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Composition dependent Synthesis, Structural and Optical Properties of Dy^{3+} Doped Fluoroborate Glasses

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Abstract

The spectroscopic properties of Dy^{3+} ions doped fluoroborate glasses with the composition $39B_2O_3$ + 20ZnO + 20NaF + $20M_2O$ + $1Dy_2O_3$ (where M=Na, K and Ca) have been prepared by melt quenching technique and investigated through XRD,FTIR, absorption and luminescence. The x-Ray diffraction pattern reveals the amorphous nature of the prepared glasses. FTIR results showed the presence of various stretching and bending vibrational modes of the functional groups. From the absorption spectra, the bonding nature of the Dy^{3+} ions with the ligand field environment is found to be ionic. The variation in hypersensitive transitions with the change in Na, K and Ca content have been studied and discussed. JO intensity parameter have been calculated from absorption spectral intensities and are used to calculate ratiative properties such as the effective bandwidth ($\Delta\lambda_{eff}$, nm), transition probability (A), calculated and experimental branching ratios (β_R) and stimulated emission cross-section (σ_P^E) for the $^4F_{9/2} \rightarrow {}^6H_{15/2}$ and $^4F_{9/2} \rightarrow {}^6H_{13/2}$ transitions of the Dy^{3+} ions in prepared fluoroborate glasses.

Keywords: FTIR, Oscillator strength, JO parameters, Radiative properties, White light emission

1. Introduction

Recent years, the trivalent rare earth (RE) ions doped fluoroborate glasses have been received much attention in development of new optoelectronic devices due to their high ionic conductivity short range order around the boron network [1-3]. The borate based glasses provide interesting structural and optical properties. The borate glass structure is not a random distribution of BO_3 triangles and BO_4 tetrahedrals, but all these units form a clear and stable borate groups like diborates, triborates and tetraborates that forms a three dimensional random glass network [4]. These structural changes usually occur due to the chosen chemical composition, involvement of various types of modifiers and conditions applied during glass preparation [5-7]. Generally fluoroborate glasses possess the advantages of both oxides and fluorides such as intermediate phonon energy with higher mechanical strength and chemical durability which helps to improve the luminescence intensity and quantum efficiency. Among the RE^{3+} ions, Dy^{3+} ions are promising for application in solid state visible lasers [8,9], up converters and optical amplifiers [9], commercial display devices [10]. Further the appropriate combination of yellow to blue emission of Dy^{3+} ion could generate white light emission [11]. Shanmugavelu and Kumar [12] investigated the luminescence behavior of Dy^{3+} ions in bismuth zinc borate glasses for white light generation and laser applications. Xiong et al. [13] explored the high-efficiency fluorescence radiation of Dy^{3+} ions in alkaline earth borate glasses for laser application. the spectroscopic properties of Dy^{3+} ions in borate [14], oxyfluoride [15], fluorophosphate [16], zinc–lead–

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phosphate [17], Ge–Ga–As–S–CsBr [18], chalcohalides [19] and Li_2O–LiF–B_2O_3–CdO [20] glasses have been investigated and the energy transfer between Dy^{3+}–Dy^{3+} ions through various cross-relaxation were discussed. The present work reports the composition dependent spectroscopic investigations of Dy^{3+} ions doped fluoroborate glasses for white light emission. The aim of the present paper is to (i) examine the structure of the prepared glass through XRD; (ii) identify the structural groups from FTIR; (iii) calculate the bonding parameter form absorption spectra ; (iv) find the oscillator strengths for each absorption transitions (v) evaluate and compare the JO parameters with the other reported Dy^{3+} doped glasses, and (vi) derive the radiative properties and compare the results with reported literature.

2. Experimental

The appropriate amount of high quality analytical grate (99.9) chemicals such as B_2O_3, NaF, Na_2Co_3, K_2Co_3, Ca_2Co_3Zno and Dy_2O_3 from sigma Aldrich for the prepared Dy^{3+} doped fluoroborate glasses by melt quenching technique. The glass code and the composition are given below.

BZKD1:39B_2O_3+20ZnO+20NaF+20K_2O+1Dy_2O_3

BZCD1:39B_2O_3+20ZnO+20NaF+20Ca_2O+1Dy_2O_3

BZND1:39B_2O_3+20ZnO+20NaF+20Na_2O+1Dy_2O_3

The relevant chemicals were mixed and grained in an agate mature as a 12 g batch. The batch in the crucible was melted in an electronic furnace at 950°C. The melt was quenched by pouring in it on a preheated brass plate and then annealing at 350°C for removing the strains and improve the mechanical strength. The glasses were slowly cooled at room temperature and polished for the optical measurements. The optical absorption spectra were measured in the spectral region 200–2000 nm by JASCO V-670 UV–Vis–NIR spectrophotometer. The luminescence spectra were recorded in the LS 45 Perkin-Elmer paragon spectrometer. The density of the glass samples were measured by Archimedes principle with xylene as an immersion liquid. The refractive indices of the prepared glasses were measured using Abbe refractometer at sodium wavelength using 1-bromonaphthaline as a contact liquid. The physical properties of the prepared glasses were calculated and presented in Table 1.

Table 1: Physical properties Density ρ (g/cm³), Refractive index n_d (589.3 nm), Rare earth ion concentration N(×10^{20} ions/cm³), Polaron radius r_p(A˚), Interionic distance r_i (A˚), Field strength F (10^{14} cm^{-2}), Electronic polarizability(α_e) 10^{-22} cm^3, Molar refractivity R_m (cm³), Dielectric constant (ε) and Reflection losses R (%)of the prepared Dy^{3+}: BZKD, BZND and BZCD1glasses

Properties	BZKD	BZND	BZCD
ρ	2.9882	2.9896	3.1838
n_d	1.5	1.5	1.575
N	0.7455	0.8111	0.8778
r_p	5.2519	5.1161	4.9829
r_i	1.3050	1.2697	1.2366
F	1.7591	1.8608	1.9615
α_e	1.5645	1.4381	1.4928
R_m	7.8900	7.2522	9.0345
ε	2.25	2.25	2.4806
R	4	4	4.9863

3. Results and discussion

3.1. XRD and FTIR spectra

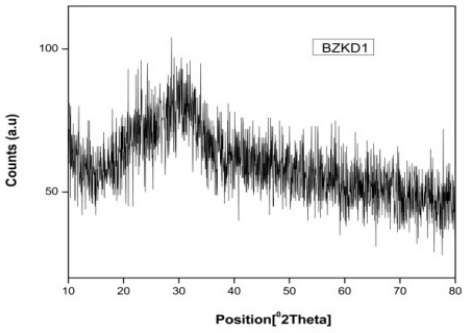

Figure 1. XRD pattern of the Dy^{3+}: BZKD 1 glass.

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Figure 1 shows the XRD spectrum of the BZKD glass and exhibits the broad scattering at lower angles the long range structural disorder. The absence of the sharp peaks in the XRD confirms the amorphous nature of the prepared glass.

FTIR spectra have been recorded in the region 400-4000 and is shown in figure 2. The band position and their assignments are presented in table 2. The band around 3437 cm^{-1} can be attributed to the fundamental stretching of hydroxyl groups. The band at 2926 cm^{-1} is assigned to the hydrogen bond [3]. The band around 1446 cm^{-1} is attributed to the asymmetric mode of B-O stretching vibrations in BO_3 units in boroxol rings. The band at 1227 cm^{-1} is arises due to the B-O stretching vibration of BO_3 units in boroxol rings. The peaks around 1019 cm^{-1} is attributed to the stretching of BO_4 group and the broad bands around 717 cm^{-1} are due to the bending of B-O-B linkages in the borate network. Zn–O stretching vibrations exhibit a band around 466 cm^{-1}.

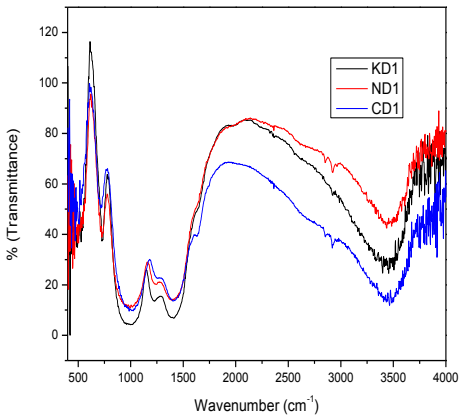

Figure 2. Infrared spectra of Dy^{3+}: BZKD1, BZND1 and BZCD1 glasses

Table 2. Band positions (in cm^{-1}) of FTIR spectra of the Dy^{3+}doped Fluoroborate glasses

BZKD1	BZND1	BZCD1	Assignments
3439	3434	3437	stretching of OH groups
2920	2923	2922	hydrogen bonding
2836	2827	2831	hydrogen bonding
1412	1455	1437	B-O vibrations
1227	1226	1226	Tetrahedral BO4 units
1019	1016	1017	stretching of BO4 units
717	712	715	bending of B-O-B linkages
466	466	462	Zn-O stretching vibration

3.2. Optical analysis

3.2.1. Absorption and Bonding parameters

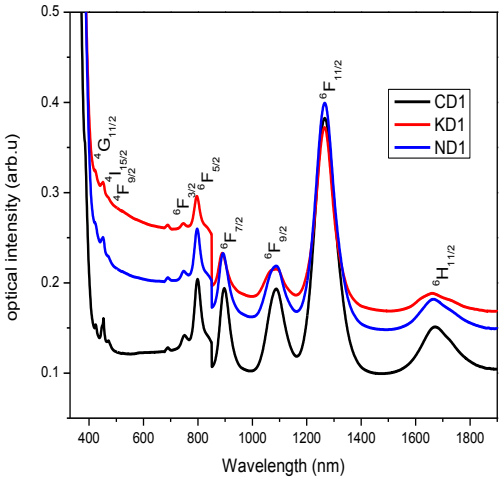

Figure 3. Absorption spectra of Dy^{3+}: BZKD1, BZND1 and BZCD1 glasses

The absorption spectra of the prepared glasses are shown in figure 3 and recorded in the wave length region 400-1800 nm. The absorption spectra exhibits nine absorption bands counterseal at around 1661, 1265, 1089, 889, 796, 746, 471, 451, and 417 nm. Corresponding to that various excited state such as $^6H_{11/2}$, $^6F_{11/2}$, $^6F_{9/2}$, $^6F_{7/2}$, $^6F_{5/2}$, $^6F_{3/2}$, $^4F_{9/2}$, $^4I_{15/2}$ and $^4G_{11/2}$ the absorption band position for the prepared glassesoccurs almost the wave length slight variation in the intensity peaks can be observed for the various compositions. The transition from the ground $^6H_{15/2}$ state to ^6H to ^6F terms are spin allowed

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(Δs=0). Moreover, the transition with in the 6H term are also allowed by the orbital angular momentum selection rule, ΔL=0 and hence, this transitions lying in the NR region are intense [3]. The position and spectral intensities of certain transitions of rare earth ions are found to be sensitive to the environment of the rare earth ion. They follow the selection rules ΔJ≤2, ΔL≤2 and ΔS=0 and such transitions are known as hypersensitive transitions. For Dy^{3+} ion the transition $^6H_{15/2}\rightarrow{}^6F_{11/2}$ is identified to be a hypersensitive transition and is found to be more intense than the other transitions. The bonding parameter (δ) is calculated by using nephelauxetic ratio and is written as δ =(1-β̄)/ (β̄)×100, where β̄ = $\Sigma_n\beta/N$, β is the nephelauxetic ratio [4]. Depending upon the environmental field, δ may be positive or negative indicating the covalent or ionic nature [21]. The negative values of bonding parameters indicate ionic nature of the prepared glasses.

Table 3. Absorption band positions (in cm^{-1}), β and δ values of the Dy^{3+}doped fluoro borate glasses

Transition $^6H_{15/2}\rightarrow$	BZKD1	BZND1	BZCD1	Aquo ion [13]
$^4G_{11/2}$	23981	23753	23585	23400
$^4I_{15/2}$	22173	22173	22075	22100
$^4F_{9/2}$	21231	21142	21231	21100
$^6F_{3/2}$	13405	13369	13333	13250
$^6F_{5/2}$	12563	12547	12516	12400
$^6F_{7/2}$	11249	11211	11148	11000
$^6F_{9/2}$	9183	9200	9191	9100
$^6F_{11/2}$	7905	7899	7899	7700
$^6H_{11/2}$	6020	6006	5984	5850
β̄	1.01633	1.01377	1.01111	
δ	-1.6070	-1.3590	-1.0989	

3.2.2. Oscillator Strength and J-O Intensity Parameter

The intensities of an absorption bands are expressed in terms of their oscillator strengths using the expression as $f_{exp}= 4.318\times10^{-9}\int\varepsilon(v)dv$. The experimental oscillator strength (f_{exp}) of the absorption bands is calculated from the relative areas under the absorption bands of the individual transition in the absorption spectra of the RE ions doped materials. The experimental and calculated oscillator strength ($\times10^{-6}$) of the Dy^{3+}doped fluoroborate glasses are presented in table 4, with rms (σ) deviation values. The JO analysis for the Dy^{3+}ions is in good agreement with (f_{exp}) and (f_{cal}) for some of the energy levels and there is an amount of moderate deviation in case of weak transition. Judd–Ofelt (J–O) model gives the theoretical estimation of the intensities of intra configurational f–f transitions of RE^{3+} ions as following the procedure in the liteture [3]. The J-O theory has been applied to evaluate the intensity parameters $\Omega_\lambda(\lambda = 2, 4, 6)$ from the measured spectral intensities of absorption bands.

Table 4. Experimental and Calculated oscillator strengths ($\times10^{-6}$) of the Dy^{3+}doped fluoroborate glasses

$^6H_{15/2}\rightarrow$	BZKD		BZND		BZCD	
	f_{exp}	f_{cal}	f_{exp}	f_{cal}	f_{exp}	f_{cal}
$^4G_{11/2}$	0.058	0.022	0.054	0.032	0.026	0.013
$^4I_{15/2}$	0.108	0.141	0.198	0.213	0.094	0.074
$^4F_{9/2}$	0.024	0.465	0.023	0.078	0.040	0.027
$^6F_{3/2}$	0.092	0.053	0.105	0.092	0.041	0.031
$^6F_{5/2}$	0.400	0.283	0.716	0.487	0.220	0.162
$^6F_{7/2}$	0.644	0.611	0.973	1.026	0.350	0.348
$^6F_{9/2}$	0.717	0.721	1.189	1.183	0.419	0.426
$^6F_{11/2}$	2.582	2.576	2.999	2.995	1.187	1.185
$^6H_{11/2}$	0.381	0.423	0.588	0.621	0.202	0.219
N	9		9		9	
Rms	0.048		0.082		0.023	

The JO parameters play an important role in studying the coordination environment around the trivalent rare-earth ions and moreover on the covalency between the rare-earth and oxygen ions in any host glass. The intensity parameter Ω_2 has been identified as associated with the asymmetry and the covalency of the lanthanide sites. Ω_4 and Ω_6 are related to the bulk property and rigidity of the samples respectively. However, the Ω_2 values are found to be higher than the Ω_4 and Ω_6 intensity

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parameter values in the prepared glasses. All the prepared glasses follow the trend as $\Omega_2>\Omega_6>\Omega_4$ presented in table 5 and compared with the similar studies. The Ω_2 ($\times10^{-20}$ cm^2) values are found to be 3.3138, 3.3375 and 1.276 for the prepared glasses BZKD1, BZND1 and BZCD1 respectively.

Table 5. JO intensity parameters ($\Omega_\lambda\times10^{-20}$ cm^2), spectroscopic quality factor (Ω_4/Ω_6) of the Dy^{3+}doped fluoroborate glasses

Glasses	Ω_2	Ω_4	Ω_6	Ref
BZKD1	3.138	0.6008	0.7234	Present
BZND1	3.337	0.9129	1.2382	
BZCD1	1.276	0.3430	0.3892	
PSB glass d2	2.27	0.94	1.31	22
Lead borate	4.90	0.94	2.07	23
BZBDy05	4.72	0.58	1.99	12
BZBDy2	4.29	1.05	1.72	12
ZBLA(1)	3.22	1.35	2.38	24
1DPTFB	7.381	1.654	2.306	25

3.3. Luminescence studies and Radiative Properties

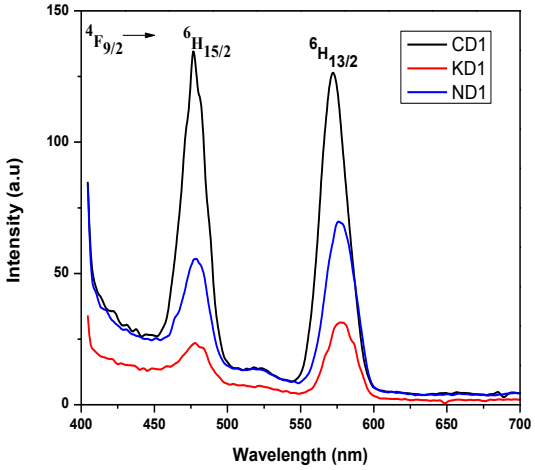

Figure 4. Emission spectra of Dy^{3+}: BZKD1, BZND1 and BZCD1 glasses

The luminescence spectra of the Dy^{3+}: fluoroborate glasses were recorded with 384 nm excitation and is shown in Figure 4. The luminescence spectra exhibit two emission band peaks at 476 nm and 571 nm corresponding to $^4F_{9/2}\rightarrow{}^6H_{15/2}$ (blue) and $^6H_{13/2}$ (yellow) transitions respectively as shown in Figure 4. Among the transitions, $^4F_{9/2}\rightarrow{}^6H_{13/2}$ electric dipole transition is found to have a higher intensity than the $^4F_{9/2}\rightarrow{}^6H_{15/2}$ magnetic dipole transition. The luminescence intensity of the $^4F_{9/2}\rightarrow{}^6H_{13/2}$ transition is hypersensitive because it is more sensitive to the environment around the Dy^{3+} ion site when compared to the $^4F_{9/2}\rightarrow{}^6H_{15/2}$ transition. The radiative properties such as the transition probability (A), branching ratios (β_R) and stimulated emission cross-section ($\sigma_P^E\times10^{-22}$ cm^2) for the emission transition $^4F_{9/2}\rightarrow{}^6H_{15/2}$ and $^4F_{9/2}\rightarrow{}^6H_{13/2}$ of the Dy^{3+} ions have been calculated using the expression given in the literature [1,13]. The calculated radiative properties of the prepared glasses are presented in table 6. The branching ratios (β_R) and stimulated emission cross-section ($\sigma_P^E\times10^{-22}$ cm^2) values are important parameters for the potential laser applications. The higher magnitude of the stimulated emission cross-section ($\sigma^E{}_P$) is an indication for efficient laser emission. Among the prepared glasses, BZND1 glass possesses higher ($\sigma^E{}_P$) value (35.1475×10^{-22}cm^2) correspond to the $^4F_{9/2}\rightarrow{}^6H_{13/2}$ (electric dipole) emission transition and higher than the reported glasses such as M.V. Vijayakumar et al. [26] and CH. Basavapoornima et al. [27] respectively.

The optical gain bandwidth and optical gain these are the important laser parameters for the development of new optical devices and the optical gain band width [$\sigma_P^E\times\Delta\lambda_{eff}$] and optical gain [$\sigma_P^E\times\tau_R$] values of the prepared glasses have been calculated and presented in table 6. It is observed from the tabulated results that among the prepared glasses, BZND glass is found to be more suitable for developing visible lasers and fiber optic amplifiers since it exhibit higher magnitude of A, β_R, σ_P^E and

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$\sigma_P^E \times \Delta\lambda_{eff}$ stimulated emission cross section and branching ratio values.

Table 6. The effective bandwidth ($\Delta\lambda_{eff}$, nm), transition probability (A), calculated and experimental branching ratios (β_R) and stimulated emission cross-section ($\sigma_P^E \times 10^{-22}$ cm^2) for the $^4F_{9/2} \rightarrow {}^6H_{15/2}$ and $^4F_{9/2} \rightarrow {}^6H_{13/2}$ transitions of the Dy^{3+}doped fluoroborate glasses

Transition	Parameters	BZKD	BZND	BZCD
$^4F_{9/2} \rightarrow {}^6H_{15/2}$	λ_p	478	478	476
	$\Delta\lambda_{eff}$	4.6434	5.6981	4.4546
	A	51.23	86.53	32.37
	σ_P^E	3.3968	4.6754	1.9955
	β_R(cal)	0.2848	0.3227	0.4742
	β_R(exp)	0.118	0.1671	0.1217
	$\sigma_P^E \times \Delta\lambda_{eff}$	1.5772	2.6641	0.8893
$^4F_{9/2} \rightarrow {}^6H_{13/2}$	λ_p	577	576	572
	$\Delta\lambda_{eff}$	4.9550	5.5320	3.6886
	A	157.57	299.51	126.89
	σ_P^E	33.977	35.147	19.699
	β_R(cal)	0.7151	0.6772	0.5257
	β_R(exp)	0.5968	0.5785	0.4770
	$\sigma_P^E \times \Delta\lambda_{eff}$	16.836	19.443	7.2662

4. Conclusion

Composition dependent structural and optical properties of Dy^{3+} doped fluoroborate glasses have been studied for photonic applications. Structural analysis has been carried out using XRD and FTIR specta. The ionic nature of the Dy^{3+} metal ligand bonding was explored from the absorption spectral measurement. The higher Ω_2 values indicate the higher asymmetry around Dy3+ ion site. Two emission band peaks corresponding to $^4F_{9/2} \rightarrow {}^6H_{15/2}$ and $^6H_{13/2}$ transitions were observed from emission spectra. Among the prepared glasses, BZND1 glass possess higher (σ^{E_P}) value correspond to the $^4F_{9/2} \rightarrow {}^6H_{13/2}$ (electric dipole) emission transition which is an indication for efficient laser emission.

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