

Concentration effect of Eu^{3+} ion doped lithium zinc borosilicate glasses for laser applications

N. Jaidass¹, C. Krishnamoorthi², A. Mohan Babu³, M. Reddi Babu⁴ and P. Chandra Sekhar⁵

^{1,2} Center for Nanotechnology Research, Vellore Institute of Technology, Vellore - 632014

³Department of Physics, Audisankara college of Engineering and Technology, Gudur, A.P

⁴Department of Physics, Chadalawada Ramanamma Engineering college, Tirupati, A.P

⁵Department of Physics, S.V.University, Tirupati-517502

Abstract: This paper reports the concentration effect of Europium (Eu^{3+}) ion doped lithium zinc borosilicate (LZBS) glasses for laser applications. Through the melt quenching process, these glasses were prepared. Different physical properties such as oxygen packing density (O), average molecular weight (M), rare earth ion concentration (N), inter-ion distance (r_i), field strength (F), molar volume (Vm), molar refractivity (Rm), reflection loss (R) and metallization (R) were calculated in the prepared LZBS glasses. The oscillator strengths experimental (f_{exp}) and calculated (f_{cal}) of the absorption bands originating from the ground ($^7\text{F}_0$) and the first excited ($^7\text{F}_1$) states of Eu^{3+} ion are discussed. The Judd - Ofelt intensity (J-O) parameters (Ω_2 , Ω_4 , and Ω_6) were assessed from the absorption spectra by applying thermal correction to the doped glasses of Eu^{3+} . For the transition $^5\text{D}_0 \rightarrow ^7\text{F}_j$, $j = 0, 1, 2, 3$, and 4 of the Eu^{3+} ion, the J-O parameters (Ω_2 , Ω_4 , and Ω_6) are further used to measure radiative parameters such as transition probabilities (A_R), branching ratio (β_R), stimulated emission cross-section (σ_e) and radiative life period (τ_R). For the prepared glasses, decay analysis was performed for different concentrations of Eu^{3+} ions.

Keywords: Europium glasses, Judd - Ofelt parameters, Absorption spectra and Decay analysis

I. INTRODUCTION

In order to improve optical devices such as optical detectors, solid state lasers, waveguide lasers, optical fibre amplifiers, infrared to visible up-converters, field emission displays, etc., rare earth ion doped glasses are very useful. Current research focuses on Ln-doped glasses for infrared optical devices as well as visible optical devices [1-4]. Borosilicate glasses have outstanding optical properties in the present work and physical properties include greater chemical resilience and stronger mechanical properties. Borosilicate glass is a kind of glass with the major glass-forming components being silica and boron trioxide. Borosilicate glasses are known for their very low thermal expansion coefficients. In this glass system, lithium is more electropositive and glass former, lithium zinc borosilicate glasses consist of heavy metals, such as lithium and zinc. Due to its low transition temperature, high refractive index and low phonon energy, borosilicate glasses are more desirable hosts of rare-earth ion doped glasses for photonic applications [5-7]. The europium (Eu^{3+}) ion is the best candidate among Ln ions to be used as a spectroscopic probe to investigate the local structure in condensed matter around Ln ions. This is because the lower-lying energy level scheme of Eu^{3+} ions is simple and it is easy to apply the fluorescence line narrowing technique to evaluate shifts from site to site by energy level scheme analysis between $^7\text{F}_0$ and $^5\text{D}_0$ non-degenerate levels [8]. In photonic applications, Eu^{3+} doped materials are also widely used as red emitting phosphors for field emission technology because of the narrow and monochromatic emitting existence of Eu^{3+} ions due to the transition from $^5\text{D}_0$ to $^7\text{F}_2$.

The spectroscopic properties of Eu^{3+} ions in LZBS glasses are reported in this paper. By using the Judd-Ofelt (J-O) principle, the transition strength can be determined. A set of three intensity parameters Ω_λ ($\lambda = 2, 4$ and 6), which are sensitive to the Ln ion environment, is described by this theory. These intensity parameters are used to calculate the probability of radiative transition, excited state radiative lifetime, fluorescence branching ratios (that predict laser transition fluorescence intensity) and emission cross-sections for different transitions from the excited state. The best composition of the ion-host can be optimized by using these parameters to increase the laser efficiency for particular electronic transitions. All of these examined parameters are useful for accessing the visible region's emission properties of Eu^{3+} ion glasses. By controlling the $^5\text{D}_0$ to $^7\text{F}_2$ (616 nm) transition, decay times of the $^5\text{D}_0$ stage have been calculated.

II. EXPERIMENTAL PROCEDURE

2.1 Glass composition

LZBS glasses with chemical compositions of $(30-x)\text{H}_3\text{BO}_3 : 25\text{SiO}_2 : 10\text{Al}_2\text{O}_3 : 30\text{LiF} : 5\text{ZnO} : \text{xEu}_2\text{O}_3$ have been prepared using the conventional melt quenching technique at different Eu_2O_3 concentrations of 0.1, 0.5, 1.0 and 2.0 mol percent and are classified as LZBS Eu0.1, LZBS Eu0.5, LZBS Eu1.0 and LZBS Eu2.0 as shown in **Table I**. About 10 g of the batch chemicals were thoroughly mixed and melted to produce a homogeneous mixture and heated for 3 hours at 1200°C in an electric furnace and then the melt was poured onto a pre-heated brass plate. The glasses were annealed at 350°C for 7 hours after casting, to eliminate thermal strains and stress.

TABLE I: GLASS COMPOSITIONS AND CODING OF LZBS Eu^{3+} :GLASS

Glass code	H_3BO_3 (mol %)	SiO_2 (mol%)	Al_2O_3 (mol%)	LiF (mol%)	ZnO (mol%)	Eu_2O_3 (mol%)
LZBSEu0.0	30	25	10	30	5	0.0
LZBSEu0.1	29.9	25	10	30	5	0.1
LZBSEu0.5	29.5	25	10	30	5	0.5
LZBSEu1.0	29	25	10	30	5	1.0
LZBSEu2.0	28	25	10	30	5	2.0

2.2 Physical and optical measurements

The refractive indices (n) of the LZBS XEu glasses ($x = 0.0, 0.1, 0.5, 1.0$ and 2.0) were measured by using the Abbe refract metre with sodium vapour lamp, The densities (ρ) were determine by the Archimedes's principle and other physical parameters such as ionic concentration (N), inter ionic distance (r_i) etc.[9] were determined for LZBS Eu1.0 glasses as shown in **Table II**. Using the JASCO V-770 UV-VIS - NIR spectrophotometer, optical absorption spectra were recorded from 300 to 2500 nm, and photoluminescence, excitation and decay measurements were recorded using the FLS-3 spectro fluorimeter.

TABLE II : CERTAIN PHYSICAL PARAMETERS OF LZBS EU1.0 GLASS

Parameters	LZBS Eu1.0
Density (d)	2.646
Refractive index (n),	1.615
Concentration(c)	0.453
Ionic concentration (N) in 10^{20}	5.45
Oxygen packing density (O)	7.92
Inter atomic distance (r_i) \AA^0	12.24
Polaron radius (r_p) \AA^0	4.93
Field strength (F) in 10^{24}	12.34
Molar volume (V_m)	22.1
Molar refractivity (R_m)	7.69
Dielectric constant (ϵ)	2.608
Reflection loss (R)	5.53
Electronic polarizability (α_e) $\times 10^{-24}$	3.047
Metalizaion factor (M)	0.652

III. RESULT AND DISCUSSION

3.1 Optical absorption spectrum

Fig 1.(a), (b) and (c) shows that the optical absorption spectrum of LZBS Eu1.0 glass in the range of 300-2500 nm (UV-VIS & NIR) along with the absorption bands of Eu^{3+} ions. The absorption takes place from the ${}^7\text{F}_0$ ground as well as the next ${}^7\text{F}_1$ state, excited and thermally populated. The characteristic $4f^6-4f^6$ optical transitions of Eu^{3+} ions between ground and excited levels are due to certain absorption bands. The majority of transitions in the visible absorption spectrum of Eu^{3+} ion are very weak which is shown in the LZBS Eu1.0 absorption spectrum for UV, visible and near infrared (NIR) regions. The Eu^{3+} ion energy level structure can be partly deduced from both the absorption and the emission spectrum.

The absorption bands of Eu^{3+} ions originating from the ${}^7\text{F}_0$ ground state were centered at 361.5 nm (${}^5\text{D}_4$), 376 nm (${}^5\text{G}_3$), 381.5 nm (${}^5\text{G}_2$), 393.5 nm (${}^5\text{L}_6$), 464 nm (${}^5\text{D}_2$), 526 nm (${}^5\text{D}_1$), 578 nm (${}^5\text{D}_0$) and 2095 nm (${}^7\text{F}_6$), while at 400 nm (${}^5\text{L}_6$), 413.5 nm (${}^5\text{D}_3$), 532 nm (${}^5\text{D}_1$), 589 nm (${}^5\text{D}_0$) and 2205 nm (${}^7\text{F}_6$) the bands originating from the ${}^7\text{F}_1$ level were centred. Among all the absorption bands, the transition from ${}^7\text{F}_0$ to ${}^5\text{L}_6$ is found to be more extreme than any other transitions, even though it is forbidden by the S and L selection rules but allowed for the J selection rule. It is clear from the figures that 13 absorption bands have been observed, which are almost present in the ultraviolet and visible regions, and two more bands in the infrared region have been observed. Since the spin forbidden ${}^7\text{F}_j \rightarrow {}^5\text{D}_j$ absorption transitions are very weak [10], the ${}^7\text{F}_0 \rightarrow {}^5\text{D}_0$ transition is forbidden to spin and its strength is very weak and therefore not clearly observed in LZBSEu1.0 glass, the ${}^7\text{F}_0 \rightarrow {}^5\text{L}_6$ transition located at 393.5 nm (violet) is forbidden by the selection rules of ΔS and ΔL , but permitted by the selection rule of ΔJ and is more extreme than the other transitions.

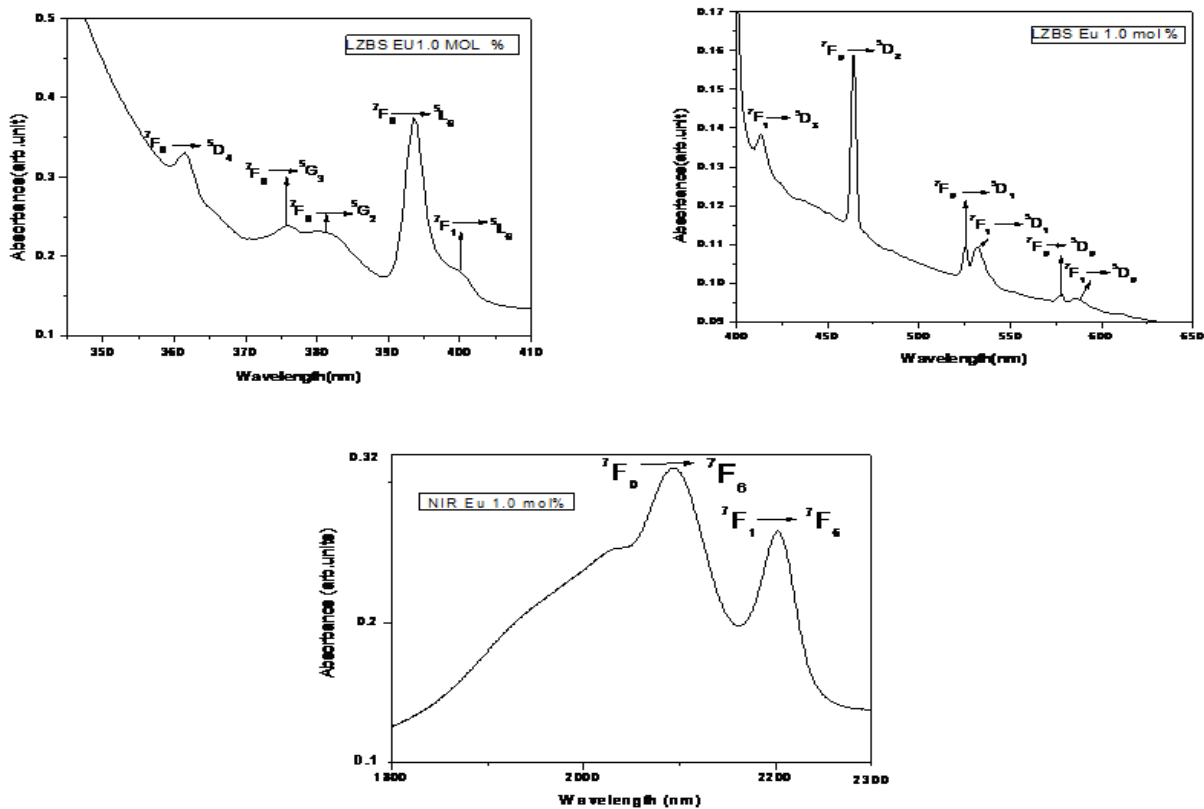


Fig1: Optical absorption spectrum of LZBSEu1.0glass (a) ultra violet (b) visible and (c) infrared regions

3.2 J-O intensity parameters

The absorption takes place both from the ground 7F_0 state as well as thermally coupled next excited 7F_1 state to higher excited states in the case of Eu^{3+} ions at room temperature. This peculiar feature allows various authors to use different procedures and carried out J-O analysis for Eu^{3+} ion [11]. For example, Ebendorff - Heidepriem and Ehrh [12] carried out JO analysis by considering absorption transitions of ${}^7F_0 \rightarrow {}^5D_2$, ${}^7F_0 \rightarrow {}^5D_4$ and ${}^7F_0 \rightarrow {}^5L_6$ transitions and calculated Ω_2 , Ω_4 and Ω_6 , respectively, and also by considering emission transitions of ${}^5D_0 \rightarrow {}^7F_2$ and ${}^5D_0 \rightarrow {}^7F_4$ transitions, calculated Ω_4 and Ω_6 , respectively. The ${}^5D_0 \rightarrow {}^7F_6$ transition found to be very weak and unable to determine Ω_6 value from emission spectra. These J-O parameters are in turn used and analyzed the radiative properties for the fluorescent levels. However, Balda et al.[13], completely ignored the ${}^7F_0 \rightarrow {}^5D_2$, ${}^7F_0 \rightarrow {}^5D_4$ and ${}^7F_0 \rightarrow {}^5L_6$ transitions due to uncertainties involved in it and carried out JO analysis by considering remaining absorption transitions. On the other hand, Pisarski [14], using absorption as well as excitation spectra, determined Ω_2 , Ω_4 and Ω_6 . Intensities of unnoticed transitions from absorption have been calculated from excitation spectra that are normalized with respect to absorption spectrum intensities. The J-O [15-16] study was therefore carried out in the present investigation using the observed absorption spectra, with and without thermal correction, as well as from the emission spectrum. The experimental (f_{exp}) and calculated (f_{cal}) oscillator strength are determined by following equations

$$f_{\text{exp}} = 4.32 \times 10^{-9} \int \varepsilon(\nu) d\nu$$

where $\varepsilon(\nu)$ is the molar extinction coefficient and

$$f_{\text{cal}} = \left[\frac{8\pi^2 m c \nu}{3\hbar(2J+1)} \right] \chi \times \sum_{\lambda=2,4,6} \Omega_{\lambda} \left(\psi J \parallel U^{\lambda} \parallel \psi' J' \right)$$

where m is the mass of the electron, c is velocity of light, \hbar is Planck's constant, ν is the mean energy of the transition. The Ω_{λ} ($\lambda = 2, 4, 6$) are the Judd-Ofelt intensity parameters and $\left\| U^{\lambda} \right\|^2$ are the squared reduced matrix elements of the unit tensor operator of the rank λ , which are calculated from the intermediate coupling approximation for a transition $\psi J \rightarrow \psi' J'$. The experimental oscillator strengths (f_{exp}) determined from the absorption spectra and the calculated oscillator strength (f_{cal}) obtained by the least square fit are presented in **Table III**. The small value of root mean square deviation (δ_{rms}) of $\pm 0.46 \times 10^{-6}$ signifies the best fit between the experimental and calculated oscillator strengths. The evaluated J-O intensity parameters of LZBSEu1.0 glass are compared with those of other Eu^{3+} doped glasses [17-20] in the **Table IV**.

TABLE III : ASSIGNMENTS OF ABSORPTION TRANSITIONS, THEIR ENERGIES (CM⁻¹), EXPERIMENTAL (F_{EXP}) AND CALCULATED (F_{CAL}) OSCILLATOR STRENGTHS (X10⁶) OF WITH AND WITHOUT THERMAL CORRECTION OF ABSORPTION BANDS OF EU³⁺ IONS LZBSEU1.0 GLASS

Transition	wave num(cm ⁻¹)	Oscillator strengths			
		Without thermal correction		With thermal correction	
⁷ F ₀ → ⁵ D ₄	27662	0.35	0.42	0.60	0.78
⁷ F ₀ → ⁵ G ₃	26595	1.02	0.39	1.92	1.20
⁷ F ₀ → ⁵ G ₂	26212	1.15	0.51	2.01	1.10
⁷ F ₀ → ⁵ L ₆	25412	1.48	0.34	2.59	0.72
⁷ F ₁ → ⁵ L ₆	25000	0.29	0.06	0.65	0.09
⁷ F ₁ → ⁵ D ₃	24183	0.23	0.21	0.50	0.46
⁷ F ₀ → ⁵ D ₂	21551	0.85	0.54	1.48	1.21
⁷ F ₀ → ⁵ D ₁	19011	0.10	0.12	0.19	0.16
⁷ F ₁ → ⁵ D ₁	18779	0.45	0.50	1.04	1.12
⁷ F ₀ → ⁵ D ₀	1730	0.11	0.12	0.13	0.15
⁷ F ₁ → ⁵ D ₀	16977	0.09	0.10	0.11	0.17
⁷ F ₀ → ⁷ F ₆	4769	0.38	0.35	0.67	0.77
⁷ F ₁ → ⁷ F ₆	4537	0.26	0.29	0.64	0.63
		Rms=0.462,		Rms=0.694	

TABLE IV : COMPARISON OF JUDD-OFELT INTENSITY PARAMETERS (Ω_J X 10⁻²⁰ CM²) AND THEIR TREND OF EU³⁺ IONS IN LZBS EU1.0GLASS ALONG WITH SOME OTHER REPORTED GLASSES

System	Ω ₂	Ω ₄	Ω ₆	Trend	Reference
LZBS Eu0.1	5.25	4.39	0.32	Ω ₂ > Ω ₄ > Ω ₆	Present work
LZBS Eu0.5	15.31	7.45	0.75	Ω ₂ > Ω ₄ > Ω ₆	Present work
LZBS Eu1.0	13.72	5.42	0.42	Ω ₂ > Ω ₄ > Ω ₆	Present work
LZBS Eu2.0	6.13	0.62	-	Ω ₂ > Ω ₄ > Ω ₆	Present work
PbFPEu10 glass (c)	3.51	0.47	0.00	Ω ₂ > Ω ₄ > Ω ₆	[17]
Fluoride phosphate	4.1	1.2	0.00	Ω ₂ > Ω ₄ > Ω ₆	[18]
PbO B ₂ O ₃ (b)	1.39	1.49	1.22	Ω ₄ > Ω ₂ > Ω ₆	[19]
PbO B ₂ O ₃ (d)	1.38	0.24	0.84	Ω ₂ > Ω ₆ > Ω ₄	[20]
PKSA Eu10(a)	6.34	5.67	0.68	Ω ₂ > Ω ₄ > Ω ₆	[21]
LTT Eu20	3.29	6.3	0.00	Ω ₄ > Ω ₂ > Ω ₆	[22]

The trend is followed by all J-O parameters as Ω₂ > Ω₄ > Ω₆. The magnitude of Ω₂ the parameter depends on the symmetry and determines the hypersensitivity of the active ion. The higher degree of symmetry around the active ion and greater covalence of the active ion-oxygen bond are shown by the lower value of Ω₂ the parameter. The structure-dependent and associated viscosity and rigidity of the medium in which the ions are located the parameters Ω₄ and Ω₆ [21 -22]. The significantly higher magnitude of the Eu³⁺ ion indicates a lower degree of symmetry and a relatively weaker covalence of the active ion-oxygen bond in the current study. Various radiative properties such as radiative transition probability (A_R), radiative lifetime (τ_R) and branching ratios (β_R) for the ⁵D₀ → ⁷F_J (J= 0-4) transitions of Eu³⁺ion have been evaluated under various constraints by using the J-O intensity parameters and the refractive index and are presented in **Table V**. Table 5 shows that the A_R of the magnetic dipole transition of ⁵D₀ → ⁷F₁ is independent of the magnitude of the J-O parameters.

TABLE V: RADIATIVE PARAMETERS SUCH AS EMISSION BAND POSITIONS (λ_P), EFFECTIVE BAND WIDTHS (Δλ_{EFF}), RADIATIVE TRANSITION PROBABILITIES (A_R), CALCULATED AND EXPERIMENTAL BRANCHING RATIOS(B_R & B_{EXP}), PEAK STIMULATED EMISSION CROSS SECTION (Σ_E) FOR EMISSION TRANSITIONS OF EU³⁺ IONS IN LZBS EU1.0 GLASS

Transitions	parameters	Present work	[23]	[24]	[25]
⁵ D ₀ → ⁷ F ₀	λ _p (nm)	578	582	579	578
	Δλ _{eff} (nm)	8.64	5.38	3.26	2.87
	A _R (S ⁻¹)	0	0	76.5	0
	σ _e (×10 ⁻²² cm ²)	0	0	11.71	0.00
	β _R	0.03	0.03	0.02	0.00
	β _{exp}	0.02	0.00	0.00	0.02
⁷ F ₁	λ _p (nm)	591	594	592	592
	Δλ _{eff} (nm)	14.72	7.99	11.71	12.68
	A _R (S ⁻¹)	50	64.07	0.00	60
	σ _e (×10 ⁻²² cm ²)	0.12	4.87	12.80	0.30
	β _R	0.15	0.201	0.19	0.22

	β_{exp}	0.13	0.199	0.12	0.28
7F_2	λ_p (nm)	612	616	613	612
	$\Delta\lambda_{eff}$ (nm)	12.02	6.33	11.21	11.38
	A_R (S^{-1})	354	250.37	210.2	142
	$\sigma_e(\times 10^{-22}cm^2)$	18.12	27.69	14.71	0.90
	β_R	0.63	0.783	0.65	0.52
	β_{exp}	0.61	0.778	0.69	0.64
7F_3	λ_p (nm)	654	565	653	563
	$\Delta\lambda_{eff}$ (nm)	9.79	6.8	8.79	10.61
	A_R (S^{-1})	20	41	49.5	0
	$\sigma_e(\times 10^{-22}cm^2)$	0	0	5.69	0.00
	β_R	0.02	0	0.02	0.00
	β_{exp}	0.01	0.018	0.00	0.01
7F_4	λ_p (nm)	702	705	702	702
	$\Delta\lambda_{eff}$ (nm)	16.02	4.81	12.88	13.74
	A_R (S^{-1})	18.09	7.43	0.00	71
	$\sigma_e(\times 10^{-22}cm^2)$	0.2	1.86	0.00	0.64
	β_R	0.11	0.023	0.12	0.26
	β_{exp}	0.09	0.028	0.09	0.05

3.3 Photoluminescence studies

It is important to know the right excitation wavelength of activator ions for the study of the optical properties of glasses. The excitation spectrum of LZBSEu1.0glass reported by 616 nm emission monitoring is illustrated in Fig. 2. At 361.5, 382, 393, 413, 464, 525 and 532 nm, the spectrum showed seven bands corresponding to the ${}^7F_0 \rightarrow {}^5D_4$, ${}^7F_1 \rightarrow {}^5L_1$, ${}^7F_0 \rightarrow {}^5L_6$, ${}^7F_1 \rightarrow {}^5D_3$, ${}^7F_1 \rightarrow {}^5D_2$, ${}^7F_1 \rightarrow {}^5D_1$, ${}^7F_1 \rightarrow {}^5D_0$ transitions, respectively. Among these excitation transitions, the ${}^7F_0 \rightarrow {}^5L_6$ transition (393 nm) is most intense and hence 393 nm has been used for recording the emission spectra.

The emission spectra in the 570-750 nm regions for different concentrations of Eu^{3+} doped LZBSEu glasses are shown in Fig 3. Five typical emission bands corresponding to ${}^5D_0 \rightarrow {}^7F_0$ (578 nm), ${}^5D_0 \rightarrow {}^7F_1$ (591 nm), ${}^5D_0 \rightarrow {}^7F_2$ (612 nm), ${}^5D_0 \rightarrow {}^7F_3$ (654 nm) and ${}^5D_0 \rightarrow {}^7F_4$ (702 nm) transitions were found in the emission spectra of LZBSEu1.0 glasses. The ${}^5D_0 \rightarrow {}^7F_2$ is more extreme among these five transitions and the other four ${}^5D_0 \rightarrow {}^7F_{0,1,3,4}$ transitions are very weak. It is noticed that the concentration quenching has been observed in the intensities of the emission bands, and also observed that the intensities of emission peaks increased upto 1.0 mol% and decreased for 2.0 mol% of Eu^{3+} ions concentrations. Table.5 [23-25] presents radiative parameters such as emission band positions (λ_p , nm), effective bandwidths ($\Delta\lambda_{eff}$, nm), radiative transition probabilities ($A_R(S^{-1})$), peak stimulated cross-sections (σ_e), calculated (β_R) and experimental (β_{exp}) branching ratios of the LZBSEu1.0 glass emission bands compared to the reported systems.

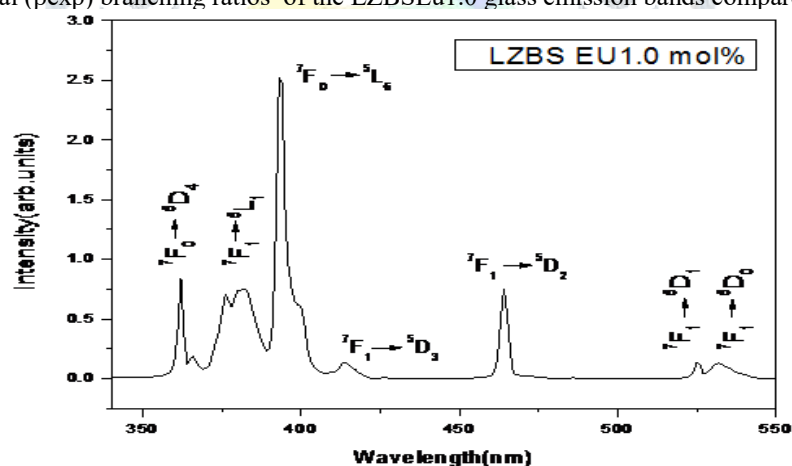


Fig 2: excitation spectra of LZBSEu10glass

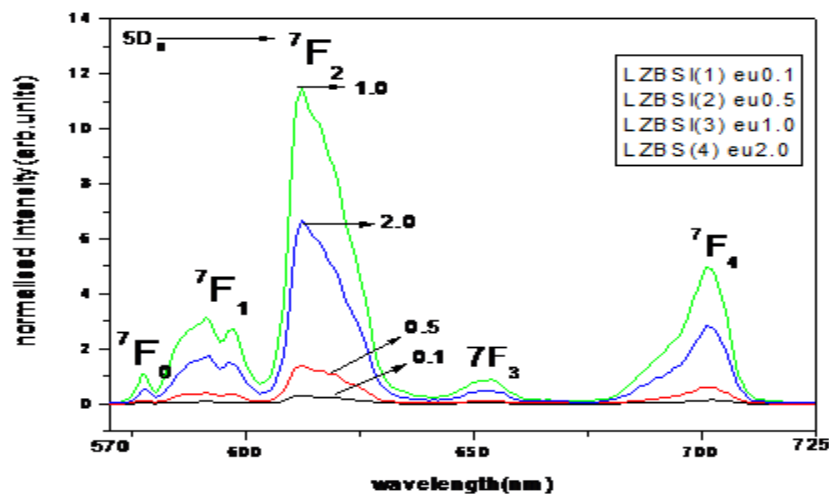


Fig 3: Photoluminescence of LZBSEu³⁺ doped glasses

3.4 Decay analysis

The decay rates for the ⁵D₀ fluorescent level of Eu³⁺ ion in title glasses were obtained by exciting the samples with 393 nm (⁷F₀ → ⁵L₆ transition) of xenon arc lamp and tracking the luminescence at 612 nm (⁵D₀ → ⁷F₂ transition) and are shown in Fig. 4. For all the concentrations of Eu³⁺ ions in the title glasses, the decay rates are identified to be single exponential. It is found that decay time decreases as the concentration of europium ions increases. The experimental decay time of ⁵D₀ fluorescent level of Eu³⁺ ions in LZBSEu0.1, LZBSEu0.5, LZBSEu1.0 and LZBSEu2.0 glasses is found to be 3.72, 3.64, 3.51 and 3.49 ms respectively, which is almost independent of Eu³⁺ ion concentration. It is obvious that the decay time decreases with increase of Eu³⁺ ion concentration due to the decrease in symmetry around Eu³⁺ ion.

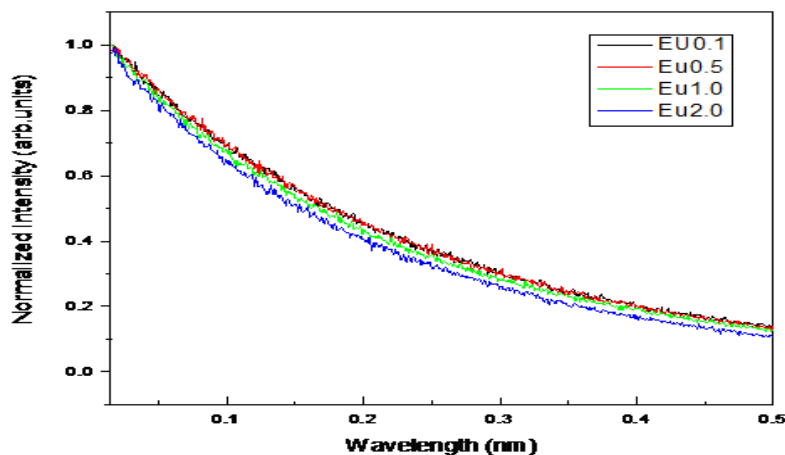


Fig 4: Decay rates for the ⁵D₀ level of Eu³⁺ : LZBS glasses

IV. CONCLUSIONS

Lithium Zinc Borosilicate glasses doped with Eu³⁺ ions have been prepared and studied through their spectral properties. The J-O parameters have been derived for the 1.0 mol% Eu³⁺ doped LZBS glass by analyzing its absorption spectrum. A relatively higher value of Ω_2 indicates higher rigidity and/or higher asymmetry around Eu³⁺ ions in the present host. Large stimulated emission cross-section, high branching ratios and quantum efficiencies are the usual characteristics of the good material for getting efficient laser light. It is observed that, as the europium ion concentration increases decaytime decreases. Overall, the strong red emission recorded at 612 nm corresponding to ⁵D₀ → ⁷F₂ transition of Eu³⁺ ions in the LZBSEu1.0 glass appears to be a good candidate for red laser source applications.

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