OPTICAL AND LUMINESCENCE PROPERTIES OF Dy^{3+} (0.5%) DOPED BISMUTH BORATE (BiB) GLASS

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Abstract In this work binary glasses with the composition $40Bi_2O_3$-$(60-x)B_2O_3$: xDy_2O_3 for x=0 and 0.5 mol % were prepared by the melt-quenching technique. The prepared glass samples were characterized by Photoluminescence and optical absorption spectra and were recorded at temperature. In order to confirm the amorphous nature of the glass samples, XRD analysis was done. Physical properties of the prepared glass samples were also measured and presented in this paper. From the obtained absorption edges optical band gap, the Urbach energies were calculated, the optical band gap is found to decrease with Dy_2O_3. The luminescence spectra exhibited blue and greenish-yellow emission bands at around 482 nm (broad with less intensity) and 562 nm (sharp with high intensity) corresponds to the $^4F_{9/2} \rightarrow {}^6H_{15/2}$ and $^4F_{9/2} \rightarrow {}^6H_{13/2}$ transitions respectively. The emission spectrum was characterized through Commission International d'Eclairage (CIE) 1931 chromaticity diagram to explore its suitability for display applications. CIE chromaticity diagram for the prepared BiBDy0.5 glass (λ_{ex} =280 nm) and the values of the (x, y) color coordinates are found to be (0.393, 0.604) with CCT 4663K. It is noted from figure that, the (x, y) color co-ordinate values are found to lie in the greenish-yellow region, which is useful for white light generation. The same prepared glass sample, BiBDy0.5 shows chromaticity co-ordinates (0.479, 0.520) with CCT 3995K which is in yellow region in the chromaticity diagram under 387nm excitation wavelength, which can be useful for display applications.

*Keywords***:** Trivalent ion, dysprosium, glass, annealing, melt-quenching, optical, luminescence

I. INTRODUCTION

Glass is not a single composition but a state of matter. It is a subset of solid state. Glass is a network of atoms bonded to each other through covalent bonds with oxygen atoms. It is usually tetrahedral bonded together in a random arrangement. The glass transition from solid glass to the viscous liquid glass is an important property. Basically glass is an elastic solid below the transformation region and a viscous liquid above it. The structure of the solid has all the attributes of a liquid except that solid does not flow on any meaningful time scale. If glass is cooled from the melt faster, the overall glass structure will have a large volume (lower density) than one that is cooled slowly. Glass is an elastic solid without the structural periodicity and long range order of crystalline material. It looks like a liquid but behaves like a solid. Generally thermodynamically glass systems have higher potential energy than crystalline material due to random arrangement.

Recent development of optical devices was based on rare earth ions doped materials is one of the interesting field of research. Rare earth doped glasses were used as optical device materials, sensors, solar concentrators, flat panel displays, fluorescent lamps, white LEDs etc. [1-4]. Glasses doped with rare earth ions are proving to be luminescence materials as they have high emission efficiencies. These emissions correspond to 4f-4f and 4f-5d electronic transitions in the rare earth^{n+}. The 4f-4f transition gives sharp fluorescence pattern from the UV to the infrared region. This is due to shield effects of the outer 5s and 5p orbital's on the 4f electrons [5]. The Judd-Ofelt (JO) theory was helpful to estimate the intensities of the transitions for rare earth ions. This theory defines three intensity parameters, Ω_λ (λ= 2, 4, 6) that are sensitive to the local environment of the rare earth ions. The lanthanum group doped materials is important because of their potential applications in the fields of optical device technology, optoelectronic devices, infrared to visible up-converters and phosphors [6]. Therefore luminescence properties of rare earth doped different glass hosts are being prepared and investigated with the purpose to know their utility for luminescence applications.

The visible luminescence of trivalent dysprosium (Dy^{3+}) mainly consists of narrow lines in the blue (470–500 nm, $^4F_{9/2} \rightarrow {}^6H_{15/2}$) and yellow (570–600 nm, $^4F_{9/2} \rightarrow {}^6H_{13/2}$) region [7]. The later one belongs to the Hyper sensitive transition (L = 2, J = 2), which is strongly influenced by the environment. Out of the 14 lanthanide elements, dysprosium in its trivalent state (Dy^{3+}) is an efficient emitter in the visible region and it is the only ion that emits two intense colors that on combining in appropriate proportions yields white light. At an appropriate yellow-to-blue (Y/B) intensity ratio, Dy^{3+} particle emits white light. Thus, light emitting materials doped with Dy^{3+} ion are used for generation of w-light in glasses and phosphors. By adjusting the Y/B intensity ratio, it is possible to obtain near white light emission in Dy^{3+} activated luminescent nanophosphors [8].

The present work reports optical and luminescence properties of Dy^{3+} doped bismuth borate glass is characterized through optical absorption, excitation and emission spectral measurements. The intensities of the transitions for rare earth ions have been investigated using Judd-Ofelt (JO) theory. The characteristics of the emission color were examined through CIE 1931 chromaticity diagram.

II. MATERIALS AND METHODS

2. 1 Glasses preparation

The glass samples were prepared by the standard melt quenching method with the following compositions. $(60-x)B_2O_3$-$40Bi_2O_3$: xDy_2O_3 where x= 0 and 0.5 mol%. These glasses can be designated as BiBDy0 and BiBDy0.5 depending on the Dy^{3+} ion concentration respectively. About 10 gm of the batches of composition were taken and grounded completely using an agate mortar to get homogenised mixture. The homogeneous mixture was then taken into a ceramic crucible and heated at 1000 ºC in an electrical furnace for 10 min until the

homogeneous melt was obtained. This soften was then poured quickly on a brass mildew and ironed quickly with another brass mildew to get circular formed glass samples with uniform thickness. The glass samples thus prepared were annealed at 400 ºC in order to make them free from thermal strains.

2. 2 Measurements

The optical absorption spectra were recorded on a JASCO UV-VIS-NIR spectrophotometer (model V-670) at room temperature within the range 200–1400 nm. The emission and excitation spectra of the prepared glasses were recorded at room temperature using Shimadzu RF-5301 PC-Spectrofluorophotometer with a spectral resolution 0.5 nm. XRD patterns were obtained using a Bruker D5005 diffractometer with a constant 12mm footprint on the sample. All runs were over a 2θ range of 10-80 at 0:02 increments using a copper Kα radiation source at 40 kV-30 mA. CIE colour co-ordinates were drawn using Radiant Imaging software version 2.0.

III RESULTS AND DISCUSSION
3.1. X-ray powder diffraction (XRD)

To confirm the amorphous nature of the prepared samples, XRD patterns were recorded. Figure 1 presents the XRD pattern of the BiBDy0 sample containing 60% B_2O_3 and 40% Bi_2O_3. The bands detected at approximately 25–30° 2θ and 45–50° 2θ, are typical of a borate glass structure where BO_3 and BO_4 units coexist. This is the clear indication of amorphous nature inside the resolution limit of XRD instrument. From the XRD pattern of the BiBDy0.5 sample containing 59.5% B_2O_3, 40% Bi_2O_3 and the dopant 0.5% Dy_2O_3, the prepared sample show no sharp Bragg's peak. This is the clear indication of amorphous nature and homogeneous in nature. Moreover, the broad humps indicate that still there is an existence of short range order in the glass network.
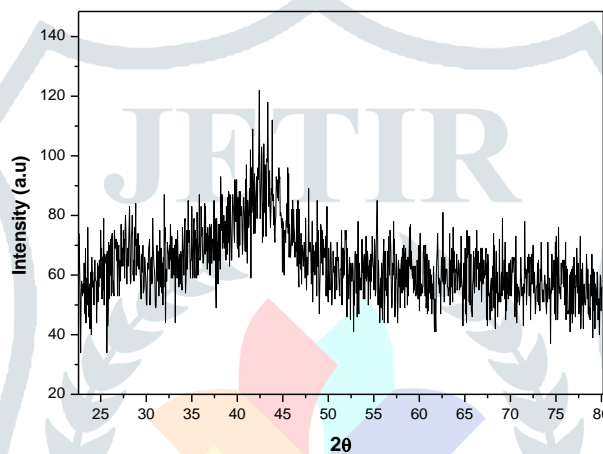

Fig. 1 XRD pattern of BiBDy0 glass

3.2 Physical Properties

Density of glass is generally explained in terms of competition between the masses and the volumes of the various structural groups present in it. Therefore, density is related to how tightly the atoms and atomic groups are placed together in the glass network. For the glasses considered in this article, the density (ρ) increases systematically by substituting B_2O_3 with Dy_2O_3, as reported in Table 1.

Table 1 Physical Properties of the synthesized samples

S.No	Parameter	Sample code	
		BiBDy0	BiBDy0.5
1	Density (ρ) g/cm^3	5.8064	5.8567
2	Molar volume (V_m) cm^3/vol	1.2885	1.2846
3	Dopant ion concentration (N_i)x10^{20}	--	7.6794
4	Inter ion separation (R_i) x10^{-7}	--	2.3526
5	Polaron radius (R_p) x10^{-7}	--	0.9479
6	Field strength (F_i)x10^{14}	--	3.3385
7	Reflection Loss (R)	0.0240	0.0245
8	Dielectric constant (ε)	2.4025	2.4217
9	Molar refractivity (R_m)	12.517	12.609
10	Electronic polarizability x10^{-22}	--	1.0001
11	Average Molecular weight	228.154	229.671
12	Refractive Index (n_d)	1.55	1.5562

3.3 Optical absorption spectra and energy level analysis

From the optical absorbance spectrum of Dysprosium doped at 0.5% concentration in BiB glass, (Figure is not presented) in the range from 250 – 550 nm recorded at room temperature, consists of six inhomogeneous absorption bands due to the absorption transitions of Dy^{3+} ions. These absorptions are assigned from ground state $^6H_{15/2}$ to various excited state of Dy^{3+} ions. From the figure we observed absorption peaks at 350, 365, 387, 426, 452 and 471nm. Among these 350 and 387nm are the strongest absorption peaks. These bands are attributed to $^6H_{15/2} \rightarrow {}^6P_{7/2}$, $^6H_{15/2} \rightarrow {}^6P_{5/2}$, $^6H_{15/2} \rightarrow {}^4F_{7/2}$, $^6H_{15/2} \rightarrow {}^4G_{11/2}$, $^6H_{15/2} \rightarrow {}^4I_{15/2}$ and $^6H_{15/2} \rightarrow {}^4F_{9/2}$ transitions respectively.

The optical absorption spectra of BiBDy0 and BiBDy0.5 glasses are shown in Fig. 2. The spectra consist of BiBDy0 sample does not show any absorption bands where as BiBDy0.5 sample shows in homogeneous absorption bands due to the transitions assigned from ground state, $^6H_{15/2}$ to various excited states of Dy^{3+} ions. From the spectrum it is also observed that various absorption levels corresponding to

various transitions between ground state and excited state are observed. From the spectra the levels of $^4I_{13/2}$ + $^4F_{7/2}$, $^4G_{11/2}$, $^4I_{15/2}$, are not observed. In this present work, the absorption spectrum of Dy^{3+} ions shows totally six absorption bands located at 754, 803, 903, 1092, 1274 and 1687 nm due to the $^4f_9$ electronic transitions of the Dy^{3+} ions from the $^6H_{15/2}$ ground level to the various excited states such as $^6F_{3/2}$, $^6F_{5/2}$, $^6F_{7/2}$, ($^6H_{7/2} \rightarrow {}^6F_{9/2}$), ($^6H_{9/2} \rightarrow {}^6F_{11/2}$) and $^6H_{11/2}$ transitions, respectively are observed and well resolved [9]. The absorption transitions such as $^6H_{15/2} \rightarrow {}^6F_{11/2}$ and $^6H_{15/2} \rightarrow {}^6H_{11/2}$ are found to posses higher intensity which are highly sensitive to host environment known as hypersensitive transitions and obeys the selection rule $|\Delta S|=0$, $|\Delta L| \leq 2$ and $|\Delta J| \leq 2$.
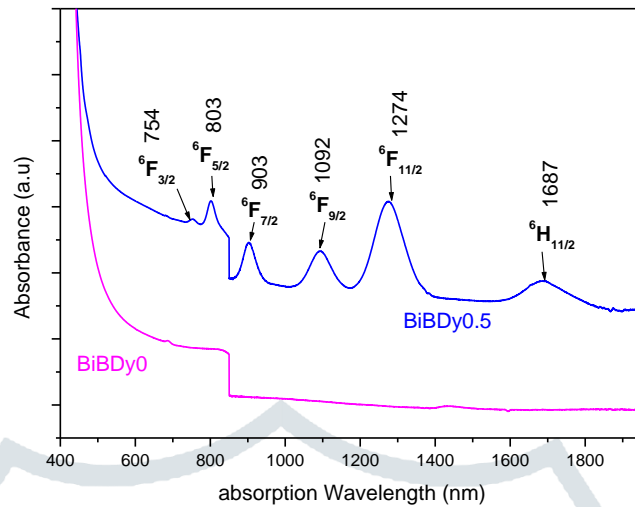

Fig. 2 Optical absorption spectra of BiBDy0 and BiBDy0.5 glasses

3.4 Band gap and Urbach's energy analysis

The optical band gap is a vital parameter within the field of photonics which provides data regarding the electronic structure of amorphous materials. The band gap energy values (E_{opt}) of the crystalline, amorphous materials are often evaluated with the help of the fundamental absorption edges through direct and indirect allowed transitions. Initially, the absorption edges shift towards the upper wavelengths side with the rise in Dy^{3+} particle content, ie the absorption edges exhibit red shift indicating the fact that the band gap values decreases. The Mott and Davis theory was used to derive a relation between the band gap and the coefficient of absorption (α) and also the same is expressed using the equation, $(\alpha h\upsilon)^n = B(E-E_g)$ where α is the absorption coefficient, E is the energy of the applied photon, h is the plank's constant, υ is the frequency of the applied photon, B is the band tailing parameter, E_g is the band gap and n is equal to 2 or 1/2 which signifies direct or indirect allowed transitions. Fig. 4(a) and Fig. 4(b) are the graphs plotted between E and $(\alpha h\upsilon)^n$ referred as Tauc's plot and Tauc's plot for the title glass BiBDy0. The linear portions of the Tauc's plot are often workout to zero absorption and also the intersection of E provides the optical band gap value. The calculated band gap value of the prepared glass is presented in Table 2.
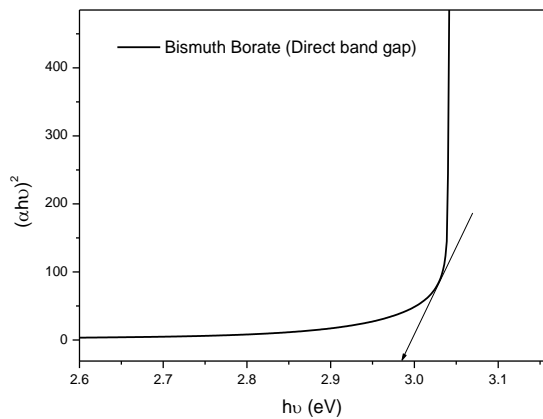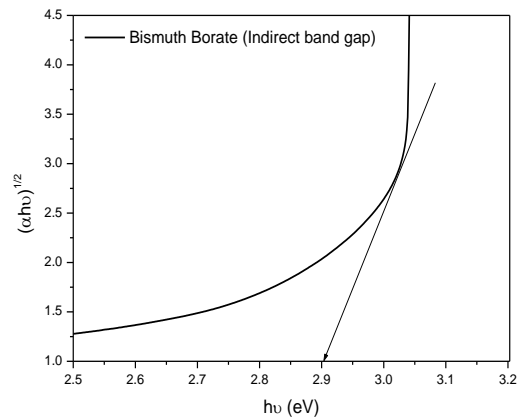

Fig. 4(a) Direct bandgap of BiB glass   Fig. 4(b) indirect bandgap of BiB glass

Table 2 The fundamental absorption edge (λ_{edge}), Optical band gap (E_{opt}) and Urbach's (ΔE) energy of the Dy^{3+} ions doped bismuth borate glass

S.No	Absorption Edge (nm)	Optical Bandgap E (eV)		Urbach Energy ΔE (eV)
		Direct	Indirect	
1	408	2.98	2.90	0.08
2	404	3.01	2.95	0.06

A typical plot of $(\alpha h\upsilon)^2$ versus $h\upsilon$ for the BiB glass having 40% Bi_2O_3 and 60% B_2O_3 is presented in Figure 4(a). Extrapolation of this plot to $a^2 = 0$ provides the optical band gap E_g for direct transition. We have estimated the optical band gap value 2.98 eV for direct transition. The value of E_g for indirect transition is obtained by extrapolation of $(\alpha h\upsilon)^{1/2}$ versus $h\upsilon$ plot to $a^{1/2} = 0$ as shown in the Figure 4(b). The optical band for indirect transition is 2.9 eV. This can be attributed to the structural changes that are taking place with the introduction of rare

earth ions. Inclusion of rare earth ions in borate structure may create some defect states in the midgap, which is responsible for the decrease of bandgap. The broadness of the absorption edge may be due to the presence of localized state in the band tails.

Here 'E' is the Urbach energy which indicates the width of the band tails of the localized states and depends on temperature, induced disorder, static disorder and on average photon energies. Hence, Urbach energy provides a measure for the disorder in amorphous and crystalline materials. The nature of disorder is different for crystalline and amorphous solids. In amorphous solids, the static atomic structural disorder dominates and can be due to presence of defects like dangling bonds or non-bridging oxygens in glasses. It shows the structural disorder of the system. Smaller is the value of Urbach energy, greater is the structural stability of the glass system. It is observed that the BiBDy0.5 glass has low value of structural disorder, which indicates the strong structural stability.

3.5 Judd-Ofelt analysis

The J–O theory has been applied to the experimentally evaluated oscillator strengths to find the J–O intensity parameters (Ω_2, Ω_4 and Ω_6) by least square fit analysis. The J–O intensity parameters and Spectroscopic quality factor ($\chi=\Omega_4/\Omega_6$) for the titled glasses are presented in Table 2 along with the reported J–O parameters for other glasses [11]. The J–O intensity parameter are necessary to review the native structure and bonding nature of rare earth ions that indicates the co-valence of metal ligand bond and also on the asymmetry of ion sites in the neighbourhood of rare earth ion. The bulk properties like viscosity and rigidity are often identified through the magnitudes of Ω_4 and Ω_6 parameters measured with the help of the J–O theory. From Table 3, it is observed that Ω_2 is higher than Ω_4 and Ω_6 and follows the trend $\Omega_2 > \Omega_6 > \Omega_4$ [12].

Table 3 Judd-Ofelt intensity parameters (Ω_λ x10^{-20} cm^2), spectroscopic quality factor (Ω_4/Ω_6) in BiB glasses doped with dysprosium ions

Sample code		J-O intensity parameters			Ω_4/Ω_6	Trend
		Ω_2	Ω_4	Ω_6		
BiBDy0.5	[present]	4.00	3.80	2.10	1.795	$\Omega2 > \Omega6 > \Omega4$
DPTFB	[13]	10.21	1.73	2.12	0.814	$\Omega2 > \Omega6 > \Omega4$
DLCZFB	[14]	11.25	2.45	5.16	0.475	$\Omega2 > \Omega6 > \Omega4$
LB	[15]	10.43	2.19	3.53	0.620	$\Omega2 > \Omega6 > \Omega4$

3.6 Luminescence properties

In order to analyze luminescence properties of the BiBDy0.5 glass, it is essential to know the suitable excitation wavelengths of Dy^{3+} ions. The luminescence behaviour of Dy^{3+} doped bismuth borate glasses have been studied throughout the emission spectral measurement. Generally the luminescence spectra exhibited by Dy ion is the conventional blue, yellow and red emission bands at around 482 nm, 575 nm and 662 nm corresponds to the $^4F_{9/2} \rightarrow {}^6H_{15/2}$, $^4F_{9/2} \rightarrow {}^6H_{13/2}$ and $^4F_{9/2} \rightarrow {}^6H_{11/2}$ transitions respectively. The yellow to blue emission bands has been studied. Luminescence results were well matched with the other glass matrix prepared by the various researchers [16 -19].

Figure 5 is the 575nm wavelength monitored PL excitation spectrum of the Dy (0.5%) doped Bi$_2$O$_3$-B$_2$O$_3$ glass named as BiBDy0.5 is measured using Xenon as excitation source at room temperature. From the figure several bands were observed in the visible region at 351, 365, 387, 426, 453 and 472nm. Among these 387 and 453nm peaks are strongest one and the transitions were assigned to $^6P_{7/2}$, $^6P_{5/2}$, $^4F_{7/2}$, $^4G_{11/2}$, $^4I_{15/2}$ and $^4F_{9/2}$ respectively.

Figure 6 is the emission spectrum of BiBDy0.5 glass measured at 280nm excitation wavelength. From the figure two emission bands were observed in the visible region one is broad band range from 375 – 500nm peaking at 468nm which belongs to blue and the second one is a sharp band at 562nm belongs to yellow. These two emission bands are the characteristic emissions of Dy^{3+} ion and were assigned to the $^4F_{9/2} \rightarrow {}^6H_{15/2}$ and $^4F_{9/2} \rightarrow {}^6H_{13/2}$ respectively. Finally the glass emits cyan colour which is very close to the white light.

Figure 7 is the emission spectrum of BiBDy0.5 glass measured at 387nm excitation wavelength. From the figure one emission band along with a small hump were observed in the visible region one is a sharp band at 575nm belongs to yellow and a small band at 662nm (red). These emission bands are the characteristic emissions of Dy^{3+} ion and were assigned to the $^4F_{9/2} \rightarrow {}^6H_{13/2}$ (yellow) and $^4F_{9/2} \rightarrow {}^6H_{15/2}$ (red) respectively. The emission intensity of yellow is highest where as the red emission intensity is negligible when compared to yellow. The Y/B ratio can't be measured because of the absence of blue emission. But from the emission spectrum under 387nm excitation glass emits yellow colour which we can see in the CIE diagram shown in the section 3.6.
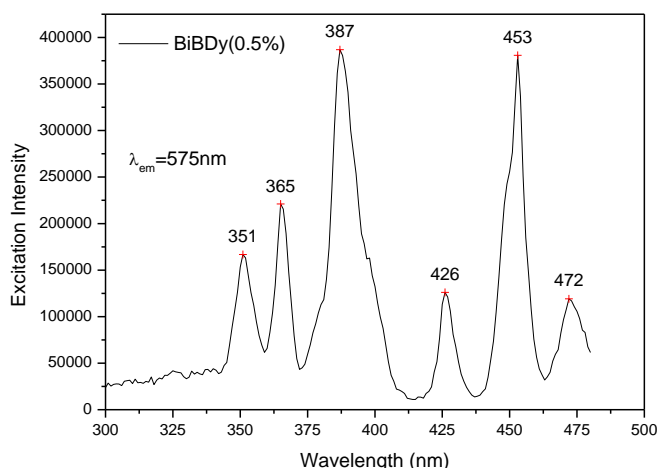

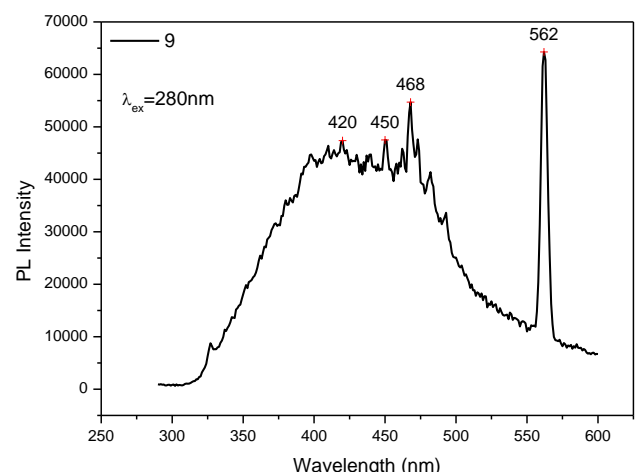Fig. 5 Excitation spectrum of BiBDy0.5 glass monitored at

Fig. 6 Emission spectrum of BiBDy0.5 glass monitored at

575nm wavelength   280nm wavelength
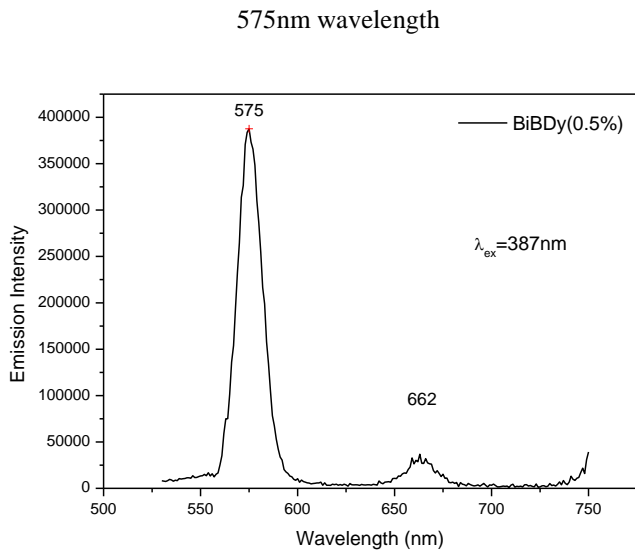

Fig. 7 Emission spectrum of BiBDy0.5 glass monitored at 387nm wavelength
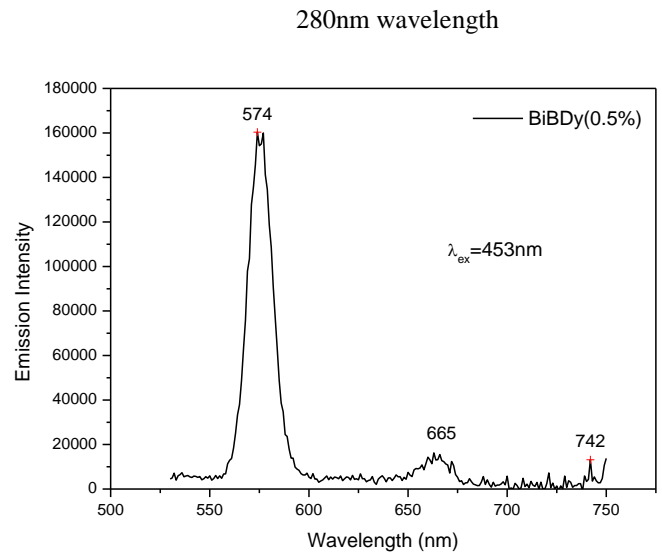

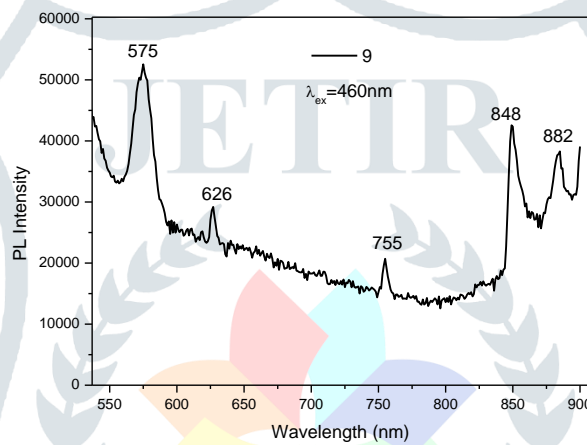Fig. 8 Emission spectrum of BiBDy0.5 glass monitored at 453nm wavelength

Fig. 9 Emission spectrum of BiBDy0.5 glass monitored at 460nm wavelength

Figure 8 is the emission spectrum of BiBDy0.5 glass measured at 453nm excitation wavelength. From the figure the same emission bands were observed when the glass is excited with 387. The emission intensity under 453nm excitation is lesser than the emission intensity under 387nm excitation. The Y/B ratio can't be measured because of the absence of blue emission. But from the emission spectrum under 387nm excitation glass emits yellow colour.

Figure 9 is the PL emission spectrum of the BiBDy0.5glass measured at 460nm as excitation source at room temperature. The PL emission spectrum was measured range from 550nm to 900nm wavelength to know the emissions were useful for LED applications. It exhibit emission peaks at 575nm, 627nm in the visible region and emissions at 755, 848 and 882nm in the IR region.

3.7 Commission Internationale de l'Elcairage (CIE) study

Figure 10 represents the CIE chromaticity with colour coordinates of the glass sample. The CIE chromaticity coordinates is measured using emission spectra under the excitation of 387 nm.
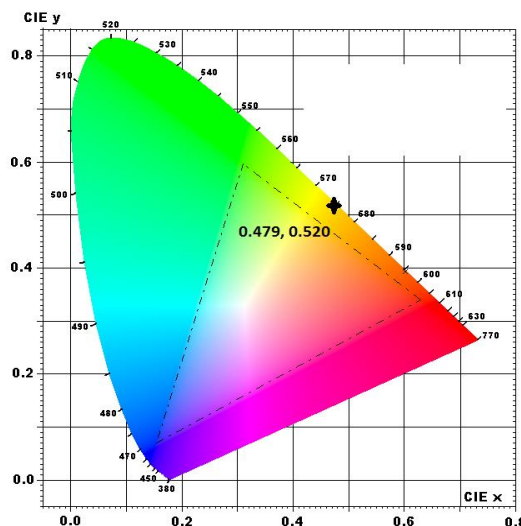

Fig. 10 CIE Co-ordinates of BiBDy0.5 glass at 387 nm excitation depicted on 1931 chart

The prepared glass sample, BiBDy0.5 shows having chromaticity co-ordinates X=0.479 and Y=0.520 with CCT value 3995 K, which is in yellow region in the chromaticity diagram. From the CIE plot, it is observed that prepared glass sample emit yellow light under the excitation of 387 nm which is useful for display applications. Generally the ratio of ED transition to the MD transition is a measure of symmetry orientation of the local environment around the RE ion site. The calculated Yellow/Blue (Y/B) intensity ratio $(^4F_{9/2} \rightarrow {}^6H_{13/2})$ / $(^4F_{9/2} \rightarrow {}^6H_{15/2})$ value is infinity. Y/B ratio depicts the local environment site symmetry and the electro-negativity of the surrounding ligands.

The CIE chromaticity co-ordinates for the prepared BiBDy0.5 glass (λ_{ex} =280 nm) and the values of the (x, y) color coordinates are found to be (0.393, 0.604) with CCT 4663K. Figure is not shown and it is noted from figure that, the (x, y) color co-ordinate values are found to lie in the greenish-yellow region, which is useful for white light generation.

IV. CONCLUSIONS

The following conclusions were obtained

1. The BiBDy0.5 and pure BiBDy0 glasses were prepared and characterized for their optical and luminescence properties using melt quenching technique successfully.
2. By doping Dy_2O_3 ions at low concentration 0.5% in substitution of Bi_2O_3 does not disturb the glass structure, transparency and density is preserved.
3. From the value of Urbach energy, it is observed that the BiBDy0.5 glass has low value of structural disorder, which indicates the strong structural stability.
4. Using J-O theory spectroscopic quality factor were calculated and compared with different glass systems doped with Dy^{3+} ions and found that our glass sample followed the different trend ($\Omega2 > \Omega6 > \Omega4$).
5. The absorption spectra consist of six absorption bands corresponding to the transitions from the ground $^6H_{15/2}$ ground level to the various excited states such as $^6F_{3/2}$, $^6F_{5/2}$, $^6F_{7/2}$, $^6F_{9/2}$, ($^6H_{9/2} \rightarrow {}^6F_{11/2}$) and $^6H_{11/2}$ transitions, states of the Dy^{3+} ions.
6. The emission spectra were studied under different excitation wavelengths to check the applicability in display devices.
7. The emission band at 575nm is observed from $^4F_{9/2} \rightarrow {}^6H_{13/2}$ transition under 387nm excitation wavelength, which belongs yellow band is sharp with high intensity. The blue band is absent so the Y/B intensity ratio is infinity.
8. The emission band at 482 and 562nm were observed from $^4F_{9/2} \rightarrow {}^6H_{15/2}$, $^4F_{9/2} \rightarrow {}^6H_{13/2}$ transitions respectively. From the emission spectrum under 280nm excitation wavelength, the blue band is less in intensity where as the greenish-yellow band is sharp with high intensity.
9. The BiB glass doped with 0.5 mol% of Dy_2O_3 gives the good result for luminescence properties.
10. The (x,y) chromaticity coordinates of the prepared glass consist in the greenish-yellow light region of the CIE1931 color chromaticity diagram indicating the glass is useful in display applications.
11. The Optical and Luminescence results were well matched with the other glass matrix prepared by the various researchers.

V. ACKNOWLEDGEMENTS

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