with high crystallinity and uniform nanoscale particle distribution, overcoming conventional limitations and making it suitable for biomedical applications. Therefore, this approach has been widely selected. In this case, the fuel content strongly affects the structural characteristics and physicochemical properties of the systems [7-9]. Batista et al., [7] observed a high degree of crystallization, up to 82%, in HA/ β -TCP nanoparticles prepared by the solution combustion method using urea as the fuel. Khal et al. [8] applied solution combustion to synthesize nanosized HA powder with a high specific surface area (48 m²/g) for removing contamination Pb²⁺ ions from an aqueous solution. Meanwhile, Wagner et al. [9] demonstrated that HA:Eu nanowhisker phosphors could be prepared via microwave-assisted combustion, indicating that this method is suitable for bioimaging applications. In addition, the final products exhibit high crystallinity, narrow morphological distribution, high chemical purity, and low in vitro cytotoxicity [9]. Furthermore, G. Ramakrishna et al., [10] observed that the photothermoluminescence properties of Y₂SiO₅ nanopowder significantly depend on the fuel materials (sugar, urea, and DFH), which affect particle size because the fuels behave differently. Moreover, Upasani et al., [11] investigated the broad greenish-yellow emission of Ce³⁺-doped YAG prepared via the solution combustion method using glycine–urea fuels. They found that the obtained phosphor could be a promising material for WLED applications. Recent studies indicate that different fuels and fuel ratios strongly affect the structure, morphology, and emission characteristics of phosphors. Furthermore, The solution combustion method is widely employed due to its various advantages, including simple synthesis, energy efficiency, high purity, and good crystallinity [10, 11]. However, to date, the effect of urea fuel content on the structure and optical properties of HA/ β -TCP:Er/Yb/Li phosphors prepared via the solution combustion method has not been well documented.

In this work, we report the effect of urea fuel content on the structure and UC emission of HA/ β -TCP:Er, Yb, Li composite phosphors prepared via the solution combustion method, followed by heat treatment at 900 °C in air. Moreover, the effect of fuel content on the physicochemical properties of the phosphors is explained in detail.

2. Experimental

2.1. Material

Samp5Er/10Yb/5Li tri-doped HA/ β -TCP composite phosphors were synthesized via the solution combustion method using urea as the fuel. The fuel contents were 10, 20, 30, and 40 mol% relative to the Ca²⁺ content, and the corresponding samples were labeled S10, S20, S30, and S40, respectively. High-purity precursor materials, such as Ca(NO₃)₂ (Aldrich, 99.9%), NH₄H₂PO₄ (Aldrich, 99.9%), Er(NO₃)₃ (Merck, 99.99%), Yb(NO₃)₃ (Merck, 99.99%), LiCl (Aldrich, 99.9%), and urea were used to prepare UC phosphors.

2.2. Synthesis

The precursor salts were dissolved in deionized water to obtain aqueous solutions containing Ca^{2+} (1 M), H_2PO_4^- (1 M), Er^{3+} (0.05 M), Yb^{3+} (0.05M), and Li^+ (0.05M). The cation-mixing solution was prepared by mixing Ca^{2+} , Er^{3+} , Yb^{3+} , and Li^+ , followed by stirring for 45 min. Afterward, the H_2PO_4^- solution was slowly added to the cation solution, and the mixture was stirred for 1 h until a homogeneous solution was obtained. Next, urea with different contents (10, 20, 30, and 40 mol%) was added to the system, and the mixture was stirred at 80 °C for 4 h. Then, the mixed solution was subjected to a combustion reaction at 400 °C for 4 h in a corundum crucible [12]. Finally, the phosphor powder was annealed at 900 °C for 2 h.

2.3. Characterizations

The structural characteristics were analyzed by X-ray diffraction (XRD, Bruker D8 Advance). The surface morphology of the phosphors was determined using field-emission scanning electron microscopy (FE-SEM, JEM 1010, JEOL Techniques, Tokyo, Japan). The photoluminescence properties of the systems were characterized using a NANO LOG spectrophotometer (Horiba, USA) with a 975 nm laser diode (MDL-III-975-1W) as the excitation source.

3. Results and Discussion

3.1. Crystal Structure

Figure 1 shows the XRD patterns of the investigated samples with urea fuel contents ranging from 10 to 40 mol%, followed by annealing at 900 °C in air. As a result, most of the diffraction peaks correspond to the main hexagonal HA phase (PDF #024-0033), with the presence of a secondary β -TCP phase with an orthorhombic structure. The typical planes corresponding to the HA phase are clearly observed, such as (002), (211), (300), (1016), and (413). The diffraction peaks are intense, indicating a high degree of crystallinity in the phosphors. These observations could significantly affect the emission efficiency of the phosphors.

Notably, the phase percentages depend on the urea content (Table 1). The percentage of HA increases with increasing urea content, while the percentage of the β -TCP phase decreases. The mixed phase in the obtained phosphors form biphasic calcium phosphate (BCP), which combines the advantageous properties of HA and β -TCP and is widely used in biomedical applications [13,14]. The presence of the β -TCP phase could reduce content of quenching centers (OH^-) in HA, thereby contributing to the improved emission intensity of the phosphors [14]. The results confirm that the fuel content affects the degree of crystallinity. Furthermore, the degree of crystallinity was calculated to explain its influence on the optical properties of the phosphors, which can be expressed as [11]:

$$X_c = 1 - \frac{V_{112/300}}{I_{300}} \quad (1)$$

where the I_{300} and the $V_{(112/300)}$ correspond to the peak intensity of the (300) plane and the space intensity of the (112) and (300) reflections (this effect is not observed in noncrystalline samples) [11], the calculation results are shown in Table 2, indicating high crystalline of the obtained phosphors, as ranging of urea contents. Notably, the degree of crystallinity increases with increasing urea content from 88.08 to 91.40%, thereby significantly enhancing their emission efficiency [14, 15].

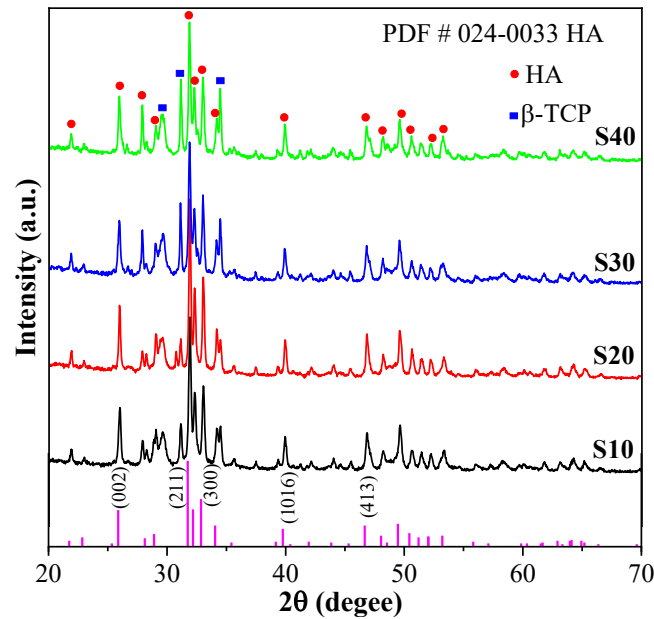


Figure 1. XRD pattern of all samples with different fuel contents.

Table 1. Phases percentage composition of all the samples

Samples	S0	S1	S2	S3	S4
% HA	58.2	65.1	70.9	79.4	82.4
% β -TCP	41.8	34.9	29.1	20.6	17.6

Table 2. Crystallinity degree of the HA phase of S10, S20, S30, and S40 samples

Sample	$2\theta^\circ$	Variable	Intensity	% X_C
S10	32.28	I_{300}	1973	88.08
	33.01	$V_{112/300}$	235	
S20	32.27	I_{300}	2286	89.76
	33.00	$V_{112/300}$	234	
S30	32.25	I_{300}	2030	90.98
	32.98	$V_{112/300}$	183	
S40	32.25	I_{300}	2528	91.40
	32.98	$V_{112/300}$	218	

3.2. SEM Images

Figures 2a-2d display the SEM images of samples with different fuel content and treatment at 900 °C in air. All samples show an agglomeration of spherical-like particles and a number of rod-like particles. Notably, with higher fuel content, the samples exhibit uniform grains with well-defined grain boundaries. In addition, the histogram distribution of grain size was analyzed, as presented in the set of all Figures. As a result, the average particle size increases from 118.7 to 306.6 nm with increasing urea content. The observations on morphology and particle size are significantly changing. Notably, the increased grain size of the sample led to a decrease in energy loss associated with photon scattering and absorption during light irradiation, thereby improving the emission efficiency [16].

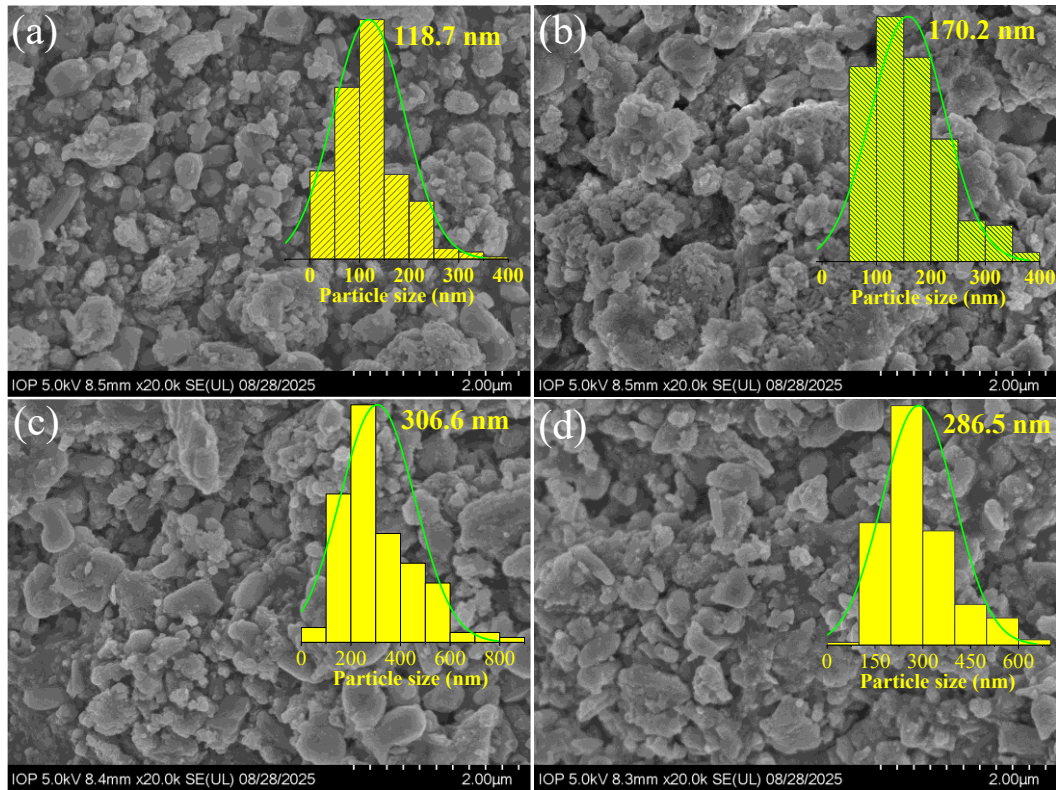


Figure 2. SEM images of all samples investigated.

3.3. UC Emission Properties

Figure 3a shows the photoluminescence spectra of samples S10, S20, S30, and S40 under 975 nm excitation. The phosphors exhibit strong green emission at 560 nm ($^4S_{3/2} \rightarrow ^4I_{15/2}$ transition) and weak red emission at 664 nm ($^4F_{9/2} \rightarrow ^4I_{15/2}$ transition). The emission intensity depends on the urea content and reaches a maximum value at 30 mol% urea (Figure 3b). This phenomenon is attributed to the increase in urea content, which lead to enhanced crystallinity, as confirmed by the XRD analysis (Figure 1) and the calculated (X_c) value (Table 2). Furthermore, the dominance of green emission was evaluated using the green-to-red intensity ratio (Figure 3c), which confirms the effect of fuel content on the selective enhancement of green UC emission. The change in the Commission Internationale de l'Eclairage (CIE) coordinates of all samples was investigated, as shown in Figure 3d, suggesting that the luminescence emission behavior of the phosphors is affected by the increasing fuel content (urea). When the urea content exceeds the optimal value, it may enhance energy transfer between Er^{3+} ions, promoting nonradiative relaxation (NR) and resulting in luminescence quenching [17].

Table 3. CIE coordinates and color purity values of all samples

Samples	Chromaticity coordinates (x, y)	Color purity (%)
S10	(0.346, 0.566)	59.21
S20	(0.328, 0.579)	62.44
S30	(0.338, 0.600)	67.78
S40	(0.326, 0.552)	55.60

Furthermore, the CIE coordinates of all samples are shown in Table 3 to confirm the effect of urea content on phosphor emission behavior. Moreover, the color purity was also calculated based on putting the CIE values (Table 3) into the equation [18]

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

with (x, y) is the color coordinates of the resulting samples, and $(x_i = 0.3333, y_i = 0.3333)$ and (x_d, y_d) are attributed to the color coordinates of the white light and the dominant wavelength, respectively [18]. As a result of Table 3, the color purity increases with the enhancement of urea contents, which is in good agreement with the PL emission observation in Figure 3a.

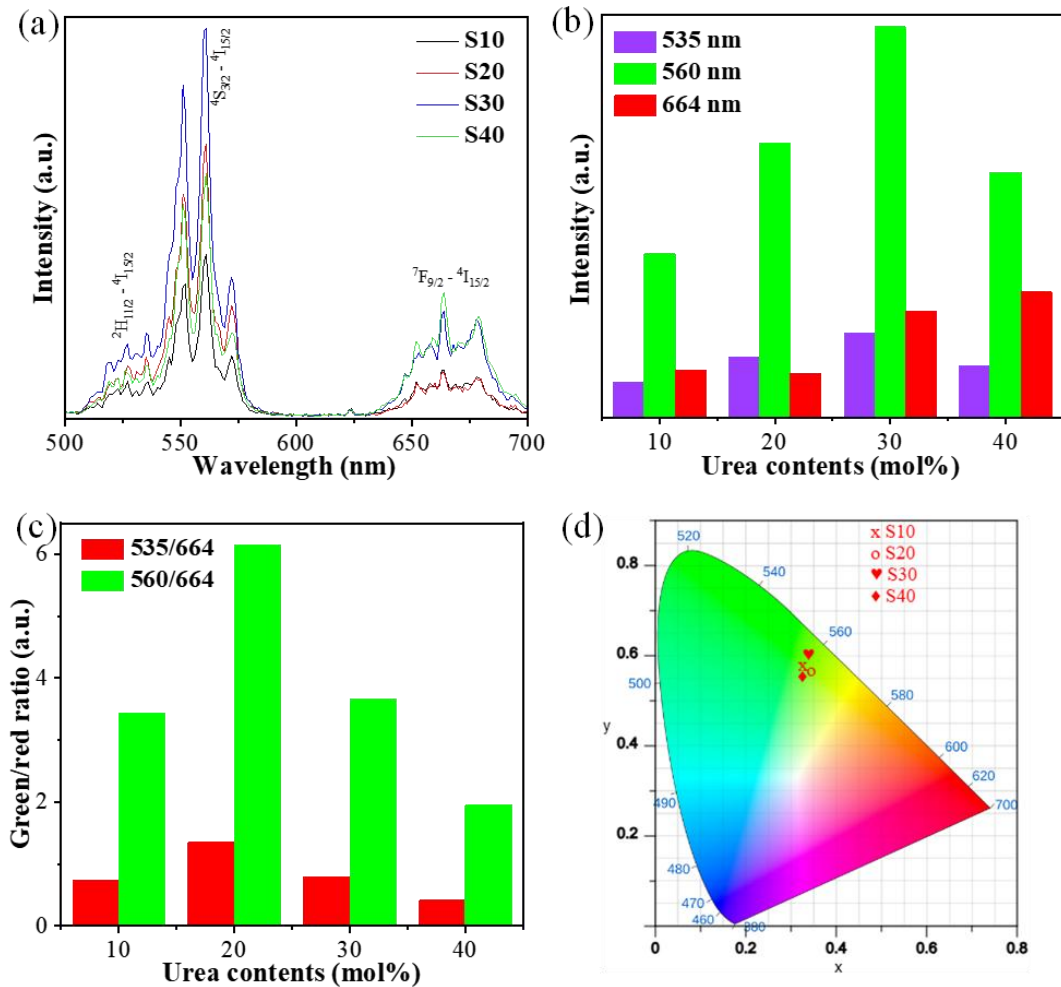


Figure 3. (a) UC emission spectra of all samples under excitation at 975 nm, (b) green and red emission intensity dependent on urea contents, (c) green–red intensity ratios of all samples, and (d) CIE coordination of phosphors.

3.4. UC Emission Mechanism

The mechanism of upconversion (UC) emission can be clarified by examining the dependence of UC emission intensity on pump power. In this study, the emission intensity was measured across a range

of laser powers from 200 mW to 600 mW. The relationship between UC emission intensity (I) and pump power (P) can be expressed as $I \propto P^n$, where n represents the number of photons involved in the UC process. Accordingly, based on the linear fitting results, the n values associated with the green emissions (535/560 nm) are 1.51 and 1.48, while that for the red emission (664 nm) are 1.39 (Figure 4a). These values are relatively close to 2, suggesting that the green and red UC emissions contributed to the two-photon absorption process. The green emissions indicate higher n values than red emissions, indicating that the green emission intensity increases more rapidly than red emissions as the pump power increased [19]. The values obtained were used to propose an energy-level diagram for evaluating the UC emission mechanism, as illustrated in Figure 4b.

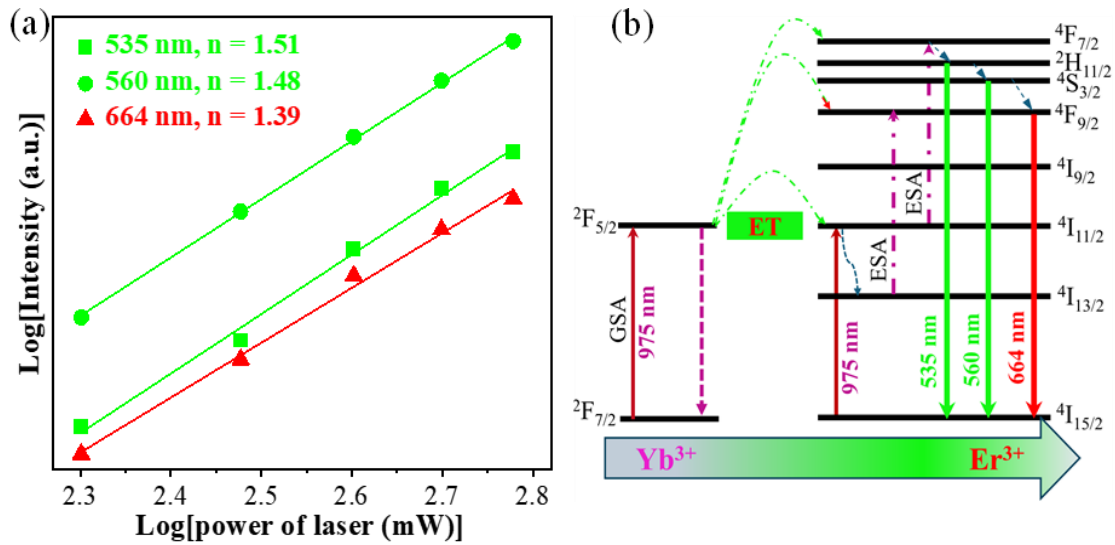


Figure 4. (a) The log-log plots of UC emission intensity as function of power of laser: (b) Energy levels diagram of UC phosphor.

Consequently, the proposed energy level diagram for the dominant green UC emission is shown in Figure 4b. The UC emission mechanism can be explained as follows: electron at the $^2F_{7/2}$ level (ground state) of Yb^{3+} can be moved to the $^2F_{5/2}$ level (excited state) by absorbing one photon, which via ground state absorption (GSA); notably, these energies at this level can resonate with excited energy levels of Er^{3+} (such as $^4I_{11/2}$, $^4F_{9/2}$, and $^4F_{7/2}$ levels). Thus, effective energy transfer from the Yb^{3+} ion to the Er^{3+} is obtained. After that, the electron in the $^4I_{11/2}$ level of Er^{3+} absorbs the second photon to move to the $^4F_{7/2}$ level via excited state absorption (ESA) [20]. It is known that, due to the instability of this state ($^4F_{7/2}$), these electrons go down from $^4F_{7/2}$ to $^2H_{11/2}$ and $^4S_{3/2}$ via the nonradiative relaxation process [21]-finally, those down to the $^4I_{15/2}$ level (ground state) to obtain strong green emission. Significantly, the $^4F_{7/2}$ and $^4F_{9/2}$ levels exhibit a substantial energy gap, leading to minimal nonradiative relaxation from $^4F_{7/2}$ to $^4F_{9/2}$ and weak red UC emission [22].

4. Conclusion

The intense green upconversion emission of Er/Yb/Li-doped HA/ β -TCP composite phosphors prepared by the solution combustion method was successfully investigated as a function of fuel content. XRD results confirm that the urea content influences the crystallinity and phase composition of the HA/ β -TCP mixture. Under 975 nm excitation, the phosphors show intense green UC emission at 560

nm and weak red UC emission at 664 nm, respectively. The emission intensity depends on the urea content and reaches its maximum at 30 mol%. This phenomenon is attributed to the higher crystallinity obtained at the optimal urea content. Overall, the phosphor shows intense green UC emission with a relatively uniform submicron particle size, making it suitable for biomedical applications.

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