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Research Article

INVESTIGATIONS OF LUMINESCENCE PROPERTIES OF NEODYMIUM IN LITHIUM BORATE GLASSES FOR NEAR INFRARED LASER APPLICATIONS

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ABSTRACT

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Key Words:

Lithium borate glass; Neodymium; Judd-Ofelt theory; Near infrared; Raman spectrum. Nd³⁺ doped lithium borate (LB) glasses with compositions (in mol %) (60-x) B₂O₃+ 20LiF + 10MgF₂ + 10ZnO + xNd₂O₃ (where x = 0.1, 0.5, 1.0, 1.5 and 2 mol%) were prepared by melt quenching technique and have been investigated using structural measurements, optical absorption, luminescence, and decay kinetic techniques as well as Judd–Ofelt (J–O) analysis. The structure of host glass system can be accessed from XRD and Raman graphs. For these glasses, optical absorption, emission and decay measurements were carried out. From the optical absorption spectra, the spectral intensities (f_{cal}) of both calculated and experimental (f_{exp}) and Judd-Ofelt intensity parameters, (= 2, 4, 6) have been evaluated. From the photoluminescence spectra and using Judd-Ofelt intensity parameters, radiative transition probabilities (A_R), experimental branching ratios ($_{exp}$) and stimulated emission cross-sections ($_{e}$) are calculated. The luminescence behavior of doping effect of the Nd³⁺ into the lithium borate glasses was discussed. To investigate luminescence efficiency of $^{4}I_{9/2}$ level of Nd³⁺ doped different lithium borate glasses, lifetimes ($_{exp}$) are predicted from decay curve analysis for all the prepared Nd³⁺ doped lithium borate glass matrices. Finally, these observed results were discussed and compared with the literature data and suggested for the near infrared laser applications.

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INTRODUCTION

Nowadays, luminescent lanthanide doped materials have attracted much interest since they possesses unique optical properties for their application as optical materials. Owing to the intrinsic spectroscopic properties of the rare earth ions are widely used in the development of new luminescent materials for exhibiting monochromatic emission in several applications such as optical markers, optoelectronic devices, biological labels and sensor devices. The combination of spectroscopic properties of these ions associated to the unique physical and chemical properties of glasses became more attractive in the last decade [1, 2].

The optical properties can be decided mainly by its primary properties such as refractive index, dispersion and transmission. Again these properties depend on secondary properties such as chemical composition, chemical durability, melting process and subsequent thermal treatment. By following these procedures, glass structure can be changed and their applications in different fields. Today, telecommunication fields utilize large quantities which exhibit emission of optical fibers through the development of optical glasses. However new type of glasses especially, below vacuum ultraviolet and infrared regions above 4000 nm requires a glass chemistry in systems like phosphate, fluoro-phosphate, silicate, tellurite, borate and chalcoagenide glasses[3,4].

Among different families of glasses, borate glasses have a few properties such as high thermal stability, low dispersion and refractive index. By addition of network modifiers like alkali oxides, alkaline oxides and zinc oxide glass network become more compact and depolymorization of non-bridging oxygen's takes place. In order to decrease the phonon energy of borate host glass matrix fluoride content is also added instead of oxides.

In rare earth ions family, Nd^{3+} ions containing hosts have been extensively studied mainly due its large cross-sections of optical absorption near 800 nm (${}^{4}I_{9/2} \, {}^{2}H_{9/2} + {}^{4}F_{5/2}$), which turn easiest the excitation by diode laser, and the high quantum efficiency of the ${}^{4}F_{3/2} \, {}^{4}I_{11/2}$ emission near 1060 nm. Moreover, as the ${}^{4}I_{11/2}$ energy level is 2000 cm⁻¹ above the ground state, this turn the system as four level one, which it is well known to favour the population inversion avoiding the reabsorption effect [5]. Lasing has been obtained in different bulk glasses doped with Nd³⁺ ions.

In this work, we report the preparation and characterization of Nd3+ doped lithium borate glass matrix with different concentrations. The method of preparation is melt quenching

technique. The characterization techniques include XRD, FTIR, optical absorption, emission and decay profiles.

Experimental

Lithium borate (LB) glass compositions with 0.1, 0.5, 1.0, 1.5 and 2.0 mol% of Nd³⁺ ions were prepared by melt quenching technique, and the precursor powders boric acid (B₂O₃), lithium fluoride (LiF), magnesium fluoride (MgF₂), zinc oxide (ZnO), dysprosium oxide (Nd₂O₃) (purity 99.9%) obtained from Himedia were used. The details of prepared glass composition as follows

LBNd01: $59.9B_2O_3 + 20LiF + 10 MgF_2 + 10ZnO + 0.1Nd_2O_3$ LBNd05: $59.5B_2O_3 + 20LiF + 10 MgF_2 + 10ZnO + 0.5Nd_2O_3$ LBNd10: $59.0B_2O_3 + 20LiF + 10 MgF_2 + 10ZnO + 1.0Nd_2O_3$ LBNd15: 58.5B₂O₃+ 20LiF + 10 MgF₂ + 10ZnO + 1.5Nd₂O₃ LBNd20: 58.0B₂O₃+ 20LiF + 10 MgF₂ + 10ZnO + 2.0Nd₂O₃

The powders were weighed in stoichiometric amount and grinded in agate mortar. The grinded powders were taken in porcelain crucible and placed in electric furnace at 1100 °C for heating about 1 hour. After that, obtained liquid is poured on a preheated brass plate. The synthesized glass samples were subsequently annealed at 250 °C to remove thermal strains to do spectral measurements. Refractive index (n) measurements were performed using an Abbe refractometer at sodium wavelength (583.3 nm) with one-bromonapthalene as contact liquid. The density (d) was measured by Archimedes's principle using water as an immersion liquid.

The prepared glasses were characterized for structural properties by using an X-ray diffractometer (XRD, SEIFERT). FTIR spectrum was taken using a series of Perkin Elmer spectrum One FT-IR spectrophotometer. Raman spectrum was obtained in backscattering geometry (Lab Ram HR800) with 514 nm line of Ar⁺ laser as an excitation source. Optical properties of the lithium borate glasses were analyzed by measuring absorption, excitation, and emission spectra. Absorption spectra were measured by using a UV-VIS-NIR spectrophotometer (ELICO SL 218). Emission spectra and decay profiles were measured by using FLS 980 Edinburg excited with 808 nm laser diode. All the spectral measurements were carried out at room temperature.

RESULTS AND DISCUSSIONS

X-ray diffractogram (XRD)

The XRD diffractogram of the lithium borate host glass system is shown in Fig. 1. Obviously, the amorphous character of this host glass matrix is evident from XRD pattern. Only a broad peak is observed in the region around $20^{\circ} < 2 < 30^{\circ}$ for a glass sample free from neodymium and indicating that these lithium borate glass samples are composed of glassy phase.

FTIR spectrum

The FTIR spectrum of the lithium borate host glass system is shown in Fig. 2. The band nearly at 700 cm⁻¹ related to bending vibration of B-O-B in [BO₃] triangles. The infrared spectral band nearly at 974 cm⁻¹ is typical for the stretching vibration of [BO₄] units. The existence of [BO₄] units indicates that the addition of modifier oxides into the glass causes a conversion of $[BO_3]$ units to $[BO_4]$ units. The band nearly at 1350 cm⁻¹ is

assigned to the stretching vibration of B-O-B in [BO₃] triangles. The band nearly at 2350 cm-1 is due to hydrogen bonding. As shown in Fig. 2, the signal at around 1820 cm⁻¹ is due to the deformation modes of O-H groups and absorbed water molecules (H–O–H). The band nearly at 2100 and 2350 cm⁻¹ are ascribed to the stretching vibrations of hydroxyl groups [6,7].



Fig.1 XRD spectrum of lithium borate host glass matrix

Raman spectrum

Structural assessment can be studied by Raman spectral measurement. The Raman spectrum of host glass matrix is recorded in the region 300-2000 cm⁻¹ and is shown in Fig.2.



Fig. 2 FTIR spectrum of lithium borate host glass matrix

In order to know the exact structural units, Raman spectrum is deconvoluted into peaks by Gaussian fitting. Based on width, position and no of bands, it is fitted well. In Fig.3, black line is original spectrum, red line is after fitting process to Gaussian line and greens lines are deconvoluted bands. It is observed that the red line is coincided well with original line and indicating that the fitting process is reliable and less deviation. Raman band at 492 cm⁻¹ is ascribed to penta, tetra and diborate rings. The band at 755 cm⁻¹ arises due to the symmetric stretching vibrations of di-triborates, triborates, tetraborates, and pentaborates, and of the various six-membered ditriborate rings. The bands at about 1230 cm^{-1} indicate that presence of symmetric stretching vibration of B-O-B bridges and terminal B-O pyroborate units are present in the structure of borate glasses. If an oxide-modifier content is less than 50 mol%, the Raman shift at 1468 cm⁻¹ originates also from the contribution

of asymmetric stretching triangles of various borate groups [8,9].



Fig. 3 Raman spectrum of lithium borate host glass matrix

Optical absorption analysis

Absorption spectrum of 0.5 mol% of Nd³⁺ doped lithium borate glass sample in the region of wavelength 350-920 nm is displayed in Fig. 4. The observed thirteen absorption bands, which originated from the ground state ${}^{4}I_{9/2}$ to higher states assigned to different transitions of Nd³⁺ ions by comparing the peak positions with reported literature [10]. The position of these absorption transitions of Nd05 lithium borate glass matrix located at 350, 430, 471, 526, 583, 621, 681, 746, 801 and 873 originates from the ${}^{4}I_{9/2}$ ground state to the various excited states of Nd³⁺ such as ${}^{2}I_{11/2} + {}^{4}D_{3/2} + {}^{4}D_{5/2}$, ${}^{2}P_{1/2}$, ${}^{2}D_{3/2} + {}^{2}G_{9/2} + {}^{2}K_{13/2}$, ${}^{4}G_{7/2}$, ${}^{4}G_{5/2} + {}^{2}G_{7/2}$, ${}^{2}H_{11/2}$, ${}^{4}F_{9/2}$, ${}^{4}S_{3/2} + {}^{4}F_{7/2}$, ${}^{4}F_{5/2} + {}^{2}H_{9/2}$ and ${}^{4}F_{3/2}$ respectively. The absorption band of Nd³⁺ located at ~580 nm corresponds to the hypersensitive transition which is dominated in intensity than other transitions of Nd³⁺ ions. It is noted that, in the UV-VIS-NIR region, the absorption bands of different transition levels overlap each other due to the presence of closed energy levels in Nd³⁺ ions and this difficulties arises can be resolved by addition of both the dipole strengths and the squared reduced matrix elements of overlapping transitions. The corresponding reduced matrix elements have been also added.



Fig. 4 UV-VIS-NIR absorption spectrum of 0.5 mol% of Nd³⁺ doped lithium borate glass matrix

The Judd–Ofelt (J-O) theory [11, 12] is used to characterize radiative transitions for Nd^{3+} doped glasses. It gives three set of intensity parameters, (=2, 4 and 6), that are sensitive to the around the environment of the neodymium ions. The

experimental oscillator strengths, $f_{\text{exp}},$ of the absorption bands can be calculated by

 $f_{exp} = 4.32 \times 10^{-9} \int \varepsilon(v) dv$

where $\varepsilon(v)$ is the molar extinction coefficient. According to the Judd-Ofelt theory (J- O), the spectral intensity of an electric dipole absorption transition can be defined from the initial state, aJ to the final state, bJ['].

$$f_{cal}(aJ, bJ') = \frac{8f^2 mc^{\hat{}}}{3hc^2(2J+1)} \left[\frac{(n^2+2)^2}{9n} \right] \sum_{\beta=2,4,6} \Omega_{\beta} \left| < aJ \right| U^{\beta} \left\| bJ' > \right|^2$$

In the above equation, all symbols are as usual meaning. The Judd-Ofelt intensity parameters Ω_{λ} are obtained from the experimental absorption band. The root mean square (RMS) deviation value of 0.66×10^6 is obtained between the experimental and calculated oscillator strengths, indicates that good fit between two magnitudes and also good consistency of J-O intensity parameters. These results are shown in Table 1. It is observed that order of Ω_{λ} parameters is $\Omega_2 > \Omega_6 > \Omega_4$ for the Nd05 glass matrix. High Ω_2 parameter and low $\Omega_{4,6}$ parameters related to covalency and rigidity respectively is also observed.

Table 1 Experimental (f_{exp}) and calculated (f_{cal}) spectralintensities $(x10^{-6})$ of different absorption bands and Judd-Ofelt intensity parameters (, $x10^{-20} \text{ cm}^2)$ for LBNd05lithium borate glass matrix

S.NO	Transition	f _{exp}	f _{cal}	
1	${}^{2}I_{11/2} + {}^{4}D_{3/2} + {}^{4}D_{5/2}$	0.81	0.82	
2	${}^{2}\mathbf{P}_{1/2}$	0.62	0.62	
3	${}^{2}K_{13/2} + {}^{2}G_{9/2} + {}^{2}D_{3/2}$	1.95	1.99	
4	${}^{4}G_{7/2}$	3.96	4.10	
5	${}^{4}G_{5/2} + {}^{2}G_{7/2}$	21.10	22.11	
6	${}^{2}H_{11/2}$	0.11	0.12	
7	${}^{4}F_{9/2}$	2.10	2.15	
8	${}^{4}S_{3/2} + {}^{4}F_{7/2}$	6.90	6.99	
9	${}^{4}F_{5/2} + {}^{2}H_{9/2}$	6.35	6.98	
10	${}^{4}F_{3/2}$	1.34	1.10	
F	RMS deviation		±0.66	
	2	6.01		
	4	4.01		
	6	5.14		

J-O intensity parameters are further used to evaluate the different radiative properties like radiative transition probability (A_R), radiative lifetime ($_{R}$) and branching ratios ($_{R}$), of certain excited states are calculated for LBNd05 lithium borate glass matrix and are collected in Table 2 for ${}^{4}F_{3/2}$ state of Nd³⁺.

Table 2 Radiative transition probabilities (A_R) (s⁻¹), totaltransition probabilities (A_T) , branching ratios $(_R)$, andradiative lifetimes $(_R)$ (μ s) of certain excited levels forLBNd05 lithium borate glass matrix

SLJ	S'L'J'	AR	R	
	${}^{4}F_{15/2}$	15	0	A _ 2402
${}^{4}F_{3/2}$	${}^{4}I_{13/2}$	324	9	$A_{\rm T} = 3403$ $_{\rm R} = 293$
Г _{3/2}	${}^{4}I_{11/2}$	1703	51	$_{\rm R} = 293$
	${}^{4}I_{9/2}$	1361	40	

The radiative lifetimes ($_{\rm R})$ and branching ratios ($_{\rm R})$ can be calculated using the equations

$$\ddagger_{rad} = \frac{1}{\sum_{bJ} A_{rad} (aJ, bJ')^{-1}}$$

$$S_{R}(aJ, bJ') = \frac{A_{rad}(aJ, bJ')}{\sum_{bJ'} A_{rad}(aJ, bJ')}$$

It is noticed (from Table 3) that the ${}^{4}F_{3/2} {}^{4}I_{11/2}$ transition has more A_R magnitude and branching ratios ($_{cal}$).

Table 3 Emission band positions $\{\}_p$, nm), effective bandwidths ($_{eff}$, nm), radiative transition probabilities (*A*, s⁻¹), peak stimulated emission cross-sections ($_p$, x10⁻²⁰cm²) and branching ratios (S_{exp}, %) of emission transitions for LBNd05 lithium borate glass matrix

Parameters	${}^{4}\mathrm{F}_{3/2}$ ${}^{4}\mathrm{I}_{9/2}$	${}^{4}\mathbf{F}_{3/2}$ ${}^{4}\mathbf{I}_{11/2}$	${}^{4}\mathbf{F}_{3/2}$ ${}^{4}\mathbf{I}_{13/2}$
$_p$	894	1059	1333
eff	45	29	55
A_R	1361	1703	324
р	1.05	2.36	0.71
exp	36	59	5

Near infrared emission properties

Fig. 5, shows the luminescence emission spectra of Nd³⁺ doped lithium borate glass matrix for LBNd01, LBNd03, LBNd05, LBNd10, LBNd15 and LBNd20 glasses in the range of wavelength 800–1300 nm under the excitation of 808 nm laser diode. In the present study, three emission peaks are observed at 894, 1059 and 1333 nm corresponding to ${}^{4}F_{3/2}$ ${}^{4}I_{9/2}$, ${}^{4}I_{11/2}$ and ${}^{4}I_{13/2}$ transitions, respectively. From the emission spectra, it is clearly observed that emission intensities increases upto 0.5 mol% of neodymium and then decreases with the increase of Nd³⁺ concentration. This is due to concentration quenching. It is occur due to the increasing of non-radiative (NR) energy transfer through cross relaxation and resonant energy channels among Nd³⁺ ions [13].



Fig 5 Emission spectra of Nd³⁺ doped lithium borate glass matrix with different concentrations

From Fig.5, the most intense emission is observed for the ${}^{4}F_{3/2}$ ${}^{4}I_{11/2}$ near infrared transition than other emission transitions. From the emission spectra, the intensity of probable lasing transition can be assessed, like the effective line widths ($_{eff}$), branching ratio ($_{exp}$), from area under the emission band, and peak stimulated emission cross-sections ($_{P}$) for the certain transitions, ${}^{4}F_{3/2}$ ${}^{4}I_{J}$ (J =9/2, 11/2 and 13/2).

The peak stimulated emission cross-section (σ_P)

$$\dagger_{p}(aJ, bJ') = \frac{\rbrace_{p}^{4}}{8fcn^{2}\Delta\rbrace_{eff}} A_{rad}(aJ, bJ')$$

where λ_P is emission peak wavelength and Δ_{eff} , is the effective linewidth.

The higher $_{\rm P}$ is very important property for laser application. From the three emission transitions of Nd³⁺ ion, $^4F_{3/2}$ $^4I_{11/2}$ transition has higher $_{\rm P}$ than rest of transitions and among all concentrations of Nd³⁺, LBNd05 lithium borate glass matrix shows higher $_{\rm P}$ i.e. 2.36×10^{-20} cm². The value of $_{\rm P}$ for the $^4F_{3/2}$ $^4I_{11/2}$ near infrared transition of the LBNd05 glass matrix located at 1.06 μm is found to be high. Hence, the glass LBNd05 glass matrix might be useful for near infrared lasing material through $^4F_{3/2}$ $^4I_{11/2}$ transition. The is one of the important and attractive parameter to

The is one of the important and attractive parameter to determine lasing power of a particular transition and it is well established that an emission transition having the values of is greater than 50% is can be considered as more as potential for laser emission. The experimental branching ratios ($_{exp}$) are measured under the relative areas of the each individual emission transitions and it is found to be higher for ${}^{4}F_{3/2}$ ${}^{4}I_{11/2}$ transition. In the present study, it is observed that there is a good agreement between $_{exp}$ and $_{R}$.

Decay kinetics

With 808 nm excitation, into the ${}^{4}I_{9/2}$ ${}^{4}F_{5/2}+{}^{2}H_{9/2}$ absorption (excitation) band and then the ${}^{4}I_{13/2}$ ${}^{4}F_{9/2}$ transition is more favourable way to depopulate nonradiatively through the multiphonon relaxation (MPR) due to the fact that the energy gap between them is low. No emissions from other energy levels are expected. The MPR from the ${}^{4}F_{9/2}$ level is negligible owing to fact that the next level (${}^{6}F_{1/2}$) lies lower by about ~6900 cm⁻¹. This wide energy gap between the ${}^{4}F_{9/2}$ ${}^{6}F_{1/2}$ levels leads to a high quantum efficiency of the ${}^{4}F_{9/2}$ emitting level [6] and emission takes place from this level as shown in Fig. 6 energy level diagram.



Fig.6 Energy level scheme of Nd³⁺ doped lithium borate glass matrix

The fluorescence decay profiles for ${}^{4}F_{3/2}$ level of Nd³⁺ doped lithium borate glasses with the variation of Nd³⁺ content while maintaining with a fixed emission at 1.06 µm, which corresponds to the electronic transition (${}^{4}F_{3/2}$ ${}^{4}I_{11/2}$) and excitation at 808 nm is displayed in Fig. 7. Fluorescence lifetimes were measured form e-folding time of the emission intensities. For the decay curves which are bi-exponential behavior, the resultant decay lifetime can be calculated using the formula [14]

$$I(t) = A_1 \exp(\frac{-t}{t_1}) + A_2 \exp(\frac{-t}{t_2})$$

where 'I' is the photoluminescence intensity at any time 't' after switching off the excitation illumination, $_1$ and $_2$ are fast and slow decay times respectively, A_1 , A_2 are respective fitting (weighing) parameters. The first term in the equation refers to excited state absorption and second term refers to the energy transfer (ET) process or in other words radiative and non-radiative decays.



Fig.7 Decay profiles of Nd³⁺ doped lithium borate glass matrix with different concentrations

The experimental lifetimes ($_{\rm exp})$ have been evaluated using the relation

$$\ddagger_{exp} = \frac{A_1 \ddagger_1^2 + A_2 \ddagger_2^2}{A_1 \ddagger_1 + A_2 \ddagger_2}$$

It is observed that the decay lifetimes decreases with increasing neodymium content in the lithium borate glass system. The lifetimes were found to be 912, 897, 801, 746, 695 and 585 μs for the LBNd01, LBNd05, LBNd10, LBNd15 and LBNd20 lithium borate glasses respectively. The decay lifetime decreases from 912 µs to 585 µs. This gradual decrease in lifetime is consistent with the steady state luminescence behaviour which is due to the increase in non-radiative transition processes to excited states of Nd³⁺ ions. However, the lifetime value drops to 580 µs when Nd³⁺ content increases to 2.0 mol% which might be due to the larger amount of activator ions in the glass system. Except LBNd01and LBNd05 glasses, all lithium borate glasses of decay profiles are fitted nonexponential function at short times. This type of activity is due to energy transfer (ET) between two Nd³⁺ atoms and concentration quenching. In order to investigate the process involved in the ET mechanism, the non-exponential decay curves for the ${}^{4}F_{9/2}$ level of Nd³⁺ ions have been analyzed using Inokuti-Hirayama (I-H) model [15].

$I(t) = I_0 exp\{-t/_0 - Q(t/_0)^{3/8}\}$

where t is the time, $_{\rm O}$ is the intrinsic decay time of donors in the absence of acceptors, energy transfer parameter (Q). The Q value linked to variable interaction parameter, S and the gamma function (x) parameters. is 1.77 (for S=6, dipole–dipole), 1.43 (for S=8, dipole–quadrupole) and 1.33 (for S=10, quadrupole–quadrupole). In co-doped system, decays were fitted to S=6. From this model it is assessed that ET occurs through dipole–dipole (d-d) interaction.

CONCLUSIONS

In the present study, neodymium doped six series of lithium borate glass composition with different concentrations (0.1, 0.5, 1.0, 1.5 and 2.0 mol%) were prepared by melt quenching method, and their luminescence has been investigated. Structural properties were accomplished from XRD (X-Ray Diffractometer), FTIR and Raman spectrum. Spectroscopic properties were investigated by measuring optical absorption spectrum, excitation spectrum, emission spectra and decay profiles. Neodymium environment in lithium borate host glass matrix can be accessed by Judd-Ofelt (J-O) theoretical approach. It is observed that order of Ω_{λ} parameters is $\Omega_2 > \Omega_6 > \Omega_4$ for the lithium borate glasses. High Ω_2 parameter and low Ω_4 parameter related to covalency and rigidity respectively is observed. J-O intensity parameters are used to further to evaluate the different radiative properties like radiative transition probability (A_R) , radiative lifetime (R) and branching ratios () of certain excited states. It is noticed that the ${}^{4}F_{3/2}$ ${}^{4}I_{11/2}$ transition has more A_R magnitude and branching ratios (cal). Luminescence parameters such as effective bandwidth (eff), stimulated emission cross-sections (_p) and branching ratios (_{exp}) have been studied through photoluminescence spectra. By adjusting the doping concentration in glass system, the quenching was observed in emission intensity. The photoluminescence spectra exhibit three prominent transitions. Of which, the near infrared transition located at 1.06 µm has high emission intensity. Furthermore, it is also noticed that the present glass (LBNd05) sample shows higher branching ratios, transition probability, and emission cross-sections for ${}^4F_{3/2} \quad {}^4I_{11/2}$ transition. Further, decay time constants have been estimated from the decay profiles of Nd³⁺ doped different lithium borate glasses. PL decay lifetimes are determined by fitting the decay data with the mono exponential decay equation. The decay lifetimes decreases with increasing of Nd³⁺ concentration. This gradual decrease in lifetime is consistent with the steady state luminescence behavior which is due to the increase in nonradiative transition processes to excited states of Nd³⁺ ions. This approach shows the present prepared lithium borate glasses useful towards the development of near infrared lasing materials.

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