



## VUV PROPERTIES OF $\text{Eu}^{3+}$ - DOPED $\text{YBO}_3$ PHOSPHOR PREPARED VIA ALDO-KETO AND SOLID-STATE PROCESS

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### ABSTRACT

The  $\text{Eu}^{3+}$  doped  $\text{YBO}_3$  was prepared by the novel aldo-keto method. The structure and VUV luminescence properties of the title compound were studied and compared to the corresponding properties of the materials prepared by a conventional solid state reaction. The use of novel aldo-keto method in preparation lowered the reaction threshold temperature by c.a. 300°C. The  $\text{YBO}_3$  phosphor from the aldo-keto method look like evenly sized spherical structures, whereas those from the solid-state process look like some agglomerates of little spheres. The  $\text{Eu}^{3+}$ -doped  $\text{YBO}_3$  phosphors prepared by both methods showed the red emission with peak around 592, 611 and 627 nm at excitations wavelengths 147 and 172 nm, which corresponds to the transitions from the excited  $^5\text{D}_0$  level to the  $^7\text{F}_j$  ( $j = 1, 2, 3, 4$ ) levels of  $\text{Eu}^{3+}$  activators. However, the emission intensity of the  $\text{Eu}^{3+}$  doped  $\text{YBO}_3$  from the aldo-keto system is about 2 times as much as that from the solid-state process.

### KEYWORDS

Aldo-keto; solid-state; VUV luminescent; Yttrium borates

### Academic Discipline And Sub-Disciplines

Physics; VUV luminescence Materials.

### SUBJECT CLASSIFICATION

Physics

### TYPE (METHOD/APPROACH)

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## 1. INTRODUCTION:

Rare-earth doped  $\text{YBO}_3$  have been studied widely due to their excellent luminescent properties of high vacuum ultraviolet (VUV) transparency, exceptional optical damage thresholds, chemical and environmental stability [1-5].  $\text{Eu}^{3+}$ -doped  $\text{YBO}_3$  has been also widely used as a red color material for plasma display panels (PDP) because of its high quantum efficiency and good color coordinates under 147 nm VUV irradiation [4–6]. Thus, phosphors based on rare-earth doped  $\text{YBO}_3$  are demanded for the development of plasma display panels (PDP) and Hg-free fluorescent lamps [7, 8]. Various methods have been used to prepare  $\text{LnBO}_3$  phosphor materials, for example, conventional solid-state reaction [1, 6, 9], co-precipitation [10, 11], microwave heating [12], spray pyrolysis [13], sol-gel [10], combustion [14], and hydrothermal method [15–17]. The phosphor of  $\text{YBO}_3:\text{Eu}^{3+}$  has been produced through a high temperature solid state method in industry for many years. Although this method was proved to be an effective technique in large scale production in industry, there are some shortcomings in the high temperature solid-state reactions. The solid state reactions are involved in several process steps as raw materials blending, high temperature calcinations and milling. High calcination temperature for long time can make the phosphor particles easily agglomerated to form a particle block, therefore, milling and grinding are required to achieve desirable particle size [18]. However, the surface of phosphors suffers from damage in milling and grinding processes. Recent studies indicated that the penetration depth of VUV is about 100–200nm consequently, the surface damage resulting from grinding and milling processes will significantly degrade the luminescence properties of phosphors. Furthermore, due to the limitation of solid-state route, the resulting product prepared by this method is very difficult to achieve compositional homogeneity and the doped ions are not able to be uniformly dispersed in the crystal lattice, thus, several impurities are more likely to be introduced and the luminescence efficiency is lowered. For these disadvantages of solid-state method, several solution-based synthesis techniques, such as sol-gel, co-precipitation, spray pyrolysis and hydrothermal method have been developed. Since the starting materials are reacted at high level in solution-based synthesis, the resulting products exhibit better purity and high-degree compositional homogeneity. In this work, we report a novel chemical route for the preparation of phase-pure porous  $\text{YBO}_3$  using an aqueous solution of metal nitrate, benzaldehyde and acetone. In this process, aldehyde and ketone are used as a complexing agent to keep Y ions in homogeneous solution. The process involves dehydration of a Y ion–aldoketo complex solution, followed by the decomposition of organic substrate. Complete dehydration of the complex solution to dryness produces a black precursor mass. Heat treatment of the dried precursor mass in a furnace air environment results in a voluminous, crushable, highly fragile phase-pure  $\text{YBO}_3$  powder. In this process, aldehyde and ketone is used as a polydentate ligand that decomposes exothermally at a low ignition temperature (500°C), leaving behind only a small amount of carbon residue. Decomposition of reaction mixture generates excess heat and huge amounts of gases that help to produce the porosity in the final product. The  $\text{YBO}_3:\text{Eu}^{3+}$  derived from low temperature synthesis methods i.e. aldo-keto (AK) were investigated and compared with those of the high temperature methods i.e. solid state diffusion (SSD). The prepared phosphors were investigated under high-energetic VUV excitation using synchrotron radiation as a light source [19]. The effect of the preparation conditions on the morphology and VUV excited luminescence of the phosphors was notable.

## 2. EXPERIMENTAL

### 2.1 Solid State Diffusion

All the chemical reagents were analytically pure and used without further purification. Stoichiometric proportions of raw materials namely, Yttrium Oxide ( $\text{Y}_2\text{O}_3$ ), Europium Oxide ( $\text{Eu}_2\text{O}_3$ , 1 mole %) and Boric acid ( $\text{H}_3\text{BO}_3$ ) of assay 99.9% were used as starting materials and grinded in an agate motor and pestle, mixed and compressed into a alumina crucible with oven dried at 40°C for 1 h. The mixture was heated in a resistive furnace at different elevated temperature (400, 600, 800 and 1000°C) for 2 h at each step with intermittent grindings. The white powder of  $\text{YBO}_3:\text{Eu}^{3+}$  so obtained was used for characterization.

### 2.2 Alko-Keto Method

The phosphor  $\text{YBO}_3:\text{Eu}^{3+}$  was prepared for the first time by a novel method of gelation named as aldo-keto gel method; offering a comparatively low temperature route. The starting chemicals  $\text{Y}(\text{NO}_3)_3$ ,  $\text{Eu}(\text{NO}_3)_3$  (1 mole %) were mixed together in a china clay basin in stoichiometric amounts. A clear solution of nitrates was obtained by adding small quantity of double distilled water. To the solution of metal nitrates a solution of  $\text{H}_3\text{BO}_3$  (AR) in water was added to obtain the homogeneous mixture. Acetone (2M, AR) and benzaldehyde (2M, AR) were added to the nitrate. The aldehyde and ketone were introduced as a novel chelating agent. Then the pH was adjusted to 1 by drop wise addition of diluted nitric acid for 4 h. The pale brownish yellow mixture obtained was stirred continuously and slowly heated to 130°C. Due to the addition of dilute Nitric acid aldehyde and ketone underwent oxidative decomposition and the –OH and –COOH groups of the decomposed products promote binding of Ytria ions. Subsequent heating at 130°C for 2 h on a hot-plate stirrer caused the mixture become dark brownish yellow and then dark reddish brown with evolution of brownish gases. The process of gelation started at near about 130°C with the evolution of dark yellowish brown fumes. The mixture was then allowed to cool. Red gel was formed after cooling. It was further heated slowly to 300°C to obtain a dark red foam mass with evolution of yellowish brown fumes. On further slow heating, pyrolysis of foam was started at 450°C and shining black foam was formed at 450°C. During the calcination process, the polymeric matrix was decomposed to gases such as  $\text{CO}_2$  and  $\text{H}_2\text{O}$  accompanied with large amount of heat. These produced gases prevent the agglomeration of calcined powder. During calcination, existed carbon in the black precursor powder was burned out and a white colour powder was formed. Fig. 1 shows detail of  $\text{Y}_{0.99}\text{BO}_3:\text{Eu}^{3+}_{0.01}$  powder synthesis in a flow chart.

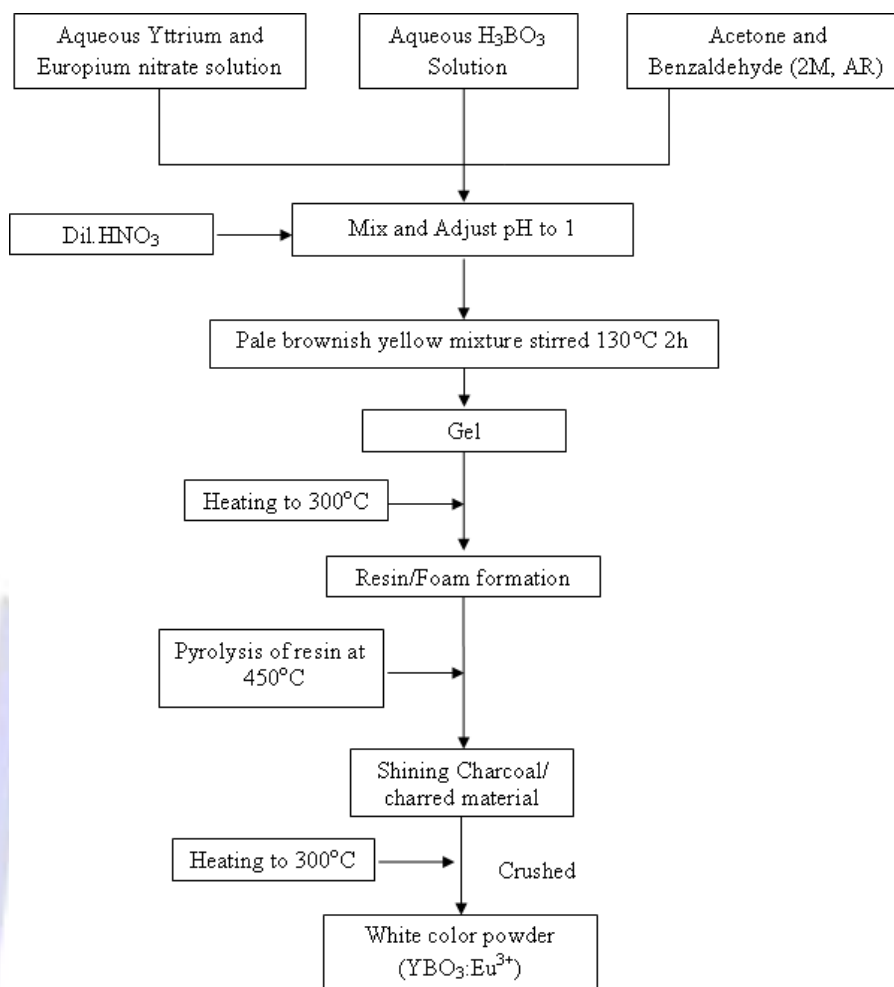


Fig. 1: Flow chart of  $Y_{0.99}BO_3:Eu^{3+}$  synthesized by Aldo-Keto method

### 3. CHARACTERISATION

The X-ray diffraction (XRD) pattern of host sample of  $YBO_3:Eu$  was recorded on Rigaku miniflex X-ray diffractometer with scan speed 2.000 deg/min and with  $Cu\ K\alpha$  radiation. The morphology of the phosphor particles was studied by using Hitachi model S-4800 type-2 field emission scanning electron microscope and elemental analysis by Bruker EDS. The VUV spectra were recorded at Department of Physics S.G.B. Amravati University, Amravati by using remote access mode of Beamline 4B8 in Beijing synchrotron radiation facilities (BSRF) [20] under dedicated synchrotron mode (2.5 GeV, 150-60mA). A 1 m Seya monochromator (1200 g/mm, 120–350nm, 1 nm bandwidth) was used for the synchrotron radiation excitation spectra measurement, and an Acton SP-308 monochromator (600 g/mm, 330–900nm) was used for the emission spectra measurement. The signal was detected with a Hamamatsu H8259-01 photon counting unit. The vacuum in the sample chamber was about  $1 \times 10^{-5}$  mbar. The effect of the experimental set-up response on the relative VUV excitation intensities of the samples were corrected by dividing the measured excitation intensities of the samples with the excitation intensities of sodium salicylate ( $\sigma-C_6H_4OHCOONa$ ) measured simultaneously in the same excitation conditions. The region of excitation spectra was from 100 nm to 300 nm and the emission spectra recorded under 147 and 172 nm excitation.

### 4. RESULT AND DISCUSSION:

#### 4.1 X-ray diffraction

The formation of the crystalline phase of as-prepared products of solid state diffusion and aldo-ketto method was confirmed by X-ray diffraction patterns of  $YBO_3$  (as shown in Figure 2) to verify the phase purity and crystal structure. The X-ray pattern of both method samples indicated a pure phase of the standard  $YBO_3$  and all the peaks are in good agreement with the (ICDD, 00-016-0277). There were no additional peaks found as the concentration of Eu ion was increased to 5%. Thus it seen that pure phase of  $YBO_3$  could be achieved using Aldo-keto method at low temperature compared to solid state diffusion (SSD) synthesis, which require higher temperature and time for synthesizing  $YBO_3$ . The high intensity peaks i.e., 20.16, 27.24, 34.11, 48.15, 49.19, and 52.68 corresponding to (0 0 2), (1 0 0), (1 0 2), (1 0 0), (1 0 4) and (1 1 2) respectively from ICDD file shows exact matching with the XRD pattern of phosphor prepared by Aldo-



keto method. This agreement indicates that the phosphor has been successfully prepared by using the Aldo-keto method. It is also noticed that the crystal diffraction intensity of  $\text{YBO}_3$  obtained by SSD is lower than that obtained from the Aldo-Keto system.

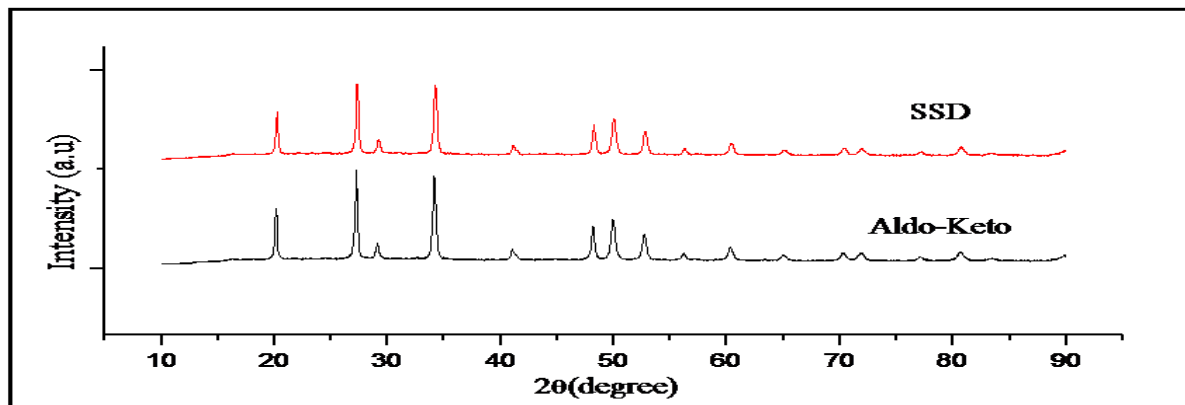


Fig. 2 Powder X-ray diffraction patterns of  $\text{YBO}_3:\text{Eu}^{3+}$  synthesized by Aldo-Keto and Solid-State method.

## 4.2 Scanning Electron Microscopy

The morphologies of  $\text{YBO}_3:\text{Eu}^{3+}$  powders prepared by conventional solid-state route and Aldo-keto technique are shown in Fig. 3(a) and (b), respectively. The  $\text{YBO}_3:\text{Eu}^{3+}$  particles prepared by conventional solid-state route have an irregular shape, coarse surface, wide size distribution, and are highly aggregated, whereas the  $\text{YBO}_3:\text{Eu}^{3+}$  particles prepared by Aldo-keto technique have spherical shape, smooth surface, narrow size distribution in 500nm range, and are lowly aggregated. The morphology difference above mentioned should originate from different preparation conditions and post-treatment techniques. In conventional solid-state reaction route, a high-temperature calcinations is required for obtaining the phosphor materials with high crystallinity, however, high-temperature calcinations makes the phosphor particles large and easily agglomerated, thus, milling and grinding appear necessary to obtain suitable particle size for application, but these post-treatment techniques significantly damage the surface quality of phosphor particles [21], as shown in Fig. 3(a). Compared with solid-state reaction route, the solution-based Aldo-keto route needs a lower calcination temperature for the host crystallization and no milling and grinding processes. The growth of individual phosphor particles is effectively controlled with low calcination temperature, and the smooth surface is obtained due to without milling and grinding processes as shown in Fig. 3(b). It is known that the morphology of phosphor particles plays an important role in improving the performance of flat panel displays. The phosphor particles with spherical shape are able to increase the screen brightness and improve the resolution [22] because of lower scattering of evolved light and higher packing densities than irregularly shaped particles obtained by conventional solid-state route. Furthermore, recent studies revealed that the VUV energy is absorbed in a very thin layer at the surface of the phosphor particles [23, 24], so the surface quality of phosphor particles seems to be very important for the luminescent efficiency. Milling and grinding processes are introduced in the phosphor prepared by conventional solid-state route, thus, the surface quality of phosphor is degraded, which is expected to influence the absorption of VUV energy significantly. As shown in Fig. 3(b), the phosphor particles obtained from Aldo-keto route have excellent morphology and homogeneous size distribution, therefore, it is predicted that the phosphor particles obtained from Aldo-keto route have higher luminance than those obtained from conventional solid-state reaction.

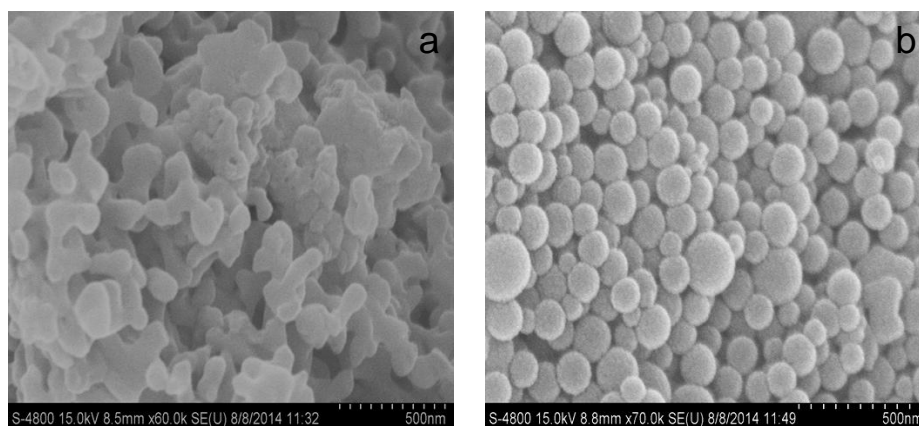
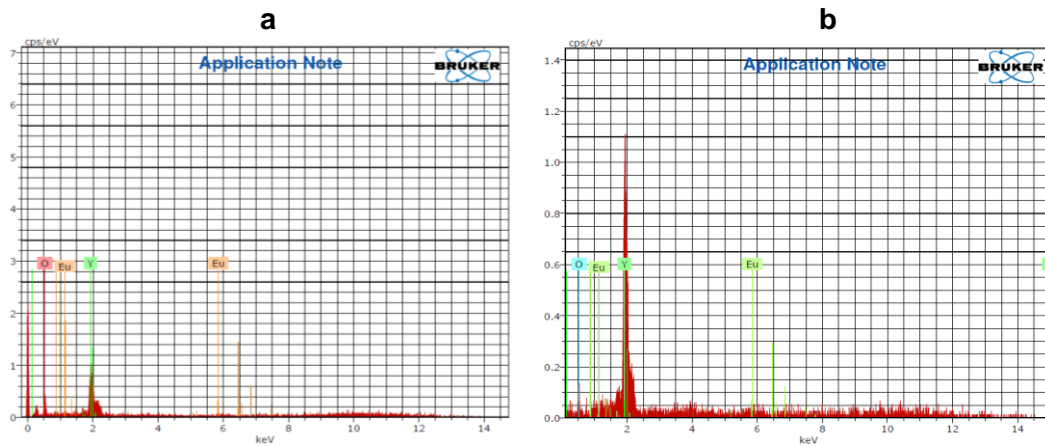


Fig.3. SEM patterns of  $\text{YBO}_3:\text{Eu}$  phosphor particle prepared by solid-state (a) and Aldo-keto (b) routes.

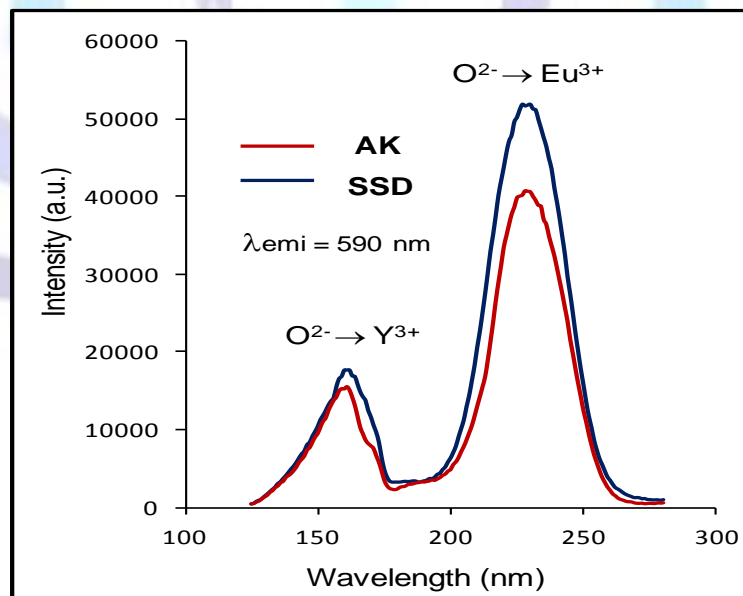


**Fig.4. EDX spectrum of YBO<sub>3</sub>:Eu<sup>3+</sup> synthesized by a. Aldo-keto and b. Solid state method.**

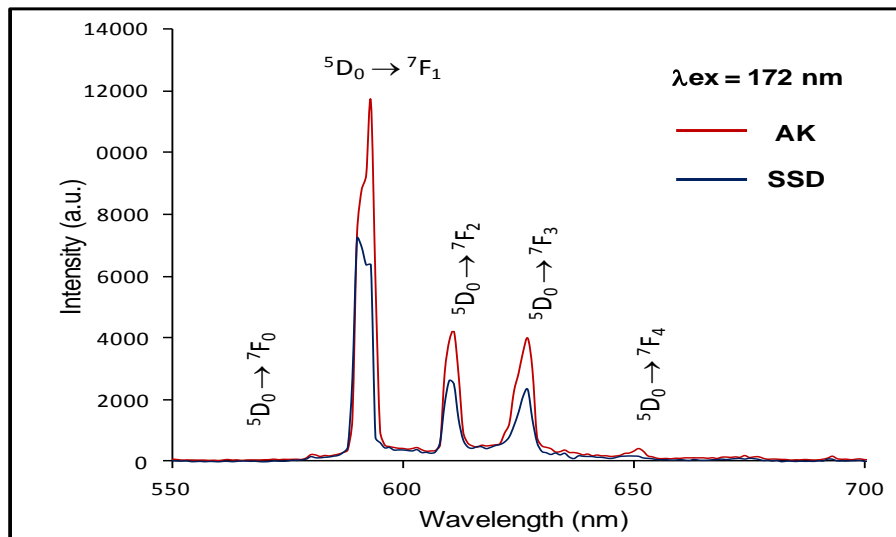
**Fig. 4a** and **b** shows the EDX spectrum of YBO<sub>3</sub>:Eu<sup>3+</sup> particles synthesized by the Aldo-keto and solid state method respectively. The EDX spectrum of YBO<sub>3</sub>:Eu<sup>3+</sup> particle shows the presence of yttrium, boron, oxygen and europium. EDX quantitative micro analysis indicates the presence of 1% europium in YBO<sub>3</sub>:Eu<sup>3+</sup> phosphor.

### 4.3 VUV Luminescence Studies

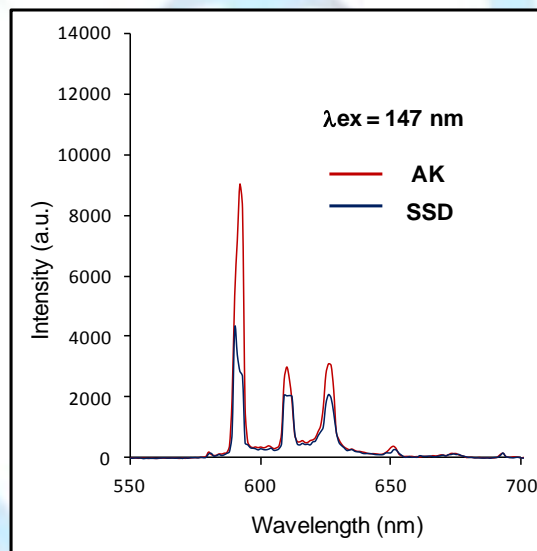
The excitation spectra measured for Y<sub>0.99</sub>Eu<sub>0.01</sub>BO<sub>3</sub> upon Eu<sup>3+</sup> emission at 590 nm prepared by Aldo-keto (AK) and solid state (SSD) method are shown in Fig. 5. There are two main broad excitation peaks, one is ascribed to absorption of the host lattice that overlapped with CT band of O<sup>2-</sup>→Y<sup>3+</sup> and the second is assigned to O<sup>2-</sup>→Eu<sup>3+</sup> charge transfer [25–28]. Therefore, when Eu doped YBO<sub>3</sub> is excited at wavelengths 147 and 172 nm, the excitation is absorbed first by the host and then transferred to Eu<sup>3+</sup> ions, leading to an indirect excitation into Eu<sup>3+</sup>. As a case, the luminescent efficiency of the Eu<sup>3+</sup> under the excitation at wavelength 147 and 172 nm depends strongly on the absorption efficiency of the host. As mentioned above, the phosphor powders obtained from solution based Aldo-keto route have higher luminance than those obtained from solid-state route, since they have higher absorption efficiency of energy due to their excellent morphology.



**Fig.5. Room temperature excitation spectra of Y<sub>0.99</sub>Eu<sub>0.01</sub>BO<sub>3</sub> prepared by Aldo-keto (AK) and solid state (SSD) (monitoring wavelength 590 nm) by using synchrotron radiation. The relative intensities are arbitrary.**



**Fig.6.** The emission spectra of  $Y_{0.99}Eu_{0.01}BO_3$  crystals synthesized from Aldo-Keto (AK) and Solid State Diffusion (SSD) at  $\lambda_{ex}=172$  nm.



**Fig.7.** The emission spectra of  $Y_{0.99}Eu_{0.01}BO_3$  crystals synthesized from Aldo-Keto (AK) and Solid State Diffusion (SSD)  $\lambda_{ex}=147$  nm.

**Figure 6** and **7** shows the emission spectrum of  $Eu^{3+}$ -doped  $YBO_3$  excited by 172 nm and 147 nm respectively. The  $Eu^{3+}$ -doped  $YBO_3$  is prepared via two different methods i.e. Aldo-keto (AK) and Solid state diffusion (SSD). The emission spectrum of  $Eu^{3+}$ -doped  $YBO_3$  prepared by low temperature method i.e. Aldo-keto (AK) shows very similar characteristics with that from high temperature i.e. solid state diffusion (SSD) with difference in emission intensity. The intense emission sharp peaks, associated with the transitions  ${}^5D_0 \rightarrow {}^7F_J$  ( $J=1, 2, 3, 4$ ) characteristic for  $Eu^{3+}$  ions, are observed. The intensity ratio of  ${}^5D_0 \rightarrow {}^7F_1$  to  ${}^5D_0 \rightarrow {}^7F_2$  bands varies with the changing of the preparation method at different excitation. It is interesting to note that the emission intensity of  $Eu^{3+}$ -doped  $YBO_3$  crystals prepared by the Aldo-keto method is about 2 times as much as that by the Solid-state method. The difference in the emission intensity of  $Eu^{3+}$ -doped  $YBO_3$  crystals prepared by different methods can be explained if we consider the morphology of crystals and the phase formation during the processes. The particles of the  $YBO_3$  prepared via the high temperature method were agglomerated (Fig.3a) and the extent of crystallization of  $YBO_3$  prepared from the high temperature process is lower than that obtained from the low temperature Aldo-keto method (Fig.3b). As mentioned earlier in the process of photoluminescence of  $Eu^{3+}$ -doped  $YBO_3$  crystals, the energy was absorbed by the host lattice  $YBO_3$  first and was transferred to the activator  $Eu^{3+}$  ions then lead to the luminescence. The absorbed energy by the host lattice in the agglomerated  $Eu^{3+}$ -doped  $YBO_3$  particles would be lower than that in the  $YBO_3$  crystals with uniform shape. Furthermore, there would be more defects in the  $Eu^{3+}$ -doped  $YBO_3$  crystals with lower crystallization obtained from the high temperature system. A part of the absorbed energy will be absorbed by the defects in  $YBO_3$  crystals and thus the efficiency of energy transfer will be reduced. The lower absorbed energy and the lower efficiency of energy transfer in the  $Eu^{3+}$ -doped  $YBO_3$  crystals prepared by the high temperature method would be the reasons that lead to the decrease of emission intensity [29].



## 5. CONCLUSION

Solid-state and Aldo-keto methods are used to synthesize the red-emitting phosphor for PDP applications. By comparison, advantages of low temperature novel Aldo-keto are summarized as follows: lower calcinations temperature, excellent particle morphology, and higher compositional homogeneity.  $\text{YBO}_3:\text{Eu}$  phosphors have strong excitation bands in VUV range, indicating high VUV energy absorption. The phosphors prepared by low temperature novel Aldo-keto route have superior luminescence, since they have excellent particle morphology and high compositional homogeneity, compared with those prepared by solid-state reaction route.

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## Author' biography with Photo



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