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Spectral studies of praseodymium doped heavy metal borate glass systems

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ABSTRACT : *Praseodymium doped HMO glasses are fabricated with the following compositions using conventional melt quenching technique. The compositions of the glass systems are 12 ZnO + 33 B_2O_3 + (50-x) PbO + (x+10) CaO + 4 Al_2O_3 + 1 Pr_6O_{11} where (x = 0,10,20,30 and 40 mol %.). Certain physical properties of these systems have been evaluated and reported. Spectral data for all these systems were recorded for X-ray diffraction, Optical absorption and Fluorescence properties. The Judd-Ofelt intensity parameters Ω_λ (λ = 2,4,6) were evaluated from the spectral data and in turn employed to evaluate the lasing parameters of Pr^{3+} HMO glass systems such as radiative transition probabilities (A), radiative life-times (τ_R), branching ratios (β_R) absorption cross-sections (σ_a) and Stimulated emission cross-sections (σ_e). The experimental and calculated branching ratios (β_R) for the lasing transitions $^3P_0 \rightarrow {}^3H_4$, $^3P_0 \rightarrow {}^3H_6$, and $^3P_0 \rightarrow {}^3F_2$ are found to be in good agreement in the present work.*

KEY WORDS*: FT-IR, HMO glasses, Optical absorption, Praseodymium, XRD.*

I. INTRODUCTION

The present study of this article is to study the spectral properties of Pr^{3+} doped glass systems due to their potential wide range of applications for memory devices, solid state lasers, solid state- batteries, optical wave guides, up converters, luminescent materials, optical telecommunication, non-linear optical materials and optical fibers [1-8]. Laser action in visible region has been reported in literature for the transition $^1D_2 \rightarrow {}^3H_4$ [9-11] of the Pr^{3+} ion. The potential Laser transition of $^1G_4 \rightarrow {}^3H_5$ at ~ 1.3μm of Pr^{3+} find its applications for the development of fiber amplifier for communication purpose [12] in the telecom window. The Judd–Ofelt (JO) [13, 14] theory has been proved to be a powerful tool to analyses optical spectra of RE ions in various media. The spectral studies give valuable information about the structure and bonding of the system along with their lasing properties. Therefore such systems are required as tailor made devices to develop optical devices for laser action.

The present work reports on the preparation and characterization of RE doped HMO glass systems for lasing materials. The host matrix composes of PbO, a glass modifier/ glass farmer a heavy metal oxide along with ZnO, B_2O_3, Al_2O_3 and CaO as their constituents. The conventional Judd-Ofelt theory [15, 16] is reported in literature to analyze the absorption spectra of Pr^{3+} ions in different host glass matrices. The physical properties for the RE HMO glass systems of the present studies are evaluated through the procedure outlined in the literature [17]. The lasing parameters such as radiative transition probabilities (A), branching ratio (β), , radiative life time (τ_R) absorption cross section (σ_a) and stimulated emission cross section (σ_e) are evaluated using J.O intensity parameters (Ω_λ , λ = 2,4,6). The HMO such as PbO in the glass composition increases the thermal stability and decreases the maximum phonon energy of the host in which it is present. Thus it provides an effective way to tailor the lasing properties of the system and therefore it prompted us to carry out of the present study.

II. EXPERIMENTAL

Heavy metal oxide glasses (HMO) with the molar compositions of (50-x) PbO + (0+x) CaO + 12ZnO + 33B_2O_3 + 4Al_2O_3 + 1 Pr_6O_{11} with x=10, 20, 30 and 40 mol% were prepared using Melt quenching technique and designated as BZCAP: 1-5 respectively. 10g batch of each chemical composition were weight accurately, mixed and grinded in an agate mortar and then transferred into a silica porcelain crucible. It is kept at 400^{o}C for 30 min to remove impurities in the base material and melted in an electric furnace in the temperature range of 950-1000 ^{o}C for 1 hour. The melt is then poured on to a preheated brass plate and air quenched to get a good optical quality glasses. The samples are annealed at about 400^{o}C for 5 hours to remove thermal strains and then polished before measuring their optical properties. The amorphous nature of the samples was confirmed by XRD spectra obtained by using Shimadzu-XD3ADiffractometer. XRD spectra were recorded for one sample

with different chemical compositions to confirm their amorphous nature. PerkinElmer Lambda 950 UV-Vis-NIR spectrophotometer is used to record the absorption spectra at room temperature in the wave length range of 190-2500 nm at room temperature. The FT-IR spectra were recorded using KBr pellet method on Thermo Nicolet-5700 FT-IR Spectrophotometer in the wave number range 4000– 400 cm^{-1}. Refractive indices (n) were evaluated using conventional methods. Densities were measured by the Archimedes method using Xylene as an immersion liquid.

III. RESULTS & DISCUSSION

3.1 Physical Properties

Various physical properties such as density (ρ), average molecular weight (g), ion concentration molar refractivity (Rμ), Reflection loss (R), Electric susceptibility (χ_e), Numerical aperture (NA), polaron radius (r_P), inter-ionic distance (ri), and field strength (F) are require for computation of radial properties of the glasses are evaluated following the expressions available in the literature [18-20] and are given in the Table 1. All these glasses exhibit good optical efficiencies.

Table-1. Various physical properties of Pr^{3+}: BZCAP 1-5 glasses

S.No.	Parameter	BZCAP 1	BZCAP 2	BZCAP 3	BZCAP 4	BZCAP 5
1	Refractive index, n	1.616	1.676	1.736	1.796	1.856
2	Density d (gm/cm^3)	3.267	3.588	3.853	3.915	4.004
3	Average molecular weight (g)	196.63	202.88	208.36	213.26	217.58
4	Molecular volume (V_M) (cm^3)	60.24	56.59	54.12	54.52	54.38
5	Optical path length(cm)	0.296	0.294	0.302	0.294	0.294
6	Pr^{3+} conc.(10^{20} ions /cc)	2.707	2.774	2.800	2.684	2.600
7	Optical dielectric constant, ε	2.614	2.811	3.016	3.228	3.447
8	Reflection loss R (%)	5.547	6.383	7.238	8.107	8.985
9	Molar refractivity Rμ (cm^3)	24.444	24.386	24.632	26.069	27.194
10	Inter-ionic distance ri (Å)	15.503	15.752	15.852	15.418	15.093
11	Polaron radius r_P (Å)	7.179	7.121	7.099	7.199	7.276
12	Field strength F (10^{+15} cm^{-2})	2.894	2.871	2.862	2.902	2.933
13	Electric susceptibility (χ_e)	0.130	0.146	0.162	0.179	0.197
14	Numerical aperture (NA)	0.25	0.26	0.27	0.27	0.28

3.2. Analysis of Absorption Spectra

The absorption spectra of all the five melt quenched Pr^{3+}: BZCAP1-5 glasses are shown in Fig (1a) & (1b).

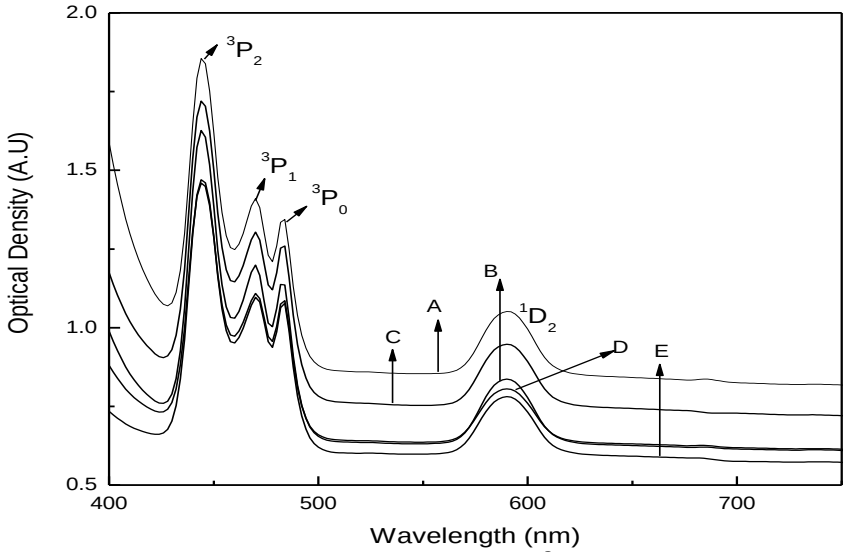

Fig-1 (a) Optical absorption spectra of Pr^{3+}: BZCAP 1-5 glasses

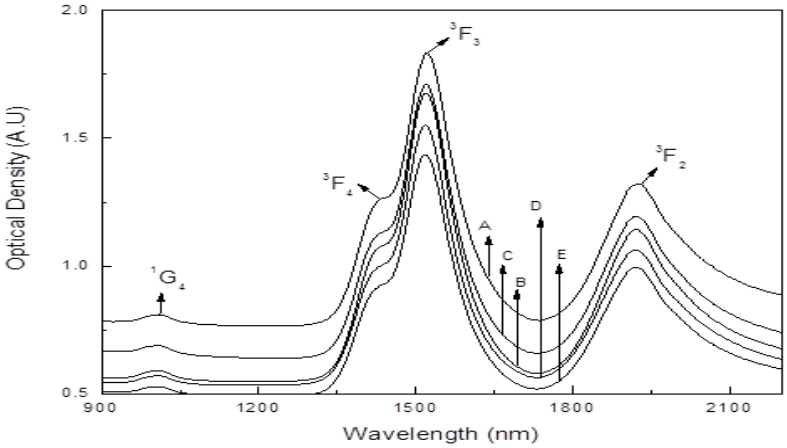

Fig-1 (b) Optical absorption spectra of Pr^{3+}: BZCAP 1-5 glasses

Eight absorption peaks were observed approximately at 5196, 6562, 7006, 9829, 16963, 20693, 21268 and 22499 cm^{-1} and their assigned as them as $^3F_2$, $^3F_3$ $^3F_4$, $^1G_4$, $^1D_2$, $^3P_0$, $^3P_1$ and $^3P_2$ levels respectively. The theoretical energies are evaluated [21, 22] by applying the Taylor series expansion as and reported in Table 2.

$$E_J = E_{0J} + (\sum dE_0/dP_K)\ \Delta P_K \longrightarrow \quad (1)$$

Where E_{0J} is the zero order energy of the chosen band and $_\Delta P_K$ are the changes to be effected in the free ion Racah (E^1, E^2 and E^3), Spin-orbit coupling ($\xi 4f$), and Configuration interaction (α, β, γ) parameters were evaluated using the partial derivatives reported in literature [23-25]. The experimental energy values (E_{expt}) are satisfactorily correlated with the theoretical energies (E_{cal}) and are given in Table-2. The rms deviation have been evaluated formed to

$$\sigma_{rms} = [\ \sum \Delta E^2/ N\ ]^{1/2} \longrightarrow \quad (2)$$

Table-2. Experimental and calculated energies of Pr^{3+}: BZCAP1-5 glasses

Transition from $^3H_4$	**BZCAP 1**		**BZCAP 2**		**BZCAP 3**		**BZCAP 4**		**BZCAP 5**	
	$\mathbf{E_{exp}}$	$\mathbf{E_{cal}}$	$\mathbf{E_{exp}}$	$\mathbf{E_{cal}}$	$\mathbf{E_{exp}}$	$\mathbf{E_{cal}}$	$\mathbf{E_{exp}}$	$\mathbf{E_{cal}}$	$\mathbf{E_{exp}}$	$\mathbf{E_{cal}}$
$^3\mathbf{P_2}$	22499	22491	22499	22501	22479	22474	22499	22499	22499	22485
$^3\mathbf{P_1}$	21268	21273	21289	21277	21249	21243	21268	21256	21229	21239
$^3\mathbf{P_0}$	20693	20695	20694	20705	20733	20744	20693	20706	20674	20677
$^1\mathbf{D_2}$	16963	16964	16982	16982	16924	16925	16963	16964	16963	16965
$^1\mathbf{G_4}$	9829	9828	9925	9913	9970	9958	9925	9910	9829	9827
$^3\mathbf{F_4}$	7006	7009	7006	7039	6998	7030	6990	7029	7006	7011
$^3\mathbf{F_3}$	6562	6571	6579	6575	6570	6774	6588	6587	6588	6603
$^3\mathbf{F_2}$	5196	5185	5204	5186	5205	5180	5205	5181	5213	5194
RMS Deviation	**± 20**		**±117**		**± 125**		**± 166**		**± 56**	

Where Δ E is the difference between the values of measured and calculated energies and N is the number of energy levels. The Racah (E^1, E^2 and E^3), Spin-orbit coupling ($\xi 4f$), and Configuration interaction (α, β, γ) parameters along with other spectroscopic parameters are given in Table 3. From the table it is observed that that the ratios of Racah parameters E^1/E^3 and E^2/E^3 do not vary much from the hydrogenic ratios.

The bonding parameter for all the glasses are calculated using the following expression [26, 27] and are given in the Table 3.

Bonding parameter (δ) = $(1 - \bar{\beta})/\bar{\beta}$ ⟶ (3)

Where $\bar{\beta} = \nu_a/\nu_b$ and ν_a and ν_b are the energies of corresponding transitions in the glass and aquo ions respectively.

The negative values obtained for all the glasses under study indicate ionic nature.

Table-3. Spectroscopic parameters of Pr^{3+}: BZCAP 1-5 glasses

Parameter	BZCAP 1	BZCAP 2	BZCAP 3	BZCAP 4	BZCAP 5
$E^1(cm^{-1})$	4964.99	5104.58	6304.42	5580.35	5401.19
$E^2(cm^{-1})$	22.19	22.41	26.44	24.21	23.91
$E^3(cm^{-1})$	468.66	476.04	557.08	509.19	498.61
E^1/E^3	10.56	10.72	11.32	10.96	10.83
E^2/E^3	0.04	0.04	0.04	0.04	0.04
ξ_{4f} (cm^{-1})	738.47	740.48	740.87	747.12	748.13
α (cm^{-1})	16.97	28.04	150.86	79.30	64.26
β (cm^{-1})	-1185.92	-1454.41	-4412.00	-2681.51	-2350.58
F_2 (cm^{-1})	316.50	322.51	386.02	348.64	340.61
$F_4(cm^{-1})$	51.37	53.19	66.18	58.06	55.67
F_6 (cm^{-1})	343.91	349.46	410.86	374.48	366.45
$F4/F2$	0.16	0.16	0.17	0.16	0.16
$F6/F2$	1.08	1.08	1.06	1.07	1.07
F^2 (cm^{-1})	71213.01	72565.7	86855.5	78444.25	76637.85
F^4 (cm^{-1})	55938.58	57921.28	72068.38	63222.7	60626.01
F^6 (cm^{-1})	2531711	2572622	3024635	2756794	2697680
F^2 / F^4	0.028	0.028	0.028	0.028	0.028
F^2 / F^6	0.022	0.023	0.024	0.023	0.022
ΣF^K	2658862	2703109	3183559	2898461	2834944
Δ	0.0017	0.0002	0.0007	0.0006	0.0018
$\bar{\beta}$	0.992006	0.992479	0.992214	0.993008	0.992885
$b^{1/2}$	0.001998	0.00188	0.001946	0.001748	0.001779

3.3 Spectral intensities

The experimental oscillator strengths f_{expt} of the measured absorption energy levels have been computed by performing a Gaussian curve analysis from the following equation.

$$f_{expt} = 4.318 \times 10^{-9} \int \varepsilon(\upsilon)\, d\upsilon \quad \longrightarrow \quad (4)$$

Where $\varepsilon(\upsilon)$ is the molar extinction coefficient. The intensities of the experimental and calculated energy levels are presented in the Table 4. It is observed from the table that the rms deviation is very low which suggests good least square fitting method employed in the present study. The Judd-Ofelt parameters (Ω_λ, λ = 2, 4, 6) parameters are evaluated following Krupke [28] from the observed spectral intensities of absorption levels and are given in Table 5.

Table-4. Experimental and calculated oscillator strengths (f x 10^{-6}) with rms deviation of BZCAP 1-5 glass systems

Transition from $^3H_4$	BZCAP 1		BZCAP 2		BZCAP 3		BZCAP 4		BZCAP 5	
	f_{exp}	f_{cal}	f_{exp}	f_{cal}	f_{exp}	f_{cal}	f_{exp}	f_{cal}	f_{exp}	f_{cal}
$^3F_2$	2.06	0.75	2.05	0.77	2.12	0.88	2.12	0.85	1.94	0.72
$^3F_3$	3.67	3.65	3.76	3.77	3.84	3.88	3.87	3.76	3.67	3.68
$^3F_4$	1.19	1.86	1.39	2.00	1.61	2.17	1.24	2.01	1.34	1.98
$^1G_4$.	1.68	0.17	1.41	0.18	1.56	0.19	1.32	0.18	1.07	0.18
$^1D_2$	1.46	0.61	1.25	0.63	1.25	0.66	1.25	0.63	1.43	0.62
^3P_O	1.43	2.17	1.43	1.99	1.34	1.73	1.34	1.92	1.01	1.88
$^3P_1$	2.87	3.17	2.63	2.96	2.30	2.65	2.15	2.87	2.69	2.80
$^3P_2$	8.17	1.95	8.31	2.05	8.39	2.16	8.17	2.05	8.62	2.02
rms deviation	±2.06		± 1.29		± 1.48		± 1.45		± 1.23	

The variation trend of J.O parameters observed in the glasses BZCAP 1-5 are as follows:

BZCAP 1, BZCAP 2, BZCAP 4, BZCAP 5 : $\Omega_2 < \Omega_6 < \Omega_4$

BZCAP 3 : $\Omega_2 < \Omega_4 < \Omega_6$

The higher values obtained for the environment sensitive and covalency related Ω_2 parameter [29, 30] in all the glasses studied suggests more or less similar asymmetry provided around the rare-earth ion site.

3.4 Fluorescence Spectra

The most intense band in the absorption spectra around 492 nm $^3P_0 \rightarrow {}^3H_4$ has been used for the excitation of Pr^{3+} ion to record photo luminescence spectra. As a consequence of which, three fluorescence bands around 492, 532, 611 and 647 nm have been observed in the wavelength region 475–675 nm, which on energy considerations, have been assigned to the transitions $^3P_0 \rightarrow {}^3H_4$,$^3P_1 \rightarrow {}^3H_5$, $^3P_0 \rightarrow {}^3H_6$ and $^3P_0 \rightarrow {}^3F_2$, respectively. Hence band appearing at 599 nm has been assigned to $^3P_0 \rightarrow {}^3H_6$ only.

The fluorescence peak wavelength (λp), the effective line width of the fluorescence peak ($\Delta\lambda_{eff}$) along with the peak intensities and stimulated emitted cross section ($\sigma_{e)}$ in arbitrary units (a. u) of the observed fluorescence bands for different glass specimens have been collected in Table-6 and shown inn Fig 2.

Table-5. Judd-Ofelt intensity parameters (Ω_λ, λ=2, 4, 6)(x10^{-20} cm^2) Pr^{3+}: BZCAP 1-5 glasses.

Parameter	BZCAP1	BZCAP 2	BZCAP 3	BZCAP 4	BZCAP5
Ω_2	0.70	0.79	1.06	1.13	0.72
Ω_4	2.32	2.12	1.84	2.44	2.00
Ω_6	1.83	2.00	2.22	2.40	1.99
Ω_4/Ω_6	1.27	1.06	0.83	1.02	1.01
$\Omega_2+\Omega_4+\Omega_6$	4.85	4.91	5.12	5.97	4.71

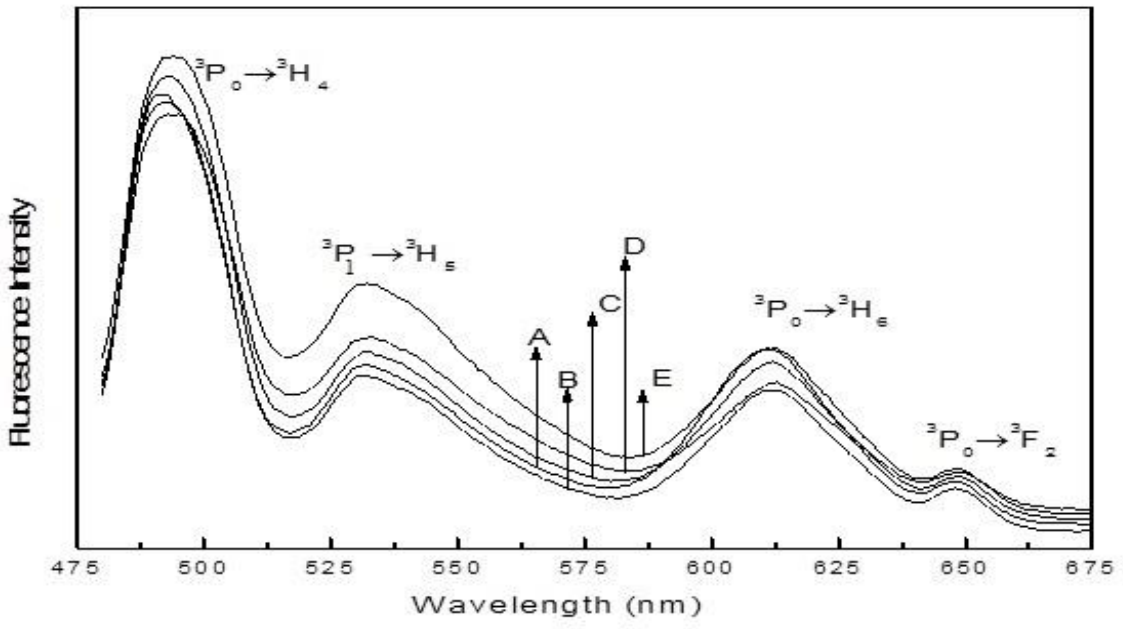

Fig.2. Emission spectra of Pr^{3+}: BZCAP 1-5 glasses

Table-6. Emission peak wavelengths ($\lambda_{P,}$ nm), effective line widths ($\Delta\lambda_{eff}$, nm) and stimulated emission cross sections ($\sigma_{e,}$ $x_{10}^{-20}cm^2$) of Pr^{3+} BZCAP 1-5 doped glasses.

Transition	BZCAP 1				BZCAP 2				BZCAP 3				BZCAP 4				BZCAP 5			
	λ_P	$\Delta\lambda_{eff}$	σ_e	A	λ_P	$\Delta\lambda_{eff}$	σ_e	A	λ_P	$\Delta\lambda_{eff}$	σ_e	A	λ_P	$\Delta\lambda_{eff}$	σ_e	A	λ_P	$\Delta\lambda_{eff}$	σ_e	A
$^3P_0 \rightarrow {}^3H_4$	492	23.4	264	20626	493	23.4	244	18906	492	20.8	209	16403	494	20.8	282	21743	494	20.8	206	17828
$^3P_1 \rightarrow {}^3H_5$	532	46.9	99	11337	532	46.9	94	10795	531	44.3	81	9952	532	44.3	102	12530	533	46.9	71	10306
$^3P_0 \rightarrow {}^3H_6$	611	26.0	950	3464	611	26.0	104	3798	612	26.0	102	4202	613	31.2	933	4546	613	28.6	755	3777
$^3P_0 \rightarrow {}^3F_2$	647	15.6	258	4487	648	18.2	251	5071	648	18.2	299	6815	648	18.2	320	7295	649	13.0	255	4652

3.5 Lasing parameters

The lasing parameter can also be obtained from the J.O parameters derived from the optical absorption spectral data [31]. This is an advantageous feature to evaluate the lasing potency of the glasses through a simple technique which is also reported earlier in the literature. In the present work the spontaneous emission cross section (σ_e) of $^3P_0 \longrightarrow {}^3H_4$ transition. The spontaneous emission cross section (σ_e) for $^3P_0 \longrightarrow {}^3H_4$ transition shows higher values among the other $^3P_0 \longrightarrow {}^3H_4$, $^3P_0 \longrightarrow {}^3H_6$ and $^3P_0 \longrightarrow {}^3F_2$ transition of all the glass systems studied and similar results can be found in literature [32]. However, the other transitions $^3P_0 \longrightarrow {}^3H_4$, $^3P_0 \longrightarrow {}^3H_6$ and $^3P_0 \longrightarrow {}^3F_2$ can also be employed for good lasing action relevant favorable condition.

The rate of energy extraction indicating parameter emission cross section (σ_e) shows the following tendency in all glass systems studied.

BZCAP -1: $^3P_0 \longrightarrow {}^3H_4 > {}^3P_0 \longrightarrow {}^3H_6 > {}^3P_0 \longrightarrow {}^3F_2$

BZCAP 2-5: $^3P_0 \longrightarrow {}^3H_4 > {}^3P_0 \longrightarrow {}^3F_2 > {}^3P_0 \longrightarrow {}^3H_6$

The branching ratios computed from the absorption data and the experimental data obtained from photoluminescence studies are reported in Table 7

Table.7 Branching ratios of some important lasing transitions of Pr^{3+}: BZCAP 1-5 glasses.

Glass systems	Transition	Branching ratios (β_R)	
		$\beta_{R\ (exp)}$	$\beta_{R\ (cal)}$
BZCAP 1	$^3P_0 \rightarrow {}^3H_4$	0.60	0.62
	$^3P_0 \rightarrow {}^3H_6$	0.31	0.33
	$^3P_0 \rightarrow {}^3F_2$	0.03	0.04
BZCAP 2	$^3P_0 \rightarrow {}^3H_4$	0.61	0.65
	$^3P_0 \rightarrow {}^3H_6$	0.32	0.36
	$^3P_0 \rightarrow {}^3F_2$	0.03	0.04
BZCAP 3	$^3P_0 \rightarrow {}^3H_4$	0.61	0.63
	$^3P_0 \rightarrow {}^3H_6$	0.31	0.35
	$^3P_0 \rightarrow {}^3F_2$	0.03	0.05
BZCAP 4	$^3P_0 \rightarrow {}^3H_4$	0.63	0.67
	$^3P_0 \rightarrow {}^3H_6$	0.30	0.31
	$^3P_0 \rightarrow {}^3F_2$	0.03	0.04
BZCAP 5	$^3P_0 \rightarrow {}^3H_4$	0.60	0.62
	$^3P_0 \rightarrow {}^3H_6$	0.30	0.34
	$^3P_0 \rightarrow {}^3F_2$	0.03	0.05

It is a quite interesting to note that with results the experimental and calculated β values are in good agreement which reflects the validity of the lasing potencies evaluate in the present study. It is quite obvious that the $^3P_0 \rightarrow {}^3H_4$ transition in all the glass systems reported exhibit higher potencies and therefore these transitions can be employed for lasing activities in the material preparation.

3.6 FT-IR Analysis

The FT-IR spectra of all the five glasses of the present study are presented in Fig.3. Following Tarte [33] and Condrate [34] the spectral data was analyzed and the characteristic absorption levels of PbO [35], and vitreous B_2O_3 [36, 37] with their assignments are presented in the Table 8.

Table-8. Assignments of FT- IR in Pr^{3+}: BZCAP 1-5 glasses.

Wavenumber(cm^{-1})	**FT- IR assignment**
476, 491	stretching vibration in PbO_4 [2], B-O-B and Pb-O-B bending vibration as well as borate ring deformation
693	Bending of B-O-B linkage
1014	stretching of B-O bond stretching of tetrahedral BO_4 units
1405	B-O stretching vibrations of BO_3 units in chain and ring type metaborate groups [2]
1632	Bending modes of OH groups [2]

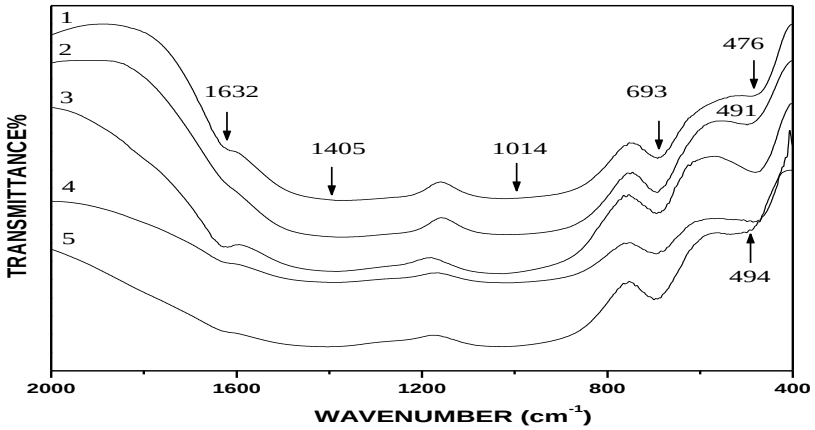

Fig.3. FT-IR spectra of BZCAP 1-5 glass systems

3.7 XRD Spectra

The XRD pattern of Pr^{3+} doped Heavy Metal Oxide (HMO) glass systems (BZCAP 1) has confirmed their amorphous nature and shown in Fig.4.

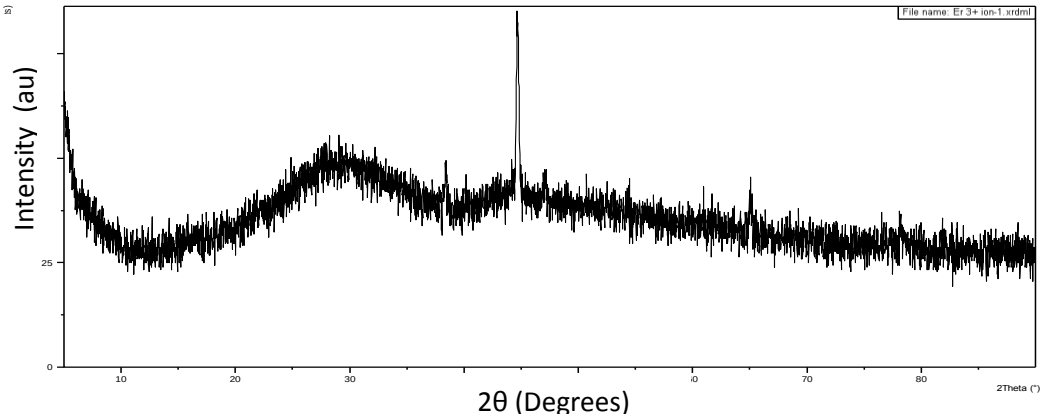

Fig-4: XRD pattern of Pr^{3+}: BZCAP 1 glass

4. Conclusions

The Pr^{3+} doped lead based HMO multi component glass systems have been fabricated to study their laser and other properties. J-O theory is successfully implemented for the analysis of optical absorption spectra. The branching ratios (β_R) estimated through the optical absorption data are in good agreement with the experimental results obtained in the photo luminescence studies. The $^3P_0 \rightarrow {}^3H_4$ lasing transition appears to be potential in all the five glass system studied.

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