Luminescence properties of Dy\(^{3+}\) doped lithium zinc borosilicate glasses for photonic applications

N. Jaidass\(^{a,b}\), C. Krishna Moorthi\(^a\), A. Mohan Babu\(^{a,b,c,}\ast\), M. Reddi Babu\(^b\)

\(^a\)School of Advanced Sciences, V I T University, Vellore, India
\(^b\)Department of Physics, Chadalawada Ramanamma Engineering College, Tirupati, India
\(^c\)Department of Physics, Institute of Aeronautical Engineering, Hyderabad, India

\ast\)Corresponding author.
E-mail address: mohanphy57@gmail.com (A. Mohan Babu).

Abstract

Different concentrations of Dy\(^{3+}\) ions doped lithium zinc borosilicate glasses of chemical composition \((30-x)\) B\(_2\)O\(_3\) - 25 SiO\(_2\) - 10 Al\(_2\)O\(_3\) - 30 LiF - 5 ZnO - x Dy\(_2\)O\(_3\) (x = 0, 0.1, 0.5, 1.0 and 2.0 mol\%) were prepared by the melt quenching technique. The prepared glasses were investigated through X-ray diffraction, optical absorption, photoluminescence and decay measurements. Intensities of absorption bands expressed in terms of oscillator strengths (f) were used to determine the Judd-Ofelt (J-O) intensity parameters \(\Omega_l\) (\(l = 2, 4\) and 6). The evaluated J-O parameters were used to determine the radiative parameters such as transition probabilities (\(A_R\)), total transition probability rate (\(A_T\)), radiative lifetime (\(\tau_R\)) and branching ratios (\(\beta_R\)) for the excited \(^4F_{9/2}\) level of Dy\(^{3+}\) ions. The chromaticity coordinates determined from the emission spectra were found to be located in the white light region of CIE chromaticity diagram.

Keywords: Condensed matter physics, Engineering, Materials science
1. Introduction

The development of rare earth (RE$^{3+}$) ions doped glasses with good thermal, mechanical and chemical durability are highly essential for the design of high performance lasers, optical amplifiers and white light emitting diodes (LEDs) [1, 2, 3, 4, 5, 6]. Different multi-component borate glasses have been considered for technological applications in many areas and among them, the lithium zinc borosilicate (LZBS) glasses have attracted the researchers for the development of visible lasers and amplifiers due to their good optical transmission, low phonon energy, low glass transition temperature, high refractive index, high dielectric constant and high thermal expansion coefficient [7, 8, 9, 10].

Among various RE$^{3+}$ ions, the dysprosium (Dy$^{3+}$) ion exhibits intense yellow (574 nm) and blue (481 nm) emissions corresponding to the $^4F_{9/2} \rightarrow ^6H_{13/2}$ and $^4F_{9/2} \rightarrow ^6H_{15/2}$ transitions, respectively together with a feeble red emission (665 nm) due to $^4F_{9/2} \rightarrow ^6H_{11/2}$ transition. Thus, the Dy$^{3+}$ - doped glasses are more suitable to produce two primary colors as well as white light emission. The $^4F_{9/2} \rightarrow ^6H_{13/2}$ (yellow) transition is a host sensitive and the $^4F_{9/2} \rightarrow ^6H_{15/2}$ (blue) transition is host insensitive. Thus, the yellow to blue (Y/B) intensity ratios can be modulated by varying the coordination environment of the host material and/or by changing the concentration of Dy$^{3+}$ ions.

The aim of this work is to investigate the optical absorption, photoluminescence (PL) and decay properties of $^4F_{9/2}$ level of Dy$^{3+}$ ions in lithium zinc borosilicate (LZBS) glasses. Optimization of concentration of Dy$^{3+}$ ions for efficient luminescent properties and the quenching in PL and decay time has been explained. The aptness of the studied glasses for solid state lasers as well as white LEDs has also been discussed.

2. Experimental

2.1. Materials and method of preparation

Different concentrations of Dy$^{3+}$ ions doped LZBS glasses with molar compositions of $(30 - x)$ B$_2$O$_3$ + 25 SiO$_2$ + 10 Al$_2$O$_3$ + 30 LiF + 5 ZnO + x Dy$_2$O$_3$ (where $x = 0, 0.1, 0.5, 1.0$ and $2.0$ mol %) were prepared by the conventional melt quenching method. About 10 g batch compositions of homogeneous mixtures of high purity B$_2$O$_3$, SiO$_2$, Al$_2$O$_3$, LiF, ZnO and Dy$_2$O$_3$ (99.99%) chemicals were taken into clean and dry alumina crucible and then heated at 1200 °C continuously for 1 h until to get molten state. The melts were poured on a preheated brass mould and annealed at 350 °C for 7 h and then cooled to room temperature in order to strengthen the glasses and also to remove the thermal strains during the preparation of glasses. Based on Dy$^{3+}$ ions concentration ($x = 0, 0.1, 0.5, 1.0$ and $2.0$ mol %), the glasses were labelled as LZBSDy0.0, LZBSDy0.1, LZBSDy0.5, LZBSDy1.0 and LZBSDy2.0, respectively as shown in the Table 1. The prepared LZBSDyx glasses are shown in the Fig. 1.
Table 1. Glass composition and labelling of LZBSDyx glasses (x = 0.0, 0.1, 0.5, 1.0 and 2.0 mol %).

| Glass code | B2O3 (mol %) | SiO2 (mol %) | LiF (mol%) | Al2O3 (mol%) | ZnO (mol%) | xDy2O3 (mol%) |
|------------|--------------|--------------|------------|--------------|------------|--------------|
| LZBSDy0.0  | 30           | 25           | 30         | 10           | 5          | 0.0          |
| LZBSDy0.1  | 29.9         | 25           | 30         | 10           | 5          | 0.1          |
| LZBSDy0.5  | 29.5         | 25           | 30         | 10           | 5          | 0.5          |
| LZBSDy1.0  | 29.0         | 25           | 30         | 10           | 5          | 1.0          |
| LZBSDy2.0  | 28.8         | 25           | 30         | 10           | 5          | 2.0          |

2.2. Physical and optical measurements

The thickness and refractive indices of the prepared glasses play an important role in the estimation of luminescence parameters. So, the refractive indices (n) were determined by the Abbe’s refractometer using sodium vapour lamp as a source. The optical absorption spectra were recorded using Jasco V-770 UV-VIS-NIR spectrophotometer. The luminescence and decay measurements were performed using FLS-980 fluorescence spectrometer with xenon flash lamp as a source. All the spectral measurements were carried out at room temperature.

3. Results and discussion

3.1. Absorption spectrum and spectral analysis

The optical absorption spectrum of LZBSDy0.5 glass recorded in the region 500—2000 nm is shown in Fig. 2. The spectrum exhibited twelve absorption bands and are assigned based on the earlier work done by Carnall et al. [11]. The absorption bands located at 347, 364, 386, 426, 451, 471, 752, 797, 895, 1084, 1264 and 1670 nm are assigned to the $^6H_{15/2} \rightarrow ^6P_{7/2}$, $^6P_{5/2}$, $^4I_{13/2}$, $^4G_{11/2}$, $^4I_{15/2}$, $^4F_{9/2}$, $^6F_{3/2}$, $^6F_{5/2}$, $^6F_{7/2}$, $^6F_{9/2}$, $^6F_{11/2}$ and $^6H_{11/2}$ transitions, respectively. To find the nature of the covalence between the rare earth ion and its ligands as well as the radiative properties of the Dy$^{3+}$ ions in the prepared glasses, the experimental oscillator strength ($f_{exp}$) of the absorption bands were determined by using the Eq. (1) [12].

Fig. 1. Prepared LZBSDyx glasses (x = 0.1, 0.5, 1.0 and 2.0 mol %).
\[ f_{\text{exp}} = 4.32 \times 10^{-9} \int \varepsilon(\nu) \, d\nu \]  

where \( \varepsilon(\nu) \) is the molar absorptivity of a band at a wavenumber \( \nu \) (cm\(^{-1}\)) which can be calculated from the Beer-Lambert’s law. The calculated oscillator strengths \( (f_{\text{cal}}) \) and the Judd-Ofelt (J-O) intensity parameters \( (\Omega_{\lambda} = 2, 4 \text{ and } 6) \) are obtained from the experimental oscillator strength \( (f_{\text{exp}}) \) by the least square fitting method using the Eq. (2) \[13, 14\].

\[ f_{\text{cal}}(\Psi', \Psi'') = \frac{8\pi^2 mc}{3\hbar(2J + 1)} \left[ \frac{(n^2 + 2)^2}{9n} S_{\text{ed}}(\Psi', \Psi'') + n S_{\text{md}}(\Psi', \Psi'') \right] \]  

where \( c \) is the speed of light, \( \hbar \) is the Planck’s constant, \( n \) is refractive index of the glass, \( (n^2 + 2)^2 / 9n \) is the Lorentz local field correction factor and accounts for dipole-dipole correction and \( (2J + 1) \) is the degeneracy of the ground state.

The electric \( (S_{\text{ed}}) \) and magnetic \( (S_{\text{md}}) \) dipole strengths can be calculated using the Eqs. (3) and (4) \[11\].

\[ S_{\text{ed}}(\Psi', \Psi'') = e^2 \sum_{\lambda, \lambda'} \Omega_{\lambda, \lambda'} \left| \langle \Psi' \| U^J \| \Psi'' \rangle \right|^2 \]  

\[ S_{\text{md}}(\Psi', \Psi'') = \frac{e^2 \hbar^2}{16 \pi^2 m c^2} \left| \langle \Psi' \| (L + 2S) \| \Psi'' \rangle \right|^2 \]

where \( e \) is the charge of electron, \( \Omega_{\lambda} (\lambda = 2, 4 \text{ and } 6) \) are the J-O intensity parameters and \( \| U^J \| \) are the doubly reduced matrix elements of rank ‘\( \lambda \)’. While calculating the

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**Fig. 2.** Optical absorption spectrum of LZBS Dy0.5 glass in the UV-VIS-NIR regions.
intensity parameters, the oscillator strengths of $^6\text{H}_{15/2} \rightarrow ^6\text{P}_{3/2}$ transitions were not taken into consideration due to their weak intensities. In order to know the quality of fit between the $f_{\text{exp}}$ and $f_{\text{cal}}$ values, it is necessary to find the root mean square deviation ($\delta_{\text{rms}}$). In the present case, the obtained $\delta_{\text{rms}}$ value is $\pm 0.35 \times 10^{-6}$, which indicates the best fit between the experimental and calculated oscillator strengths. For LZBS Dy0.5 glass, the evaluated experimental ($f_{\text{exp}}$) and calculated ($f_{\text{cal}}$) oscillator strengths are shown in Table 2 and from which it is observed that, the intensity of $^6\text{H}_{15/2} \rightarrow ^6\text{F}_{11/2}$ hypersensitive transition which is very sensitive to the host environment is higher when compared to the other transitions obeying the selection rules $||\Delta S|| = 0$, $||\Delta L|| \leq 0$ and $||\Delta J|| \leq 0$.

The evaluated J-O intensity parameters for the LZBS Dy0.5 glass are $\Omega_2 = 11.75 \times 10^{-20}$ cm$^2$, $\Omega_4 = 3.90 \times 10^{-20}$ cm$^2$, $\Omega_6 = 3.61 \times 10^{-20}$ cm$^2$ and follows the trend $\Omega_2 > \Omega_4 > \Omega_6$. Similar trend has been observed for other Dy$^{3+}$ doped host matrices such as L6BD [15], Ge-Ga-Se [16], PKBFAD [17] and Dy:KLTB [18] as compared in Table 3. The higher magnitude quantity of $\Omega_2$ parameter indicates the lower degree of symmetry around the active ion and stronger covalence of active ion-oxygen ligand bond. Whereas, the $\Omega_4$ and $\Omega_6$ parameters are related to the bulk properties such as viscosity, stability and rigidity of the host medium in which the ions are doped [19, 20, 21]. In the present investigation, considerably higher magnitude of $\Omega_2$ indicates higher degree of asymmetry around the Dy$^{3+}$ ions and relatively weaker covalence of active ion-oxygen bond.

### 3.2. Photoluminescence

The luminescence spectra of LZBS Dyx ($x = 0.1, 0.5, 1.0$ and $2.0$ mol %) glasses recorded by exciting with $385$ nm wavelength are shown in Fig. 3. The emission

| Transition from $^6\text{H}_{15/2}$ | Oscillator strengths ($\times 10^{-6}$) |
|-------------------------------------|---------------------------------------|
|                                     | $f_{\text{exp}}$ | $f_{\text{cal}}$ |
| $^4\text{I}_{13/2}$                | 0.50             | 0.90             |
| $^4\text{G}_{11/2}$                | 0.27             | 0.15             |
| $^4\text{I}_{15/2}$                | 0.73             | 0.82             |
| $^4\text{F}_{9/2}$                 | 0.22             | 0.29             |
| $^6\text{F}_{3/2}$                 | 0.47             | 0.32             |
| $^6\text{F}_{5/2}$                 | 2.64             | 1.72             |
| $^6\text{F}_{7/2}$                 | 3.66             | 3.72             |
| $^6\text{F}_{9/2}$                 | 4.58             | 4.59             |
| $^6\text{F}_{11/2}$                | 12.00            | 11.97            |
| $^6\text{H}_{11/2}$                | 2.07             | 2.27             |
| $\delta_{\text{rms}}$             | $\pm 0.39 \times 10^{-6}$            |
spectra revealed three characteristic emission bands of Dy$^{3+}$ ions in the blue (481 nm), yellow (574 nm) and red (665 nm) regions which are assigned to the $^4F_{9/2}$/$^6H_{15/2}$, $^4F_{9/2}$/$^6H_{13/2}$ and $^4F_{9/2}$/$^6H_{11/2}$ transitions, respectively. The intensity of emission bands increased from 0.1 to 0.5 mol% then decreased with increase of Dy$^{3+}$ ions concentrations. From this, it is concluded that, the optimum concentration as well as efficient luminescence of Dy$^{3+}$ ions is obtained for LZBSDy0.5 glass. The concentration quenching exhibited by Dy$^{3+}$ ions at higher concentrations could be due to the energy transfer among the excited Dy$^{3+}$ ions. The variation of intensities of $^4F_{9/2}$/$^6H_{15/2}$ and $^4F_{9/2}$/$^6H_{13/2}$ emission transitions as a function of Dy$^{3+}$ ions concentration is illustrated in Fig. 4.

Table 3. Comparison of J-O intensity parameters of LZBSDy0.5 glass with other Dy$^{3+}$ doped glasses.

| Glass system          | J–O intensity parameters ($\times 10^{-20}$ cm$^2$) |
|-----------------------|---------------------------------------------------|
|                       | $\Omega_2$  | $\Omega_4$  | $\Omega_6$  |
| LZBSDy0.5 [Present work] | 11.75       | 3.90        | 3.61        |
| L6BD [15]             | 12.83       | 3.47        | 3.43        |
| Ge-Ga-Se [16]         | 11.61       | 2.79        | 1.11        |
| PKBFAD [17]           | 10.41       | 2.29        | 2.07        |
| Dy:KLTB [18]          | 9.86        | 3.39        | 2.41        |

Fig. 3. Photoluminescence spectra for different concentrations of LZBSDyx glasses (x = 0.1, 0.5, 1.0 and 2.0 mol %).
From the luminescence spectra, it is noticed that, the emission intensity of $^4F_{9/2} \rightarrow ^6H_{15/2}$ (481 nm) transition is slightly higher than that of $^4F_{9/2} \rightarrow ^6H_{13/2}$ (574 nm) transition. Similar results were reported for Dy$^{3+}$ ion doped alkali lead tellurofluoroborate glasses [18]. Additionally, the optical properties of rare earth doped materials are influenced by the coordination environment around the active ions. The $^4F_{9/2} \rightarrow ^6H_{13/2}$ transition is electric dipole ($\Delta J = \pm 2$), which is host sensitive whereas the transition $^4F_{9/2} \rightarrow ^6H_{15/2}$ is magnetic dipole ($\Delta J = 0, \pm 1$ but $0 \rightarrow 0$ forbidden) and is host insensitive. Thus, the yellow to blue (Y/B) intensity ratio has been used to determine the Dy$^{3+}$-O$^{2-}$ bond covalence. In the present study for all the prepared glasses, the values of Y/B ratios are calculated and presented in the Table 4.

![Graph showing variation of emission intensities of $^4F_{9/2} \rightarrow ^6H_{15/2}$ and $^4F_{9/2} \rightarrow ^6H_{13/2}$ transitions with the increase of Dy$^{3+}$ ions concentration in LZBS glasses.](image)

**Fig. 4.** Variation of emission intensities of $^4F_{9/2} \rightarrow ^6H_{15/2}$ and $^4F_{9/2} \rightarrow ^6H_{13/2}$ transitions with the increase of Dy$^{3+}$ ions concentration in LZBS glasses.

**Table 4.** CIE chromacity color coordinates (x, y), correlated color temperature (CCT) values and Y/B ratios for the LZBSDy$x$ glasses (x = 0.1, 0.5, 1.0 and 2.0 mol%).

| Glass code  | Chromacity coordinates | CCT  | Y/B ratio |
|-------------|------------------------|------|-----------|
|             | x          | Y          |            |
| LZBSDy0.1   | 0.321      | 0.347      | 6002       | 2.35 |
| LZBSDy0.5   | 0.319      | 0.363      | 6040       | 2.45 |
| LZBSDy1.0   | 0.318      | 0.357      | 6102       | 2.65 |
| LZBSDy2.0   | 0.327      | 0.358      | 5722       | 2.39 |
3.3. Color perception

In order to know the emission color perception of LZBSDy (x = 0.1, 0.5, 1.0 and 2.0 mol %) glasses, the Commission International de l’Eclairage in 1931 (CIE) color coordinates (x, y) have been calculated from the intensities of emission spectral profiles [22, 23]. The evaluated CIE chromaticity coordinates (x, y) for LZBSDy0.1, LZBSDy0.5, LZBSDy1.0 and LZBSDy2.0 glasses are (0.321, 0.347), (0.319, 0.363), (0.318, 0.357) and (0.327, 0.358), respectively and are found to be located in the white region of CIE diagram as shown in Fig. 5. From these results, one can conclude that the Dy³⁺ doped LZBS glasses are more suitable for the white light generation.

To know the spectral behaviour of near white light produced by the broadband light sources can be characterised by the correlated color temperature (CCT) values which can be calculated by using the Eq. (5) and is familiar as Mc Camy’s formula [24].

\[
\text{CCT} = 449 \, n^3 + 3525 \, n^2 + 6823.3 \, n + 5520.33
\]

where \(n = \frac{(x-0.3320)}{(0.1858-y)^7}\).

As per the CCT ratings for a light source if CCT values below 3200 K are referred as “warm sources” and above 4000 K are considered as “cool in appearance” [24].

Fig. 5. CIE chromaticity diagram showing the color coordinates (x, y) for LZBSDy glasses (x = 0.1, 0.5, 1.0 and 2.0 mol %).
The CCT values obtained for LZBSDy0.1, LZBSDy0.5, LZBSDy1.0 and LZBSDy2.0 glasses are 6002, 6040, 6102 and 5722 K and also presented in Table 4. It is observed that, all the CCT values are above 4000 K and can be concluded that the prepared glasses are usually considered as “cool” in appearance [24].

### 3.4. Radiative properties

From the emission spectra of LZBSDyx (x = 0.1, 0.5, 1.0 and 2.0 mol%) glasses, various radiative properties such as transition probabilities \( A_R \), total radiative transition rate \( A_T \), radiative lifetimes \( \tau_R \), luminescence branching ratios \( \beta_R \) and emission cross-sections \( \sigma_e \) are determined for the \( \Psi J \rightarrow \Psi'J' \) emission transitions using the Eqs. (6), (7), (8), (9) and (10) [25].

\[
A_R(\Psi J \rightarrow \Psi'J') = \frac{64 \pi^4 v^3}{3h (2J + 1)} \left[ \frac{n (n^2 + 2)^2}{9} S_{ed}(\Psi J \rightarrow \Psi'J') + n^3 S_{md}(\Psi J \rightarrow \Psi'J') \right]
\]  

(6)

\[
A_T(\Psi J) = \sum_{\Psi'J'} A_R(\Psi J \rightarrow \Psi'J')
\]  

(7)

\[
\tau_R(\Psi J) = \frac{1}{\sum_{\Psi'J'} A_R(\Psi J \rightarrow \Psi'J')}
\]  

(8)

\[
\beta_R(\Psi J \rightarrow \Psi'J') = \frac{A_R(\Psi J, \Psi'J')}{A_T(\Psi J)}
\]  

(9)

The experimental branching ratios \( \beta_{exp} \) are determined from the relative areas under the emission peaks and stimulated emission cross-sections \( \sigma_e \) between \( \Psi J \) and \( \Psi'J' \) levels are given by [25].

\[
\sigma_e(\Psi J \rightarrow \Psi'J') = \frac{\lambda_p^4}{8 \pi c n^2 \Delta \lambda_{eff}} A_R(\psi J \rightarrow \psi'J')
\]  

(10)

where \( \lambda_p \) is the emission peak wavelength and \( \Delta \lambda_{eff} \) is the effective linewidth of the emission band. Various radiative parameters of \( ^4F_{9/2} \rightarrow ^6H_{15/2, 13/2 \& 11/2} \) transitions in LZBS Dy0.5 glass are presented in Table 5. The values of \( \sigma_e \) and \( (\sigma_e \times \tau_R) \) for \( ^4F_{9/2} \rightarrow ^6H_{13/2} \) transition are found to be \( 20.15 \times 10^{-22} \, \text{cm}^2 \) and \( 9.87 \times 10^{-25} \, \text{cm}^2 \), respectively and are in good agreement (Table 6) with other Dy\(^{3+}\) doped glass hosts [26, 27, 28, 29]. Relatively higher values of the gain bandwidth \( (\sigma_e \times \Delta \lambda_p) \) and optical gain \( (\sigma_e \times \tau_R) \) parameters for the \( ^4F_{9/2} \rightarrow ^4H_{13/2} \) transition are critical to predict the amplification of the medium and so it is confirmed that the LZBS Dy0.5 glass is a suitable candidate for photonic applications.
3.5. Luminescence decay analysis

The decay profiles of $^4F_{9/2}$ excited level in LZBSDy $x$ ($x = 0.1, 0.5, 1.0$ and $2.0$ mol %) glasses are obtained by exciting with $385$ nm wavelength and monitoring the emission at $574$ nm. The experimental ($\tau_{exp}$) lifetime values are found to be $0.58, 0.39, 0.36$ and $0.32$ ms for the LZBSDy0.1, LZBSDy0.5, LZBSDy1.0 and LZBSDy2.0 glasses, respectively. The decrease of $\tau_{exp}$ values of $4F_{9/2}$ emission level with the increase of Dy$^{3+}$ ions concentrations is shown in the inset of Fig. 6. This could be due to the energy transfer through non-radiative decay rates ($W_{NR}$). One way of evaluating the $W_{NR}$ is using the Eq. (11) [25]:

$$W_{NR} = \frac{1}{\tau_{exp}} - \frac{1}{\tau_R}$$  \hspace{1cm} (11)

where $\tau_R$ and $\tau_{exp}$ are the radiative and experimental lifetimes, respectively. The evaluated values of $\tau_R$, $\tau_{exp}$ and $W_{NR}$ values for LZBSDy0.1, LZBSDy0.5, LZBSDy1.0 and LZBSDy2.0 glasses are presented in Table 7. Moreover, the fluorescence quantum
efficiency \((\eta)\) of an emission level \(^{4}F_{9/2}\) can be obtained using the Eq. (12) [25] and are presented in the Table 7.

\[
\eta = \frac{\tau_{\text{exp}}}{\tau_{R}}
\]  

4. Conclusions

In the present study, different concentrations Dy\(^{3+}\) ions doped LZBS glasses of high optical quality were prepared and characterized through XRD, optical absorption,
emission and decay techniques. The intensity analysis of absorption levels has been done using the Judd-Ofelt theory. The evaluated intensity parameters follow the trend as $\Omega_2 > \Omega_4 > \Omega_6$ and are further used to evaluate the radiative parameters such as transition probabilities ($A_R$), total transition probability rate ($A_T$), branching ratios ($\beta_R$), radiative lifetimes ($\tau_R$) and stimulated emission cross sections ($\sigma_e$) for all the emission transitions of Dy$^{3+}$ doped LZBS glasses. The PL spectra recorded for different concentration of Dy$^{3+}$ ions in LZBS glasses exhibited two strong intense emissions in the blue and yellow regions. Reasonably, large stimulated emission cross-section of the $^4\text{F}_{9/2} \rightarrow ^6\text{H}_{13/2}$ transition confirmed that the present glasses are good hosts for laser active materials. From the evaluated chromaticity coordinates, it is concluded that the present glasses are also useful for the generation white light.

**Declarations**

**Author contribution statement**

N. Jaidass: Conceived and designed the experiments; Performed the experiments; Wrote the paper.

C. Krishna Moorthi and A. Mohan Babu: Contributed reagents, materials, analysis tools or data.

M. Reddi Babu: Analyzed and interpreted the data.

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**Competing interest statement**

The authors declare no conflict of interest.

**Additional information**

No additional information is available for this paper.

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