Optical absorption and photoluminescence investigations Dy$^{3+}$ doped oxyfluoride phosphate glass system for active laser medium and solid-state lighting materials

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Abstract. This article reports on the optical, photoluminescence, and radiative properties of Dy$^{3+}$ doped oxyfluoride glass. The composition of glass is: 20Li$_2$O–10AlF$_3$–69P$_2$O$_5$–1Dy$_2$O$_3$ (LAPD). The glass has been prepared using the melt-quenching technique at 1100°C with a total mass of 15 grams in an alumina crucible. Judd–Ofelt analysis was used to determine the intensity parameters and radiative properties of Dy$^{3+}$ ion in glass. The CIE 1931 chromaticity diagram coordinate is calculated to define the color coordinate of emission in a glass sample. Based on research, it is expected to produce glass material that is applied as an active laser medium and solid-state lighting materials.

Keywords: Dysprosium oxide, Oxyfluoride glass, Luminescence, J-O parameters, White light emission.

1. Introduction

Lasers are increasingly being developed into applications industrial, medical, military, scientific, etc. The laser is a gain medium that emits light in the visible range to the infrared region which is excited by ultraviolet. Glass is a material under investigation for its multi-functional characteristic behavior when treated with rare earth oxides. Optical studies reveal the radiative properties of RE$^{3+}$ doped glasses, these properties strongly depends on the host matrix and can be modified by proper network formers as well as modifiers. The phosphate glass possess low melting point, good thermal stability, mechanical, transmission in the visible to infrared region, high transparency, long decay lifetime, and rare earth (Ln$^{3+}$) solubility. The oxyfluoride glasses can be prepared relatively easily and have good thermal stability and insensitivity to moisture. Moreover fluoride compounds present in phosphate glasses can keep phonon energies of the host glass by increasing the resistance to moisture and remove OH group from phosphate glasses. With the addition of metal alkaline Li$_2$O and AlF$_3$ in glass, the host can improve the chemical and mechanical stability, decrease the melting point, and low phonon energy[1][2][3][4]. Among RE ions, the Dy$^{3+}$ (4f$^0$) ion is a most fascinating ions to explore for luminescence properties, color display phosphors and white light emissions, since the Dy$^{3+}$ ion typically emitting two intense emissions in the yellow (~576 nm) and blue (~482 nm) regions that are attributed to the $^4F_{9/2}$$rightarrow$$^4H_{13/2}$ and $^4F_{9/2}$$rightarrow$$^4H_{15/2}$ transitions[5]. Dysprosium doped glass has been investigated for producing white light is the combination of a blue LED with yellow emitting phosphorous[6][7][8]. The current work focuses on the characterization of optical, luminescence properties, judd-ofelt parameter, and CIE of Dy$^{3+}$ doped oxyfluoride phosphate glass system. The applicability of these glasses are used for solid state yellow laser devices and white LEDs applications.
2. Experiment
The oxyfluoride phosphate glass doped Dy³⁺ was prepared by melt-quenching technique with chemical composition 20Li₂O–10AlF₃–69P₂O₅–1Dy₂O₃. The mixtures 15 g of compounds was taken in a porcelain crucible and melted at 1100°C for 2 h, then the melted sample was poured into preheated stainless steel molds. The glass sample was annealed at 350°C for 3 h for eliminated internal mechanical stress before slowly cooled down to room temperature. The glass sample was transparent in Figure 1.

The optical spectra of the glass was measured with UV-Vis-NIR spectrophotometer (Shimadzu, UV-3600) in the range 200-2000 nm. The Photoluminesence and luminescence decay time was measured with a spectro-fluorophotometer (Cary-Eclipse) with xenon light source. All characterizations have been conducted at room temperature. Judd-Ofelt analysis has been applied to determine the spectroscopic properties. The radiative properties of sample was obtained by theoretical calculation. The CIE 1931 chromaticity diagram coordinate is calculated to define the color coordinate of emission in glass sample.

![LAPD](image)

**Figure 1.** Dysprosium oxide doped Oxyfluoride phosphate glass system

3. Results and Discussion

3.1 Optical Properties
The absorption spectra of LAPD glass is indicated in the range of 300-2000 nm shown in Figure 2. The spectra consist of thirteen absorption band transitions initiated from the ⁴H₁₅/₂ state to the various excited state, i.e., 325 nm (⁶P₃/₂+⁴M₁₇/₂), 351 nm (⁶P₇/₂+⁴M₁₅/₂), 364 nm (⁴I₁₁/₂+⁶P₃/₂), 387 nm (⁴I₁₃/₂+⁴F₉/₂+⁴M₂₁/₂+⁴K₁₇/₂), 425 nm (⁴G₁₁/₂), 475 nm (⁴F₉/₂), 475 nm (⁴F₉/₄), 804 nm (⁶F₅/₂), 901 nm (⁶F₇/₂), 1096 nm (⁴F₉/₂+⁴H₇/₂), 1278 nm (⁴F₁₁/₂+⁴H₉/₂) and 1695 nm (⁶H₁₁/₂). The assignments of the absorption transitions have been explained as detailed in previous work [9][10][11]. The highest peak at 351 nm (suitable to the ⁴H₁₅/₂ → ⁶P₃/₂ transition) in the UV-Vis region and at 1278 nm (suitable to the ⁴H₁₅/₂ → ⁴F₁₁/₂, ⁴H₉/₂ transition) in the NIR region, this is the hypersensitive transition (The 4f transition that very sensitive to the environment) that obey the selection rules, |ΔJ|≤2, |ΔL|≤2 and ΔS = 0). [6]
3.2 Luminescence Properties

The photoluminescence spectra of 1 mol% Dy$^{3+}$ doped lithium fluorophosphate glass was recorded in the excitation spectra range from 275 to 500 nm by emission wavelength of 572 nm shown in Figure 3. The excitation band in Figure 3 indicate peaks located at 296, 325, 338, 351, 364, 387, 426, 452, 474 nm and related to the transitions from ground state level $^6$H$_{15/2}$ to the excited states $^4$K$_{13/2}$+$^4$G$_{13/2}$, $^6$P$_{3/2}$+$^4$M$_{17/2}$, $^4$I$_{9/2}$+$^4$F$_{7/2}$, $^6$P$_{3/2}$+$^4$I$_{11/2}$, $^4$F$_{7/2}$+$^4$I$_{13/2}$+$^4$K$_{17/2}$+$^4$M$_{21/2}$, $^4$G$_{11/2}$, $^4$I$_{15/2}$, $^4$F$_{9/2}$. These excitation wavelengths are in good agreement with the wavelengths of the commercially available UV LEDs having emission ranges from 350 to 420 nm and blue LEDs having emission ranges from 450 to 470 nm. The intensity of the excitation band observed at 351 nm corresponding to the $^6$H$_{15/2}$→$^6$P$_{7/2}$ transition is found to be higher when compared to other transitions. The emission spectra range from 400 to 800 nm by excitation wavelength of 351 nm shown in Figure 4. Four emission peaks were identified from the transition $^4$F$_{9/2}$→$^4$H$_{15/2}$ at 483 nm the blue (B) emission, transition $^4$F$_{9/2}$→$^4$H$_{11/2}$ at 572 nm the yellow (Y) emission, transition $^4$F$_{9/2}$→$^4$H$_{11/2}$ at 663 nm and transition $^4$F$_{9/2}$→$^4$H$_{9/2}$ at 751 nm. The blue emission (magnetic dipole) and yellow emission (electric dipole) were investigated as a result glass sample is more asymmetry in nature [12][13][14]. The Figure 5 shown the energy level scheme for the luminescence of LAPD.

![Figure 2](image2.png)

**Figure 2.** Absorption spectra of LAPD glass

![Figure 3](image3.png)

**Figure 3.** Excitation spectra of LAPD glass

![Figure 4](image4.png)

**Figure 4.** Emission spectra of LAPD glass
3.3 Judd-Ofelt

The Judd-Ofelt parameter has been calculated in this work. The experimental oscillator strength experimental \((f_{\text{exp}})\) and theoretical \((f_{\text{cal}})\) values of Dy\(^{3+}\) doped lithium fluorophosphate glass shown in Table 1. The best absorption in the NIR Region which is located at 1278 nm has the highest value of oscillator strength. The small root-mean-square deviation value \((\delta_{\text{rms}})\) of \(\pm 0.96 \times 10^{-6}\) obtained between the \(f_{\text{exp}}\) and \(f_{\text{cal}}\) values indicate the good fit.

Table 1. Experimental \((f_{\text{exp}})\) and theoretical \((f_{\text{cal}})\) oscillator strength values \((\times 10^{-6})\) of LAPD glass

| Transitions    | \(\lambda_{\text{abs}}\) (nm) | Energy \((\text{cm}^{-1})\) | \(f_{\text{exp}}\) | \(f_{\text{cal}}\) |
|----------------|-----------------|-----------------|--------|--------|
| \(^{4}I_{15/2}\rightarrow^{6}P_{3/2}+^{4}M_{17/2}\) | 325 | 30769.23 | 1.2137 | 1.3070 |
| \(^{6}P_{7/2}+^{4}M_{15/2}\) | 351 | 28490.03 | 2.9593 | 2.8131 |
| \(^{4}I_{11/2}\rightarrow^{4}P_{5/2}\) | 364 | 27472.53 | 0.9574 | 0.0808 |
| \(^{4}I_{13/2}\rightarrow^{4}F_{7/2}+^{4}M_{21/2}+^{4}K_{13/2}\) | 387 | 25839.79 | 1.0238 | 0.3292 |
| \(^{4}G_{11/2}\) | 425 | 23529.41 | 0.1410 | 0.0707 |
| \(^{4}I_{15/2}\) | 450 | 22222.22 | 0.4515 | 0.6792 |
| \(^{4}F_{9/2}\) | 475 | 21052.63 | 0.2230 | 0.2587 |
| \(^{6}F_{7/2}\) | 755 | 13245.03 | 0.7338 | 0.3134 |
| \(^{6}F_{5/2}\) | 804 | 12437.81 | 1.6932 | 1.6656 |
| \(^{6}F_{7/2}\) | 901 | 11098.78 | 3.1032 | 3.3482 |
| \(^{6}F_{9/2},^{4}H_{7/2}\) | 1096 | 9124.09 | 3.3456 | 0.1399 |
| \(^{6}F_{11/2},^{4}H_{9/2}\) | 1278 | 7824.73 | 6.9588 | 6.9584 |
| \(^{6}H_{11/2}\) | 1695 | 5899.71 | 1.8834 | 1.8892 |

\[\delta_{\text{rms}} = \pm 0.955\]

The result J–O parameters are \(\Omega_{2} = 7.62 \times 10^{20} \text{ cm}^2\), \(\Omega_{4} = 1.95 \times 10^{20} \text{ cm}^2\), \(\Omega_{6} = 4.20 \times 10^{20} \text{ cm}^2\). Table 2 compares the JO parameters obtained in the present work with those obtained for the other glass systems. For LAPD glass, the magnitude of parameters follows the trend as \(\Omega_{2} > \Omega_{4} > \Omega_{6}\). The
higher magnitude of $\Omega_2$ in the present glass suggests a higher degree of covalency between doped RE ions and an oxygen atom and the magnitude of $\Omega_4$, $\Omega_6$ are indicated that viscosity and rigidity. Generally, the JO parameters allow information on the nature of bonding between RE ions and surrounding ligands as well as the symmetry of the environment around the Ln ions.

### Table 2. Judd-Ofelt parameters ($x10^{20}$ cm$^2$) and spectroscopic quality factor of LAPD glass

| Label Glass     | $\Omega_2$ | $\Omega_4$ | $\Omega_6$ | Trend           | Reference |
|-----------------|------------|------------|------------|-----------------|-----------|
| C2              | 7.62       | 1.95       | 4.20       | $\Omega_2 > \Omega_6 > \Omega_4$ | This work |
| Dy:PBiNaGd4     | 1.10       | 0.34       | 0.47       | $\Omega_2 > \Omega_6 > \Omega_4$ | [1]       |
| PbFPF Dy10      | 7.12       | 1.59       | 2.20       | $\Omega_2 > \Omega_4 > \Omega_6$ | [11]      |
| Li: 49.5P$_2$O$_5$–10AlF$_3$–10BaF$_2$–10SrF$_2$–10PbO–10Li$_2$O–0.5Dy$_2$O$_3$ | 6.83 | 3.14 | 1.60 | $\Omega_2 > \Omega_4 > \Omega_6$ | [15] |
| BGGD1.00        | 3.65       | 0.65       | 1.57       | $\Omega_2 > \Omega_6 > \Omega_4$ | [16] |
| LLCZFB:Dy glass | 11.58      | 6.17       | 4.93       | $\Omega_2 > \Omega_4 > \Omega_6$ | [17] |
| NKL10B          | 14.55      | 8.04       | 8.03       | $\Omega_2 > \Omega_4 > \Omega_6$ | [18] |
| SLBP Dy10       | 6.37       | 0.34       | 2.16       | $\Omega_2 > \Omega_6 > \Omega_4$ | [19] |
| B5Te2: 0.5Dy    | 14.88      | 4.57       | 6.32       | $\Omega_2 > \Omega_4 > \Omega_6$ | [20] |
| 0.5 Dy:B$_2$O$_3$–WO$_3$–ZnO–Li$_2$O–Na$_2$O | 26.62 | 6.21 | 17.19 | $\Omega_2 > \Omega_6 > \Omega_4$ | [21] |
| CNGG            | 5.04       | 1.81       | 1.53       | $\Omega_2 > \Omega_4 > \Omega_6$ | [22] |
| 50B$_5$O$_3$–30PbO–20SrO–xDy | 8.48 | 3.54 | 3.17 | $\Omega_2 > \Omega_4 > \Omega_6$ | [23] |

In this section, some important spectroscopic parameters including total radiative transition probabilities ($A_R$), radiative lifetimes ($\tau_R$), branching ratios ($\beta_R$) and absorption cross-sections ($\sigma_\alpha$) were presented in Table 3. It was found that different emission levels, the $^6$H$_{15/2}$ level exhibits higher radiative transition probabilities, branching ratio and emission cross-sections, be in accordance with the highest peak emission [24][25][26].

### Table 3. Radiative properties, emission peak wavelength ($\lambda_p$), effective bandwidth ($\Delta \lambda_{\text{eff}}$), emission cross-section ($\sigma_e(\lambda_p) \times 10^{21}$), experimental branching ratio ($\beta_{\text{exp}}$), calculated branching ratio ($\beta_{\text{cal}}$), radiative transition probability ($A_R$), and radiative lifetime ($\tau_R$) of LAPD glass

| Transition of emission | $\lambda_p$ (nm) | $\Delta \lambda_{\text{eff}}$ (nm) | $\sigma_e(\lambda_p)$ (cm$^2$) | $\beta_{\text{exp}}$ (%) | $\beta_{\text{cal}}$ (%) | $A_R$ (s$^{-1}$) | $\tau_R$ (ms) | $\nu$ |
|------------------------|------------------|-----------------------------------|--------------------------------|--------------------------|--------------------------|-----------------|------------|------|
| $^4$F$_{9/2} \rightarrow$ |                  |                                   |                                |                          |                          |                 |            |      |
| $^6$H$_{15/2}$         | 483              | 14.41                             | 38.68                          | 0.3570                   | 0.2491                   | 200.12          |            |      |
| $^6$H$_{11/2}$         | 572              | 13.66                             | 2.65                           | 0.6102                   | 0.6663                   | 660.83          | 0.905      | 0.450 |
| $^6$H$_{11/2}$         | 663              | 12.58                             | 56.74                          | 0.0222                   | 0.0653                   | 72.13           |            |      |
| $^6$H$_{9/2}$          | 751              | 11.98                             | 28.94                          | 0.0106                   | 0.0193                   | 21.28           |            |      |
Table 4. The radiative properties for transition $^6\text{H}_{13/2}$ of LAPD glass compared with other Dy-doped glass

| Label Glass          | $A_R$ (s$^{-1}$) | $\sigma_e (\lambda_p)$ (cm$^2$) | $\eta$ (%) | Reference |
|----------------------|------------------|---------------------------------|------------|-----------|
| LAPD                 | 660.8            | 2.65                            | 0.497      | This work |
| Dy$^{3+}$ doped different BiP | -                | 9.19                            | 98.2       | [13]      |
| NKLB10               | 760              | 1.66                            | 1.73       | [18]      |
| PKFSADy10            | 540              | 3.354                           | -          | [24]      |
| Dy$^{3+}$ doped NAP  | -                | 3.68                            | 0.63       | [26]      |
| PKANbDy              | 2245             | 6.400                           | -          | [27]      |
| KFP10Dy              | 2514             | 0.434                           | 1.30       | [28]      |

The observed $A_R$, $\sigma_e (\lambda_p)$, and $\eta$ values for transitions $^6\text{H}_{13/2}$ were presented in Table 4. As can be referenced from the table, all of these parameters fall within the range of values commonly reported for the Dy$^{3+}$ doped glass system.

3.4 Lifetime

The Lifetime of Dy$^{3+}$ doped oxyfluoride phosphate glass for an emission wavelength of 572 nm under the excitation wavelength 351 nm shown in Figure 5. The effective decay time ($\tau_f$), was found through the relation:

$$\tau_f = \int tl(t) dt / \int I(t) dt$$

The value of 0.45 ms was obtained for the $^4\text{F}_{9/2}$ level decay time in the LAPD glass. In Dy$^{3+}$ singly-doped phosphors the non-exponential decay of the $^4\text{F}_{9/2}$ level emission has been related to cross-relaxation process from Dy$^{3+}$ ions. From table 5 comparing the lifetime of this work the range of values commonly reported for Dy$^{3+}$ doped glass.

![Figure 6. The lifetime of LAPD glass](image-url)
Table 5. The lifetime of LAPD glass compared with other Dy-doped glass

| Sample    | Lifetime | References |
|-----------|----------|------------|
| LAPD      | 0.450 ms | This work  |
| G1        | 0.539 ms | [10]       |
| G2        | 0.540 ms | [10]       |
| NKLB10    | 0.520 ms | [18]       |
| GdCaSiBD  | 0.455 ms | [29]       |
| LGBaBDy1.0| 0.693 ms | [30]       |
| LGBiBDy1.0| 0.423 ms | [31]       |

3.5 CIE
To determine the suitability of the color of light emitted by the glass sample, the x, y color coordinates of emission spectra has been analyzed in the framework of CIE 1931 chromaticity diagram. The results show that the x,y color coordinates of emission spectra of LAPD glass is found at the point (0.37, 0.42). Figure 9 presents the location of x,y color coordinate of the emission light of the glass samples on CIE 1931 chromaticity diagram which is fall in the white region, the white light emission of LAPD glass also shown in photograph in Figure 9 when exciting the samples by 365 nm UV lamp (4 W, 0.16 A). The CIE color coordinates for various host glass falling in white region, near to white center (x=0.333, y=0.333) [18] are given in Table 6.

Figure 7. (a) The CIE 1931 diagram of the glass sample; (b) White emission excited by 365 nm UV lamp (4 W, 0.16 A) of LAPD glass
Table 6. CIE 1931 color coordinate of LAPD glass compared with other Dy-doped glass

| Sample       | Chromaticity coordinates | Reference |
|--------------|--------------------------|-----------|
| LAPD         | 0.37 0.42                | Present work |
| LuCSB:1.0Dy  | 0.368 0.405              | [7]        |
| G1 and G2    | 0.38 0.43                | [10]       |
| PbFPDy10     | 0.31 0.34                | [11]       |
| NKLB10       | 0.36 0.40                | [18]       |
| E            | 0.33 0.43                | [21]       |

4. Conclusion
The Dy$^{3+}$ doped oxyfluoride phosphate glass has been investigated their properties through, absorption spectra, photoluminescence spectra, judd-ofelt parameter and CIE 1931 color chromaticity studies. The luminescence spectra exhibited four emission bands corresponding to $^4F_{9/2} \rightarrow ^6H_{15/2}$ at 482 nm the blue (B) emission, transition $^4F_{9/2} \rightarrow ^6H_{13/2}$ at 572 nm the yellow (Y) emission, transition $^4F_{9/2} \rightarrow ^6H_{11/2}$ at 663 nm and transition $^4F_{9/2} \rightarrow ^6H_{9/2}$ at 751 nm transitions under 351 nm excitation. The trend of JO intensity parameters for LAPD is $\Omega_2 > \Omega_4 > \Omega_6$. Based on higher stimulated emission cross-section and branching ratio (61%), it is suggested that LAPD glass of transition $^4F_{9/2} \rightarrow ^6H_{13/2}$ is might be useful for the development of lasing and other photonics devices in the visible region. The estimated CIE chromaticity coordinates fallen within white light region.

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