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Spectral, Thermal and Raman Analysis of Dy³⁺ ions doped Phosphate Glasses with Large Thermal Stability Relation

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ABSTRACT: Glass of the system: (20-x)P₂O₅: 10ZnO: 10Li₂O 10K₂O: 10Nb₂O₅: 10WO₃: 10Sb₂O₃:20Y₂O₃:xDy₂O₃. (where x=1, 1.5,2 mol %) have been prepared by melt-quenching technique. The amorphous nature of the prepared glass samples was confirmed by X-ray diffraction. The thermal parameters were calculated using DTA Thermogram. Optical absorption and fluorescence spectra were recorded at room temperature for all glass samples. Judd-Ofelt intensity parameters Ω_λ ($\lambda=2, 4$ and 6) are evaluated from the intensities of various absorption bands of optical absorption spectra. The radiative properties like spontaneous emission probability (A), branching ratio (β), radiative life time(τ_R),stimulated emission cross-section(σ_p)and thermal properties have been evaluated.

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

I. INTRODUCTION

The glasses doped with rare earth ions have several potential applications such as fiber lasers, optical amplifier, solid state lasers, [1-5]. Among different host matrices, phosphate glasses exhibit very important properties such as high refractive index, high thermal expansion coefficient, low melting point, low non-linear refractive index, good stability, excellent color purity, high thermal stability, high transparency and low phonon energy [6-10]. Phosphate glasses are very well known for their suitable excellent applications in telecommunications, visible and near infrared optical lasers. Phosphate glasses are very important because of their application in fiber optic large bandwidth, optoelectronic, optic device fields, lasers and solar cells [11-13]. They have low melting temperature, low glass transition temperature and low softening temperature. Due to their good chemical durability, Dysprosium doped phosphate glasses are attractive materials due to their good physical, optical and chemical properties. Glasses contain WO₃ and Nb₂O₅ have received increased attention due to their application in the field of glass ceramics, reflecting windows, thermal and mechanical sensors [14-16].The addition of network modifier (NWF) Li₂O and is to improve both electrical and mechanical properties of such glasses. Zinc oxide is added in the glass matrix to increase glass thermal stability and to ensure low rates of crystallization in the glass system .Recently, rare earth doped phosphate glass has attracted much interest because of high rare earth ion solubility, good structure, optical and thermal properties. [17-20].

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 absorption, Excitation, emission and thermal spectra of Dy³⁺ doped yttrium zinc lithium potassiumniobate tungsten antimony phosphate glasses. 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 using DTA Thermogram. 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 , $\lambda=2,4$ and 6).

II. EXPERIMENTAL TECHNIQUES

Preparation of glasses

The following Dy³⁺doped phosphate glass samples (20-x)P₂O₅: 10ZnO: 10Li₂O 10K₂O: 10Nb₂O₅: 10WO₃: 10Sb₂O₃:20Y₂O₃: xDy₂O₃. (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 , K_2O , Nb_2O_5 , WO_3 , Sb_2O_3 , Y_2O_3 and Dy_2O_3 . They were thoroughly mixed by using an agate pestle mortar. then melted at $1050^\circ C$ 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 $350^\circ C$ 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 %)
YZLPNTAP (UD)	20 P_2O_5 : 10 ZnO : 10 Li_2O 10 K_2O : 10 Nb_2O_5 : 10 WO_3 :10 Sb_2O_3 :20 Y_2O_3
YZLPNTAP DY(1.0)	19 P_2O_5 : 10 ZnO : 10 Li_2O 10 K_2O : 10 Nb_2O_5 : 10 WO_3 :10 Sb_2O_3 :20 Y_2O_3 : 1 Dy_2O_3 .
YZLPNTAP DY(1.5)	18.5 P_2O_5 : 10 ZnO : 10 Li_2O 10 K_2O : 10 Nb_2O_5 : 10 WO_3 :10 Sb_2O_3 :20 Y_2O_3 :1.5 Dy_2O_3 .
YZLPNTAP DY(2.0)	18 P_2O_5 : 10 ZnO : 10 Li_2O 10 K_2O : 10 Nb_2O_5 : 10 WO_3 :10 Sb_2O_3 :20 Y_2O_3 :2 Dy_2O_3 .

YZLPNTAP (UD) -Represents undoped yttrium zinc lithium potassiumniobate tungsten antimony phosphate glass specimens.

YZLPNTAP (DY) -Represents Dy^{3+} doped yttrium zinc lithium potassiumniobate tungsten antimony phosphate glass specimens.

III.THEORY

3.1 Oscillator Strength

The intensity of spectral lines are expressed in terms of oscillator strengths using the relation [21].

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

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 [22], using the modified relation:

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

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

3.2. Judd-Ofelt Intensity Parameters

According to Judd [23] and Ofelt [24] 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:

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

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

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

where, the sum is over all terminal manifolds.

The radiative life time is given by

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

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

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

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

IV. RESULT AND DISCUSSION

4.1 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.

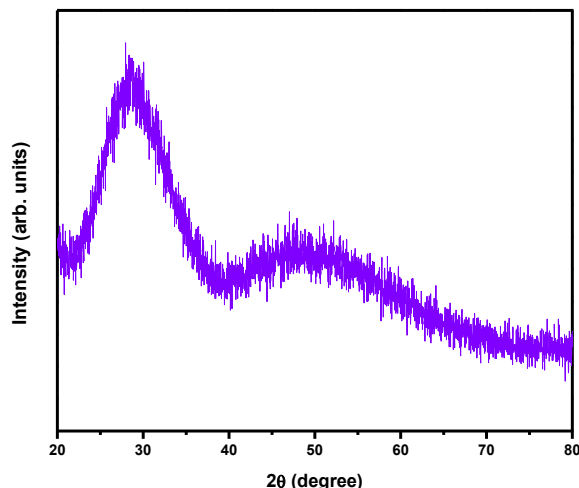


Fig. 1: X-ray diffraction pattern of YZLPNTAP DY(1.0) glass.

4.2 Thermal Property

Differential thermal analysis checks the heat absorbed by glass samples during heating or cooling. Fig. 2 depicts the DTA thermogram of powdered YZLPNTAP DY(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's Parameter (K_s) were calculated. Balaji-Shankar relation also calculated by using eq. (14). All the determined thermal parameters are given in table 2.

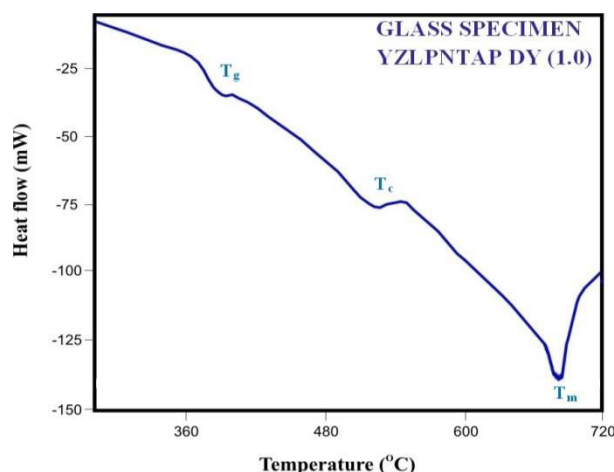


Fig.2: DTA curve of YZLPNTAP DY(1.0) glass.

Table.2. Thermal parameters determined from the DTA curve of YZLPNTAP DY glasses.

Sample Name	$T_g(^{\circ}\text{C})$	$T_c(^{\circ}\text{C})$	$T_p(^{\circ}\text{C})$	$T_m(^{\circ}\text{C})$	$T_s(^{\circ}\text{C})$	$B_p(^{\circ}\text{C})$	$H_R(^{\circ}\text{C})$	$K_s(^{\circ}\text{C})$	$T_{rg}(^{\circ}\text{C})$	$B_p \times K_s$
YZLPNTAP DY (1.0)	375	511	550	685	136	3.487	0.224	34.546	0.547	120.462
YZLPNTAP DY (1.5)	380	512	552	688	132	3.300	0.227	33.767	0.552	111.431
YZLPNTAP DY (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 Hruby'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 is given by [25].

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

4.3 Raman Spectrum

The Raman spectrum of the YZLPNTAP DY(1.0) glass specimen is recorded and shown in Fig.3. 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 $(\text{P}_2\text{O}_7)_4$ units.

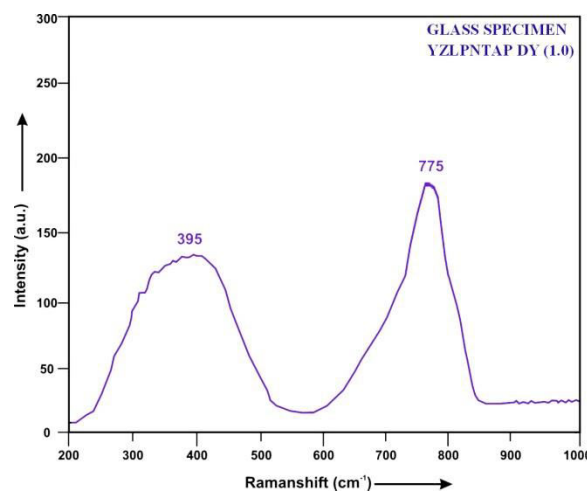


Fig.3: Raman Spectrum of YZLPNTAP DY(1.0) glass .

4.4 Absorption Spectrum

The absorption spectra of Dy^{3+} doped YZLPNTAP DY(1.0) glass specimen has been presented in Figure 4 in terms of Intensity versus wavelength. Thirteen absorption bands have been observed from the ground state $^6\text{H}_{15/2}$ to excited states $^6\text{H}_{13/2}$, $^6\text{H}_{11/2}$, $^6\text{H}_{9/2} + ^6\text{F}_{11/2}$, $^6\text{H}_{7/2} + ^6\text{F}_{9/2}$, $^6\text{F}_{7/2} + ^6\text{H}_{5/2}$, $^6\text{F}_{5/2}$, $^6\text{F}_{3/2}$, $^6\text{F}_{9/2}$, $^4\text{I}_{15/2}$, $^4\text{G}_{11/2}$, $^6\text{F}_{7/2} + ^4\text{I}_{13/2}$, $^6\text{M}_{19/2} + 4(\text{P}, \text{D})_{3/2}$ and $^4\text{G}_{9/2} + ^6\text{P}_{3/2}$ for Dy^{3+} doped YZLPNTAP DY(1.0) glass.

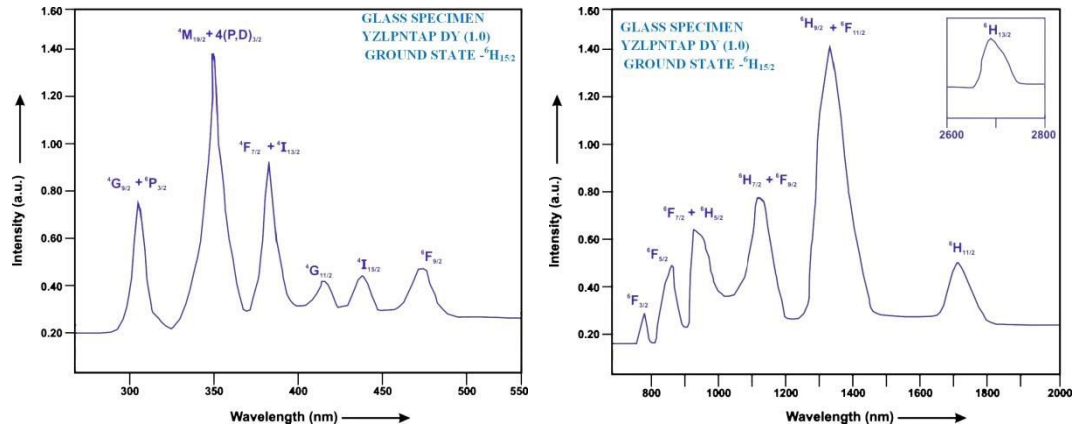


Fig. 4: Absorption spectrum of YZLPNTAP DY(1.0) glass.

The experimental and calculated oscillator strength for Dy^{3+} ions in YZLPNTAP glasses are given in Table 3.

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

Energy level from $6H_{15/2}$	Glass YZLPNTAP DY(1.0)		Glass YZLPNTAP DY(1.5)		Glass YZLPNTAP DY(2.0)	
	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$
$6H_{13/2}$	2.15	2.43	2.12	2.42	2.08	2.40
$6H_{11/2}$	1.50	2.06	1.48	2.04	1.45	2.01
$6H_{9/2} + 6F_{11/2}$	10.34	10.25	10.31	10.21	10.28	10.18
$6H_{7/2} + 6F_{9/2}$	5.62	5.31	5.59	5.28	5.56	5.25
$6F_{7/2} + 6H_{5/2}$	4.75	3.84	4.72	3.80	4.69	3.75
$6F_{5/2}$	1.38	1.74	1.36	1.72	1.33	1.69
$6F_{3/2}$	0.36	0.33	0.34	0.32	0.32	0.32
$6F_{9/2}$	0.40	0.29	0.38	0.29	0.35	0.28
$4I_{15/2}$	0.35	0.71	0.32	0.71	0.30	0.69
$4G_{11/2}$	0.29	0.17	0.27	0.17	0.25	0.17
$6F_{7/2} + 4I_{13/2}$	3.56	3.74	3.53	3.71	3.50	3.67
$6M_{19/2} + 4(P,D)3/2$	7.98	10.02	7.95	10.00	7.93	9.99
$4G_{9/2} + 6P_{3/2}$	1.70	2.12	1.68	2.10	1.65	2.07
r.m.s. deviation	0.6785		0.6817		0.6875	

The values of Judd-Ofelt intensity parameters are given in Table 4.

Table 4: Judd-Ofelt intensity parameters for Dy^{3+} doped YZLPNTAP glass specimens

Glass Specimen	$\Omega_2(pm^2)$	$\Omega_4(pm^2)$	$\Omega_6(pm^2)$	Ω_4/Ω_6	Ref.
YZLPNTAP DY(1.0)	2.961	1.717	1.500	1.145	P.W.
YZLPNTAP DY(1.5)	2.947	1.719	1.480	1.161	P.W.
YZLPNTAP DY(2.0)	2.928	1.728	1.452	1.190	P.W.
ZLCMBP (DY)	2.790	1.715	1.354	1.267	[26].
ZLSLAS (SM)	4.743	4.482	4.104	1.092	[27].
BACMBSY(ER)	2.390	1.323	1.310	1.010	[28].
PKAPN(HO)	8.680	3.040	2.370	1.283	[29].
ZLCMBP(EU)	4.614	3.493	3.263	1.070	[30].

4.5 Excitation Spectrum

The Excitation spectra of Dy^{3+} -doped YZLPNTAP DY(1.0) glass specimen has been presented in Figure 5 in terms of Excitation Intensity versus wavelength. The excitation spectrum was recorded in the spectral region 315–480 nm fluorescence at 575nm having different excitation band centered at 321,354, 365, 385, 425, 455 and 473 nm are attributed to the $^6\text{P}_{3/2}$, $^6\text{P}_{7/2}$, $^4\text{P}_{3/2}$, $^4\text{I}_{13/2}$, $^4\text{G}_{11/2}$, $^4\text{I}_{15/2}$ and $^4\text{F}_{9/2}$ transitions, respectively. The highest absorption level is $^4\text{I}_{13/2}$ and is at 385nm. So this is to be chosen for excitation wavelength.

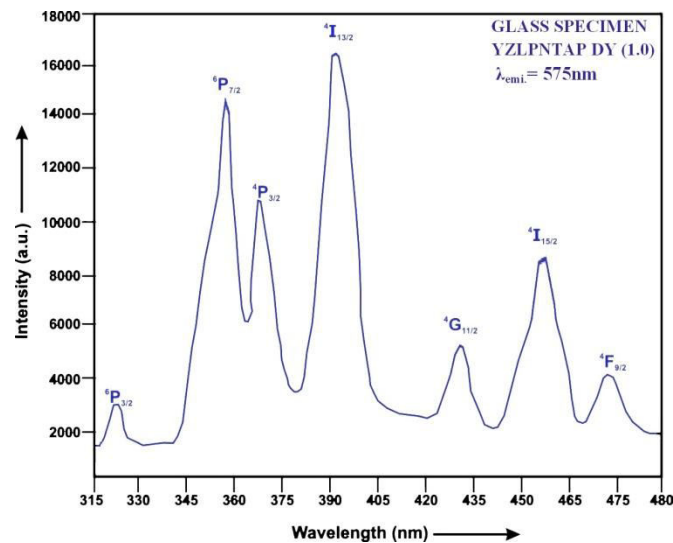


Fig. 5: Excitation spectrum of YZLPNTAP DY(1.0) glass.

4.6 Fluorescence Spectrum

The fluorescence spectrum of Dy^{3+} -doped in yttrium zinc lithium potassiumniobate tungsten antimony phosphate glass is shown in Figure 6. There are four broad bands observed in the Fluorescence spectrum of Dy^{3+} -doped yttrium zinc lithium potassiumniobate tungsten antimony phosphate glass. The wavelengths of these bands along with their assignments are given in Table 5. The peak with maximum emission intensity appears at 485nm, 575 nm, 665 nm and 752 nm and corresponds to the ($^4\text{F}_{9/2} \rightarrow ^6\text{H}_{15/2}$), ($^4\text{F}_{9/2} \rightarrow ^6\text{H}_{13/2}$), ($^4\text{F}_{9/2} \rightarrow ^6\text{H}_{11/2}$) and ($^4\text{F}_{9/2} \rightarrow ^6\text{H}_{9/2}$) transition.

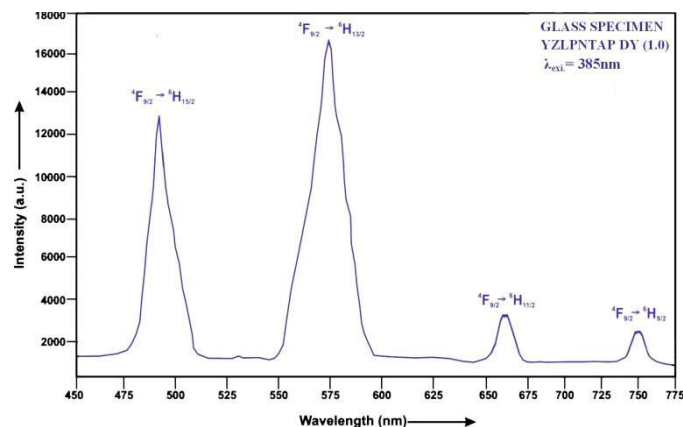


Fig. 6: Fluorescence spectrum of YZLPNTAP DY(1.0) glass.

Table5: 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 Dy3+ doped YZLPNTAP glasses.

Transition	λ_{max} (nm)	YZLPNTAP DY (1.0)				YZLPNTAP DY (1.5)				YZLPNTAP DY (2.0)			
		$A_{rad}(s^{-1})$	β	σ_p ($10^{-20}cm^2$)	$\tau_R(\mu s)$	$A_{rad}(s^{-1})$	β	σ_p ($10^{-20}cm^2$)	$\tau_R(\mu s)$	$A_{rad}(s^{-1})$	β	σ_p ($10^{-20}cm^2$)	τ_R ($10^{-20}cm^2$)
$^4F_{9/2} \rightarrow ^6H_{13/2}$	485	79.81	0.1993	0.143	2496.63	79.08	0.1985	0.139	2510.48	78.04	0.1975	0.134	2530.78
$^4F_{9/2} \rightarrow ^6H_{13/2}$	575	269.98	0.6740	1.002		268.67	0.6745	0.970		266.76	0.6751	0.943	
$^4F_{9/2} \rightarrow ^6H_{11/2}$	665	28.41	0.0709	0.129		28.31	0.0711	0.127		28.17	0.0713	0.124	
$^4F_{9/2} \rightarrow ^6H_{9/2}$	752	22.34	0.0668	0.139		22.26	0.0559	0.136		22.17	0.0561	0.134	

V. CONCLUSION

In the present study, the glass samples of composition (20-x)P2O5: 10ZnO: 10Li2O 10K2O: 10Nb2O5: 10WO3: 10Sb2O3:20Y2O3:xDy2O3 (where x =1, 1.5and 2mol %) have been prepared by melt-quenching method. The value of Balaji-Shankar relation is found to be greater than 100 OC, which indicates that the prepared glass samples have good thermal stability and useful for thermionic emission applications. The value of stimulated emission cross-section (σ_p) is found to be maximum for the transition ($4F_{9/2} \rightarrow 6H_{13/2}$) for all glass specimens. This shows that ($4F_{9/2} \rightarrow 6H_{13/2}$) transition is most probable transition.

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