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Structural, Thermal and Spectral features of Ho^{3+}doped Borophosphate Glasses for Photonic and Laser Materials Applications

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Abstract: ***Glasses samples containing Ho^{3+} in Ytterbium Zinc Lithium Cadmium Potassium niobate Borophosphate Glasses (25-x)P_2O_5: 10ZnO: 10Li_2O: 10CdO: 10K_2O: 10Nb_2O_5:10Y_2O_3 :15B_2O_3 :xHo_2O_3 (where x=1, 1.5,2 mol %) have been prepared by melt-quenching method. The amorphous nature of the prepared glass samples was confirmed by X-ray diffraction. DTA curve was analysed to evaluate the glass transition temperature, crystallization temperature and melting temperature. Optical absorption, Excitation and fluorescence spectra were recorded at room temperature for all glass samples. Judd-Ofelt intensity parameters Ω_λ (λ=2, 4 and 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 (A), branching ratio (β), radiative life time (τ_R) and stimulated emission cross–section (σ_p) of various emission lines have been evaluated.***
Keywords: YZLCPNBP Glasses, Optical Properties, Judd-Ofelt Theory, Radiative Properties.

I. INTRODUCTION

Rare earth glasses have attracted much attention, because they have potential applications such as optical fiber amplifiers, up-conversion lasers, waveguide laser, optical fiber amplifiers, electro-luminescent devices and memory devices [1–5].Among different glasses borate glasses have some important properties. They have high refractive index high transparency and low dispersion rates with good physical, chemical and thermal stability [6-8].The borate glasses is also compressed because of their relatively large phonon energy. Borate glasses can be a good matrix for fiber lasers because they exhibit good mechanical property [9-12].The addition of network modifier (NWF) Li_2O is to improve optical, electrical and mechanical properties of such glasses. Zinc oxide is added in the glass matrix to increase glass forming ability and to ensure low rates of crystallization in the glass system. Among active rare-earth ions Ho^{3+} exhibits high solubility in ceramic glasses, which also possess excellent physical, optical and Thermal properties [13-15].

The present work reports on the preparation and characterization of rare earth doped heavy metal oxide (HMO) glass systems for lasing materials. I have studied on the Optical absorption, Excitation, fluorescence and DTA spectra of Ho^{3+} doped ytterbium zinc lithium cadmium potassium niobate borophosphate glasses. The intensities of the transitions for the rare earth ions have been estimated successfully using the Judd-Ofelt theory, The laser parameters such as radiative probabilities(A),branching ratio (β), radiative life time(τ_R) and stimulated emission cross section(σ_p) are evaluated using J.O.intensity parameters(Ω_λ, λ=2,4 and 6).

II. EXPERIMENTAL TECHNIQUES

A. Preparation of glasses

The following Ho^{3+}doped borophosphate glass samples (25-x)P_2O_5: 10ZnO: 10Li_2O: 10CdO: 10K_2O: 10Nb_2O_5:10Y_2O_3 :15B_2O_3:xHo_2O_3. (where x=1,1.5 and 2 mol%) have been prepared by melt-quenching method. Analytical reagent grade chemical used in the present study consist of P_2O_5, ZnO, Li_2O, CdO, K_2O,Nb_2O_5,Y_2O_3, B_2O_3 and Ho_2O_3. They were thoroughly mixed by using an agate pestle mortar. then melted at 1060^0C by an electrical muffle furnace for 2h., After complete melting, the melts were quickly poured in to a preheated stainless steel mould and annealed at temperature of 250^0C 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.

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Table 1.

Chemical composition of the glasses

Sample	Glass composition (mol %)
YZLCPNBP (UD)	$25Bi_2O_3$: 10ZnO: $10Li_2O$: $10Al_2O_3$: 10CdO: 10CaO: $10Na_2O$:$15B_2O_3$
YZLCPNBP HO (1.0)	$24Bi_2O_3$: 10ZnO: $10Li_2O$: $10Al_2O_3$: 10CdO: 10CaO: $10Na_2O$:$15B_2O_3$:$1Ho_2O_3$
YZLCPNBP HO (1.5)	$23.5Bi_2O_3$: 10ZnO: $10Li_2O$: $10Al_2O_3$: 10CdO: 10CaO: $10Na_2O$:$15B_2O_3$:1.5 Ho_2O_3
YZLCPNBP HO (2.0)	$23Bi_2O_3$: 10ZnO: $10Li_2O$: $10Al_2O_3$: 10CdO: 10CaO: $10Na_2O$:$15B_2O_3$:2 Ho_2O_3

YZLCPNBP (UD) -Represents undoped Ytterbium Zinc Lithium Cadmium Potassium niobate Borophosphate glass specimen.
YZLCPNBP (HO)-Represents Ho^{3+} doped Ytterbium Zinc Lithium Cadmium Potassium niobate Borophosphate glass specimens.

III. THEORY

A. *Oscillator Strength*

The intensity of spectral lines are expressed in terms of oscillator strengths using the relation [16].

$$f_{expt.} = 4.318 \times 10^{-9} \int \varepsilon(\nu)\, d\nu \qquad (1)$$

where, ε (*v*) is molar absorption coefficient at a given energy *v* (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 [17], using the modified relation:

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

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

B. *Judd-Ofelt Intensity Parameters*

According to Judd [18] and Ofelt [19] theory, independently derived expression for the oscillator strength of the induced forced electric dipole transitions between an initial J manifold $|4f^N (S, L) J>$ level and the terminal J' manifold $|4f^N (S',L') J'>$ is given by:

$$\frac{8\Pi^2 mc\bar{\upsilon}}{3h(2J+1)} \frac{1}{n}\left[\frac{(n^2+2)^2}{9}\right] \times S(J, J') \qquad (3)$$

Where,

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

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

In the above equation m is the mass of an electron, c is the velocity of light, *v* 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 (λ=2,4and 6) are known as Judd-Ofelt intensity.

C. *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 $|4f^N (S', L') J'>$ to a final manifold $|4f^N (S, L) J>|$ is given by:

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

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 $|4f^N (S', L') J'>$ to a final many fold $|4f^N (S, L) J>$ is given by

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

S L J

where, the sum is over all terminal manifolds.

The radiative life time is given by

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

S L J

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

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

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

D. 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 [20, 21]. 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 value of bonding parameter ($b^{1/2}$) is given by

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

IV. RESULT AND DISCUSSION

A. XRD Measurement

Figure 1 presents the XRD pattern of the sample contain – P_2O_5 which is show no sharp Bragg's peak, but only a broad diffuse hump around low angle region. This is the clear indication of amorphous nature within the resolution limit of XRD instrument.

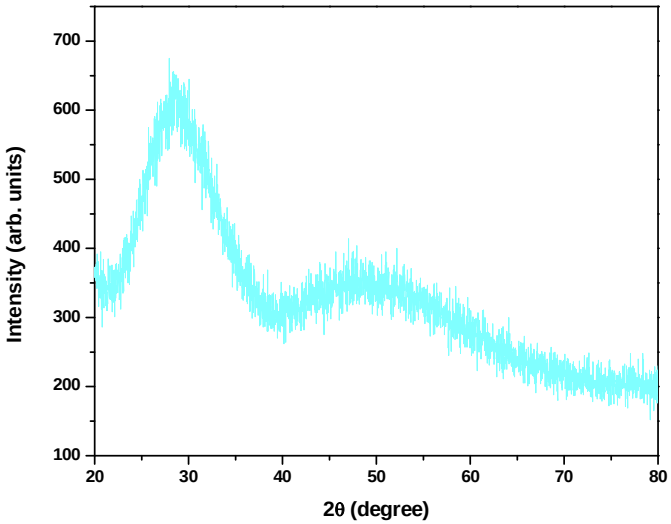

Fig. 1: X-ray diffraction pattern of YZLCPNBP HO (1.0) glass.

B. Raman Spectrum

The Raman Spectrum of ytterbium zinc lithium cadmium potassium niobate borophosphate YZLCPNBP HO (1.0) glass specimen is recorded and shown in Fig.2.The specimen peaks located at 395 and 775 cm-1.The band 395 cm^{-1} is related to the bending motion of phosphate polyhedral PO_4 units .The broad band at 775 cm^{-1} is due to symmetric stretching of P-O-P bridging oxygen bonds in $(P_2O_7)_4$ units.

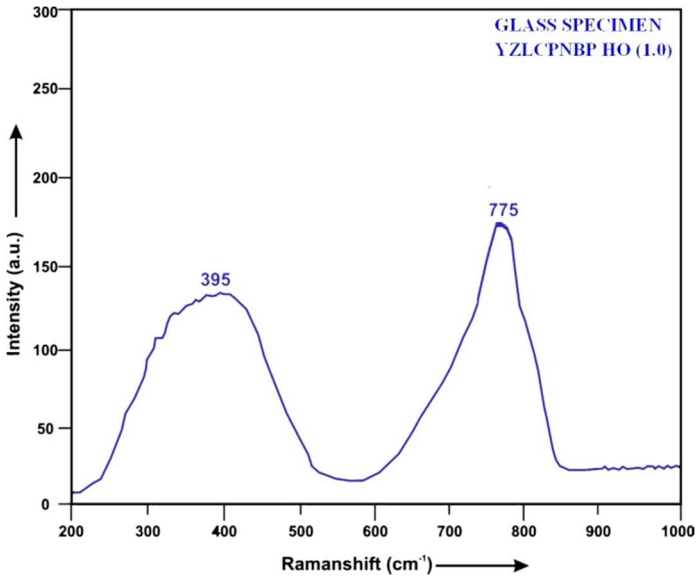

Fig.2: Raman Spectrum of YZLCPNBP HO (1.0) glass.

C. Thermal Property

Differential thermal analysis checks the heat absorbed by glass samples during heating or cooling. Fig. 3 depicts the DTA thermogram of powdered YZLCPNBP HO(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) and reduced glass transition temperature (T_{rg}) were calculated. Shankar's parameter also calculated by using eq. (15). All the determined thermal parameters are given in table 2.

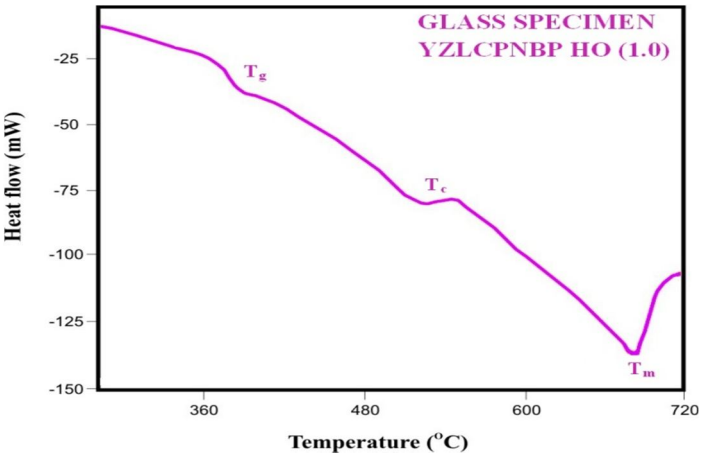

Fig.3: DTA curve of YZLCPNBP HO (1.0) glass.

Table 2. Thermal parameters determined from the DTA traces of YZLCPNBP HO glasses.

Sample Name	T_g(^{0}C)	T_c(^{0}C)	T_p(^{0}C)	T_m(^{0}C)	T_s(^{0}C)	B_P(^{0}C)	H_R(^{0}C)	K_S(^{0}C)	T_{rg}(^{0}C)
YZLCPNBP HO (1.0)	375	510	548	685	135	3.553	0.217	34.489	0.547
YZLCPNBP HO (1.5)	380	511	550	688	131	3.359	0.220	33.702	0.552
YZLCPNBP HO (2.0)	385	513	556	692	128	2.977	0.240	33.110	0.556

The thermal stability of the glass samples can be calculated by difference between onset crystallization temperature and transition temperature [22].

Thermal Stability (T_s)= T_c-T_g (11)

Balaji Parameter can be calculated using [22].

Balaji Parameter (B_P) = [(T_c-T_g)/(T_p-T_c)] (12)

Hruby's criterion is calculated using the Hurby's relation [22].

Hruby's criterion(H_R)=[(T_p-T_c)/(T_m-T_c)] (13)

Reduced glass transition temperature is given as [22].

Reduced glass transition temperature (T_{rg}) =T_g/T_m (14)

Thermal Parameter is given as [22].

(15)

K_S= [(T_m-T_c) (T_c-T_g)/T_m]

D. Absorption Spectrum

The absorption spectra of Ho^{3+}doped YZLCPNBP HO(1.0) glass specimen has been presented in Figure 4 in terms of optical density versus wavelength. Twelve absorption bands have been observed from the ground state $^5I_8$ to excited states $^5I_5$, $^5I_4$, $^5F_5$, $^5F_4$, $^5F_3$, $^3K_8$, $^5G_6$, (^5G,^3G)$_5$, $^5G_4$, $^5G_2$, $^5G_3$, and$^3F_4$ for Ho^{3+} doped YZLCPNBP HO(1.0) glass.

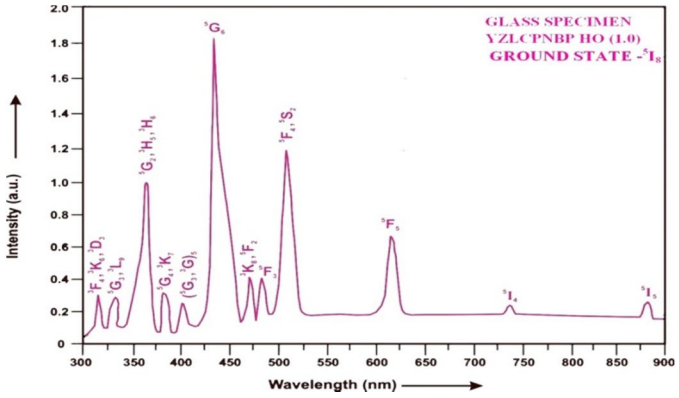

Fig. 4: Absorption spectrum of YZLCPNBP HO (1.0) glass.

The experimental and calculated oscillator strength for Ho^{3+} ions in YZLCPNBP glasses are given in **Table 3.**

Table 3: Measured and calculated oscillator strength ($P_m \times 10^{+6}$) of Ho^{3+}ions in YZLCPNBP glasses.

Energy level from $^5I_8$	Glass YZLCPNBP HO(1.0)		Glass YZLCPNBP HO(1.5)		Glass YZLCPNBP HO(2.0)	
	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$
$^5I_5$	0.48	0.25	0.45	0.25	0.41	0.25
$^5I_4$	0.07	0.02	0.06	0.02	0.04	0.02
$^5F_5$	3.82	2.89	3.78	2.87	3.72	2.84
$^5F_4$	4.78	4.48	4.75	4.45	4.70	4.40
$^5F_3$	1.62	2.49	1.57	2.47	1.52	2.45
$^3K_8$	1.45	2.06	1.41	2.03	1.36	2.00
$^5G_6$	27.25	27.23	26.36	26.37	25.45	25.49
(^5G,^3G)$_5$	3.90	1.76	3.86	1.74	3.82	1.72
$^5G_4$	0.09	0.64	0.07	0.63	0.05	0.62
$^5G_2$	5.95	5.76	5.91	5.60	5.87	5.44
$^5G_3$	1.56	1.45	1.52	1.43	1.48	1.41
$^3F_4$	1.39	4.32	1.35	4.28	1.30	4.23
r.m.s. deviation	±1.1422		±1.1420		±1.1425	

Computed values of F_2, Lande′ parameter (ξ_{4f}**),** Nephlauxetic ratio (β') and bonding parameter ($b^{1/2}$) for Ho^{3+} ions in YZLCPNBP glass specimen are given in Table 4.

Table 4: F_2, ξ_{4f}, β' and $b^{1/2}$ parameters for Holmium doped glass specimen.

Glass Specimen	F_2	ξ_{4f}	β'	$b^{1/2}$
Ho^{3+}	358.82	1258.16	0.9337	0.1821

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

Table 5: Judd-Ofelt intensity parameters for Ho^{3+} doped YZLCPNBP glass specimens.

Glass Specimen	$\Omega_2(pm^2)$	$\Omega_4(pm^2)$	$\Omega_6(pm^2)$	Ω_4/Ω_6	Ref.
YZLCPNBP HO(1.0)	6.682	1.391	2.270	0.613	P.W.
YZLCPNBP HO(1.5)	6.432	1.376	2.253	0.618	P.W.
YZLCPNBP HO (2.0)	6.181	1.359	2.227	0.610	P.W.
TB (ND)	3.340	3.860	3.540	1.090	[23].
TBZB (ND)	2.890	1.350	1.590	0.849	[24].
BSLNAD (DY)	1.290	0.350	0.420	0.833	[25].

E. Excitation Spectrum

The Excitation spectrum of YZLCPNBP HO(1.0) glass has been presented in Figure 5 in terms of Excitation Intensity versus wavelength. The excitation spectrum was recorded in the spectral region 325–525 nm fluorescence at 545nm having different excitation band centered at 349,419, 452, 473and 486 nm are attributed to the $^5G_3$, **(**^5G, $^3G)_5$, $^5G_6$, $^3K_8$ and $^5F_3$ transitions, respectively. The highest absorption level is $^5G_6$ and is at 452nm.So this is to be chosen for excitation wavelength.

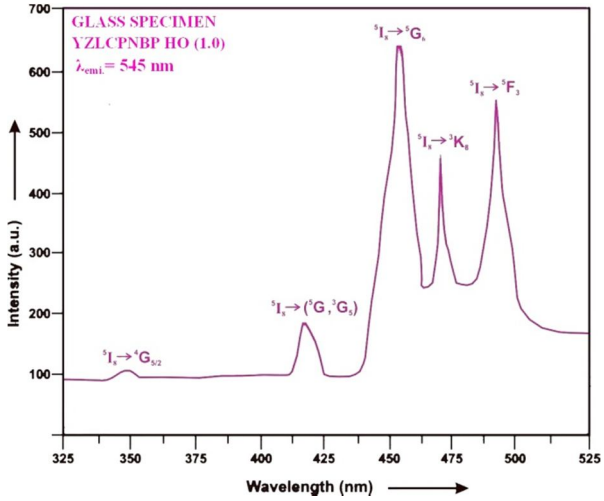

Fig. 5: Excitation spectrum of YZLCPNBP HO (1.0) glass.

F. Fluorescence Spectrum

The fluorescence spectrum of Ho^{3+}doped in ytterbium zinc lithium cadmium potassium niobate borophosphate glass is shown in Figure 6. There are eleven broad bands observed in the Fluorescence spectrum of Ho^{3+}doped ytterbium zinc lithium cadmium potassium niobate borophosphate glass. The wavelengths of these bands along with their assignments are given in Table 6. The peak with maximum emission intensity appears at 2035 nm and corresponds to the ($^5I_7 \rightarrow {}^5I_8$) transition.

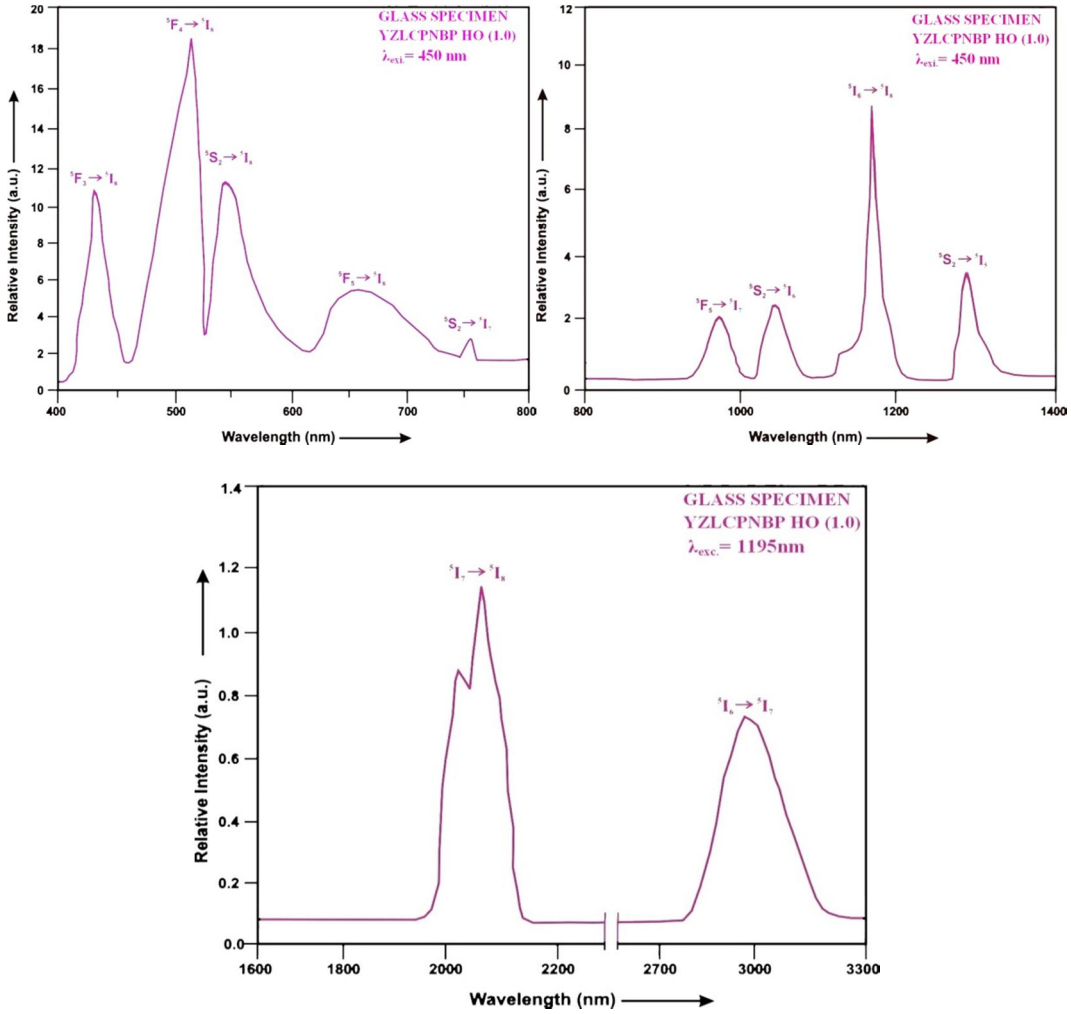

Fig. 6: Fluorescence spectrum of YZLCPNBP HO (1.0) glass.

Table6: Emission peak wave lengths (λ_p),radiative transition probability (A_{rad}),branching ratio (β),stimulated emission cross-section(σ_p) and radiative life time(τ_R) for various transitions in Ho^{3+} doped YZLCPNBP glasses.

Transition		YZLCPNBP HO (1.0)				YZLCPNBP HO(1.5)				YZLCPNBP HO(2.0)			
	λ_{max} (nm)	$A_{rad}(s^{-1})$	β	σ_p (10^{-20} cm^2)	$\tau_R(\mu s)$	$A_{rad}(s^{-1})$	β	σ_p (10^{-20} cm^2)	τ_R (μs)	$A_{rad}(s^{-1})$	β	σ_p (10^{-20} cm^2)	τ_R (10^{-20} cm^2)
$^5F_3 \rightarrow {}^5I_8$	435	4327.54	0.2483	0.549		4303.67	0.2484	0.533		4262.47	0.2485	0.512	
$^5F_4 \rightarrow {}^5I_8$	501	6872.09	0.3943	1.184		6831.11	0.3943	1.156		6764.94	0.3943	1.118	
$^5S_2 \rightarrow {}^5I_8$	555	1806.37	0.1037	0.431		1796.41	0.1037	0.419		1779.21	0.1037	0.406	
$^5F_5 \rightarrow {}^5I_8$	652	1957.56	0.1123	0.707		1944.74	0.1123	0.690		1925.62	0.1122	0.671	
$^5S_2 \rightarrow {}^5I_7$	761	1370.37	0.0786	1.064	5738.24	1362.81	0.0787	1.037	5772.48	1349.76	0.0787	1.006	5829.23
$^5F_5 \rightarrow {}^5I_7$	995	458.15	0.0263	1.148		454.04	0.0262	1.112		448.58	0.0261	1.074	
$^5I_6 \rightarrow {}^5I_8$	1032	210.24	0.0121	0.668		209.02	0.0121	0.648		206.99	0.0121	0.626	
$^5S_2 \rightarrow {}^5I_5$	1195	240.58	0.0138	1.171		238.98	0.0138	1.139		236.46	0.0138	1.103	
$^5S_2 \rightarrow {}^5I_6$	1310	64.03	0.0037	0.577		63.67	0.0037	0.555		63.06	0.0037	0.538	
$^5I_7 \rightarrow {}^5I_8$	2035	95.59	0.0055	4.383		94.93	0.0055	4.274		93.90	0.0055	4.130	
$^5I_6 \rightarrow {}^5I_7$	2925	24.41	0.0014	3.924		24.20	0.0014	3.815		23.92	0.0014	3.714	

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V. CONCLUSION

In the present study, the glass samples of composition (25-x)P_2O_5: 10ZnO: 10Li_2O: 10CdO: 10K_2O: 10Nb_2O_5:10Y_2O_3 :15B_2O_3:xHo_2O_3 (where x =1, 1.5and 2mol %) have been prepared by melt-quenching method. The large value of Balaji and Shankar parameters indicate that the prepared glass samples have good thermal stability. The value of stimulated emission cross-section (σ_p) is found to be maximum for the transition ($^5I_7 \rightarrow {}^5I_8$) for glass YZLCPNBP HO (1.0), suggesting that glass YZLCPNBP HO(1.0) is better compared to the other two glass systems YZLCPNBP HO(1.5) and YZLCPNBP HO (2.0). The large stimulated emission cross section in borate glasses suggests the possibility of utilizing these systems as laser materials. The results show that the Ho^{3+} doped bismuth borate glasses could be potential candidates for Photonic and Laser Materials Applications.

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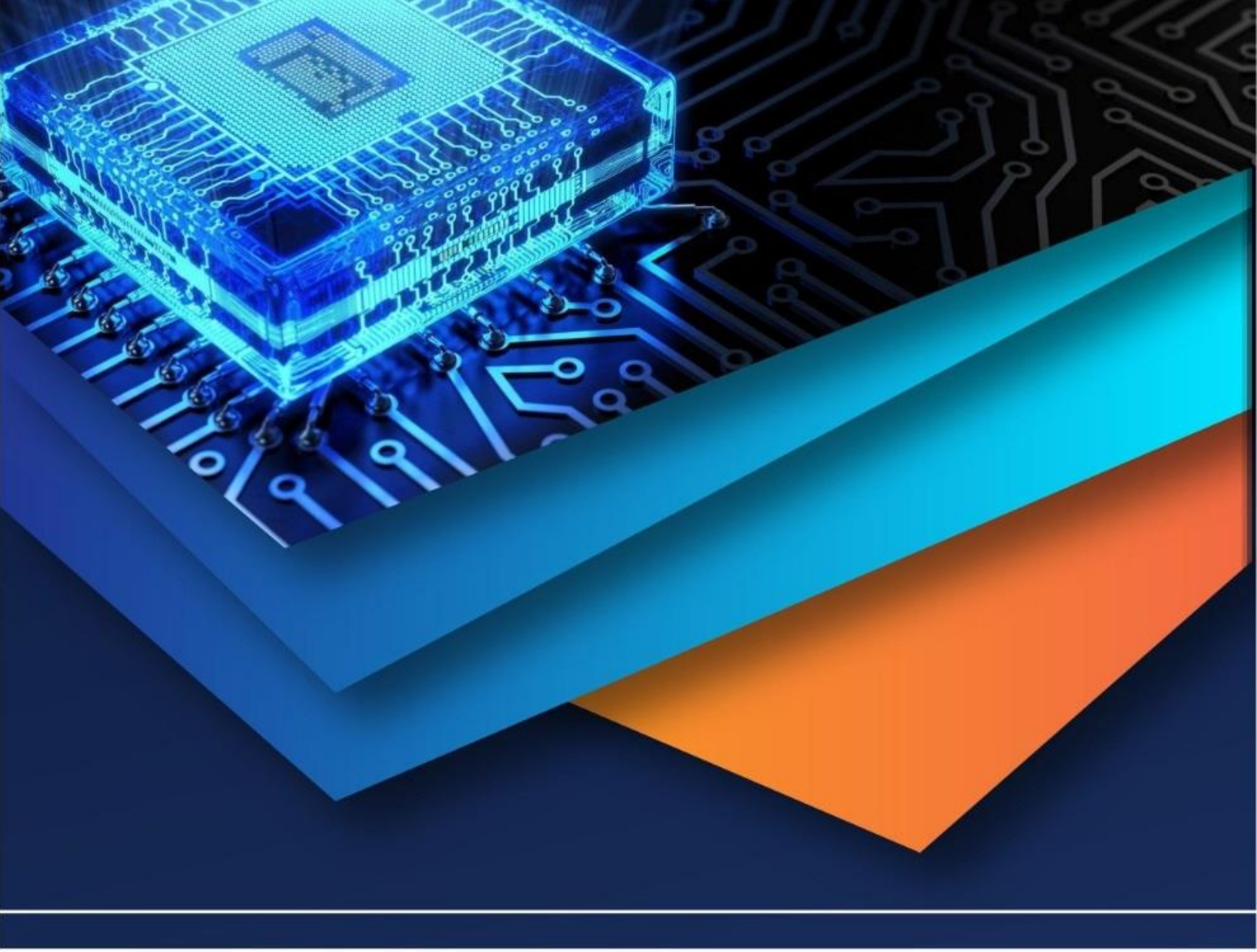

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