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Spectral and Raman Analysis of Pr^{3+} Doped Zinc Lithium Lead Sodium Sodalime Silicate Glasses

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ABSTRACT

Zinc lithium lead sodium sodalime silicate glasses containing Pr^{3+} in (35-x):SiO_2:10ZnO:10Li_2O:10PbO:10CaO:15Na_2O:xPr_6O_{11} (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 and fluorescence spectra were recorded at room temperature for all glass samples. Judd-Ofelt intensity parameters Ω_λ (λ=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: *ZLLSSLS Glasses, Optical Properties, Judd-Ofelt Theory, Rare earth ions.*

1. INTRODUCTION

Glasses doped with various rare –earth ions are important materials for fluorescent display devices, solid state lasers, optical detectors, optical fibers and optical amplifiers [1-5].Among the other heavy metal oxide glasses, silicate glasses have wide range of applications in the field of glass ceramics, layers for optical, thermal and mechanical sensors, reflecting windows [6-9]. The addition of network modifier (NWF) Li_2O is to improve both 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 [10, 11]. The past literature shows that the rare earth ions find more important application in the preparation of the laser materials [12-16].Pr^{3+} ion is the most studied among the rare earth ions and the up conversion process of this ion in various kinds of host materials has been investigated [17-20].

In this work, the spectroscopic properties of Pr^{3+} -doped (35-x):SiO_2:10ZnO:10Li_2O:10PbO:10CaO:15Na_2O:xPr_6O_{11} (where x=1, 1.5,2 mol %) glasses were investigated. The physical and optical properties, the absorption spectra, fluorescence spectra of Pr^{3+} of the glasses were investigated. The J-O intensity parameters render significant information regarding local structure and bonding in the vicinity of rare- earth ions. The parameter Ω_2 is related with the symmetry of the glass hosts while Ω_6 is a measure of the covalency in the network.

2. EXPERIMENTAL TECHNIQUES

Preparation of glasses

The following Pr^{3+} doped zinc lithium lead sodium sodalime silicate glass samples (35-x):SiO_2:10ZnO:10Li_2O:10PbO:10CaO:15Na_2O: xPr_6O_{11} (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,PbO,CaO ,$Na_2O$and Pr_6O_{11}. 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 1050^0C, for preparation of zinc lithium lead sodium sodalime silicate 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 to100^0C.While pouring; the temperature of crucible was also maintained to prevent crystallization. And annealed at temperature of 350^0C 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 %)
ZLLSSLS (UD)	35SiO_2:10ZnO:10Li_2O:10PbO:10CaO:15Na_2O
ZLLSSLS (PR 1)	34SiO_2:10ZnO:10Li_2O:10PbO:10CaO:15Na_2O:1 Pr_6O_{11}
ZLLSSLS (PR 1.5)	33.5SiO_2:10ZnO:10Li_2O:10PbO:10CaO:15Na_2O: 1.5 Pr_6O_{11}
ZLLSSLS (PR 2)	33SiO_2:10ZnO:10Li_2O:10PbO:10CaO:15Na_2O: 2 Pr_6O_{11}

ZLLSSLS (UD)—Represents undoped Zinc Lithium Lead Sodium Sodalime Silicate glass specimen.

ZLLSSLS (PR) -Represents Pr^{3+} doped Zinc Lithium Lead Sodium Sodalime Silicate glass specimens.

3. THEORY

3.1 Oscillator Strength

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

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

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, using the modified relation [22].

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

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

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

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

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

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 (λ = 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).

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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

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

where, the sum is over all terminal manifolds.

The radiative life time is given by

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

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'> 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 [25, 26]. 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

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

4. 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.

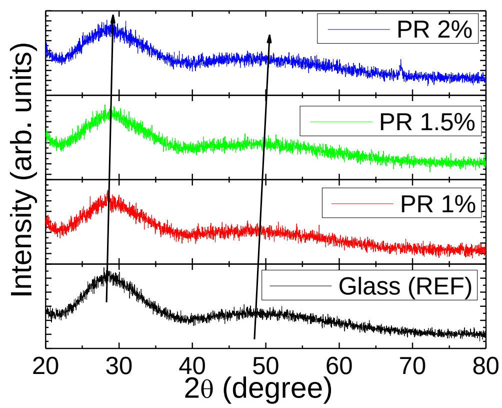

Fig.1: X-ray diffraction pattern of ZLLSSLS (PR) glasses.

4.2. Absorption spectra

The absorption spectra of ZLLSSLS (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 ZLLSSLS (PR) glasses.

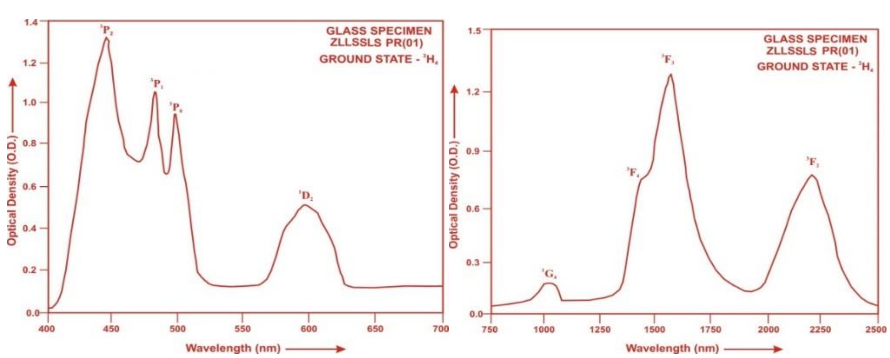

Fig.2: Vis-NIR absorption spectra of ZLLSSLS (PR) glasses.

The experimental and calculated oscillator strengths for Pr^{3+} ions in zinc lithium lead sodium sodalime silicate glasses are given in **Table 2**

Table 2. Measured and calculated oscillator strength ($P^m \times 10^{+6}$) of Pr^{3+} ions in ZLLSSLS glasses.

Energy level $^3H_4$	Glass ZLLSSLS (PR01)		Glass ZLLSSLS (PR1.5)		Glass ZLLSSLS (PR02)	
	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$
$^3F_2$	4.40	3.68	4.37	3.67	4.33	3.64
$^3F_3$	7.72	6.73	7.69	6.71	7.65	6.69
$^3F_4$	4.32	3.93	4.28	3.91	4.24	3.89
$^1G_4$	0.52	0.34	0.50	0.34	0.47	0.34
$^1D_2$	3.38	1.17	3.35	1.16	3.32	1.16
$^3P_0$	4.56	2.44	4.52	2.45	4.48	2.47
$^3P_1$	4.99	2.47	4.96	2.49	4.92	2.50
$^3P_2$	12.32	3.84	12.28	3.83	12.21	3.81
R.m.s.deviation	3.3399		3.3203		3.2908	

The various energy interaction parameters like Slater-Condon parameters F_k (k=2, 4, 6), Lande's parameter ξ_{4f} and Racah parameters E^k (k=1, 2, 3) have been computed using partial regression method and formula described elsewhere [24]. The ratio of Racah parameters E^1/E^3 and E^2/E^3 are about 9.79 and 0.048 respectively. Computed values of Slater-Condon, Lande′, Racah, nephelauexetic ratio and bonding parameter for Pr^{3+} doped ZLLSSLS glass specimens are given in **Table 3**.

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Table3. Computed values of Slater-Condon, Lande, Racah, nephelauexetic ratio and bonding parameter for Pr^{3+} doped ZLLSLBS glass specimens.

Parameter	Free ion	ZLLSLBS PR01	ZLLSLBS PR1.5	ZLLSLBS PR02
$F_2(cm^{-1})$	322.09	300.02	299.98	300.00
$F_4(cm^{-1})$	44.46	44.28	44.25	44.26
$F_6(cm^{-1})$	4.867	4.413	4.411	4.412
$\xi_{4f}(cm^{-1})$	741.00	858.28	858.81	858.48
$E^1(cm^{-1})$	4728.92	4451.45	4449.98	4450.84
$E^2(cm^{-1})$	24.75	22.01	22.01	22.01
$E^3(cm^{-1})$	478.10	454.72	454.68	454.72
F_4/F_2	0.13805	0.14758	0.1475	0.1475
F_6/F_2	0.01511	0.014709	0.014702	0.014705
E^1/E^3	9.8911	9.7894	9.7870	9.7882
E^2/E^3	0.0518	0.04840	0.04841	0.04840
β'		0.8888	0.88844	0.888646
$b^{1/2}$		0.2358	0.2362	0.2360

Judd-Ofelt intensity parameters Ω_λ (λ = 2, 4 and 6) were calculated by using the fitting approximation of the experimental oscillator strengths to the calculated oscillator strengths with respect to their electric dipole contributions. In the present case the three Ω_λ parameters follow the trend $\Omega_2 < \Omega_4 < \Omega_6$.

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

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Table 4. Judd-Ofelt intensity parameters for Pr^{3+} doped ZLLSSLS glass specimens.

Glass Specimen	$\Omega_2(pm^2)$	$\Omega_4(pm^2)$	$\Omega_6(pm^2)$	Ω_4/Ω_6
ZLLSSLS (PR01)	1.946	2.545	4.032	0.6312
ZLLSLBS (PR1.5)	1.920	2.559	4.006	0.6388
ZLLSLBS (PR02)	1.887	2.571	3.979	0.6461

4.4 Excitation Spectrum

Excitation spectra of ZLLSSLS PR (01) glass recorded at the emission wavelength 395 nm is depicted as figure 3. The excitation spectra consists of three peaks corresponding to the transitions from the ground state $^3H_4$ to the various excited states $^3P_2$, $^3P_1$ and $^3P_0$ at the wavelengths of 448, 467 and 485 nm respectively. Among these, a prominent excitation band at 448 nm has been selected for the measurement of emission spectrum of ZLLSSLS PR (01) glass.

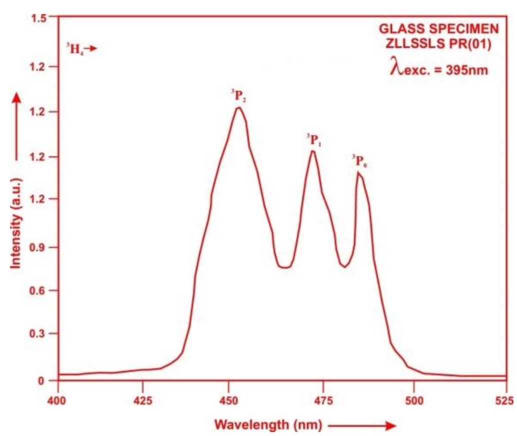

Fig.3: Excitation spectra of ZLLSSLS PR (01) glass.

4.3. Fluorescence Spectrum

The fluorescence spectrum of Pr^{3+}doped in zinc lithium lead sodium sodalime silicate glass is shown in Figure 3. There are eight broad bands ($^3P_0\rightarrow^3H_4$), ($^3P_0\rightarrow^3H_5$), ($^3P_0\rightarrow^3H_4$), ($^3P_0\rightarrow^3H_6$), ($^3P_0\rightarrow^3F_2$), ($^3P_1\rightarrow^3F_3$), ($^1D_2\rightarrow^3H_5$) and ($^3P_0\rightarrow^3F_4$) respectively for glass specimens.

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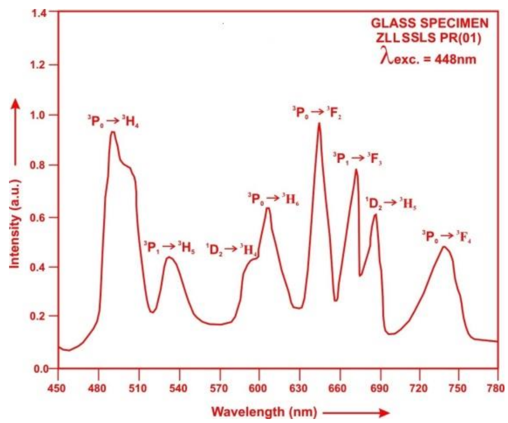

Fig.4: Fluorescence spectrum of ZLLSSLS PR (01) glass.

Table 5. 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 ZLLSSLS glasses.

Transition		ZLLSSLS PR 01				ZLLSSLS PR 1.5				ZLLSSLS PR 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)
$^3P_0 \rightarrow {}^3H_4$	485	1213.45	0.165	0.492		1221.83	0.167	0.580		1230.79	0.169	0.691	
$^3P_0 \rightarrow {}^3H_5$	532	1874.60	0.256	0.421		1882.34	0.257	0.452		1887.90	0.259	0.479	
$^3P_0 \rightarrow {}^3H_4$	599	361.48	0.049	0.160		360.58	0.049	0.166		359.53	0.0492	0.180	
$^3P_0 \rightarrow {}^3H_6$	602	294.96	0.040	0.179		293.65	0.040	0.196		292.12	0.040	0.219	

$^3P_0 \rightarrow ^3F_2$	643	1230.34	0.168	1.344	136.345	1216.91	0.166	1.522	136.540	1198.41	0.164	1.818	136.946
$^3P_1 \rightarrow ^3F_3$	676	2130.16	0.290	1.327		2117.48	0.289	1.402		2100.79	0.288	1.493	
$^1D_2 \rightarrow ^3H_5$	685	5.212	0.0007	0.0064		5.24	0.0007	0.0074		5.254	0.0007	0.00846	
$^3P_0 \rightarrow ^3F_4$	730	224.15	0.0306	0.189		225.84	0.0308	0.0206		227.36	0.0311	0.218	

5. CONCLUSION

In the present study, the glass samples of composition (35-x):SiO_2:10ZnO:10Li_2O:10PbO:10CaO:15Na_2O:xPr_6O_{11} (where x =1, 1.5, 2 mol %) have been prepared by melt-quenching method. The value of stimulated emission cross-section (σ_p) is found to be maximum for the transition ($^3P_0 \rightarrow ^3F_2$) for glass ZLLSSLS (PR 02), suggesting that glass ZLLSSLS (PR 02) is better compared to the other two glass systems ZLLSSLS (PR01) and ZLLSSLS (PR1.5).

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