IJCPS Vol. 10, No. 4 , Jul-Aug 2021
www.ijcps.org
DOI Prefix:10.30731/ijcps

International Journal of Chemical and Physical Sciences
(Peer-Reviewed Journal)
ISSN:2319-6602

Spectral and Raman Analysis of Nd^{3+} Doped Zinc Lithium Soda lime Cadmium Borosilicate Glasses

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Abstract

Glass of the system: (35-x) SiO_2:10ZnO:10Li_2O:10CaO:10Na_2O:10CdO:15B_2O_3: xNd_2O_3. (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,Excitation, fluorescence and Raman spectra have been recorded at room temperature for all glass samples.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 these parameters energies and intensities of these bands has beencalculated.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 (A),branching ratio (β_R),radiative life time (τ)and stimulated emission cross–section (σ_p)of various emission lines have been evaluated.

Keywords: ZLCBS Glasses, Optical Properties, Judd-Ofelt Theory, Raman Spectra.

Introduction:

Rare earth doped phosphate glasses are being extensively studied due to their technological importance and their applications in the field of glass ceramics, thermal and mechanical sensors, reflecting windows [1-5].Among different host matrices, silicate glasses are quite prone with the advantages such as low non-linear refractive index, good physical and chemical stability and high transparency from near Ultra Violet to mid-Infrared region [6-11].Due to their good chemical durability, neodymium-doped soda-lime silicate glasses are attractive materials for the fabrication of low-cost integrated optical amplifiers by using the ion-exchange technique.Glasses contain B_2O_3 have received increased attention due to their application in the field of glass ceramics, reflecting windows, thermal and mechanical sensors [12-14].The addition of network modifier (NWF) Li_2O is to improve both electrical and mechanical properties of such glasses. Silicate glasses also exhibit high RE ions solubility [15].Nd^{3+} doped materials have proven to be among the most efficient candidates for photonic devices, such as lasers and planer wave guides [16-20].

In the present work, Optical absorption, Excitation, fluorescence and Raman spectra have been recorded at room temperature for all glass samples. From the spectral data 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. The Judd-Ofelt theory has been applied to compute the intensity parameters Ω_λ (λ=2, 4, 6).To understand the laser efficiency of these materials, the value of spectroscopy quality factor (Ω_4/Ω_6) has been evaluated.

Experimental:

Preparation of glasses:

The following Nd^{3+} doped borosilicate glass samples (35-x) SiO_2:10ZnO:10Li_2O:10CaO:10Na_2O:10CdO:15B_2O_3: xNd_2O_3 (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, CaO,Na_2O,CdO,$B_2O_3$and Nd_2O_3. They were thoroughly mixed by using an agate pestle mortar.Then melted at 1075˚C by an electricalmuffle furnace for 2 hours. After complete melting, the melts were quickly poured in to a preheated stainless steel mould and annealed at temperature of 350˚C for 2 h to remove thermal strains and stresses. Every time fine powder of cerium oxide was used for polishing the samples. Theglass samples so prepared were of good optical quality and were transparent. The chemical compositions of theglasses with the name of samples are summarized in **Table 1.**

Table 1: Chemical composition of the glasses

Sample	Glass composition (mol %)
ZLSLCBS (UD)	35SiO_2:10ZnO:10Li_2O:10CaO:10Na_2O:10CdO:15B_2O_3:
ZLSLCBS (ND1)	34SiO_2:10ZnO:10Li_2O:10CaO:10Na_2O:10CdO:15B_2O_3:1Nd_2O_3
ZLSLCBS (ND1.5)	33.5SiO_2:10ZnO:10Li_2O:10CaO:10Na_2O:10CdO:15B_2O_3:1.5Nd_2O_3
ZLSLCBS (ND2)	33SiO_2:10ZnO:10Li_2O:10CaO:10Na_2O:10CdO:15B_2O_3:2Nd_2O_3

ZLSLCBS (UD)-Represents undopedZinc Lithium SodalimeCadmium Borosilicateglassspecimen.
ZLSLCBS (ND)-Represents Nd^{3+} doped Zinc Lithium SodalimeCadmium Borosilicate glass specimens.

Theory:

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 [22], 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.

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 Jmanifold $|4f^N (S, L) J\rangle$ level and the terminal J' manifold $|4f^N (S', L') J'\rangle$ is given by:

International Journal of Chemical and Physical Sciences
(Peer-Reviewed Journal)
ISSN:2319-6602

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

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

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

$\lambda = 2, 4, 6$

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 ($\lambda = 2, 4, 6$) are known as Judd-Ofelt intensity parameters.

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:

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

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

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

S L J where, the sum is over all terminal manifolds.

The radiative life time is given by

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

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.

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 values of bonding parameter $b^{1/2}$ are given by

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

Results and Discussion:

XRD Measurement

Figure 1presents the XRD pattern of the samples shows no sharp Bragg's peak, but only a broad diffuse humparound low angle region. This is the clear indication of amorphous nature within the resolution limit of XRD instrument.

Raman spectra

The Raman spectrum of Zinc Lithium Sodalime Cadmium Borosilicate (ZLSLCBS) glass specimens is recorded and is shown in Fig. 2. The spectrum peaks located at 602, 795 and 1204 cm^{-1}. The band at 602 and 795cm^{-1} assigned to Si–O–Si symmetric stretching and bending vibration, respectively. The band at 1204 cm^{-1} assigned to Si–O–Si asymmetric stretching.

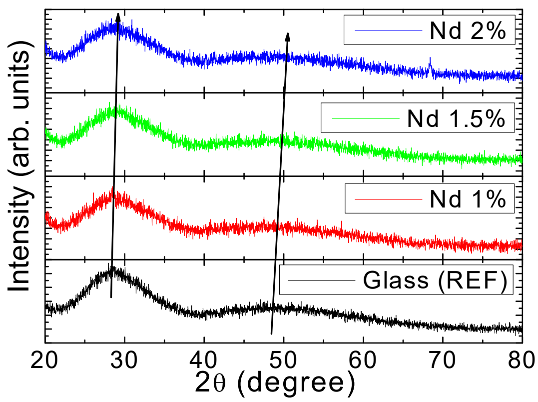


Fig.1:X-ray diffraction pattern of SiO_2:ZnO:Li_2O:CaO:Na_2O:CdO:B_2O_3:Nd_2O_3 glasses.

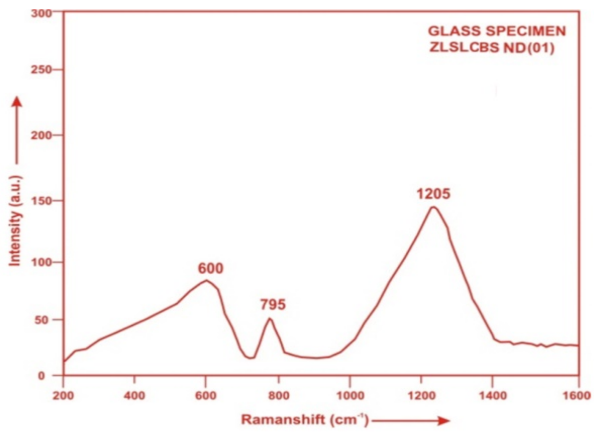


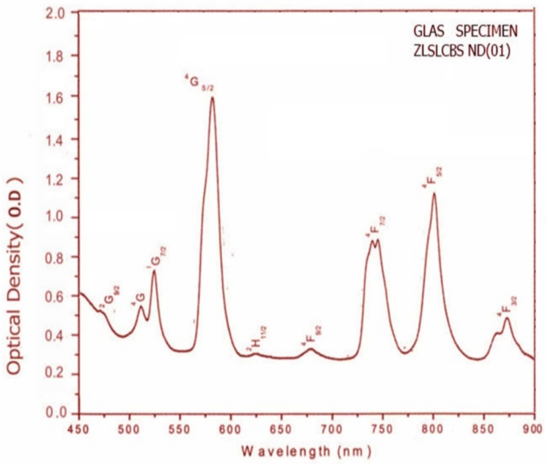
Fig.2: Raman spectrum of ZLSLCBSND (01) glass.

Absorption spectrum

The absorption spectra ofZLSLCBS (ND 01) glass,consistsof absorption bands corresponding to the absorptions from the ground state $^4I_{9/2}$ of Nd^{3+} ions.Nine absorption bands have been observed from the ground state $^4I_{9/2}$to excited states $^4F_{3/2}$,$^4F_{5/2}$,$^4F_{7/2}$,$^4F_{9/2}$,$^2H_{11/2}$,$^4G_{5/2}$,$^4G_{7/2}$,$^4G_{9/2}$, and $^2G_{9/2}$ for Nd^{3+} doped ZLSLCBS(ND 01) glass.

Excitation Spectrum

The Excitation spectra of Nd^{3+}doped ZLSLCBS ND (01) glass specimen has been presented in Figure 4 in terms of Excitation Intensity versus wavelength. The excitation spectrum was recorded in the spectral region 700–1000 nm fluorescence at 1065nm having different excitation band centered at 808 nm and 887 nmare attributed to the ($^4I_{9/2}\rightarrow{}^4F_{5/2}$) and($^4I_{9/2}\rightarrow{}^4F_{3/2}$) transitions, respectively. The highest absorption level is $^4F_{5/2}$ and is at 808 nm. So this is to be chosen for excitation wavelength.



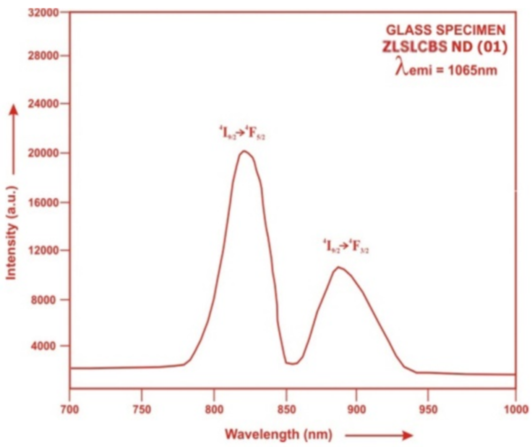


Fig.3: Vis-NIR absorption spectra of ZLSLCBS (ND 01) glass.

Fig.4: Excitation spectra of ZLSLCBSND (01) glass.

The experimental and calculated oscillator strengths for Nd^{3+} ions in zinc lithium sodalime cadmium borosilicate glasses are given in **Table 2**.

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

Energy level from $^4I_{9/2}$	GlassZLSLCBS (ND01)		Glass ZLSLCBS (ND1.5)		Glass ZLSLCBS (ND02)	
	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$
$^4F_{3/2}$	4.38	4.03	3.42	3.38	2.64	2.76
$^4F_{5/2}$	8.24	8.51	7.44	7.46	6.44	6.44
$^4F_{7/2}$	8.94	9.03	7.83	8.14	6.73	7.26
$^4F_{9/2}$	0.64	0.51	0.55	0.45	0.43	0.40
$^2H_{11/2}$	0.24	0.15	0.21	0.13	0.12	0.11
$^4G_{5/2}$	25.43	25.62	24.33	24.58	23.28	23.70
$^4G_{7/2}$	4.68	5.44	3.89	4.81	2.63	4.21
$^4G_{9/2}$	2.83	2.39	2.25	2.05	1.43	1.73
$^2G_{9/2}$	0.94	3.12	0.82	2.64	0.62	2.19
R.m.s.deviation	0.8009		0.6981		0.7811	

The various energy interaction parameters like Slater-Condon parameters F_k(k=2, 4, 6), Lande's parameter ξ_{4f},Racah parameters E^k(k=1, 2, 3), nephelauexetic ratio and bonding parameter for Nd^{3+}dopedZLSLCBS glassspecimens are given in **Table 3**.

Table 3: Computed values of Slater-Condon, Lande′, Racah, nephelauexetic ratio and bonding parameter for Nd^{3+} doped ZLSLCBS glass specimens.

Parameter	Free ion	ZLSLCBS (ND01)	ZLSLCBS (ND1.5)	ZLSLCBS (ND02)
$F_2(cm^{-1})$	331.16	327.29	327.17	327.23
$F_4(cm^{-1})$	50.71	49.91	49.94	49.93
$F_6(cm^{-1})$	5.154	5.137	5.131	5.134
$\xi_{4f}(cm^{-1})$	884.0	889.48	889.35	889.23
$E^1(cm^{-1})$	5024.0	4969.20	4967.90	4968.78
$E^2(cm^{-1})$	23.90	23.73	23.69	23.71
$E^3(cm^{-1})$	497.0	489.48	489.51	489.50
F_4/F_2	0.1531	0.1525	0.1526	0.1526
F_6/F_2	0.0155	1.569	0.01568	0.01569
E^1/E^3	10.1086	10.16	10.15	10.15
E^2/E^3	0.0481	0.0485	0.00484	0.00484
β'		0.98832 0.0076398	0.98796 0.0077583	0.988135 0.0077024
$b^{1/2}$				

Judd-Ofelt intensityparameters Ω_λ(λ = 2, 4 and 6) were calculated by using the fitting approximationof the experimental oscillator strengths to the calculated oscillator strengths with respect to their electric dipolecontributions. In the present case the three $\Omega_\lambda$parameters follow the trend $\Omega_2<\Omega_6<\Omega_4$. The values of Judd-Ofelt intensity parameters are given in **Table 4**.

Table 4: Judd-Ofelt intensity parameters for Nd^{3+} doped ZLSLCBSglass specimens.

Glass Specimen	$\Omega_2(pm^2)$	$\Omega_4(pm^2)$	$\Omega_6(pm^2)$	Ω_4/Ω_6
ZLSLCBS(ND01)	1.718	7.945	2.848	3.204
ZLSLCBS(ND1.5)	2.401	6.617	2.607	2.538
ZLSLCBS(ND02)	3.092	5.339	2.367	2.256

Fluorescence Spectrum

The fluorescence spectrum of Nd^{3+}doped in zinc lithium sodalimecadmium borosilicateglass is shown in Figure 5. There aresix broad bands ($^4G_{7/2}\rightarrow{}^4I_{9/2}$)($^4G_{7/2}\rightarrow{}^4I_{11/2}$),($^4F_{3/2}\rightarrow{}^4I_{9/2}$),($^4F_{3/2}\rightarrow{}^4I_{11/2}$), ($^4F_{3/2}\rightarrow{}^4I_{13/2}$)and ($^4F_{3/2}\rightarrow{}^4I_{15/2}$)respectively for glass specimens. The wavelengths of these bands along with their assignments are given in **Table 5**.

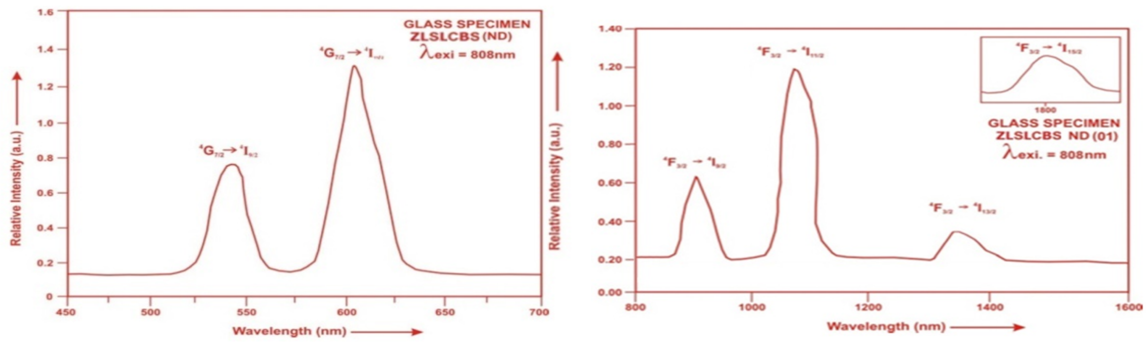


Fig.5: Fluorescence spectra of ZLSLCBSND (01) glass.

Conclusion:

In the present study, the glass samples of composition (35−x)SiO_2: 10ZnO: 10Li_2O: 10CaO: 10Na_2O:10CdO:15B_2O_3:xNd_2O_3 (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 ($^4F_{3/2}\rightarrow{}^4I_{11/2}$) forglass ZLSLCBS(ND 01), suggesting that glass ZLSLCBS (ND 01) is better compared to the other two glass systemsZLSLCBS(ND1.5) and ZLSLCBS(ND02).The large stimulated emission cross sectionin silicate glasses suggests the possibility of utilizing these systems as laser materials.

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International Journal of Chemical and Physical Sciences
(Peer-Reviewed Journal)
ISSN:2319-6602

Table 5: 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 ZLCBS glasses.

Transition		ZLCBS (ND 01)				ZLCBS (ND1.5)				ZLCBS (ND 02)			
	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	$\sigma_p(10^{-20}$ $cm^2)$	$\tau_R(10^{-20}$ $cm^2)$
${}^4G_{7/2}\rightarrow{}^4I_{9/2}$	532	3877.10	0.4422	0.494		3384.80	0.4207	0.458		2956.54	0.3965	0.414	
${}^4G_{7/2}\rightarrow{}^4I_{11/2}$	595	3263.30	0.3799	1.144		3309.47	0.4114	1.231		3370.14	0.4519	1.327	
${}^4F_{3/2}\rightarrow{}^4I_{9/2}$	905	983.69	0.1133	0.866	115.22	827.30	0.1028	0.746	124.30	676.07	0.0907	0.631	134.10
${}^4F_{3/2}\rightarrow{}^4I_{11/2}$	1075	517.28	0.0596	2.174		452.94	0.0563	2.035		389.94	0.0523	1.884	
${}^4F_{3/2}\rightarrow{}^4I_{13/2}$	1320	75.39	0.00869	0.378		69.13	0.0085	0.366		62.89	0.0084	0.345	
${}^4F_{3/2}\rightarrow{}^4I_{15/2}$	1800	1.80	0.0002	0.0224		1.65	0.0002	0.0209		1.50	0.0002	0.0194	