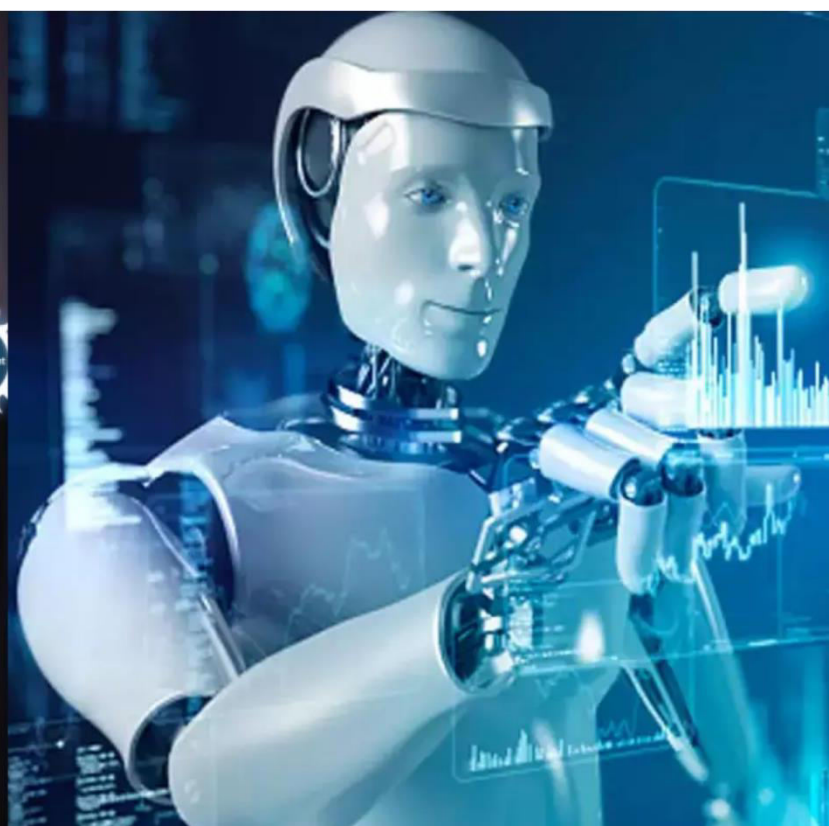




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Spectral, Transmittance and Thermal Properties of Pr^{3+} Doped Borosilicate Glasses with Large Balaji-Shankar Relation

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ABSTRACT: Yttrium Zinc lithium alumino magnesium lead borosilicate glasses containing Pr^{3+} in (25-x): SiO_2 : 10ZnO: 10 Li_2O : 10 Al_2O_3 : 10MgO: 10PbO: 10 Y_2O_3 : 15 B_2O_3 : $x\text{Pr}_2\text{O}_3$ (where $x=1, 1.5, 2$ mol %) have been prepared by melt-quenching method. The amorphous nature of the glasses was confirmed by x-ray diffraction studies. Optical absorption, Excitation spectra and fluorescence spectra were recorded at room temperature for all glass samples. The thermal parameters were calculated using DTA Thermogram. The glass transition temperature (T_g), onset crystallization temperature (T_c), crystallization temperature (T_p), melting temperature (T_m), thermal stability (T_s), Balaji Parameter (B_p), Hurbe's criterion (H_R), reduced glass transition temperature (T_{rg}) and Shankar parameter (K_s) were calculated. Judd-Ofelt intensity parameters Ω_λ ($\lambda=2, 4, 6$) are evaluated from the intensities of various absorption bands of optical absorption spectra. Using these intensity parameters various radiative properties like spontaneous emission probability, branching ratio, radiative life time and stimulated emission cross-section of various emission lines have been evaluated.

KEYWORDS: YZLAMLBS Glasses, Optical Properties, Judd-Ofelt Theory, Thermal Properties.

I. INTRODUCTION

Rare earth doped glassy materials are of great importance due to their potential application in optical fiber amplifiers, wavelength converting devices, optical fiber amplifiers, [1–5]. Silicate glasses exhibit very important physical properties such as high refractive index, large transparency, low phonon energy, better thermal stability, good mechanical, high density and chemical durability [6-10]. Pr^{3+} doped silicate glasses are very important because of the possibility of their application in optic device fields, optoelectronic, laser action, optical, electronic devices, NIR lasers and fiber amplifiers [11-14]. The addition of network modifier (NMF) Li_2O is to improve both electrical and mechanical properties of such glasses. Recently, rare earth doped silicate glass has attracted much interest because of high rare earth ion solubility, low melting point, high thermal stability, good rare earth ions solubility and high phonon energy [15-18].

In this work, the spectroscopic properties of Pr^{3+} -doped (25-x): SiO_2 : 10ZnO: 10 Li_2O : 10 Al_2O_3 : 10MgO: 10PbO: 10 Y_2O_3 : 15 B_2O_3 : $x\text{Pr}_2\text{O}_3$ (where $x=1, 1.5, 2$ mol %) glasses were investigated. The DTA, absorption, Excitation and fluorescence spectra of Pr^{3+} of the glasses were investigated. The thermal parameters are useful for thermionic emission. The J-O intensity parameters render significant information regarding local structure and bonding in the vicinity of rare- earth ions.

II. EXPERIMENTAL TECHNIQUES

Preparation of glasses

The following Pr^{3+} doped yttrium zinc lithium alumino magnesium lead borosilicate glass samples (25-x): SiO_2 : 10ZnO: 10 Li_2O : 10 Al_2O_3 : 10MgO: 10PbO: 10 Y_2O_3 : 15 B_2O_3 : $x\text{Pr}_2\text{O}_3$ (where $x=1, 1.5, 2$) have been prepared by melt-quenching method. Analytical reagent grade chemical used in the present study consist of SiO_2 , ZnO, Li_2O , Al_2O_3 , MgO, PbO, Y_2O_3 , B_2O_3 and Pr_2O_3 . All weighed chemicals were powdered by using an Agate pestle mortar and mixed thoroughly before each batch (10g) was melted in alumina crucibles in silicon carbide based an electrical furnace.

Silicon Carbide Muffle furnace was heated to working temperature of 980°C, for preparation of yttrium zinc lithium alumino magnesium lead borosilicate glasses, for two hours to ensure the melt to be free from gases. The melt was stirred several times to ensure homogeneity. For quenching, the melt was quickly poured on the steel plate & was immediately inserted in the muffle furnace for annealing. The steel plate was preheated to 100°C. While pouring, the temperature of crucible was also maintained to prevent crystallization. And annealed at temperature of 350°C for 2h to remove thermal strains and stresses. Every time fine powder of cerium oxide was used for polishing the samples. The glass samples so prepared were of good optical quality and were transparent. The chemical compositions of the glasses with the name of samples are summarized in Table 1

Table 1 Chemical composition of the glasses

Sample	Glass composition (mol %)
YZLAMLBS (UD)	25 SiO ₂ : 10ZnO: 10Li ₂ O: 10Al ₂ O ₃ : 10MgO: 10PbO: 10Y ₂ O ₃ : 15B ₂ O ₃
YZLAMLBS PR(1.0)	24 SiO ₂ : 10ZnO: 10Li ₂ O: 10Al ₂ O ₃ : 10MgO: 10PbO: 10Y ₂ O ₃ : 1 Pr ₂ O ₃
YZLAMLBS PR(1.5)	23.5SiO ₂ : 10ZnO: 10Li ₂ O: 10Al ₂ O ₃ : 10MgO: 10PbO: 10Y ₂ O ₃ : 1.5 Pr ₂ O ₃
YZLAMLBS PR(2.0)	23SiO ₂ : 10ZnO: 10Li ₂ O: 10Al ₂ O ₃ : 10MgO: 10PbO: 10Y ₂ O ₃ : 2 Pr ₂ O ₃

YZLAMLBS (UD)—Represents undoped Yttrium Zinc lithium alumino magnesium lead borosilicate glass specimen.

YZLAMLBS (PR) -Represents Pr³⁺ Lead Yttrium Zinc lithium alumino magnesium lead borosilicate glass specimens.

III. THEORY

3.1 Oscillator Strength

The intensity of spectral lines are expressed in terms of oscillator strengths using the relation [19].

$$f_{\text{expt.}} = 4.318 \times 10^{-9} \int \epsilon(\nu) d\nu \quad (1)$$

where, $\epsilon(\nu)$ is molar absorption coefficient at a given energy ν (cm⁻¹), to be evaluated from Beer–Lambert law.

Under Gaussian Approximation, using Beer–Lambert law, the observed oscillator strengths of the absorption bands have been experimentally calculated, using the modified relation [20].

$$P_m = 4.6 \times 10^{-9} \times \frac{1}{cl} \log \frac{I_0}{I} \times \Delta\nu_{1/2} \quad (2)$$

where c is the molar concentration of the absorbing ion per unit volume, l is the optical path length, $\log I_0/I$ is absorptivity or optical density and $\Delta\nu_{1/2}$ is half band width.

3.2. Judd-Ofelt Intensity Parameters

According to Judd [21] and Ofelt [22] theory, independently derived expression for the oscillator strength of the induced forced electric dipole transitions between an initial J manifold $|4f^N(S, L) J\rangle$ level and the terminal J' manifold $|4f^N(S', L') J'\rangle$ is given by:

$$\frac{8\pi^2 m c \bar{\nu}}{3h(2J+1)n} \left[\frac{(n^2+2)^2}{9} \right] \times S(J, J') \quad (3)$$

where, the line strength $S(J, J')$ is given by the equation

$$S(J, J') = e^2 \sum_{\lambda=2, 4, 6} \Omega_{\lambda} \langle 4f^N(S, L) J \| U^{(\lambda)} \| 4f^N(S', L') J' \rangle^2 \quad (4)$$

In the above equation m is the mass of an electron, c is the velocity of light, ν is the wave number of the transition, h is Planck's constant, n is the refractive index, J and J' are the total angular momentum of the initial and final level respectively, Ω_{λ} ($\lambda = 2, 4$ and 6) are known as Judd-Ofelt intensity parameters.

3.3. Radiative Properties

The Ω_{λ} parameters obtained using the absorption spectral results have been used to predict radiative properties such as spontaneous emission probability (A) and radiative life time (τ_R), and laser parameters like fluorescence branching ratio (β_R) and stimulated emission cross section (σ_p).

The spontaneous emission probability from initial manifold $|4f^N(S', L') J' \rangle$ to a final manifold $|4f^N(S, L) J \rangle$ is given by:

$$A[(S', L') J'; (S, L) J] = \frac{64 \pi^2 \nu^3}{3h(2J'+1)} \left[\frac{n(n^2+2)^2}{9} \right] \times S(J', \bar{J}) \quad (5)$$

Where, $S(J', J) = e^2 [\Omega_2 \| U^{(2)} \|^2 + \Omega_4 \| U^{(4)} \|^2 + \Omega_6 \| U^{(6)} \|^2]$

The fluorescence branching ratio for the transitions originating from a specific initial manifold $|4f^N(S', L') J' \rangle$ to a final manifold $|4f^N(S, L) J \rangle$ is given by

$$\beta[(S', L') J'; (S, L) J] = \sum_{S, L, J} \frac{A[(S', L') J'; (S, L) J]}{A[(S', L') J'; (S, L) J]} \quad (6)$$

where, the sum is over all terminal manifolds.

The radiative life time is given by

$$\tau_{rad} = \sum_{S, L, J} A[(S', L') J'; (S, L) J] = A_{Total}^{-1} \quad (7)$$

where, the sum is over all possible terminal manifolds. The stimulated emission cross-section for a transition from an initial manifold $|4f^N(S', L') J' \rangle$ to a final manifold $|4f^N(S, L) J \rangle$ is expressed as

$$\sigma_p(\lambda_p) = \left[\frac{\lambda_p^4}{8\pi c n^2 \Delta \lambda_{eff}} \right] \times A[(S', L') J'; (S, L) J] \quad (8)$$

where, λ_p the peak fluorescence wavelength of the emission band and $\Delta \lambda_{eff}$ is the effective fluorescence line width.

3.4 Nephelauxetic Ratio (β) and Bonding Parameter ($b^{1/2}$)

The nature of the R-O bond is known by the Nephelauxetic Ratio (β) and Bonding Parameters ($b^{1/2}$), which are computed by using following formulae [23, 24]. The Nephelauxetic Ratio is given by

$$\beta' = \frac{\nu_g}{\nu_a} \quad (9)$$

where, ν_a and ν_g refer to the energies of the corresponding transition in the glass and free ion, respectively. The values of bonding parameter $b^{1/2}$ are given by

$$b^{1/2} = \left[\frac{1-\beta'}{2} \right]^{1/2} \quad (10)$$

IV. RESULT AND DISCUSSION

4.1. XRD Measurement

Figure 1 presents the XRD pattern of the samples containing show no sharp Bragg's peak, but only a broad diffuse hump around low angle region. This is the clear indication of amorphous nature with in the resolution limit of XRD instrument.

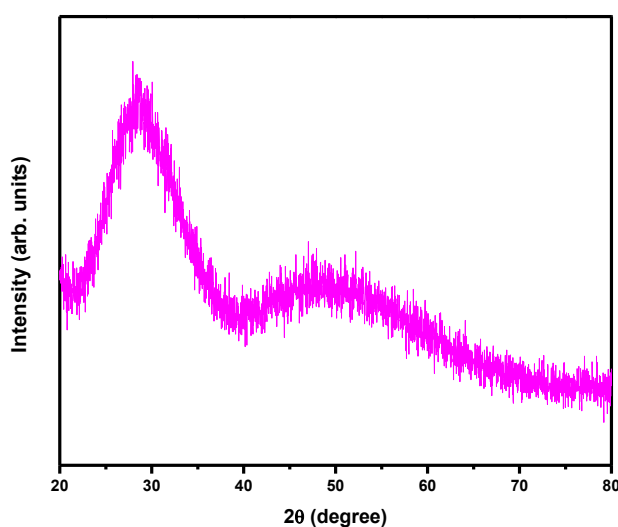


Fig.1: X-ray diffraction pattern of YZLAMLBS PR(1.0) glass.

4.2 Transmittance Spectra

The Transmittance spectra of YZLAMLBS PR(1.0) is shown in Fig.2

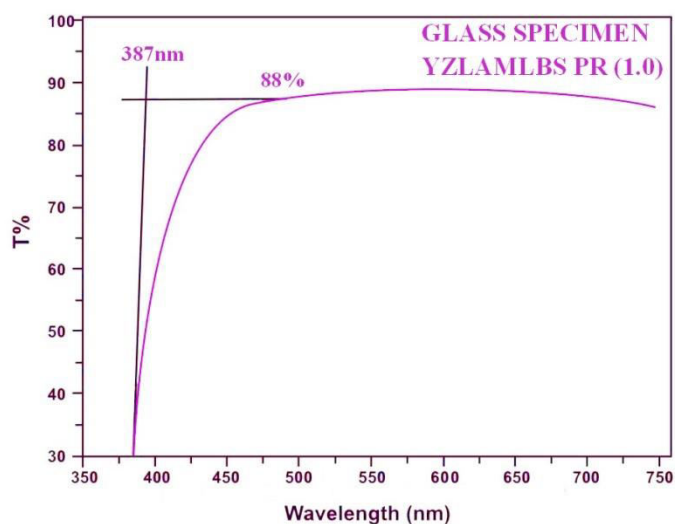


Fig.2: Transmittance spectra of YZLAMLBS PR (1.0) glass.

4.3 Thermal Property

Differential thermal analysis checks the heat absorbed by glass samples during heating or cooling. Fig. 3 depicts the DTA thermogram of powdered YZLAMLBS PR(1.0) sample. The glass transition temperature (T_g), onset crystallization temperature (T_c), crystallization temperature (T_p), melting temperature (T_m), thermal stability (T_s), Balaji Parameter (B_p), Hurbe's criterion (H_R), reduced glass transition temperature (T_{rg}) and Shankar parameter were calculated. Balaji-Shankar relation also calculated. All the determined thermal parameters are given in table 2.

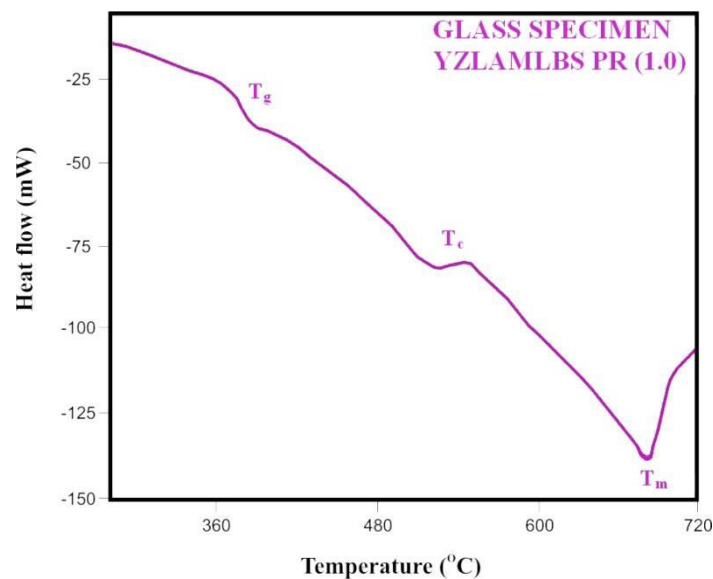


Fig.3: DTA curve of YZLAMLBS PR (1.0) glass.

Table.2. Thermal parameters determined from the DTA traces of YZLAMLBS PR glasses.

Sample Name	$T_g(^{\circ}\text{C})$	$T_c(^{\circ}\text{C})$	$T_p(^{\circ}\text{C})$	$T_m(^{\circ}\text{C})$	$T_s(^{\circ}\text{C})$	$B_p(^{\circ}\text{C})$	$H_R(^{\circ}\text{C})$	$K_s(^{\circ}\text{C})$	$T_{rg} (^{\circ}\text{C})$	$B_p \times K_s$
YZLAMLBS PR (1.0)	375	511	550	685	136	3.487	0.224	34.546	0.547	120.462
YZLAMLBS PR(1.5)	380	512	552	688	132	3.300	0.227	33.767	0.552	111.431
YZLAMLBS PR (2.0)	385	515	555	692	130	3.250	0.226	33.251	0.556	108.066

The thermal stability of the glass samples can be calculated by difference between onset crystallization temperature and transition temperature [25].

$$\text{Thermal Stability } (T_s) = T_c - T_g \quad (9)$$

Balaji Parameter can be calculated using [25].

$$\text{Balaji Parameter } (B_p) = [(T_c - T_g) / (T_p - T_c)] \quad (10)$$

Hruby's criterion is calculated using the Hurby's relation [25].

$$\text{Hruby's criterion } (H_R) = [(T_p - T_c) / (T_m - T_c)] \quad (11)$$

Reduced glass transition temperature is given as [25].

$$\text{Reduced glass transition temperature } (T_{rg}) = T_g / T_m \quad (12)$$

Shankar Parameter is given as [25].

$$K_S = [(T_m - T_c) (T_c - T_g) / T_m] \quad (13)$$

Balaji-Shankar relation [26].

$$(B_p \times K_S) = [(T_c - T_g)^2 (T_m - T_c) / T_m (T_p - T_c)] \quad (14)$$

4.4. Absorption spectra

The absorption spectra of YZLAMLBS (PR) glasses, consists of absorption bands corresponding to the absorptions from the ground state 3H_4 of Pr^{3+} ions. Eight absorption bands have been observed from the ground state 3H_4 to excited states 3F_2 , 3F_3 , 3F_4 , 1G_4 , 1D_2 , 3P_0 , 3P_1 and 3P_2 for Pr^{3+} doped YZLAMLBS (PR) glasses.

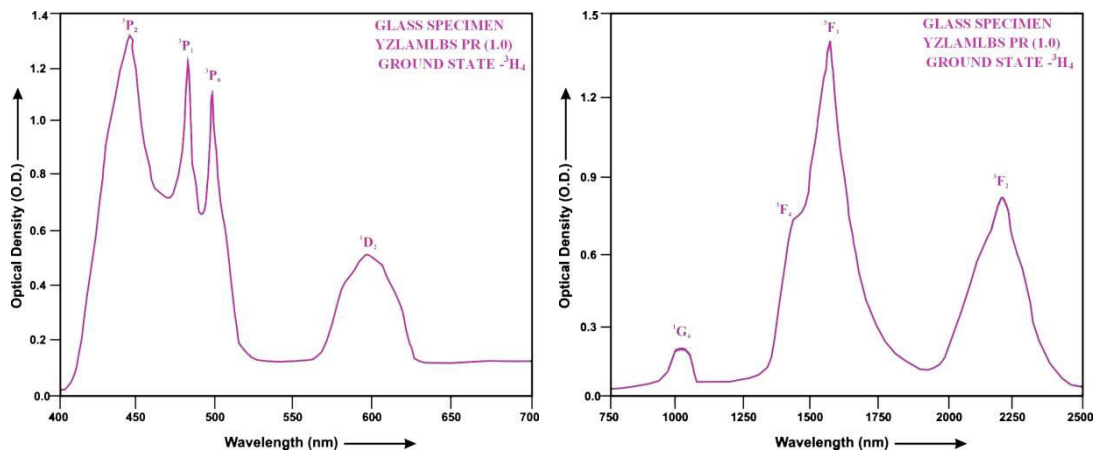


Fig.4: Absorption spectra of YZLAMLBS PR (01) glass.

The experimental and calculated oscillator strengths for Pr^{3+} ions in yttrium zinc lithium alumino magnesium lead borosilicate glasses are given in Table 3

Table.3. Measured and calculated oscillator strength ($P^m \times 10^{+6}$) of Pr^{3+} ions in YZLAMLBS glasses.

Energy level 3H_4	Glass YZLAMLBS PR(1.0)		Glass YZLAMLBS PR(1.5)		Glass YZLAMLBS PR(2.0)	
	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$
3F_2	4.47	3.76	3.64	3.06	2.55	2.09
3F_3	6.88	6.00	5.86	5.07	4.74	4.00
3F_4	4.24	3.74	3.38	3.13	2.38	2.47
1G_4	0.38	0.31	0.26	0.26	0.22	0.21
1D_2	2.38	1.07	1.88	0.90	1.24	0.71
3P_0	4.50	1.48	3.69	1.36	2.58	1.13
3P_1	4.63	2.59	3.84	2.32	2.83	1.89
3P_2	12.43	3.54	11.36	2.98	10.47	2.37
R.m.s.deviation	3.4542		3.1617		2.9487	

Computed values of Slater-Condon, Lande', Racah, nephelauxetic ratio and bonding parameter for Pr^{3+} doped YZLAMLBS glass specimens are given in Table 4.

Table4. Computed values of Slater-Condon, Lande', Racah, nephelauxetic ratio and bonding parameter for Pr^{3+} doped YZLAMLBS glass specimens.

Parameter	Free ion	YZLAMLBS PR(1.0)	YZLAMLBS PR(1.5)	YZLAMLBS PR(2.0)
$F_2(\text{cm}^{-1})$	322.09	300.02	299.99	299.99
$F_4(\text{cm}^{-1})$	44.46	44.28	44.25	44.26
$F_6(\text{cm}^{-1})$	4.867	4.413	4.411	4.412
$\xi_{4f}(\text{cm}^{-1})$	741.00	858.21	858.80	858.56
$E^1(\text{cm}^{-1})$	4728.92	4451.59	4450.28	4450.68
$E^2(\text{cm}^{-1})$	24.75	22.01	22.01	22.01
$E^3(\text{cm}^{-1})$	478.10	454.74	454.69	454.70
F_4/F_2	0.13804	0.14758	0.14749	0.14754
F_6/F_2	0.01511	0.014708	0.014704	0.014706
E^1/E^3	9.8911	9.7893	9.7876	9.7882
E^2/E^3	0.0518	0.04840	0.04842	0.04841
β'		0.8888	0.8885	0.8886
$b^{1/2}$		0.2358	0.2361	0.2360

The values of Judd-Ofelt intensity parameters are given in Table 5.

Table.5. Judd-Ofelt intensity parameters for Pr^{3+} doped YZLAMLBS glass specimens.

Glass Specimen	$\Omega_2(\text{pm}^2)$	$\Omega_4(\text{pm}^2)$	$\Omega_6(\text{pm}^2)$	Ω_4/Ω_6
YZLAMLBS PR(1.0)	2.694	2.073	5.284	0.3923
YZLAMLBS PR(1.5)	2.063	1.904	4.400	0.4327
YZLAMLBS PR(2.0)	1.199	1.578	3.479	0.4536

4.5 Excitation Spectrum

Excitation spectra of YZLAMLBS PR (1.0) glass recorded at the emission wavelength 395 nm is depicted as figure 5. The excitation spectra consists of three peaks corresponding to the transitions from the ground state $^3\text{H}_4$ to the various excited states $^3\text{P}_2$, $^3\text{P}_1$ and $^3\text{P}_0$ at the wavelengths of 448, 465 and 486 nm respectively. Among these, a prominent excitation band at 448 nm has been selected for the measurement of emission spectrum of YZLAMLBS PR (1.0) glass.

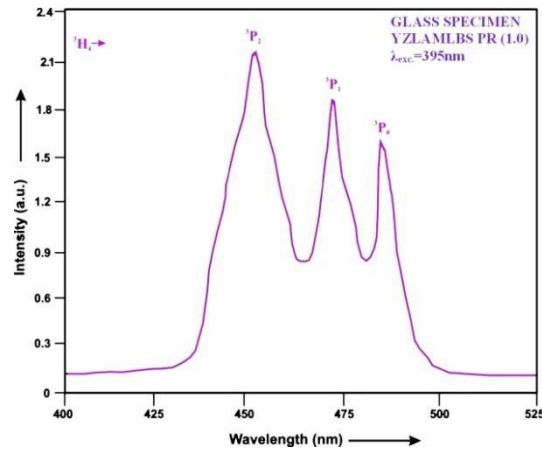


Fig.5: Excitation Spectrum of YZLAMLBS PR (1.0) glass.

4.6 Fluorescence Spectrum

The fluorescence spectrum of Pr^{3+} doped in yttrium zinc lithium aluminosilicate lead borosilicate glass is shown in Figure 6. There are eleven broad bands ($^3\text{P}_0 \rightarrow ^3\text{H}_4$), ($^3\text{P}_0 \rightarrow ^3\text{H}_5$), ($^1\text{D}_2 \rightarrow ^3\text{H}_4$), ($^3\text{P}_0 \rightarrow ^3\text{H}_6$), ($^3\text{P}_0 \rightarrow ^3\text{F}_2$), ($^3\text{P}_1 \rightarrow ^3\text{F}_3$), ($^1\text{D}_2 \rightarrow ^3\text{H}_5$), ($^3\text{P}_0 \rightarrow ^3\text{F}_4$), ($^1\text{G}_4 \rightarrow ^3\text{H}_5$), ($^1\text{G}_4 \rightarrow ^3\text{H}_6$), and ($^1\text{G}_4 \rightarrow ^3\text{F}_4, ^3\text{F}_2$), respectively for glass specimens.

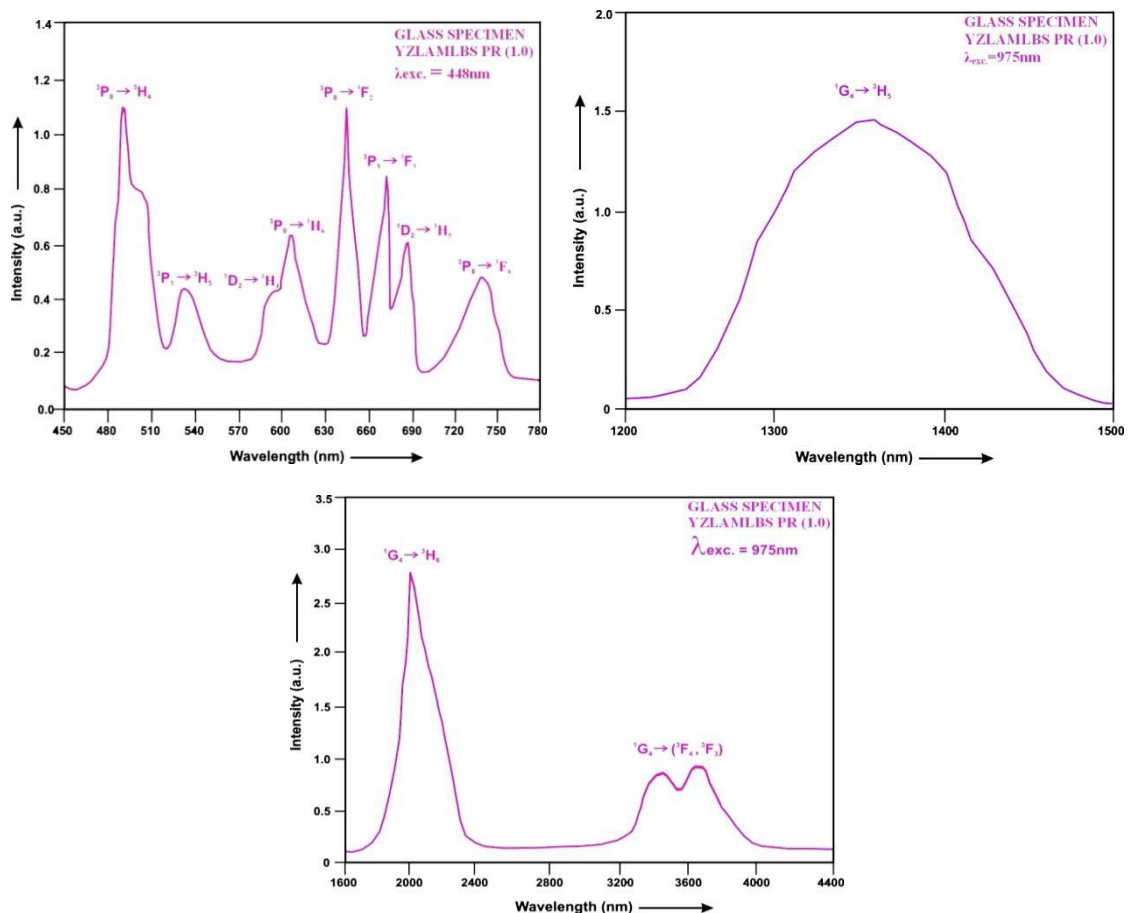


Fig.6: Fluorescence spectrum of YZLAMLBS PR (1.0) glass.

Table 6. Emission peak wave lengths (λ_p), radiative transition probability (A_{rad}), branching ratio (β_R), stimulated emission crosssection (σ_p), and radiative life time (τ) for various transitions in Pr^{3+} doped YZLAMLBS glasses.

Transiti on	λ_{ma} x (n m)	YZLAMLBS PR (1.0)				YZLAMLBS PR (1.5)				ZLATLBB PR (2.0)			
		$A_{rad}(\text{s}^{-1})$	β	$\sigma_p(10^{-20} \text{ cm}^2)$	$\tau_R(\mu\text{s})$	$A_{rad}(\text{s}^{-1})$	β	$\sigma_p(10^{-20} \text{ cm}^2)$	$\tau_R(\mu\text{s})$	$A_{rad}(\text{s}^{-1})$	β	$\sigma_p(10^{-20} \text{ cm}^2)$	$\tau_R(10^{-20} \text{ cm}^2)$
$^3P_0 \rightarrow ^3H_4$	485	1293.64	0.1168	0.405	90.2 687	1190.49	0.1290	0.383	108. 39	988.58	0.1490	0.331	150.6 9
$^3P_1 \rightarrow ^3H_5$	532	2402.45	0.2169	0.442		2119.41	0.2297	0.395		172.61	0.2601	0.329	
$^1D_2 \rightarrow ^3H_4$	599	580.02	0.0524	0.216		488.91	0.0530	0.185		387.97	0.0585	0.152	
$^3P_0 \rightarrow ^3H_6$	602	505.93	0.0457	0.245		422.11	0.0458	0.210		334.40	0.0504	0.169	
$^3P_0 \rightarrow ^3F_2$	643	2230.29	0.2013	1.904		1711.23	0.1855	1.476		996.49	0.1502	0.891	
$^3P_1 \rightarrow ^3F_3$	676	3366.96	0.3039	1.705		2691.10	0.2917	1.377		1727.87	0.2604	0.904	
$^1D_2 \rightarrow ^3H_5$	685	6.40	0.00058	0.0062		5.698	0.00062	0.0058		4.66	0.0007	0.0049	
$^3P_0 \rightarrow ^3F_4$	730	238.97	0.0216	0.1674		219.91	0.0238	0.158		182.61	0.0275	0.134	
$^1G_4 \rightarrow ^3H_5$	1350	276.12	0.0249	0.8747		231.37	0.0251	0.0074		182.18	0.0274	0.585	
$^1G_4 \rightarrow ^3H_6$	2025	157.27	0.0142	5.196		129.46	0.0140	4.333		94.78	0.0143	3.245	
$^1G_4 \rightarrow (^3F_4, ^3F_2)$	3555	20.05	0.0018	3.356		16.15	0.0018	2.719		10.60	0.0016	1.797	

V. CONCLUSION

In the present study, the glass samples of composition (25-x): SiO_2 : 10ZnO: 10Li₂O: 10Al₂O₃: 10MgO: 10PbO: 10Y₂O₃: 15B₂O₃: xPr₂O₃ (where x = 1, 1.5, 2 mol %) have been prepared by melt-quenching method. The value of Balaji-Shankar relation is found to be greater than 100 °C, which indicates that the prepared glass samples have good thermal stability. The stimulated emission cross-section (σ_p) has highest value for the transition ($^1G_4 \rightarrow ^3H_6$) in all the glass specimen doped with Pr^{3+} ion. The result shows that the glass samples are useful for thermionic emission and laser applications.

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