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Photoluminescence studies of Eu^{3+} ions doped calcium zinc niobium borotellurite glasses

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ABSTRACT

A facile melt quenching technique was employed to prepare Eu^{3+}-ions doped TRZNB glasses using commercial powders through mixing the specific weights of batches. The compositions prepared were $10TeO_2 + 15RO + 5ZnO + 10Nb_2O_5 + 59B_2O_3 + Eu_2O_3$ (where R= Mg, Ca and Sr). Under 395 nm excitation wavelength, photoluminescence (PL) and lifetime measurements of Eu^{3+}-doped TRZNB glasses were recorded and reported. The PL spectra composed of five emission bands that are originating from the $^5D_0$ metastable state to ^7F_J (J = 0 - 4) lower lying states. Using the emission intensities of $^5D_0\rightarrow{}^7F_2$ and $^5D_0\rightarrow{}^7F_4$ transitions, respectively, the Judd–Ofelt (J–O) intensity parameters such as Ω_2 and Ω_4 were calculated by considering the magnetic dipole (MD) $^5D_0\rightarrow{}^7F_1$ transition as reference. The radiative parameters such as spontaneous emission probabilities (A_R), lifetimes (τ_m), branching ratios (β_m) for different excited states were estimated theoretically. For all the glasses, the decay profiles were fitted to the single exponential equation. The obtained intense red emission at about 616 nm assigned to the $^5D_0\rightarrow{}^7F_2$ transition suggested the potentiality of present Eu^{3+} -doped TRZNB glasses as a laser host.

Keywords: Tellurite glass; europium ion; J-O theory; photoluminescence; fiber amplifier.

Introduction

Over the past, rare-earth (RE) ions doped glasses and crystals have considered great attention due to its large practical and potential applications in many fields, such as lasers, memory devices, phosphors, electro-luminescent devices, optical fiber amplifiers and flat-panel displays, etc. In recent years, RE ions doped solid-state materials have intense interest in developing compact lasers, white lighting devices and broadband amplifiers **[1]**. In the recent past few decades, the spectroscopic properties of various RE ions doped oxide glasses have been studied widely because of its potential technological and commercial applications. In general, the analysis of spectroscopic properties of glasses give the essential data like the energy level positions, cross-sections, radiative and non-radiative relaxation rates, transition probabilities, and branching ratios of various excited states for the development of lasers, up-converters, color displays, and fiber amplifiers **[2]**. The researchers have devoted to develop borate glass system due to the anomalous behavior of borates. Unlike other glass systems, the borates could form tremendous structural units by the fundamental units of BO_3 and BO_4 groups. The increase in BO_3 and BO_4 units promotes the compactness of glass structure, therefore; density of glasses increases with alkaline earth ions. Since the density changes and the remaining physical properties such as concentration, polaron radius, inter ionic distance, field strength, molar volume and refractive index may also affect and change. The composition, structure and nature of the glasses play a vital role to tune the Physical properties of glasses that in turn will be used for various applications **[3-7]**.

Among the other host glasses, the optical properties of RE doped borates have been studied extensively due to their potential use in the lasers, optical communications and fuel cells. The borates possess advantageous properties such as super hardness, high insulation, linear and nonlinear optical behavior **[8]**. In comparison to other glass materials, the high mechanical strength of oxide glasses and low phonon energy of fluoride glasses are combined to form a new class of oxyfluoride glasses that have several advantages properties for wide variety of applications. It is known that tellurite glasses have favored for optical applications against oxide glasses because of their low melting temperature, high refractive index, high chemical and thermal durability, corrosion resistance, high infrared transmittance, good mechanical strength and low phonon energies **[9]**. Among the other RE ions, the optical properties of europium ions doped glasses have attracted potential application in memory devices and phosphors **[10]**. The trivalent europium ions exhibits pure electric-dipole (ED) and magnetic-dipole (MD) transitions and the variations of these two transitions ((excited) $^5D_0$ and the $^7F_0$ (ground) state) influences the symmetry and inhomogeneity present in the glasses **[11]**.

In the present investigation, the spectroscopic studies of Eu^{3+}-doped TRZNB glasses were reported. Under 395 nm excitation of Eu^{3+} ions, the photoluminescence and lifetimes of glasses were recorded and studied in detail.

Table 1. Measured and calculated physical properties for 1.0 % Eu^{3+} -doped TRZNB glasses.

Physical quantities	TMZNB	TCZNB	TSZNB
Sample thickness (cm)	0.320	0.280	0.300
Refractive index (n)	1.53	1.49	1.55
Density (g/cc)	3.84	3.85	3.88
Concentration (mol/litre)	0.489	0.486	0.482
Concentration (ions cm^{-3} $\times 10^{20}$)	2.948	2.813	2.906
Average molecular weight (g)	90.03	92.40	99.53
Dielectric constant (ε)	2.34	2.31	2.40
Molar volume V_m (cm^3/mol)	23.45	24.32	25.65
Glass molar refractivity (cm^{-3})	7.239	7.390	8.172
Electronic polarizability α_e ($\times 10^{-24}$ cm^3)	2.871	2.930	3.241
Reflection losses R (%)	4.39	4.26	5.05
Polaron radius r_p (Å)	9.61	9.76	9.66
Inter ionic distance r_i (Å)	15.02	15.26	15.09
Field strength F ($\times 10^{14}$ cm^{-2})	3.25	3.15	3.22

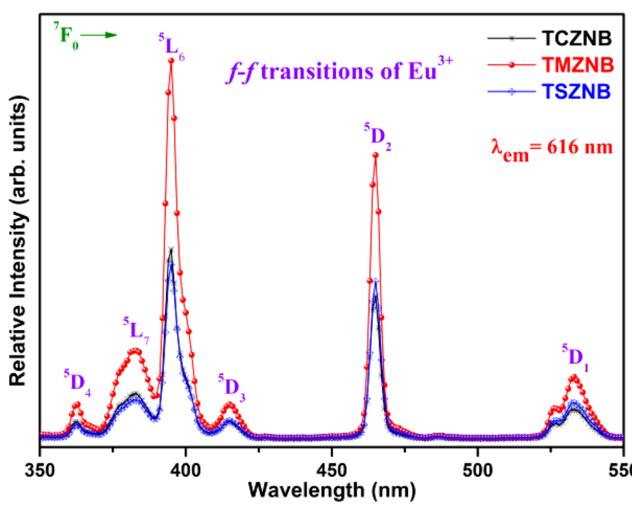

Fig. 1. Excitation spectra of 1 mol % Eu^{3+} -doped TRZNB glasses.

Experimental

To prepare trivalent europium ions doped TRZNB glasses, the conventional melt quenching technique was employed. For this, the specific weights of batches of the commercially purchased powders (analytical reagent grade) were used without further purification. The compositions used for the preparation of glasses were $10TeO_2 + 15RO + 5ZnO + 10Nb_2O_5 + 59B_2O_3 + Eu_2O_3$ (where R= Mg, Ca and Sr). First, the appropriate amounts of homogenously mixed samples were taken separately in a platinum crucible and melted in an electric furnace at 1450 °C for 90 min. Subsequentially, the melted mixtures were quenched to a steel mould in air. Thereafter, to reduce thermal strains, the glasses were calcined at 380 °C for 12 h and cooled to room temperature (RT) gradually for further processing. Prior to measure the physical and optical properties of glasses, the annealed glasses were well shaped and polished. The refractive indices of the polished glasses were estimated and are found to be 1.53, 1.49 and 1.55 for TMZNB, TCZNB and TSZNB glasses, respectively. **Table 1** shows the various measured and calculated physical properties of the Eu^{3+}-doped TRZNB glass (Eu^{3+} = 1.0 mol %). The photoluminescence excitation and emission spectra were measured on a fluorescence spectrophotometer (Jobin Vyon Fluorolog -3 spectrofluorometer) with xenon flash lamp as a source. Using the same instrument, the decay curves of the $^5D_0$ excited level for TRZNB glasses of Eu^{3+} ions were also recorded with 395 nm excitation wavelength.

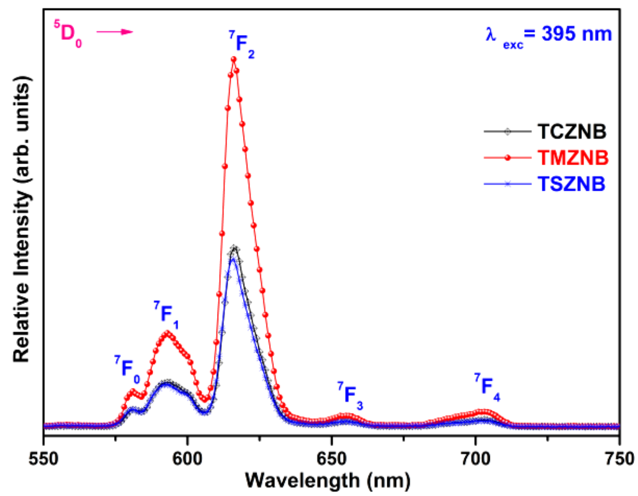

Fig. 2. Emission spectra of 1 mol % Eu^{3+} -doped TRZNB glasses.

Results and discussion

In the spectral region of 350 to 550 nm, the excitation spectra of TRZNB glasses were collected at 616 nm emission wavelength. The excitation spectra of all the glasses are shown in **Fig. 1**. The spectra consist of eight excitation bands around 362, 382, 395, 415, 465, 526 and 533 nm corresponding to the $^7F_0 \rightarrow ^5D_4$, $^7F_0 \rightarrow ^5G_2$, $^7F_1 \rightarrow ^5L_7$, $^7F_0 \rightarrow ^5L_6$, $^7F_1 \rightarrow ^5D_3$, $^7F_0 \rightarrow ^5D_2$, $^7F_0 \rightarrow ^5D_1$ and $^7F_1 \rightarrow ^5D_1$ transitions, respectively. Among them, the excitation band at 395 nm, ascribed to the $^7F_0 \rightarrow ^5L_6$ transition is more predominant, and hence; the PL spectra for all other glasses have been recorded using 395 nm excitation wavelengths **[12]**. The PL spectra of TRZNB glasses are shown in **Fig. 2**. In the range of 550 to 750 nm, the emission spectra composed of five distinct bands at about 581, 593, 616, 655, and 703 nm, corresponding to the transitions from the $^5D_0$ metastable state to the various lower lying $^7F_0$, $^7F_1$, $^7F_2$, $^7F_3$ and $^7F_4$ states, respectively. The emission spectra are appeared to similar, when the glasses are excited by other than the $^5D_0$ level. This is because of the results of quick non-radiative relaxation to this excited fluorescent level, which are having the small energy gaps between them **[13]**. In the emission of Eu^{3+} ions, the $^5D_0 \rightarrow ^7F_1$ transition belongs to MD transition and it is independent of the crystal field strength around RE ion. Apparently, this transition is used for the estimation of transition probabilities of various excited levels. Between the $^5D_0\rightarrow{}^7F_2$ and $^5D_0\rightarrow{}^7F_4$ transitions, the $^5D_0\rightarrow{}^7F_3$ transitions also become apparent. The transitions $^5D_0\rightarrow{}^7F_J$ with J = 5 and 6 are not observed as transition probabilities because of these transitions are very weak in nature. The $^5D_0\rightarrow{}^7F_J$ (J = 2, 4 and 6) transitions are ED transitions. From the figure, the narrow emission bands were noticed. This is due to the shielding effect of $^4f_6$ electrons by 5*s* and 5*p* electrons in outer shells in the Eu^{3+} ion. Among the various bands, the band at 616 nm corresponding to the transition $^5D_0\rightarrow{}^7F_2$ exhibit a strong red emission and is the hypersensitive to the host structure of the glasses with the selection rules ΔJ = 2. On the other hand, the band at 593 nm that is assigned to the transition $^5D_0\rightarrow{}^7F_1$ with ΔJ = 1 could be belong to a MD transition. This transition is independent of the crystal field strength around the Eu^{3+} ion. The nature of this transition could be used for the estimation of transition probabilities of various excited levels. Using the emission data, the Judd–Ofelt **[14, 15]** parameters (Ω_λ, λ = 2, 4 and 6) were determined **[16, 17]**. For calculating, the JO parameters, the least-square fitting method were used for all the emission bands. **Table 2** presents the estimated JO intensity (Ω_λ, λ = 2, 4, 6) parameters of Eu^{3+}-doped TRZNB glass. The larger value of Ω_2 for all three glass systems indicates stronger covalency of Eu–O bonds **[12, 13, 18, 19]**.

Table 2. Comparison of J-O intensity parameters ($\times10^{-20}$ cm^2) for Eu^{3+} ions in TRZNB glasses with different glass hosts.

Host matrix	J-O Parameters		
	Ω_2	Ω_4	Ω_6
Present work			
TMZNB	4.48	0.27	-
TCZNB	4.57	0.34	-
TSZNB	4.49	0.45	-
Reported			
LLiFB [13]	3.62	1.19	-
1EBT [18]	5.30	0.31	-
LTTEu [19]	3.29	0.63	-
PTBEu20 [12]	4.21	0.80	-

In comparison to the Ω_4 and Ω_6 parameters, the Ω_2 parameter is very sensitive to the ligand environment and is used to measure the symmetry as well as structural details around Eu^{3+} ions. The larger the value of Ω_2 stronger the covalency, while other is the symmetry **[20]**. The higher magnitude of Ω_2 was observed for the present TRZNB glasses that suggesting the ligands around the Eu^{3+} ions possess higher distortion as well as high covalent nature. The changes in the emission intensity ratios of $(^5D_0\rightarrow{}^7F_2)/(^5D_0\rightarrow{}^7F_1)$ and $(^5D_0\rightarrow{}^7F_4)/(^5D_0\rightarrow{}^7F_1)$ related to the JO parameters, Ω_2 and Ω_4 are used to describe the variation of emission spectra as a function of glass composition **[21]**. The JO intensity parameters Ω_2 and Ω_4 refer to the covalency and/or structural changes in the vicinity of the Eu^{3+} ion (short range effect) and to long-range effects, respectively. The emission intensity of ED transition ($^5D_0\rightarrow{}^7F_2$) is more intense in non-symmetric sites; whereas the MD transition ($^5D_0\rightarrow{}^7F_1$) intensity is independent nature of the host environment **[22]**.

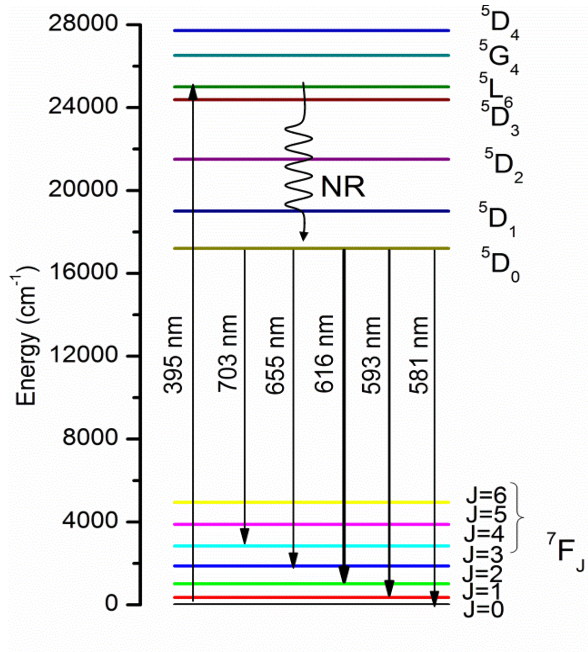

Fig. 3. Energy level diagram showing emission transitions of 1.0 mol % Eu^{3+} -doped TRZNB glasses.

Table 3. Emission properties such as peak emission wavelength (λ_p), effective linewidth, ($\Delta\lambda_p$ nm), radiative transition probabilities (A_R, s^{-1}), stimulated emission cross-section (σ_e x 10^{-22} cm^2), experimental branching ratios (β_m) and gain bandwidth parameters ((σ_e x $\Delta\lambda_p$)x 10^{-25} cm^3) for Eu^{3+} ions in LAFB glasses.

Level	λ_P	$\Delta\lambda_P$	σ_e	A_R	β_m	$\sigma_e\times\Delta\lambda_P$
			TMZNB			
$^5D_0\rightarrow{}^7F_1$	581	15.3	2.48	55	0.22	38.04
$^5D_0\rightarrow{}^7F_2$	593	12.5	33.9	516	0.70	415.64
			TCZNB			
$^5D_0\rightarrow{}^7F_1$	581	14.1	2.9	55	0.21	40.64
$^5D_0\rightarrow{}^7F_2$	593	11.8	38.9	532	0.67	457.82
			TSZNB			
$^5D_0\rightarrow{}^7F_1$	581	17.1	2.0	55	0.21	33.94
$^5D_0\rightarrow{}^7F_2$	593	12.4	29.7	512	0.67	368.81

Table 3 represents the comparison of gain cross sections (A_R), branching ratios (β_m), effective line widths ($\Delta\lambda_P$), stimulated emission cross sections (σ_e) and optical gain band widths ($\sigma_e\times\Delta\lambda_P$) for $^5D_0\rightarrow{}^7F_1$ and $^5D_0\rightarrow{}^7F_2$ transitions in all three glasses. As shown in the table, the stimulated emission cross section (σ_e)/radiative transition probability (A_R) are found to be larger in magnitudes for the $^5D_0\rightarrow{}^7F_2$ transition with the values of 34×10^{-22} cm^2/516 s^{-1}, 39×10^{-22} cm^2/532 s^{-1} and 30 $\times10^{-22}$ cm^2/512 s^{-1} for TMZNB, TCZNB and TSZNB glasses, respectively. The branching ratios (β_m) that are estimated from the areas under the emission bands are the significant parameter to determine the potentiality of the glasses for lasing action. The values of branching ratios are found to be 70, 67 and 67 % for TMZNB, TCZNB and TSZNB glasses, respectively. This indicated that the $^5D_0\rightarrow{}^7F_2$ transition is the most predominant in the present case and these Eu^{3+} -doped TRZNB glasses have potential application for laser emission at 616 nm. The noticed higher magnitudes of

spontaneous emission probability (A_R), stimulated emission cross section (σ_e) and measured branching ratio (β_m) suggests that the $^5D_0 \rightarrow {^7F_2}$ emission peak of Eu^{3+} in TRZNB glass display dominant red emission band at 616 nm. **Fig. 3** represents partial energy level diagram of 1.0 mol % Eu^{3+} -doped TRZNB glasses.

Table 4. Comparison of intensity ratio R = ($^5D_0 \rightarrow {^7F_2}$) / ($^5D_0 \rightarrow {^7F_1}$) of Eu^{3+} ions in TRZNB glasses with different hosts.

Host matrix	Intensity ratio (R)
Present work	
TMZNB	3.083
TCZNB	3.143
TSZNB	3.099
Reported	
Tellurite [25]	4.280
Zinc borate [26]	3.940
PTBEu20 [12]	2.820
ZBS2 [27]	2.690
BLEu [21]	2.410

Nogami *et al.* **[23]** reported that the emission intensities are enhanced when the covalent chemical bond is more between the Eu^{3+} ions and oxygen. In order to study the relative strength of covalent/ionic bonding between Eu^{3+} ions and the surrounding ligands and to found the degree of asymmetry in the vicinity of dopant ions, the fluorescence intensity ratio, R = ($^5D_0 \rightarrow {^7F_2}$)/ ($^5D_0 \rightarrow {^7F_1}$) was calculated. The R values are calculated from the area under various $^5D_0 \rightarrow {^7F_J}$ emission bands and are listed in **Table 4 [24]**. The obtained values for the present glasses are comparable with the reports in literature **[13, 21, 25-27]**. The high intensity ratio of 3.14 was obtained for Eu: TCZNB glass, which is suggesting the formation of strong Eu–O bond in the present glasses. It is concluded from the results of emission spectra that the Eu: TRZNB glass has higher degree of Eu–O bond covalency with relatively greater emission intensity for $^5D_0 \rightarrow {^7F_2}$ transition.

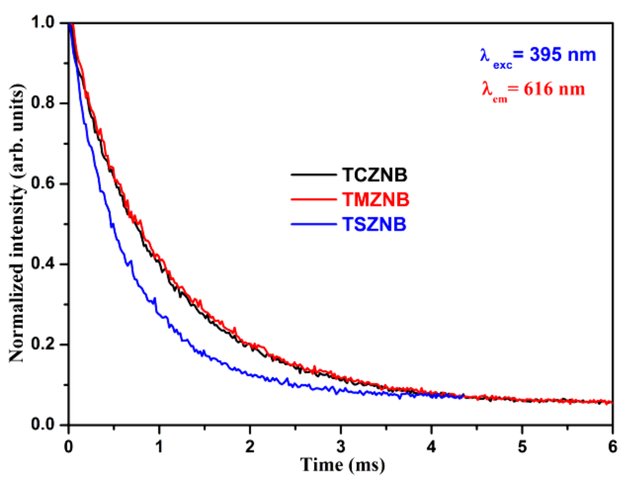

Fig. 4. The decay profiles of $^5D_0$ level of 1.0 mol % Eu^{3+} -doped TRZNB glasses.

Under the 616 nm emission, the decay profiles of the $^5D_0$ emission state of Eu^{3+} ions in TRZNB glasses were collected. To record the lifetimes of the all glasses for $^5D_0$ level of Eu^{3+}, the first e-folding times of the emission intensities were considered. **Fig. 4** shows the logarithmic plot of the experimental decay curves. The curves were well fitted to the single exponential function and the lifetime values are found to be 1.05, 1.02 and 1.0 ms for TMZNB, TCZNB, TSZNB glasses, respectively. The estimation of optical gain parameter ($\sigma_e \times \tau_{exp}$) values is crucial to obtain highly efficient and stable laser active materials **[28]**. Therefore, the obtained optical gain parameters ($\sigma_e \times \tau_{exp}$) are 3.56, 3.98 and 2.97 (10^{-25} cm^2/s) for Eu^{3+} doped TMZNB, TCZNB, and TSZNB glasses, respectively.

Conclusion

In summary, the Eu^{3+}-doped calcium zinc niobium borotellurite (TCZNB) glasses were prepared by the melt quenching method. The emission spectra exhibited the characteristic $^5D_0 \rightarrow {^7F_J}$ (J=0, 1, 2, 3, 4) transitions of Eu^{3+} at 581, 593, 616, 653 and 703 nm when excited at 395 nm. The $^5D_0 \rightarrow {^7F_2}$ red emission (616 nm) occurring through an electric dipole mechanism was found to be more intense in all the TRZNB glasses. For all glasses, the decay curves were well fitted to the single exponential function. The intense red emission at 616 nm suggested that the TRZNB glasses have potential use in the optoelectronic luminescent display devices and laser materials.

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Author Contributions
Conceived the plan: BDPR, SWJ; Performed the expeirments: PR, OR, BR; Data analysis: OR, GRD, CMR; Wrote the paper: GRD, CMR, BDPR. Authors have no competing financial interests.

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