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Spectral and FTIR Analysis of Ho^{3+} ions doped Zinc Lithium Calcium Potassiumniobate Phosphate Glasses

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Abstract
Glass of the system: (45-x)P_2O_5:10ZnO:10Li_2O:10CaO:10K_2O:15Nb_2O_5::xHo_2O_3, (where x=1, 1.5,2 mol %) have been prepared by melt-quenching method.The amorphous nature of the prepared glasssamples was confirmed by X-ray diffraction. Optical absorption,Excitation, fluorescence and FTIR spectra were recorded at roomtemperature for all glass samples.Judd-Ofeltintensity parameters Ω_λ (λ=2, 4 and6) are evaluated from theintensities of various absorption bands of opticalabsorption spectra. Using these intensity parameters variousradiative properties like spontaneous emission probability (A), branching ratio (β), radiative life time(τ_R) and stimulatedemission cross–section(σ_p)of various emission lines have been evaluated.

Keywords: *ZLCPNP Glasses,Optical Properties, FTIR Spectroscopy, Judd-Ofelt Theory.*

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I. Introduction

Glasses doped with rare earth ions have attracted a great deal of attention because of their applications in laser fusion, optical fibers, sensors, infrared detectors, marine optical communications, up-conversion lasers, optical data storage and high density memory storage devices [1–5]. Among different glasses,phosphate glasses have unique properties. They have high transparency,high thermal stability,lower phonon energy,high refractive index and high density[6-11].Addition of network modifier (NWF) Li_2O to the phosphate glasses improves both electrical and mechanical properties of such glasses[12, 13].The addition of ZnO increases both the tendency of glass formation, refractive index while decreases the optical energy band gap [14].Ho^{3+} doped glasses are very important because of the possibility of their application in optoelectronic and optic device fields, such as lasers, fiber optics and solar cells [15-21].

The present work reports on the preparation and characterization of rare earth doped heavy metal oxide (HMO) glass systems for lasing materials.We have studied on the Optical absorption,Excitation, fluorescence and FTIR spectra of Ho^{3+}doped zinc lithium calcium potassiumniobatephosphateglasses. 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

Preparation of glasses

The following Ho^{3+}doped phosphateglass samples (45-x)P_2O_5:10ZnO:10Li_2O:10CaO:10K_2O:15Nb_2O_5: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, CaO,K_2O,$Nb_2O_5$and Ho_2O_3. They were thoroughly mixed by using an agate pestle mortar. then melted at 1075^0C 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.**

Table 1.
Chemical composition of the glasses

Sample	Glass composition (mol %)
ZLCPNP(UD)	45P_2O_5:10ZnO:10Li_2O:10CaO:10K_2O:15Nb_2O_5
ZLCPNP (HO1)	44P_2O_5:10ZnO:10Li_2O:10CaO:10K_2O:15Nb_2O_5:1 Ho_2O_3
ZLCPNP(HO1.5)	43.5P_2O_5:10ZnO:10Li_2O:10CaO:10K_2O:15Nb_2O_5:1.5Ho_2O_3

ZLCPNP(HO2) $43P_2O_5$:10ZnO:10Li_2O:10CaO:10K_2O:15Nb_2O_5:2Ho_2O_3

ZLCPNP (UD) -Represents undopedZinc Lithium Calcium PotassiumniobatePhosphateglass specimens
ZLCPNP(HO)-Represents Ho^{3+}dopedZinc Lithium Calcium PotassiumniobatePhosphateglass specimens

III. Theory

3.1Oscillator Strength

The intensity of spectral lines are expressed in terms of oscillator strengths using the relation [22].

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

where, ε (*ν)* 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 [23], 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.

3.2. Judd-Ofelt Intensity Parameters

According to Judd[24] and Ofelt[25] 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

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

λ =2, 4, 6

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 (λ=2,4and 6) are known as Judd-Ofelt intensity.

3.3Radiative 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

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

where, the sum is over all terminal manifolds.

The radiative life time is given by

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

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

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

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 [26, 27]. The Nephelauxetic Ratio is given by

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

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

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

IV. Result and Discussion

4.1XRD 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.

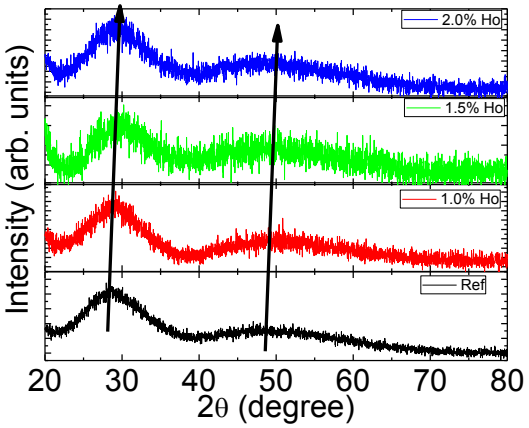

Fig. 1 X-ray diffraction pattern of ZLCPNP (HO) Glasses.

4.2 FTIR Transmission spectra

The FTIR spectrum of ZLCPNP(HO 01) glass is in the wave number range500-4000$cm^{-1}$is presented in Fig.2 and the possible mechanism bands are tabulated in Table 2.

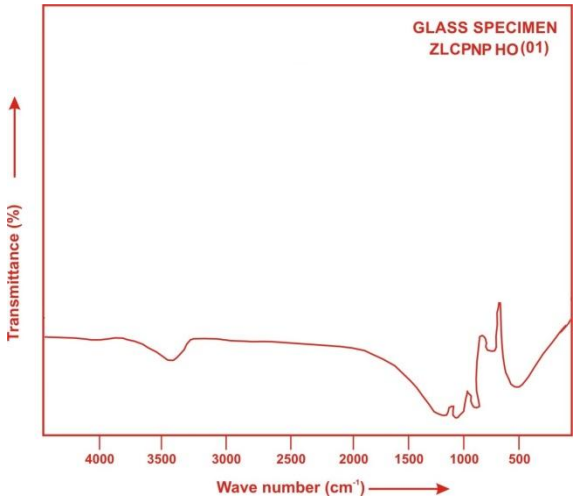

Fig. (2) FTIR spectrum of ZLCPNP HO (01) glass.

The band observed at 525 cm^{-1} is attributed to the P-O-P bending vibrations [20]. The observed band around at 750 cm^{-1} is due to the P-O-P symmetric stretching vibrations while the occurrence of band around 926cm^{-1} is assigned to the P-O-P asymmetric stretching vibrations [21, 22]. The Asymmetric stretching modes of $(PO_3)^{2-}$ groups is observed around 1110 cm^{-1}[23].The Asymmetric stretching vibration of P=O and O-P-O bonds is observed around 1235cm^{-1}[24].The observed band around at 3425 cm^{-1} is attributed to symmetric stretching vibrations of the O-H bonds [25].

Table2. Assignment of infrared transmission bands of(ZLCPNPHO 01) glass.

Peak position(cm^{-1})	**Band Assignment**
~ 525	Bending vibration of P-O-P bands
~750	Symmetric stretching vibration of P-O-P bonds
~926	Asymmetric stretching vibration of P-O-P bonds
~1110	Asymmetric stretching modes of $(PO_3)^{2-}$ groups
~1235	Asymmertic Stretching Vibration of P=O and O-P-O bonds
~3425	Symmetric stretching of the O-H bonds

4.3 Absorption Spectrum

The absorption spectra of Ho^{3+}doped ZLCPNPglass specimens have been presented in Figure 3 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+} dopedZLCPNPglasses.

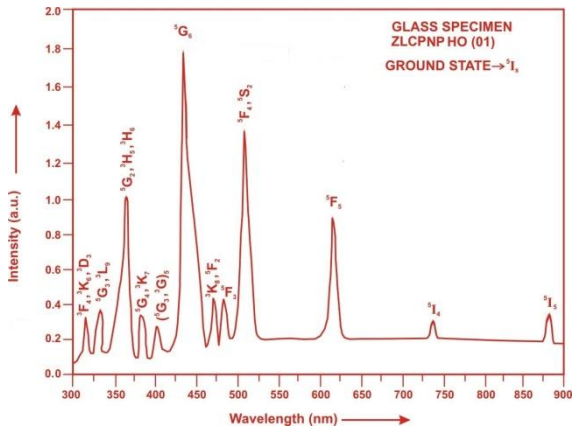

Fig. (3) Absorption spectrum of ZLCPNP HO (01) glass.

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

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

Energy level from $^5I_8$	Glass ZLCPNP(HO01)		Glass ZLCPNP(HO1.5)		Glass ZLCPNP(HO02)	
	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$
$^5I_5$	0.48	0.25	0.43	0.24	0.39	0.23
$^5I_4$	0.06	0.02	0.05	0.02	0.04	0.02
$^5F_5$	3.68	2.84	3.43	2.72	3.38	2.69
$^5F_4$	4.72	4.39	4.64	4.25	4.59	4.19
$^5F_3$	1.62	2.44	1.55	2.37	1.48	2.34
$^3K_8$	1.50	2.00	1.41	1.93	1.37	1.89
$^5G_6$	25.78	25.75	24.26	24.26	23.13	23.15
$(^5G,^3G)_5$	3.95	1.73	3.76	1.63	3.72	1.61
$^5G_4$	0.04	0.62	0.03	0.59	0.02	0.58
$^5G_2$	5.65	5.48	5.60	5.19	5.53	4.98
$^5G_3$	1.48	1.41	1.43	1.36	1.38	1.33
$^3F_4$	1.36	4.25	1.32	4.02	1.28	3.97
r.m.s. deviation	1.1335		1.0787		1.0797	

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

Table 4: F_2,$\xi_{4f,}$ 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

In theZinc Lithium Calcium PotassiumniobatePhosphateglasses (ZLCPNP)Ω_2, Ω_4 and $\Omega_6$parameters decrease with the increase of x from 1 to 2 mol%. The order ofmagnitude of Judd-Ofelt intensity parameters is $\Omega_2>\Omega_6>\Omega_4$for all the glass specimens. Thespectroscopic quality factor (Ω_4/Ω_6) related with the rigidity of the glass system has been found to lie between 0.598 and 0.617 in the present glasses.
The values of Judd-Ofelt intensity parameters are given in **Table 5.**

Table5:Judd-Ofelt intensity parameters for Ho^{3+} doped ZLCPNP glass specimens.

Glass Specimen	Ω_2(pm^2)	Ω_4(pm^2)	Ω_6(pm^2)	Ω_4/Ω_6
ZLCPNP (HO01)	6.356	1.392	2.256	0.6170
ZLCPNP (HO1.5)	5.982	1.304	2.188	0.5960
ZLCPNP HO02)	5.657	1.290	2.158	0.5978

4.4Excitation Spectrum

The Excitation spectrum of ZLCPNP (HO 01) glass has been presented in Figure 4 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 at349,419, 450, 473and 486 nmare 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 450 nm.So this is to be chosen for excitation wavelength.

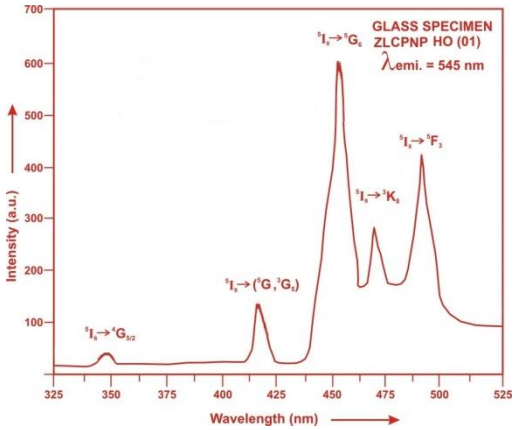

Fig. (4) Excitation spectrum of ZLCPNP HO (01) glass.

4.5 Fluorescence Spectrum

The fluorescence spectrum of Ho^{3+}doped in zinc lithium calcium potassiumniobatephosphate glass is shown in Figure 5. There are five broad bands observed in the Fluorescence spectrum ofHo^{3+}dopedzinc lithium calcium potassiumniobatephosphateglass. The wavelengths of these bands along with their assignments are given in Table 6. The peak with maximum emission intensity appears at 501nm and corresponds to the ($^5F_4 \rightarrow {}^5I_8$) transition.

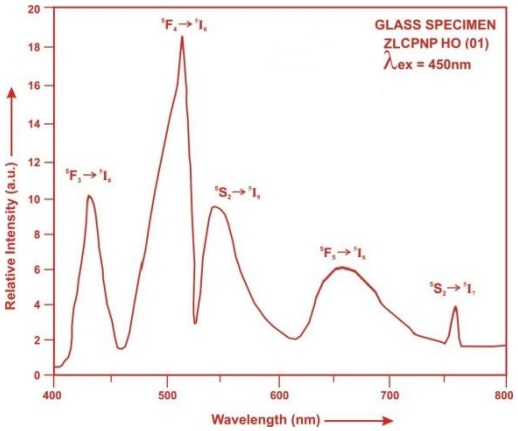

Fig. (5). Fluorescence spectrum of ZLCPNP HO (01) glass.

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 ZLCPNP glasses.

Transition		ZLCPNP(HO 01)				ZLCPNP(HO 1.5)				ZLCPNP (HO 02)			
	λ_{max} (nm)	$A_{rad}(s^{-1})$	β	σ_p (10^{-20} cm^2)	τ_R(μs)	$A_{rad}(s^{-1})$	β	σ_p (10^{-20} cm^2)	τ_R (μs)	$A_{rad}(s^{-1})$	β	σ_p(10^{-20} cm^2)	τ_R(10^{-20} cm^2)
$^5F_3 \rightarrow {}^5I_8$	435	4138.30	0.2646	0.530		4024.36	0.2657	0.506		3977.11	0.2656	0.489	
$^5F_4 \rightarrow {}^5I_8$	501	6582.29	0.4209	1.181		6363.06	0.4201	1.121		6290.86	0.4202	1.082	
$^5S_2 \rightarrow {}^5I_8$	555	1727.64	0.1105	0.403	6395.12	1679.82	0.1109	0.385	6602.25	1660.10	0.1109	0.374	6679.20
$^5F_5 \rightarrow {}^5I_8$	652	1877.33	0.1201	0.718		1804.75	0.1192	0.681		1784.39	0.1192	0.658	
$^5S_2 \rightarrow {}^5I_7$	761	1311.35	0.0839	0.970		1274.36	0.0841	0.933		1259.40	0.0841	0.904	

V. Conclusion

In the present study, the glass samples of composition (45-x)P2O5:10ZnO:10Li2O:10CaO:10K2O:15Nb2O5:xHo2O3. (where x =1, 1.5and 2mol %) have been prepared by melt-quenching method. The value of stimulated emission cross-section (σp) is found to be maximum for the transition (5F4→5I8) for glass ZLCPNP(HO 01), suggesting that glass ZLCPNP (HO 01) is better compared to the other two glass systems ZLCPNP (HO1.5) and ZLCPNP(HO02).The large stimulated emission cross section in bismuth borate glasses suggests the possibility of utilizing these systems as laser materials. The FTIR of glasses revealed the presence of characteristic bonding vibrations of different functional groups.

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