

# YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub>:Tm<sup>3+</sup>/Ho<sup>3+</sup> nanophosphors for blue-LED applications

B. C. JAMALIAH<sup>a,\*</sup>, N. LALITHA RANI<sup>b</sup>, G. V. LOKESWARA REDDY<sup>c</sup>, D. V. RAGHU RAM<sup>d</sup>, T. SRINIVASA RAO<sup>e</sup>

<sup>a</sup>Department of Physics, Rajeev Gandhi Memorial College of Engineering and Technology (Autonomous), Nandyal 518501, Andhra Pradesh, India

<sup>b</sup>Department of Physics, Sri Krishnadevaraya University, Anantapur 515003, Andhra Pradesh, India

<sup>c</sup>Department of Physics, S.C.N.R. Government Degree College, Proddatur 516360, Andhra Pradesh, India

<sup>d</sup>Department of Physics, Hindu College, Guntur 522002, Andhra Pradesh, India

<sup>e</sup>Department of Physics, Jawahar Bharati Degree College, Kavali-524201, Andhra Pradesh, India

The YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub> nanophosphors activated with different concentrations of Tm<sup>3+</sup>/Ho<sup>3+</sup> were synthesized by conventional solid-state reaction technique sintering at 1200 °C for 3 hours. Upon 359 nm near UV excitation, the YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub>:Tm<sup>3+</sup>/Ho<sup>3+</sup> nanophosphors emit intense blue luminescence through Tm<sup>3+</sup>:<sup>1</sup>D<sub>2</sub> → <sup>3</sup>F<sub>4</sub> (456 nm) and Tm<sup>3+</sup>:<sup>1</sup>G<sub>4</sub> → <sup>3</sup>H<sub>6</sub> (481 nm) transitions. A quenching in luminescence was noticed beyond xTm<sup>3+</sup>/yHo<sup>3+</sup> (x = 1.0 mol% and y = 0.5 mol%) due to energy transfer at higher Tm<sup>3+</sup> ions concentration through cross-relaxation mechanism. The experimental results show that the YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub>:xTm<sup>3+</sup>/yHo<sup>3+</sup> (x = 1.0 mol% and y = 0.5 mol%) nanophosphors excited at 359 nm near UV radiation could be the best choice to develop blue-LEDs.

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## 1. Introduction

The trivalent rare earth (RE<sup>3+</sup>) ions activated phosphors of high efficiency, low power consumption and environmental friendly nature play a key role in lighting technology as light-emitting diodes (LEDs), fluorescent lamps, flat plane displays, solid state laser and high energy radiation detectors etc. Many inorganic compounds such as aluminate [1], silicate [2], phosphate [3], borate [4], tungstate [5], molybdate [6] and vanadate [7] phosphors have been extensively studied owing to their efficient visible emissions under near ultra violet (UV) excitation.

It is well known that the generation of white light is possible in two different approaches. In the first approach, white light has been generated by combining a blue-LED chip with yellow phosphor like YAG:Ce, while in the second approach a near UV-LED chip has been combined with tri-colour (red/green/blue) phosphors [8-11]. The yttrium based phosphors act as good host materials due to their high thermal and chemical stability. The aluminate based phosphors show high fluorescence quantum efficiency due to strong UV absorption. The borate based phosphors are also exhibit excellent optical properties due to their non-linear optical properties and strong UV absorption. Thus, the YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub> phosphor is a good non-centrosymmetric host lattice for different RE<sup>3+</sup> ions due to its wide isomorphous substitutions and non-linear optical properties.

The blue emitting phosphors play significant role in the design of white light emitting phosphors. Sulphide based phosphors emit an efficient blue luminescence with

limited lifetime due to the degradation of sulphide during its operation [12]. The commercial BaMgAl<sub>10</sub>O<sub>17</sub>:Eu<sup>2+</sup> blue-phosphor has aging effect due to host lattice deformation and Eu<sup>2+</sup> ions [13]. Hence, the synthesis of thermally and chemically stable blue-emitting phosphors is not easy. Among the RE<sup>3+</sup> ions, the Tm<sup>3+</sup> ions exhibit blue luminescence through <sup>1</sup>D<sub>2</sub> → <sup>3</sup>H<sub>4</sub> (~450 nm) and <sup>1</sup>G<sub>4</sub> → <sup>3</sup>H<sub>6</sub> (~480 nm) transitions. In our previous work, a blue emitting YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub>:Tm<sup>3+</sup> phosphor was synthesized effectively and characterized [14].

The present research work reports the synthesis of different concentrations of Tm<sup>3+</sup>/Ho<sup>3+</sup> -doped YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub> phosphors for blue light applications. The morphological studies also carried out to study the agglomeration of particles. The aim of the present work is to investigate the structural and fluorescence properties of Tm<sup>3+</sup> ions in the presence of Ho<sup>3+</sup> and also the sensitization effect of Ho<sup>3+</sup> ions on the luminescence of Tm<sup>3+</sup> ions in YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub> nanophosphors.

## 2. Experimental techniques

### 2.1. Materials and method of preparation

Different concentrations of Tm<sup>3+</sup>/Ho<sup>3+</sup> co-doped YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub> nanophosphors were synthesized by solid state reaction method using Y<sub>2</sub>O<sub>3</sub> (99.99%, Aldrich-India), Al<sub>2</sub>O<sub>3</sub> (99.9%, Aldrich-India), H<sub>3</sub>BO<sub>3</sub> (≥99.5% Merk-India), Ho<sub>2</sub>O<sub>3</sub> (99.9%, Alfa Aesar-US) and Tm<sub>2</sub>O<sub>3</sub> (99.9%,

Alfa Aesar-US). The chemical composition for the studied phosphors was chosen as Y<sub>(1-x-y)</sub>Al<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub>: xTm<sup>3+</sup>/yHo<sup>3+</sup> (x = 0, 0.1, 0.5, 1.0, 3.0 mol% and y = 0.5 mol%). About 5.0 g batch composition homogeneous powders were prepared using agate mortar and pestle in the presence of acetone. About 5.0 wt% of H<sub>3</sub>BO<sub>3</sub> was added to compensate the evaporation of H<sub>3</sub>BO<sub>3</sub> during heat treatment process. The firing process was done using alumina crucibles for 3 hours at 200°C and 600°C with intermediate grinding. Finally, the powders were sintered at 1200°C for 3 hours to produce milky white YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub>: Tm<sup>3+</sup>/Ho<sup>3+</sup> nanophosphors.

## 2.2. Optical characterization

The crystalline phase of YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub>: Tm<sup>3+</sup>/Ho<sup>3+</sup> nanophosphors was confirmed by powder X-ray diffraction (XRD) studies carry out on X'Pert-Pro Materials Research Diffractometer ( $\lambda_{\text{CuK}\alpha} = 1.5406 \text{ \AA}$ ). The scanning electron microscope (SEM) image of YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub>: xTm<sup>3+</sup>/yHo<sup>3+</sup> (x = 1.0 mol% and y = 0.5 mol%) nanophosphor was recorded on Oxford instruments INCA PENTA FETx3 attached to SEM: Carl Zeiss EVO MA 15. The photoluminescence excitation and emission spectral analysis was done on Jobin YVON Fluorolog-3 spectrofluorimeter. All the measurements were carried out at room temperature.

## 3. Results and discussion

### 3.1. Structure and morphology

Fig. 1. Illustrates the XRD profiles of un-doped, 1.0 mol% Tm<sup>3+</sup>, 0.5 mol% Ho<sup>3+</sup> and 3.0 mol% Tm<sup>3+</sup>/0.5 mol% Ho<sup>3+</sup>-doped YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub> nanophosphors

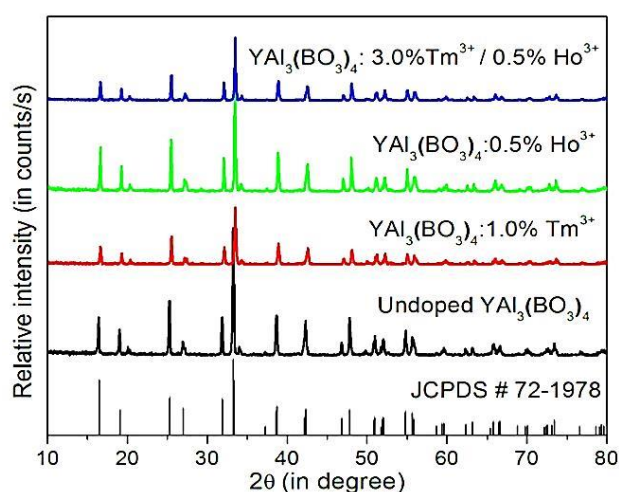


Fig. 1. XRD profiles of YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub>: Tm<sup>3+</sup>/Ho<sup>3+</sup> nanophosphors

The XRD profiles are well consistent with JCPDS Card No. 72-1978 with a rhombohedral structure and R32 space group [15]. The intensity of observed XRD peaks decrease with increase of concentration of Tm<sup>3+</sup>/Ho<sup>3+</sup> impurities and the peak positions remain unchanged.

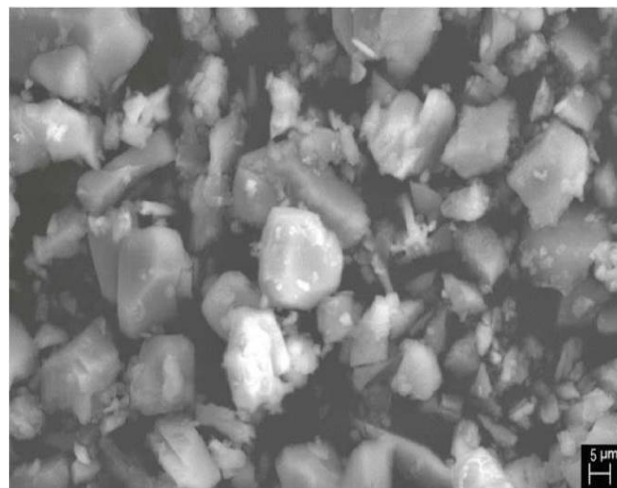


Fig. 2. SEM image of YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub>: 1.0 mol% Tm<sup>3+</sup>/0.5 mol% Ho<sup>3+</sup> nanophosphor

The average size ( $D_{\text{hkl}}$ ) of crystallites has been determined using the Scherrer's formula,  $D_{\text{hkl}} = [(0.89 \times \lambda) / (\beta_{2\theta} \cdot \cos \theta)]$ , where  $\lambda$  is the wavelength of X-rays (1.5406 Å),  $\beta_{2\theta}$  is the full width at half maximum and  $\theta$  is the angle of diffraction [18]. The value of  $D_{\text{hkl}}$  is estimated to be 56 nm and it is found very close to our previous work [14, 19]. The SEM image of YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub>: 1.0 mol% Tm<sup>3+</sup>/0.5 mol% Ho<sup>3+</sup> nanophosphor presented in Fig. 2 reveals a grain like morphology with different size and shape and appeared to be agglomerated. This agglomeration of particles affects the luminescence of RE<sup>3+</sup> ions.

### 3.2. Photoluminescence excitation

The excitation spectra of different concentrations of Tm<sup>3+</sup>/Ho<sup>3+</sup> co-doped YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub> nanophosphors have been studied to select a suitable excitation wavelength to produce intense luminescence. Fig. 3 shows the excitation spectra of x mol% Tm<sup>3+</sup>/0.5 mol% Ho<sup>3+</sup> co-doped YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub> phosphors, where (x = 0.1, 0.5, 1.0 and 3.0 mol%) when the emission is monitored at 456 nm i.e., within the Tm<sup>3+</sup> ions. These spectra reveal four excitation bands at about 263, 277, 289 and 359 nm corresponding to the <sup>3</sup>H<sub>6</sub> → <sup>3</sup>P<sub>2</sub>, <sup>3</sup>H<sub>6</sub> → <sup>3</sup>P<sub>1</sub>, <sup>3</sup>H<sub>6</sub> → <sup>1</sup>I<sub>6</sub> and <sup>3</sup>H<sub>6</sub> → <sup>1</sup>D<sub>2</sub> transitions, respectively [20]. The 1.0 mol% Tm<sup>3+</sup>-doped YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub> phosphor also exhibits similar kind of spectrum and it is illustrated as inset of Fig. 3. The Ho<sup>3+</sup> ions possess an excitation band at around 359 nm due to Ho<sup>3+</sup>: <sup>5</sup>I<sub>8</sub> → <sup>3</sup>H<sub>6</sub> transition.

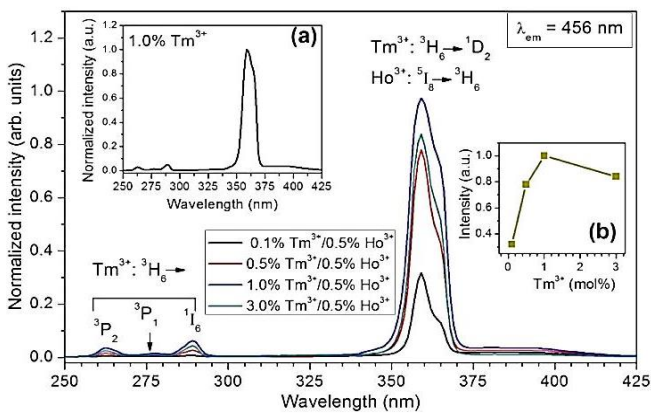


Fig. 3. Excitation spectra of  $YAl_3(BO_3)_4: Tm^{3+}/Ho^{3+}$  nanophosphors. Inset: excitation spectrum  $YAl_3(BO_3)_4: 1.0\% Tm^{3+}$  nanophosphor (a) and the variation of intensity of  $Tm^{3+}: ^3H_6 \rightarrow ^1D_2$  transition as a function of  $Tm^{3+}$  concentration

The intensity of observed excitation bands increase with increase of  $Tm^{3+}$  concentration reaches to maximum at 1.0 mol% and then decrease for further increase of its concentration in  $YAl_3(BO_3)_4: Tm^{3+}/Ho^{3+}$  nanophosphors, when the emission is monitored at 466 nm. The variation of intensity of  $Tm^{3+}: ^3H_6 \rightarrow ^1D_2$  transition as a function of  $Tm^{3+}$  concentration is also described as inset of Fig. 3. Among the observed excitation transitions, the  $^3H_6 \rightarrow ^1D_2$  transition noticed at 358 nm has maximum intensity and it has been used to investigate the emission of  $YAl_3(BO_3)_4: Tm^{3+}/Ho^{3+}$  nanophosphors.

To investigate the effect of sensitization of  $Ho^{3+}$  ions on the luminescence of  $Tm^{3+}$  ions in  $YAl_3(BO_3)_4: Tm^{3+}/Ho^{3+}$  nanophosphors, the excitation spectra has also been studied monitoring the emission at 466 nm corresponding to  $Ho^{3+}: ^5F_1 \rightarrow ^5I_8$  transition. Under this conditions, the  $YAl_3(BO_3)_4: Tm^{3+}/Ho^{3+}$  nanophosphors exhibit quite different excitation spectra and they are described in Fig. 4. These spectra contain the transitions both from  $Tm^{3+}$  and  $Ho^{3+}$  ions and they are assigned to ( $Tm^{3+}: ^3H_6 \rightarrow ^3P_2$ ), ( $Tm^{3+}: ^3H_6 \rightarrow ^3P_1$ ), ( $Tm^{3+}: ^3H_6 \rightarrow ^1I_6$ ), ( $Tm^{3+}: ^3H_6 \rightarrow ^1D_2/Ho^{3+}: ^5I_8 \rightarrow ^3H_6$ ), ( $Ho^{3+}: ^5I_8 \rightarrow ^3K_7$ ) and ( $Ho^{3+}: ^5I_8 \rightarrow ^5G_4$ ) transitions with peak maximum at about 263, 277, 289, 358, 378 and 394 nm, respectively.

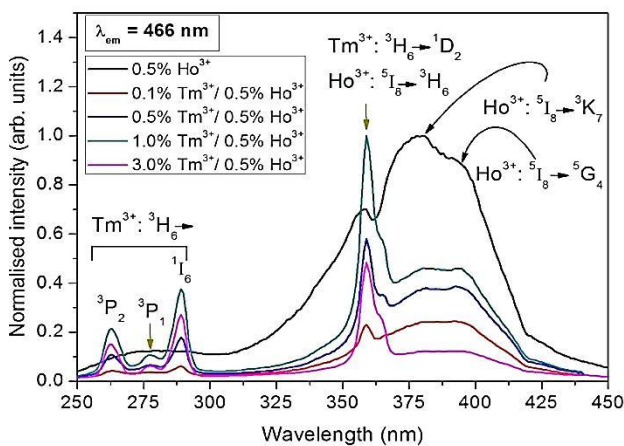


Fig. 4. Excitation spectra ( $\lambda_{em} = 466$  nm) of  $Tm^{3+}/Ho^{3+}$  codoped  $YAl_3(BO_3)_4$  nanophosphors

When the emission is monitored at 466 nm ( $Ho^{3+}: ^5F_1 \rightarrow ^5I_8$ ), the intensity of  $Tm^{3+}: ^3H_6 \rightarrow ^1D_2$  transition increases up to 1.0 mol% and then decrease for further increase of  $Tm^{3+}$  ion concentration as illustrated in inset of Fig. 3. One can notice from Fig. 4 that the excitation band noticed at 359 nm is common for  $Tm^{3+}$  and  $Ho^{3+}$  ions and its intensity increases with increase of  $Tm^{3+}$  ions concentration indicating the uniform distribution of  $Tm^{3+}$  and  $Ho^{3+}$  ions in  $YAl_3(BO_3)_4: Tm^{3+}/Ho^{3+}$  nanophosphors. Since the  $Ho^{3+}: ^5I_8 \rightarrow ^3H_6$  (359 nm) and the  $Tm^{3+}: ^3H_6 \rightarrow ^1D_2$  (359 nm) transitions have the same energy, there is a tendency for transfer of energy from  $Ho^{3+}$  and  $Tm^{3+}$  ions. The emission intensity of  $Tm^{3+}$  ions enhances due to the energy transfer between  $Ho^{3+}$  and  $Tm^{3+}$  ions in  $YAl_3(BO_3)_4: Tm^{3+}/Ho^{3+}$  nanophosphors. In the present work, the luminescence from  $YAl_3(BO_3)_4: Tm^{3+}/Ho^{3+}$  nanophosphors has been investigated by exciting the samples at 359 nm.

### 3.3. Blue luminescence

The deviation from Russell-Saunders coupling in the 4f configuration and the complicated energy level structure of  $Tm^{3+}$  results different possible emission transitions in any host matrix. The emission spectra of  $YAl_3(BO_3)_4: Tm^{3+}/Ho^{3+}$  nanophosphors under 359 nm ( $^3H_6 \rightarrow ^1D_2$ ) excitation in the spectral region from 400 nm to 500 nm are shown in Fig. 5. This spectra reveal an intense blue luminescence through  $^1D_2 \rightarrow ^3F_4$  (456 nm) transition and a feeble blue luminescence through  $^1G_4 \rightarrow ^3H_6$  (481 nm) transition.

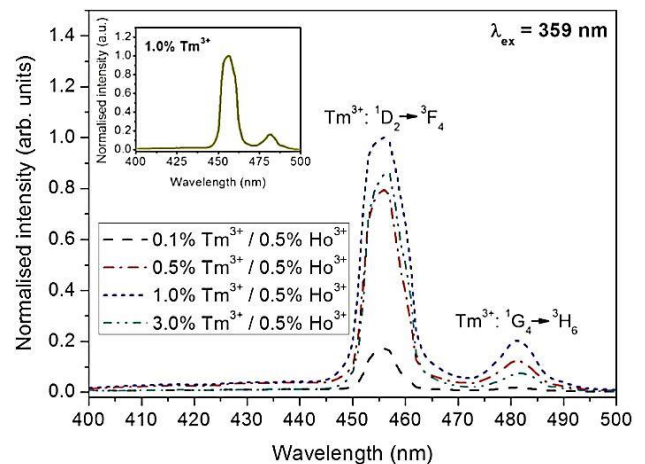


Fig. 5. Emission spectra of  $YAl_3(BO_3)_4: Tm^{3+}/Ho^{3+}$  codoped nanophosphors. Inset show the emission spectrum of 1.0 mol%  $Tm^{3+}$ -doped  $YAl_3(BO_3)_4$  nanophosphor

When excited at 359 nm, some of the  $Tm^{3+}$  ions get excited to  $Tm^{3+}: ^1D_2$  energy level along with the  $Ho^{3+}$  ions to  $Ho^{3+}: ^3H_6$  energy levels. Since the  $Tm^{3+}: ^1D_2$  level has the same energy as that of  $Ho^{3+}: ^3H_6$  energy level, a transfer of energy has been initiated from  $Ho^{3+}: ^3H_6$  to

Tm<sup>3+</sup>: <sup>1</sup>D<sub>2</sub> increasing the population of Tm<sup>3+</sup>: <sup>1</sup>D<sub>2</sub> level. From there an intense blue luminescence at 456 nm takes place through Tm<sup>3+</sup>: <sup>1</sup>D<sub>2</sub> → <sup>3</sup>F<sub>4</sub> transition along with a non-radiative transition to Tm<sup>3+</sup>: <sup>1</sup>G<sub>4</sub> energy level. Another radiative transition has also been take place through Tm<sup>3+</sup>: <sup>1</sup>G<sub>4</sub> → <sup>3</sup>H<sub>6</sub> transition producing a feeble blue (481 nm) luminescence. A partial energy level diagram shown in Fig. 6 illustrate the emission mechanism of Ho<sup>3+</sup>/Tm<sup>3+</sup> and energy transfer (ET) from Ho<sup>3+</sup> to Tm<sup>3+</sup> in YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub>: Tm<sup>3+</sup>/Ho<sup>3+</sup> nanophosphors.

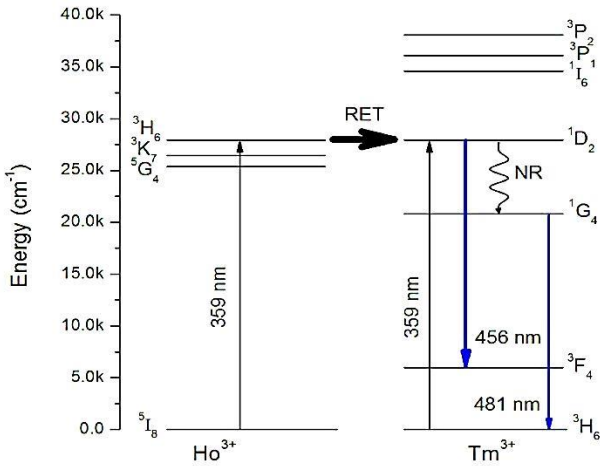


Fig. 6. Partial energy level diagram showing the emission mechanism and energy transfer in YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub>: Tm<sup>3+</sup>/Ho<sup>3+</sup> - codoped nanophosphors at 359 nm excitation

From Fig. 5 one can notice a quenching in luminescence beyond 1.0 mol% concentration of Tm<sup>3+</sup> ions in YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub>: Tm<sup>3+</sup>/Ho<sup>3+</sup> nanophosphors. The variation of intensity of Tm<sup>3+</sup>: <sup>1</sup>D<sub>2</sub> → <sup>3</sup>F<sub>4</sub> transition as a function of Tm<sup>3+</sup> concentration in Tm<sup>3+</sup>/Ho<sup>3+</sup> -codoped YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub> nanophosphors is presented in Fig. 7.

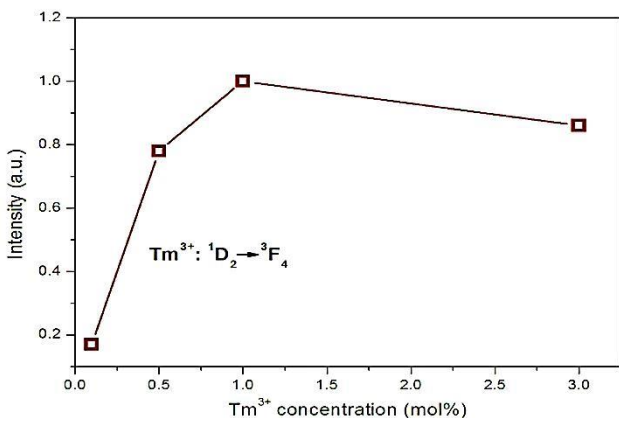


Fig. 7. Variation of luminescence intensity of Tm<sup>3+</sup>: <sup>1</sup>D<sub>2</sub> → <sup>3</sup>F<sub>4</sub> transition as a function of Tm<sup>3+</sup> concentration in Tm<sup>3+</sup>/Ho<sup>3+</sup> -codoped YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub> nanophosphors

Though there is a transfer of energy from Ho<sup>3+</sup> to Tm<sup>3+</sup> ions, no considerable enhancement in luminescence corresponding to the Tm<sup>3+</sup>: <sup>1</sup>D<sub>2</sub> → <sup>3</sup>H<sub>4</sub> transition is noticed. Because the ET among the excited Tm<sup>3+</sup> ions at higher concentrations through the [<sup>1</sup>D<sub>2</sub>, <sup>3</sup>H<sub>6</sub>] → [<sup>3</sup>F<sub>2</sub>, <sup>3</sup>H<sub>4</sub>] and [<sup>1</sup>D<sub>2</sub>, <sup>3</sup>H<sub>6</sub>] → [<sup>3</sup>H<sub>4</sub>, <sup>3</sup>F<sub>2</sub>] cross-relaxation channels dominates the resonant energy transfer (RET) from Ho<sup>3+</sup> to Tm<sup>3+</sup> [14]. Another possible reason for unfavorable luminescence is the agglomeration of particles. Thus, the YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub>:Tm<sup>3+</sup>/Ho<sup>3+</sup> nanophosphors emit blue luminescence via Tm<sup>3+</sup>: <sup>1</sup>D<sub>2</sub> → <sup>3</sup>H<sub>4</sub> and Tm<sup>3+</sup>: <sup>1</sup>G<sub>4</sub> → <sup>3</sup>H<sub>6</sub> transitions. The authors are interested to study the effect of sensitization of Tm<sup>3+</sup> ions on Ho<sup>3+</sup> ions in YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub> nanophase in near future.

### 3.4. Chromaticity analysis

In order to analyze the emitted colour from YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub>: Tm<sup>3+</sup>/Ho<sup>3+</sup> nanophosphors, the Commission International de l’Eclairagein (CIE-1931) chromaticity coordinates have been determined from the emission spectra. When excited at 359 nm near UV radiation, the YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub>:Tm<sup>3+</sup>/Ho<sup>3+</sup> nanophosphors show blue luminescence through Tm<sup>3+</sup>: <sup>1</sup>D<sub>2</sub> → <sup>3</sup>F<sub>4</sub> and Tm<sup>3+</sup>: <sup>1</sup>G<sub>4</sub> → <sup>3</sup>H<sub>6</sub> transitions. The chromaticity coordinates are found to be (0.1443, 0.0346), (0.1471, 0.0324), (0.1452, 0.0345), (0.1439, 0.0363) and (0.1457, 0.0318) for 1.0 mol% Tm<sup>3+</sup> and xTm<sup>3+</sup>/ 0.5 mol% Ho<sup>3+</sup> (x = 0.1, 0.5, 1.0 and 3.0 mol%) phosphors, respectively.

These colour coordinates are well located in the blue region of CIE-1931 chromaticity diagram and it is shown in Fig. 8. The evaluated CIE coordinates are found very close to the European Broadcasting Union (EBU: 0.150, 0.060) illuminants and the National Television Standard Committee (NTSC: 0.155, 0.070) illuminants. These are also found very close to YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub> [14], Ca<sub>2</sub>Gd<sub>8</sub>Si<sub>6</sub>O<sub>26</sub> [21] and β-NaCaPO<sub>4</sub> [22] phosphors.

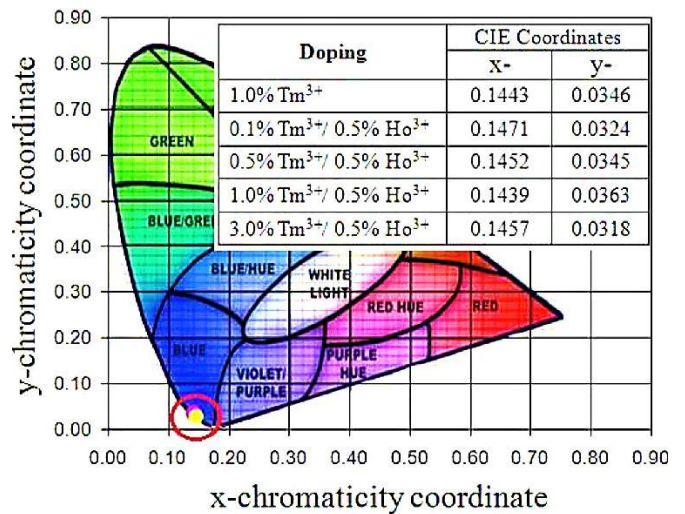


Fig. 8. CIE coordinates of Tm<sup>3+</sup> and Tm<sup>3+</sup>/Ho<sup>3+</sup> co-doped YAl<sub>3</sub>(BO<sub>3</sub>)<sub>4</sub> nanophosphors at 359 nm excitation

#### 4. Conclusions

The  $YAl_3(BO_3)_4: Tm^{3+}/Ho^{3+}$  nanophosphors were synthesized through conventional solid-state reaction method for blue-LED applications. The average size of crystallites has been estimated to be 56 nm. The SEM results show an agglomerated grain like morphology with different size and shape. The observed luminescence quenching beyond 1.0 mol%  $Tm^{3+}$ / 0.5 mol%  $Ho^{3+}$  concentration has been assigned to the energy transfer among the excited  $Tm^{3+}$  ions at higher concentration as well as agglomeration of particles. The observed results conclude that the  $YAl_3(BO_3)_4: Tm^{3+}/Ho^{3+}$  nanophosphors could be the best choice to design blue-LEDs at 359 nm near UV excitation.

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\*Corresponding author: bcjphysics@gmail.com;  
jamalaiahbc@rgmct.edu.in