Luminescence properties and energy transfer of Tb$^{3+}$, Eu$^{3+}$ co-doped YTaO$_4$ phosphors obtained via sol–gel combustion process

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Abstract

Tantalate is considered as a valuable and efficient luminescence host because of its intense absorption in the ultraviolet area and excellent chemical properties. In this work, a series of pure YTaO$_4$:Eu$^{3+}$ and/or Tb$^{3+}$ crystals were prepared via a sol–gel combustion method. The morphology, structure, and optical properties of the samples were discussed in detail. The Eu$^{3+}$, Tb$^{3+}$ co-doped YTaO$_4$ samples are consisted of small spherical particles of around 18 nm. The prepared YTaO$_4$:Tb$^{3+}$ and/or Eu$^{3+}$ samples exhibit the characteristic wide excitation band around 210–300 nm, the characteristic narrow red emission of Eu$^{3+}$ (5D$_{0}$ → 7F$_{2}$) transitions and green emission of the Tb$^{3+}$ (5D$_{4}$ → 7F$_{5}$) transitions when excited by UV light. It is focused on the energy transfer processes from the YTaO$_4$ to Tb$^{3+}$ as well as Eu$^{3+}$ ions and from Tb$^{3+}$ to Eu$^{3+}$ ions of YTaO$_4$:Eu$^{3+}$/Tb$^{3+}$ phosphors. Color-tunable emissions are realized through adjusting the types of rare earth ion (Eu$^{3+}$ and Tb$^{3+}$) and relative doping concentrations excited by a single wavelength. That is to say, the obtained Tb$^{3+}$ and Eu$^{3+}$ co-doped YTaO$_4$ phosphors have a promising prospect in lasers, white light diodes (WLED), fluorescent lamp, and field emission display devices, etc.

1 Introduction

At present, owing to the advantages of friendly environment, nice color rendering index and excellent luminescent performance, rare earth active oxide phosphors have been got quite attention in imaging, lighting, display, and other fields. In general, high temperature solid-state method is a common way to synthesize rare earth activated oxide phosphors. Unfortunately, the traditional solid reaction route is not beneficial to control the particle size and separation of components, and may lead to other problems such as long reaction times and stoichiometric losses caused by evaporation at high temperature in general [1]. Therefore, it is still necessary to develop some timesaving and low temperature methods to synthesize the target product such as the hydrothermal method, supersonic microwave co-assistance method, sol–gel method[2–6]. Among these routes, sol–gel combustion method is a good choice because it can obtain stable precursors and nanometer phosphors with homogeneous composition and high purity. Compared with bulk materials, nanophosphors have a large number of surface atoms. Therefore, the interaction between nanophosphor and the incident UV light is much higher than that of its bulk counterpart. Additionally, nanophosphors have the advantages of better absorption, high conversion efficiency, and high color rendering [7–11]. All of these advantages of reduced dimension phosphors make them easier to use in modern applications [12].

Compared with the common LED used for lighting, the deep UV LED with the wavelength of 240–280 nm has the outstanding advantages of safety, environmental protection, cold light source, pure light quality, high efficiency, rich wavelength type, easy to compound spectral energy, convenient modulation, etc., which is much suitable for civil use. In addition to being a common germicidal lamp, deep UV LED has been widely used in medical and health fields [13, 14]. At the same time, with the outbreak of the coronavirus, the research on UV-C (280–200 nm) LED with bactericidal effect is more and more urgent. Recently, rare earth ions doped tantalate phosphors have received considerable concern for their important applications in lighting fields [15–19]. YTaO$_4$ is known as an excellent chemical and photoactive host material in virtue of its luminescence
performance, high density, high irradiation hardness, strong X-ray absorption, and stable physical and chemical properties [20]. YTaO₄ has two crystal phases, one is tetragonal phase (T), namely scheelite structure, with space group I41/a [21], the other is monoclinic phase, including the pyrite phase (M) with I2/a space group and the prime phase (M′) with P2/a space group [22, 23]. The difference of the two structures is essentially on account of the different coordination number of atoms. With M structure, Ta atoms occupy tetrahedral lattice, while in M′ structure, Ta atoms exist in twisted octahedral structure [24, 25]. As reported, Rare earth-doped YTaO₄ crystal with M′ structure can present the charge transfer process more effectively and provide splendid luminescent emission [26–28].

Eu³⁺ along with Tb³⁺ ions doped phosphors have been extensively studied on account of their ideal luminescent properties [29, 30]. Besides, based on energy transfer mechanism, the phosphors doped with Eu³⁺/Tb³⁺ ions can realize tunable multicolor emission. Particularly, thanks to the ⁵D₀ → ⁷F₁,₂ transitions, the Eu³⁺-doped phosphors can emit red light [31–37]. Tb³⁺ can be used as both activator and sensitizer to achieve green light emission due to its ⁵D₄ → ⁷F₅ transitions at approximately 547 nm [38, 39] and improve the emission intensity of activator ions (Eu³⁺). In addition, Tb³⁺ ions also broaden the UV–visible absorption region when Tb³⁺ along with Eu³⁺ ions co-doping in some hosts owing to the existence of more level transitions [40–43]. It means that owing to the effective energy transfer, the characteristic emission light of Tb³⁺ and Eu³⁺ ions can be adjusted to obtain color-tunable phosphors excited by ultraviolet light. Moreover, the strong excitation band in the ultraviolet region of YTaO₄ host can effectively transfer absorbed energy to the doped activators. Thereby, it allows the energy to be more fully transmitted to rare earth ions to improve the fluorescence efficiency of the phosphors. Hence, we focus on synthesizing color adjustable phosphors by doping Tb³⁺ and Eu³⁺ in YTaO₄ host.

In this work, we prepared the phosphors with the composition of YTaO₄:Eu³⁺/Tb³⁺ by sol–gel combustion method. Photoluminescence properties, energy transfer, and adjustable color were studied closely. Due to the excellent luminescence properties and colorimetric properties of YTaO₄:Ln³⁺ (Ln³⁺ = Eu³⁺ and/or Tb³⁺) samples, there will be potential phosphors used in the fields of display and lighting.

2 Experimental section

2.1 Materials

Yttrium oxide (Y₂O₃) (99.99%), Terbium oxide (Tb₂O₇) (99.99%), Europium oxide (Eu₂O₃) (99.99%), Tantalum oxide (Ta₂O₅) (99.99%), Citric acid (CA) (Analytical reagent, AR), Nitric acid (HNO₃), Ammonia, Ammonium nitrate (NH₄NO₃) (AR) were used as starting materials. The reagents we used were analytical grade and utilized as purchased without further purification.

2.2 Preparation

The corresponding quantities of Y₂O₃, Eu₂O₃, and Tb₂O₇ were put into dilute HNO₃ solution, stirred, and heated in ambient atmosphere, and then after evaporation, crystallization, and cooling, the obtained rare earth nitrate crystals were finally configured into the corresponding aqueous solutions to obtain 0.1 mol L⁻¹ Y(NO₃)₃, 0.05 mol L⁻¹ Eu(NO₃)₃ and 0.05 mol L⁻¹ Tb(NO₃)₃ solutions. Eu³⁺-, Tb³⁺-doped YTaO₄ nanocrystals were obtained by sol–gel combustion method. The typical steps for preparing YTaO₄:0.07Tb³⁺, 0.02Eu³⁺ are as follows: First, 0.5 mmol of Ta₂O₅ was completely dissolved in HF acid (40%), and then ammonia solution (25%) was added until the pH equals to 9. The obtained precipitate was centrifuged and washed with deionized water to get rid of the redundant F⁻, and then dissolved in citric acid aqueous solution at 50 °C. Subsequently, 0.97 mmol Y(NO₃)₃, 0.01 mmol Eu(NO₃)₃, 0.02 mmol Tb(NO₃)₃ and 1.20 g of NH₄NO₃ were added into the above solution. Under vigorous stirring at 80 °C, a high viscosity gel with pale-yellow was formed. Further, the gel was heated at 180 °C for 3 h to get a highly condensed porous network dry gel. After that, it was transferred to the muffle furnace and heated to 500 °C for 3 h. Finally, grind the solid taupe mass obtained in the previous process into a fine powder and put it into the alumina crucible then for heat treatment at 1000 °C for 5 h. The target phosphors were obtained and used for further characterization. The synthesis steps of other samples are the same as that of YTaO₄:0.07Tb³⁺, 0.02 Eu³⁺ samples except for doping different concentrations of rare earth ions.

2.3 Characterization

X-ray diffraction (XRD) measurements were carried out on a Rigaku-Dmax-RA diffractometer using Cu Kα radiation (λ = 0.15406 nm), operating at a scanning speed of 10° min⁻¹ in the 2θ range from 10° to 90°. The morphologies of the samples were analyzed using a FEI-30 field emission scanning electron microscope (FE-SEM) equipped with an energy dispersive X-ray spectrometer (EDX). The photoluminescence measurements were carried out on a HITACHIF-7000 spectrophotometer equipped with a 150-W Xenon lamp as the excitation source. All the measurements were carried out at room temperature.
3 Results and discussion

3.1 Phase identification, structure, and morphology analysis

The XRD patterns of YTaO₄:Eu³⁺/Tb³⁺ samples synthesized via sol–gel combustion method are shown in Fig. 1a. We can see that all measured peak positions match with YTaO₄ standard card (PDF# 24-1425) properly. The obtained YTaO₄ powder is a monoclinic structure (M’ structure) with P2/a space group and the lattice parameters are \(a = 5.29\), \(b = 5.45\), \(c = 5.11\) Å, and \(Z = 2\). No any other impure phase is detected, suggesting that the replacement of Y³⁺ ions by Eu³⁺ and Tb³⁺ions does not affect the purity of the YTaO₄. This is because the radii of Y³⁺ (\(r = 0.908\) Å), Tb³⁺ (\(r = 0.923\) Å) and Eu³⁺ (\(r = 0.947\) Å) ions are similar, and all three ions have the same coordination number (CN = 8). Figure 1b shows the lattice structure as well as atomic coordination of YTaO₄. We can see that each tantalum ion and yttrium ion are encircled by six and eight oxygen ions in the YTaO₄ M’-type structure, respectively, to form a distorted octahedron (TaO₆) and a twisted cube (YO₈).

The morphology and elemental component of YTaO₄:0.07Tb³⁺, 0.02Eu³⁺ sample are characterized by FE-SEM and EDX, respectively. Figure 2a illuminates that the phosphors are uniform nanoparticles with a small number of agglomerates and the particle size is about 18 nm, seen from the size distribution histogram of the samples.

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Fig. 1  a XRD patterns of the as-prepared YTaO₄:Tb³⁺, YTaO₄:Eu³⁺ and YTaO₄:0.07Tb³⁺, 0.02 Eu³⁺ samples, and the corresponding standard data of YTaO₄ (PDF No. 24-1425) are given as reference, b the crystal structure of YTaO₄ host

Fig. 2  a FE-SEM image and b EDX spectrum of YTaO₄:0.07Tb³⁺, 0.02 Eu³⁺ sample. Inset of a is the size distribution histogram

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3.2 Luminescence properties

The photoluminescence excitation (PLE) and photoluminescence (PL) spectra of Tb3+ singly activated YTaO4 samples are presented in Fig. 3a. When monitored at 547 nm (5D4 → 7F5 transition of Tb3+), there is a strong broadband around 210–300 nm with a maximum at 270 nm in PLE spectrum, corresponding to the charge transfer band of Tb3+-O2− and O2−–Ta5+ in TaO43− group [13], which lead to better luminescent properties of Tb3+ ions in YTaO4 host for UV excitation [44]. It also indicates that the existence of energy transfer between the host lattice (TaO43− group) and the active centers (Tb3+ ions) [45]. In addition, the f-f transitions of Tb3+ ions from the ground state (7F6) to the 5D2, 5L10, 5G6, 5D3, 5D2, 5D1 states result in several sharp PLE peaks ranged from 320 to 380 nm [46, 47]. Upon 270 nm excitation, the representative emission peaks at 492 nm, 547 nm, 585 nm, and 621 nm in the PL spectrum are corresponded to 5D4 → 7F5, 4, 3, 2 transitions of Tb3+ ions, respectively [48]. The strongest emission peak of YTaO4:0.07Tb3+ phosphor situates at 547 nm in the green area. The PL spectra of the YTaO4:xTb3+ phosphors under 270 nm excitation illuminate that the luminescence intensity of Tb3+ reaches maximum at x = 0.07. When Tb3+ concentration exceeds 0.07, the emission intensity decreases owing to the concentration quenching effect [49, 50].

The PLE and PL spectra of the Eu3+ singly activated YTaO4 phosphor are illustrated in Fig. 4. When 613 nm (5D0 → 7F2 transition) is used as monitoring wavelength, the PLE spectrum consists of a broad band of 210—300 nm centered at 258 nm and a few of weak sharp peaks ranging from 300 to 450 nm, which are corresponded to the charge transfer band transitions of Eu3+-O2− and O2−–Ta5+ [51–53] and the f-f transitions of Eu3+ from the ground state (7F0) to the 5H6 (321 nm), 5D4 (362 nm), 5G4 (384 nm), 5L6 (395 nm), 5D3 (418 nm), 5D2 (466 nm) and 5D1 (537 nm) states [54, 55]. At 258 nm excitation, in PL spectrum, we can see the main peaks at around 578, 593, 613 and 656 nm, which correspond to the 5D0 → 7F0, 5D0 → 7F1, 5D0 → 7F2 and 5D0 → 7F3 transitions of Eu3+ [56]. In addition, the symmetry of the doping position of Eu3+ directly affects the emission intensity of 5D0 → 7F1 and 5D0 → 7F2. If the Eu3+ ion has a site with inversion symmetry, the 5D0 → 7F1 (593 nm) transition dominates, while if the Eu3+ ion holds a site with non-inversion symmetry, the 5D0 → 7F2 transition predominates. In our study, the peak at 613 nm (5D0 → 7F2) is the strongest, which shows that Eu3+ ions locate at non-inversion symmetry center in YTaO4 [57]. It can be concluded that the luminescence intensity reaches maximum at y = 0.02 from the PL spectra of the YTaO4:yEu3+ (y = 0.005–0.04) phosphors under 258 nm excitation. When Eu3+ concentration exceeds 0.02, the emission intensity reduces owing to the concentration quenching effect [38].

To obtain the multicolor tunable emission and further prove the possibility of energy transfer process of the YTaO4:Tb3+, Eu3+ phosphors, a series of YTaO4:xTb3+, 0.02Eu3+ (x = 0.01, 0.03, 0.05, 0.07, 0.09) phosphors were synthesized. The emission spectra of YTaO4:xTb3+, 0.02Eu3+ (x = 0.01, 0.03, 0.05, 0.07 and 0.09) phosphors excited by 270 nm are illustrated in Fig. 5, which shows the variation of luminescence intensity and the intense characteristic emission peaks of Tb3+ and Eu3+ ions. Because
of the concentration quenching, the emission intensity of Tb$^{3+}$ increases first and then decreases with the increase of Tb$^{3+}$ content. Although the concentration of Eu$^{3+}$ is constant, the emission of Eu$^{3+}$ (613 nm) increases gradually in a certain range with the increase of the Tb$^{3+}$ content, which due to the energy transfer from Tb$^{3+}$ to Eu$^{3+}$ ions, but then the emission of Eu$^{3+}$ (613 nm) decreases when Tb$^{3+}$ content is from 0.05 to 0.09, which due to the concentration quenching of Eu$^{3+}$, the results prove that Tb$^{3+}$ can transfer energy to Eu$^{3+}$ [30].

To further understand the energy transfer process between Tb$^{3+}$ and Eu$^{3+}$, the emission spectra of YTaO$_4$:0.07Tb$^{3+}$, yEu$^{3+}$ (y = 0.01, 0.02, 0.03, 0.04, 0.05) samples under 270 nm excitation are shown in Fig. 6. In the emission spectra of YTaO$_4$:0.07Tb$^{3+}$, yEu$^{3+}$ phosphors, we can observe the characteristic sharp peaks of Eu$^{3+}$ and Tb$^{3+}$ ions. As there is increase in doping concentration of Eu$^{3+}$ (y), the emission intensity of Eu$^{3+}$ continues to rise until y = 0.03, afterward the characteristic emission intensity of Eu$^{3+}$ begins to decrease. In addition, when the doping concentration of Eu$^{3+}$ goes up, the emission intensity of Tb$^{3+}$ declines as a result of the energy transfer from Tb$^{3+}$ to Eu$^{3+}$ in YTaO$_4$ host [42].

Figure 7 shows the fluorescence lifetime of YTaO$_4$:0.07Tb$^{3+}$, yEu$^{3+}$ (y = 0, 0.01, 0.02, 0.03, 0.04, 0.05). Test data can be fitted and processed according to the following formula:

$$I = I_0 + Ae^{-t/\tau}$$

Here I corresponds to the fluorescence intensity at $t$, $I_0$ corresponds to the fluorescence intensity of the $t = 0$, and $\tau$ is on behalf of the fluorescence lifetime. The fluorescence lifetime of Tb$^{3+}$ are 2.42, 1.73, 1.72, 1.54, 1.36 and 1.23 ms for y = 0, 0.01, 0.02, 0.03, 0.04, 0.05, respectively. With the increase of Eu$^{3+}$ doping concentration, the fluorescence lifetime of Tb$^{3+}$ decreases gradually as we can obviously see, which further proves that Tb$^{3+}$ transfers energy to Eu$^{3+}$ ions.

The energy transfer efficiency could be roughly calculated by the related luminescence intensities as the following equation:
Here $I_s$ and $I_{s0}$ correspond to the luminescence intensity of Tb$^{3+}$ ions with and without Eu$^{3+}$ ions, respectively. Figure 8 shows that the energy transfer efficiency increases gradually till about 86% of phosphors with the increase of Eu$^{3+}$ content, indicating that the effective energy transfer between Tb$^{3+}$ and Eu$^{3+}$ is occurred in YTaO$_4$ host.

According to the energy transfer theory of Dexter and Reisfeld, the possible mechanism can be analyzed by the following formula [58]:

$$I_{so}/I_s \propto C^{n/3}$$

where $I_{so}$ and $I_s$ are the luminescence intensity of Tb$^{3+}$ with or without Eu$^{3+}$, respectively. $C$ is the sum concentration of Tb$^{3+}$ and Eu$^{3+}$, and $n = 6, 8, 10$ are corresponding to the dipole–dipole, dipole–quadrupole, and quadrupole–quadrupole interactions, respectively. Plots of the value of $I_{so}/I_s$ and $C^{n/3}$ are depicted in Fig. 9. It is found that when $n = 8$, the fitting result is the best, indicating that the energy transfer mechanism is dipole-quadrupole interaction.

### 3.3 Energy transfer mechanism

Figure 10 describes the energy levels and energy transfer mechanism from Tb$^{3+}$ to Eu$^{3+}$ in YTaO$_4$ host which explain the polychromatic luminescence mechanism of YTaO$_4$:Tb$^{3+}$, Eu$^{3+}$ clearly. For YTaO$_4$:Tb$^{3+}$, Eu$^{3+}$ phosphors, both Tb$^{3+}$/Eu$^{3+}$–O$^{2-}$ charge transfer band and TaO$_4$$^{3-}$ group make contributions for the emissions of Tb$^{3+}$ and Eu$^{3+}$ [13]. In the whole process of excitation, electrons are transferred from O$^{2-}$ valence to the Tb$^{3+}$ and Eu$^{3+}$ conduction band upon the excitation of ultraviolet light. Subsequently, the electrons transfer to $^5D_3$ state of Tb$^{3+}$ ions and $^5L_6$ of Eu$^{3+}$ ions, respectively. As is known to all, electrons in excited state are unstable. Therefore, the electrons of Tb$^{3+}$ ions relax from $^5D_3$ to $^5D_4$ level through non-radiative transition, then transmit to $^7F_6$, $^7F_5$, $^7F_4$ and $^7F_3$ energy level, leading to the emission of 492 nm, 547 nm, 585 nm, and 621 nm, respectively. Similarly, the electrons of Eu$^{3+}$ relax from $^5L_6$ to $^5D_0$ state, then go back to $^7F_1$ and $^7F_2$, corresponding to the emission of 593 nm and 613 nm, respectively. In addition, the transitions from ground state $^1A_1$ of TaO$_4$$^{3-}$ group to excited levels take place and TaO$_4$$^{3-}$ group transfers energy to Tb$^{3+}$ and Eu$^{3+}$ ions leading to the characteristic emission of Tb$^{3+}$ and Eu$^{3+}$ ions. Meanwhile, the characteristic emission of
Eu³⁺ ions can be checked, owing to that some electrons of Tb³⁺ ions make a non-radiative transition from 5D₄ to the 5D₀ of Eu³⁺ ions. Due to the manageable energy transfer efficiency, the YTaO₄:Tb³⁺, Eu³⁺ samples upon excitation of ultraviolet light can show the Tb³⁺ and Eu³⁺ emissions with different intensities at the same time. Consequently, the emission color of phosphors is adjustable.

3.4 Adjustable color

The luminescence properties of YTaO₄:0.07Tb³⁺ (1), YTaO₄:0.07Tb³⁺, yEu³⁺ (y = 0.01(2), 0.02 (3), 0.03 (4), 0.04 (5) and 0.05(6)) and YTaO₄:0.02Eu³⁺ (7) samples upon 270 nm excitation have been investigated in detail, and the corresponding CIE chromaticity diagrams are shown in Fig. 11. It is worth mentioning that the CIE chromaticity coordinate of YTaO₄:0.07Tb³⁺ locates in the green emission zone with 1 point (0.3119, 0.5294). The emissive color of YTaO₄:0.02Eu³⁺ is in the red emission zone and the CIE chromaticity coordinate is calculated to be (0.4165, 0.2909) (7 point). With the change of doping amount of Eu³⁺ ion, the YTaO₄:0.07Tb³⁺, yEu³⁺ (y = 0.01–0.05) phosphors can achieve color adjustable emission from green to yellow and the corresponding CIE chromaticity coordinates are calculated to be (2, 0.368, 0.5176), (3, 0.3864, 0.5142), (4, 0.3854, 0.4838), (5, 0.2634, 0.5935), (6, 0.3754, 0.4225). The results show that multi color luminescence can be achieved through adjusting the Eu³⁺/Tb³⁺ ratio.

4 Conclusions

In summary, Eu³⁺-and/or Tb³⁺-doped M'-YTaO₄ phosphors were prepared by a sol–gel combustion method, which needs lower temperature than before. Under excitation at 270 nm, due to the typical 5D₄→7F₅ transition of Tb³⁺, the YTaO₄:Tb³⁺ phosphors show characteristic green emission. Meanwhile, the PL intensity is strongly dependent on the Tb³⁺ doping concentration and the optimal doping concentration is determined to be 0.07. YTaO₄:Eu³⁺ phosphors show excellent red emission on account of the 5D₀→7F₁,₂ transitions. The emission color of the samples is transformed from green to red by adjusting the doping ratio of Tb³⁺ and Eu³⁺. The energy transfers from YTaO₄ host to Tb³⁺ and Eu³⁺ and Tb³⁺–Eu³⁺ are systematically discussed. The energy transfer efficiency of Tb³⁺–Eu³⁺ in the YTaO₄:0.07Tb³⁺,0.05Eu³⁺ phosphor can reach up to about 86%. From the above experimental results, it can be concluded that the YTaO₄:Tb³⁺, Eu³⁺ phosphors have a broad application prospect in phosphor conversion LEDs.

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Compliance with ethical standards

Conflict of interest There are no conflicts of interest to declare.

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