



Research Article

DELVING INTO THE SPECTROSCOPIC PROPERTIES OF DY³⁺ IN DIFFERENT FLUOROPHOSPHATE GLASSES FOR MULTITUDE OF APPLICATIONS

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ABSTRACT

Dy³⁺ ion doped different fluorophosphate glasses were prepared by melt-quenching process. Structural study was conducted by XRD, FTIR, FT-Raman and 31P MAS NMR spectroscopy. The optical properties were examined using absorption and photoluminescence spectroscopy. Various structural groups that made up the glass were identified using FTIR and FT-Raman spectra. Breaking up of metaphosphate chains were detected by the decrease of Q2 tetrahedral sites paving the formation of pyrophosphate groups (Q1) as revealed by 31P MAS NMR spectroscopic study. Judd-Ofelt intensity parameters Ω_2 , Ω_4 , and Ω_6 , were calculated from absorption spectra. Radiative lifetimes (τ_R), branching ratios (β_R) integrated absorption cross-sections (Σ) and were calculated subsequently using the above said technique. Experimental branching ratios (β_{exp}) and stimulated emission cross-sections (σ_P) were calculated for all the observed emission transitions of Dy³⁺ doped prepared fluorophosphate glasses. The CIE chromaticity coordinates, correlated colour temperature (CCT) and yellow to blue (Y/B) intensity ratios were calculated to ascertain these glasses usefulness for photonic applications in lasers and white LEDs..

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INTRODUCTION

According to United Nations environmental programme (UNEP) 20% of the world's electricity production is used for lighting purposes and is responsible for 6% of CO₂ emission. It is going to increase at enormous rate of 60% by 2030. This will have far reaching consequences on the climate change. The problems associated with the conventional white light emitting diodes (WLEDs) which make use of crystalline phosphors, ultraviolet LED chip and epoxy resin are that, deterioration of the resin that encapsulates the conventional LEDs, large refractive index difference between the resin and phosphor which increases the amount of scattering of light, decrease of overall lifetime of the device to name a few. So, there is a dire need to develop energy efficient lighting systems.

Spectroscopic examination unravelled the fact that the intra 4f electronic transitions of rare earth ions play a vital role and made glasses doped with rare-earth ion as technologically suitable and superior materials for solid-state lasers, planar waveguides, microchip lasers to name a few. Low melting temperatures, ultraviolet (UV) transmission and good optical characteristics are some of the outstanding features of fluorophosphate glasses that made these glasses suitable for various applications. A range of materials can be created using these glasses including fast ion conducting materials, energy efficient solid-state lighting devices, laser hosts, and

biocompatible ones. and production of these glasses have good chemical durability which made them carve a niche among photonic glasses [1-4].

Among Ln³⁺ ions, trivalent dysprosium (Dy³⁺) doped glasses are widely studied for their applications in telecommunication and solid-state lighting devices, in the fabrication and functioning of optical amplifier systems. The study of luminescence from the 4F_{9/2} level of Dy³⁺ ions is highly significant towards this end, as its range covers the visible and NIR regions. Dy³⁺ is an excellent luminescent ion whose visible spectrum has three bands, blue (460–485 nm; 4F_{9/2} to 6H_{15/2}), yellow (570–590 nm; 4F_{9/2} to 6H_{13/2}) and red (635–655 nm; 4F_{9/2} to 6H_{11/2}). Of these 4F_{9/2}→6H_{13/2} transition is an electric dipole (E.D) and 4F_{9/2}→6H_{15/2} transition which is a magnetic dipole (M.D) transition. These two transitions correspond to yellow and blue regions respectively. The E.D transition is hypersensitive and hence that transition's intensity is highly influenced by the nature of the host. The intensity of 4F_{9/2}→6H_{15/2} transition is less sensitive to the host [5-8]. Hence, at a suitable yellow-to-blue (Y/B) intensity ratio, Dy³⁺ can emit white light. Thus, luminescent materials doped with Dy³⁺ ion are usually employed in the production of white light both in glasses and in phosphors.

In the present work, a systematic probe was carried out to arrive at some conclusions about the effect of alkali, alkaline

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and post transition metal fluorides, keeping dysprosium ion concentration as same i.e 0.5 mol%. The addition of zinc oxide makes the fluorophosphate glass moisture resistant and strengthens the glass network. Addition of alkali fluorides form new substructures which will alternate the phosphate network. These substructures behave as defects in the host glass network. The extent of depolymerization of the glass network heavily depends on the ionic radius of the modifier ion and its suitability with the ligand network. Spectroscopic properties such as intensity parameters, spectral intensities, and radiative properties namely transition probabilities (AR), radiative lifetimes (τ_R) and integrated absorption cross-sections (Σ), Emission properties such as emission intensities, branching ratios (β_{exp}) and peak stimulated emission cross-sections (σ_P) were studied. In connection with optical properties, structural properties such as X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, FT-Raman spectroscopy and ³¹P magic angle spin nuclear magnetic resonance (MAS NMR) spectroscopy were also investigated.

Experimental

Glass preparation

A series of five (59.5NH₄H₂PO₄+15ZnO+15BaF₂+10X+0.5Dy₂O₃) different fluorophosphate (X=LiF, NaF, CaF₂, SrF₂, AlF₃) glass systems were prepared by melt quenching route. Analar grade chemicals NH₄H₂PO₄, ZnO, BaF₂, LiF, NaF, CaF₂, SrF₂, AlF₃ and Dy₂O₃ of highly purified (99.9%) were used in the glass preparation. The above glass composition each of 15g pre-weighed amounts of chemicals were rigorously pulverized in an agate mortar to mix its constituents. After this process, the fine powder is taken in a porcelain crucible which can stand high temperatures and melted in an electric furnace at a temperature range of 1120-1140oC for an hour. The melted mixture was stirred and allowed to equilibrate by the release of gas bubbles. After homogenization, the molten chemical was quickly transferred into hot square brass plates to ensure glass formation. After casting, the samples were annealed at 300oC for about 120 minutes to remove any thermal stress that might exist in the samples. Then the samples were taken to room temperature slowly. The glasses were polished properly for characterization of their physical and optical measurements. Basing on cation change, the prepared glasses are named as follows:

- 59.5NH₄H₂PO₄+15ZnO+15BaF₂+10LiF+0.5Dy₂O₃ --- LFPD
- 59.5NH₄H₂PO₄+15ZnO+15BaF₂+10NaF+0.5Dy₂O₃ --- NFPD
- 59.5NH₄H₂PO₄+15ZnO+15BaF₂+10CaF₂+0.5Dy₂O₃ --- CFPD
- 59.5NH₄H₂PO₄+15ZnO+15BaF₂+10SrF₂+0.5Dy₂O₃ --- SFPD
- 59.5NH₄H₂PO₄+15ZnO+15BaF₂+10AlF₃+0.5Dy₂O₃ --- AFPD

Measurements

The densities of the manufactured glasses were measured using Archimedes’ principle with water as fluid that gives buoyancy. Refractive indices of the glasses were measured with ATAGO model 1211 of Abbe refractometer. The physical properties of the above Dy³⁺ are calculated and are shown in Table.1

XRD profiles of these glasses were recorded in the range 10-800 with RIGAKU X-ray diffractometer. The Fourier transform infrared spectra were recorded at room temperature with 4 cm⁻¹ spectral resolution between 400 and 4000 cm⁻¹

by a BRUKER FTIR spectrometer. FT-RAMAN spectra were analysed between 50 and 1500 cm⁻¹ by a BRUKER: RFS 27 spectrometer. Solid state ³¹P MAS NMR spectra were obtained at 400 MHz using a JOEL ECX400 DELTA2 NMR spectrometer with a 4 mm probe. The acquisition time was 18 ms and pulse width was 2.9μs. The ³¹P spectra were collected in 128 scans, 5s relaxation delay. The optical absorption spectra were recorded using JASCO V-570 spectrophotometer. The fluorescence spectra were recorded with FL920 in the range of 450-700 nm, which functions with xenon lamp as excitation source.

Table 1 Physical Properties of Dy³⁺ doped different fluorophosphate glasses

S. No	property	Glass				
		LFPD	NFPD	CFPD	SFPD	AFPD
1	Density, ρ (g/cm ³)	3.036	3.067	3.136	3.187	3.012
2	Refractive index, n	1.634	1.641	1.643	1.612	1.632
3	Molar volume, V _m (cm ³ /mol)	35.83	35.98	36.37	37.60	39.63
4	Ion concentration, N _i (10 ²² ions/cm ³)	0.141	0.139	0.137	0.133	0.126
5	Inter ionic distance, r _i (nm)	1.920	1.929	1.938	1.957	1.993
6	Polaron radius, r _p (Å0)	0.773	0.778	0.781	0.788	0.799
7	Thickness,(cm)	0.335	0.346	0.350	0.345	0.336

RESULTS AND DISCUSSION

X-ray diffraction (XRD)

X-ray diffraction (XRD) delineation of the prepared 0.5mol% Dy³⁺ doped different five glass samples were recorded between 100 ≤ 2θ ≤ 800 and were juxtaposed and shown in Fig.1. The manufactured glasses had shown broad diffused scattering at low angles and did not show any sharp Bragg’s peaks. The amorphous nature of glass samples was confirmed by the X-ray diffraction pattern.

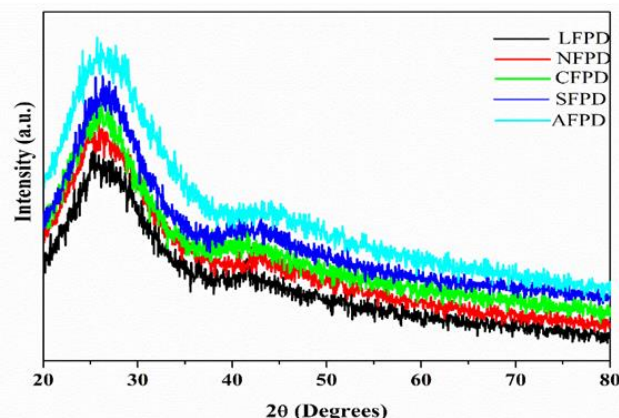


Fig 1 XRD patterns of Dy³⁺ doped different fluorophosphate glasses

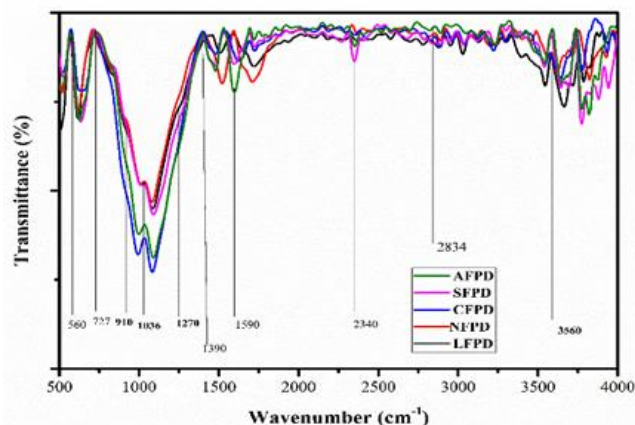


Fig 2 FTIR spectra of Dy³⁺ doped different fluorophosphate glasses

Fourier Transform infrared (FTIR) spectra

FTIR is the result of absorption of light by the vibrating molecules, it elucidates presence of various chemical units and functional groups in the prepared glass samples. FTIR spectra were recorded for the glasses and were shown in Fig. 2. The studied spectra of these glass matrices were in the wavenumber range of 500-4000 cm^{-1} . Many absorption bands around 560, 727, 910, 1036, 1270, 1390, 1590, 2340, 2834, and 3560 cm^{-1} were noticed. The ascription of the absorption bands were done by the comparison of our results with data given in the literature and the following conclusions were made. The band at 560 cm^{-1} was assigned to deformation mode in Q2 tetrahedra. The band at 727 cm^{-1} was assigned to symmetric stretching mode connections P-O bridging groups intermediate PO₂- tetrahedra Q2. The band at 910-1036 cm^{-1} was related to elongation of asymmetric links (P-O-P) between Q2 tetrahedra and elongation symmetric connections P-O terminal (Q2). The band at 1390 cm^{-1} was due to P-OH deformation. The bands in the range 2340 cm^{-1} were due to the existence of antisymmetric stretching of water molecules. The band at 2834 cm^{-1} was due to the hydrogen bonding. The bands at 3560 cm^{-1} were due to symmetric stretching vibration of hydroxyl groups (O-H) [9-13].

FT-RAMAN spectra analysis

FT Raman spectrum is due to the scattering of light by the molecules of the substance. It gives a wealth of information about the structural parameters of host glass matrix and it is shown in Fig 3 for the glasses studied.

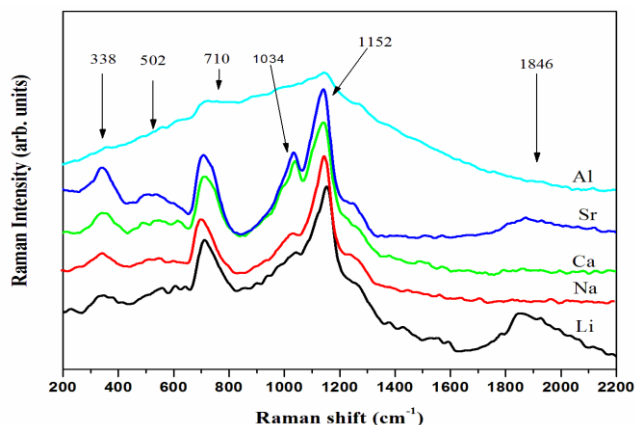


Fig 3 FT-Raman spectra of Dy³⁺ doped different fluorophosphate glasses

The scattering spectra were captured in the range of 200 cm^{-1} to 2200 cm^{-1} . In the present work six distinctive bands were noticed except for AFPD glass (very low intensities) at ~338 cm^{-1} , ~502 cm^{-1} , ~710 cm^{-1} , ~1034 cm^{-1} , ~1152 cm^{-1} , and ~1846 cm^{-1} for all the glasses. The band at 338 cm^{-1} was due to F-PO₃ scissoring mode. The band at 502 cm^{-1} was due to distortion mode of P-O and P-F vibrations. The band at 710 cm^{-1} was due to (p-o-p)sys stretching. The bands around 1030 cm^{-1} and 1152 cm^{-1} were due to symmetric stretching of nonbridging oxygen on a Q2 tetrahedron and are also due to (p-o-p)asym typical vibrational motion of PO₄ tetrahedra and the motion of cationic polyhedral. Existence of this band shows that various metal cations function as network modifiers in the fluorophosphate glass structure [14-17]. The band at 1152 cm^{-1} was the most intense one for all the glass compositions. Hence it can be inferred that addition of alkali and alkaline earth fluorides increases the (p-o-p)asym in the glass matrix.

Magic angle spin nuclear magnetic resonance (MAS NMR) analysis

³¹P MAS NMR is an invaluable tool to fathom the structures of phosphate-model glasses due to observed chemical shifts that are sensitive to the phosphorus environment. It gives information about the degree of polymerization of glass structure. The local symmetry of the atoms in the prepared/examined samples can be quantitatively studied by solid-state NMR due to its element selectivity property. The phosphate bonding is best described through Q_n species, here the superscript *n* refers to the number of bridging oxygens per tetrahedron. This categorization can be met by means of the three parameters of the chemical shift tensor. It has typical ranges of values for different Q_n groups [18-22].

Based on the bridging oxygens number with the surrounding tetrahedra, three types of tetrahedral classification can be done. They are Q₁ – phosphate tetrahedra with one bridging oxygen; Q₂ – with two bridging oxygens; and Q₃ – with three bridging oxygens. The bedrock for the phosphate glass structure is cross-linked Q₃ tetrahedron. But with the change of network modifier cations the degree of the presence of Q₃ changes leading to the formation of Q₂ and Q₁ [23-26]. This is structural changes can be studied with ³¹P NMR Spectra.

Fig. 4 shows the results of ³¹P NMR spectra of the prepared glasses. The major peaks observed are at -8.74, -22.83, -8.65, -8.65, and -22.83 for LFPD, NFPD, CFPD, SFPD and AFPD glasses respectively. In the case of LFPD glass matrix, a pronounced signal at -8.74 ppm can be assigned with Q₁ structural units. It confirms that the fluorine takes place into PO₃F tetrahedra, breaks the chains as monovalent element, and so decreases the reticulation of the tetrahedral network. Small intensity peaks observed at about -22.83 ppm are due to Q₂ pyrophosphate [27-33].

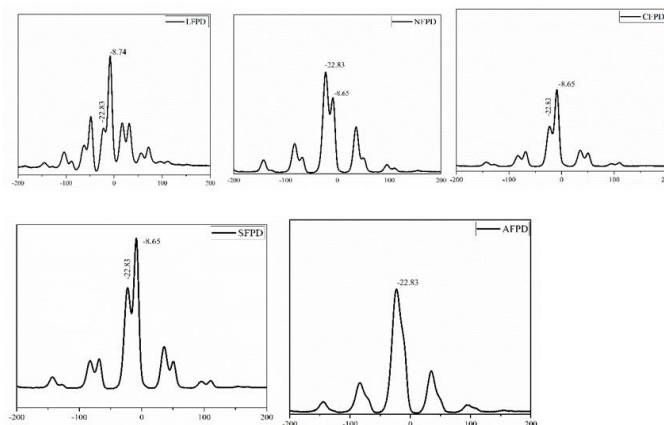


Fig 4 Chemical shifts in ³¹P MAS NMR spectra of Dy³⁺ doped different fluorophosphate Glasses.

Similar trend is observed in other two glass compositions namely CFPD and SFPD glasses. In the case of NFPD and AFPD glass matrices, major signal present at -22.83 ppm could be assigned to Q₂ tetrahedra. This clearly indicates that structural changes have happened with the change of the cations in the fluorophosphate glass network.

Absorption spectroscopy and Judd-Ofelt theory

Absorption spectral pattern of Dy³⁺ doped various fluorophosphate glass matrices were recorded in the wavelength range 300-1800 nm and were shown in two separate Figs.5(a) and 5(b). The absorption bands in Dy

3+originate from the ground state 6H15/2 to different excited states. The observed band positions were assigned using the energy levels reported by Carnal *et al* [34]. In Fig 5(a) 6P7/2, 6P5/2+6P3/2, and 4F7/2 are the dominant peaks. The spectra in the NIR region revealed that the most intense band 6F11/2+6H9/2 was centred at 1278 nm. The other notable bands are 6F3/2, 6F5/2, 6F7/2 6F9/2 +6H11/2. It was observed from Fig. 5 that, the wavelengths of the absorption peaks seldom change with cation substitution.

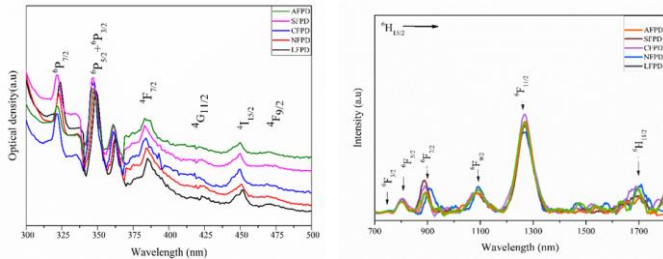


Fig 5 (a) UV-Vis and NIR optical absorption spectra of Dy³⁺ doped different fluoro phosphate glasses

Both the experimental and calculated spectral intensities (f_{exp}, f_{cal}) of 0.5 mol% of Dy³⁺ doped five glasses with root mean square deviations (δ_{rms}) were reported in Table 2. The experimental spectral intensities (f_{exp}) for the observed absorption bands were arrived at using the equation given in Ref. [35], f_{exp} positively depends on the area under absorption bands of different transitions. Whereas the theoretical spectral intensities (f_{cal}) calculations were based on Judd-Ofelt (J-O) theory [36,37]. This formula includes refractive index, Lorentz local field correction term for electric dipole transition and doubly reduced matrix elements of unit tensor operator. From Table 2 it was observed that 6F11/2 band has highest spectral intensity values 5.08, 29.00, 29.39, 29.29 and 33.06 for LFPD, NFPD, CFPD, SFPD and AFPD glasses respectively. It is also observed that among different glasses, the spectral intensities of many absorption bands were higher for AFPD glass. Rms deviation is a measure of difference between values predicted by J-O theory and the values observed via experiment. Lower is the rms deviation, good and reliable the calculation process. The rms deviations for LFPD, NFPD, CFPD, SFPD and AFPD glasses were calculated, and the values were ± 0.51 , ± 0.61 , ± 0.71 , ± 0.74 , ± 0.87 respectively. This demonstrates the accuracy of Judd-Ofelt (J-O) theory and its prediction capability.

The J-O intensity parameters, Ω_λ ($\lambda=2, 4, 6$) are widely used by the researchers to delve into short range local structure and bonding nature around rare earth ions [38]. Ω_2 gives insight about asymmetry and covalency between rare earth (RE) ions and ligand ions. Ω_4 and Ω_6 are indicative of bulk properties such as viscosity and rigidity of the glass media. When the ambience around Dy³⁺ ion has less symmetry, the field gradient environment created by ligand on the Dy³⁺ ion is felt and as a result the magnitude of induced f \rightarrow f transition is high. This results in greater spectral intensity. This greater spectral intensity is an indication of higher asymmetry and coordination environment around the Dy³⁺ ion. In order to know the influence of various cations on the local environment of Dy³⁺ ions, J-O intensity parameters were calculated by J-O theory and were presented in Table 3. It is observed that the change in the Ω_2 parameter from lithium to aluminium in these glasses is due to the change in the covalent nature bonding environment around the dysprosium and ligand

ion. It was also noticed that the decrement in the Ω_2 parameter from lithium to aluminium This decrement is due to the change of effective ionic radii of lithium (0.60nm) and aluminium(0.054nm)

All the glasses commonly have displayed larger Ω_2 values than Ω_4 and Ω_6 . The pattern observed in these glasses was $\Omega_2 > \Omega_4 > \Omega_6$ except in NFPD glass where $\Omega_2 > \Omega_6 > \Omega_4$ is noticeable. It was evident from Table 3 that in the present work i.e. for 0.5mol% of Dy³⁺ doped fluorophosphate glasses, Ω_2 parameter decreased from $18.72 \times 10^{-20} \text{ cm}^2$ to $12.04 \times 10^{-20} \text{ cm}^2$ for Li to Al in proper order. The Ω_2 values of the present work are found to be higher than CBTZnDy glasses [39], (NaPO₃)₆-TeO₂-AlF₃-LiF glasses [40] and NbLiFsDy glasses [41]. Greater Ω_2 value indicates greater covalency, hence LFPD glass has more covalent nature than the AFPD glass. Ω_4 reflects the bulk property and viscosity. The values came out to be 5.89, 3.52, 5.17, 4.9, and 5.98 with their usual unit ($\times 10^{-20} \text{ cm}^2$). For the first and the last glasses namely LFPD and AFPD were observed to have higher values. Of these AFPD has highest value. So, it is concluded that AFPD glass has high viscous nature among all glass samples. Ω_6 which gives insight into rigidity is observed to have lowest value 2.45 for AFPD glass. It is known from the literature that Ω_6 has inverse relation to viscosity [42]. Hence it can be concluded that AFPD glass has high rigidity.

The crux of the matter is that, the intensities of f-f transitions are usually insensitive to the glass environment. This is because of the shielding effect created by the closed shell 5s and 5p electrons. Despite this, there are some transitions whose intensities are highly sensitive to any change in the surrounding environment. These transitions are called “hypersensitive transitions”. HST transitions follow the selection rules $|\Delta J| \leq 2$, $|\Delta L| \leq 2$, and $\Delta S = 0$, It has large reduced matrix elements $\|U\lambda\|^2$. Usually, the changes that occur in the intensity and band shapes of HST due to the change in the environment are useful in ascertaining the changes in the structure around lanthanide ion. In the present work, 6H15/2 \rightarrow 6F11/2+6H9/2 was centred at 1278 nm, higher spectral intensity of hypersensitive transition is observed for AFPD glass among all the five prepared glass compositions. This is due to the higher magnitude of the electrostatic attraction between dysprosium ion and the ligand in the said composition.

Radiative properties & Luminescence spectra

In order to calculate radiative properties like electric dipole line strengths Sed, radiative transition probabilities, Aed, branching ratios, β and absorption cross sections, Σ of certain excited states 4I15/2, 4F9/2, 6F3/2, 6F5/2 and 6F11/2+6H9/2 of Dy³⁺, J-O theory comes handy. Using this theory, these physical quantities were assessed from the absorption data and refractive indices for all the prepared Dy³⁺ doped FP glasses using the formulae given in ref [43-46]. The branching ratios (β_R) and absorption cross-sections (Σ) of certain transitions of 0.5 mol% of Dy³⁺ doped FP sample glasses were calculated and presented in Table 4. It was found that among different transitions, 4F9/2 \rightarrow 6H13/2, transition exhibits higher branching ratio β consistently for all the samples and among all these glasses LFPD glass has highest β (83.5%) followed by CFPD glass with (76.8%).

Table 4 Certain radiative parameters of Dy³⁺ doped different fluorophosphate glasses. (Electric dipole line strengths (S_{ed}), radiative transition probabilities (A_{ed}) sec⁻¹, branching ratios (β %) and absorption cross sections (Σ (10⁸) cm²).

Glass	Transition	S _{ed}	A _{ed}	β	Σ
LFPD	4I _{15/2} →6H _{15/2}	3.19	62.67	57.00	0.06
	4F _{9/2} →6H _{13/2}	22.25	342.30	83.50	0.54
	6F _{3/2} →6H _{9/2} +6H _{7/2}	109.16	55.10	65.40	1.57
	6F _{5/2} →6H _{15/2}	156.30	73.30	46.40	1.67
	6F _{11/2} +6H _{9/2} →6H _{15/2}	469.26	190.10	91.80	3.01
NFPD	4I _{15/2} →6H _{15/2}	52.71	1036.17	55.90	1.03
	4F _{9/2} →6H _{13/2}	167.05	2613.4	67.00	4.13
	6F _{3/2} →6H _{13/2}	215.08	1405.2	32.50	7.33
	6F _{5/2} →6H _{15/2}	187.63	1755.9	37.00	5.50
	6F _{11/2} +6H _{9/2} →6H _{15/2}	3511.20	16429.5	59.60	51.45
CFPD	4I _{15/2} →6H _{15/2}	34.45	687.98	53.20	0.68
	4F _{9/2} →6H _{13/2}	176.87	2746.40	76.80	4.37
	6F _{3/2} →6H _{13/2}	70.78	466.20	25.70	2.42
	6F _{5/2} →6H _{13/2}	210.7	707.00	0.32	4.39
	6F _{11/2} +6H _{9/2} →6H _{15/2}	3631.78	1480.90	0.93	23.53
SFPD	4I _{15/2} →6H _{15/2}	52.95	978.06	63.50	0.97
	4F _{9/2} →6H _{13/2}	194.23	2847.3	74.10	4.51
	6F _{3/2} →6H _{13/2}	177.43	1097.7	41.30	5.69
	6F _{5/2} →6H _{15/2}	154.79	1365.7	43.8	4.26
	6F _{11/2} +6H _{9/2} →6H _{15/2}	3657.78	1412.5	92.8	22.36
AFPD	4I _{15/2} →6H _{15/2}	78.91	1519.48	69.00	0.50
	4F _{9/2} →6H _{13/2}	226.93	3458.10	70.90	0.17
	6F _{3/2} →6H _{13/2}	322.36	2057.40	47.50	10.75
	6F _{5/2} →6H _{15/2}	281.23	2587.20	51.90	8.09
	6F _{11/2} +6H _{9/2} →6H _{15/2}	3989.84	1582.90	91.40	25.34

The radiative transition probability (A_{ed}) values for 6F_{13/2} transition came out to be 342.3, 2613.4, 2746.4, 2847.3, and 3458.1 for LFPD, NFPD, CFPD, SFPD and AFPD glasses respectively. For the aluminium glass, A_{ed} value came out to highest. The branching ratios were consistently higher for the 4F_{9/2} → 6H_{13/2} transition [47-50].

The excitation spectra of Dy³⁺-doped different FP glass samples were documented in the wavelength range 300 to 550 nm at an emission wavelength of 570 nm. Fig.6 shows excitation spectrum of LFPD glass matrix.

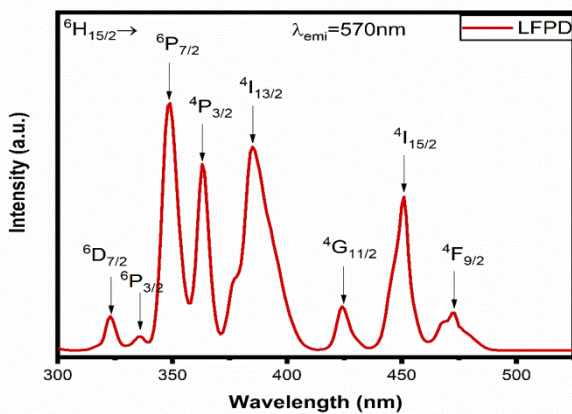


Fig 6 Excitation spectrum of Dy³⁺ doped different fluorophosphate glasses

The other spectra resembled exactly, hence we did not show them here. The excitation spectrum consists of eight inhomogeneous excitation peaks located at 325 nm, 341nm, 350 nm, 363 nm, 385 nm, 425 nm, 450 nm, and 475 nm and are related to the transitions from ground state 6H_{15/2} to the excited states 6D_{7/2}, 6P_{3/2}, 6P_{7/2}, 4P_{3/2}, 4I_{13/2}, 4G_{11/2} and 4I_{15/2} and 4F_{9/2} respectively. Among these excitation peaks, the excitation peak at 350 nm (6H_{15/2}→6P_{7/2}) was relatively high in intensity and this was further used for the measurements of luminescence spectra [51-54].

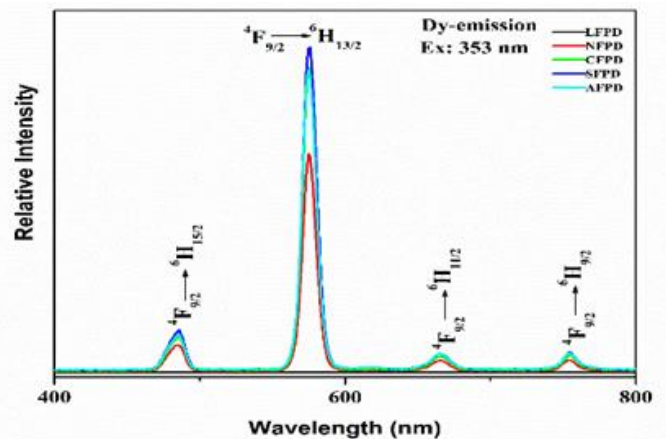


Fig 7 Emission spectra of Dy³⁺ doped different fluorophosphate glass

Table 5 Branching ratios (β_{cal}, β_{exp}) and calculated lifetimes of certain transitions

	4F _{9/2} →6H _{13/2}	
	β _{cal}	β _{exp}
LFPD	78	81
NFPD	80	75
CFPD	81	81
SFPD	82	81
AFPD	81	83

Fig. 7 show the visible photoluminescence (PL) spectra of Dy³⁺ doped different fluoro phosphate glass matrices observed in the wavelength range of 400-800 nm by excitation at a wavelength(λ_{exc}) 353 nm. The emission spectra exhibiting four remarkable emission transitions of which, three in the visible range and one in NIR region 4F_{9/2}→6H_J (J=15/2, 13/2, and 11/2) which were observed at 485, 575 and 667 nm respectively. The blue (B) emission transition (4F_{9/2}→6H_{15/2}) at 485 nm and yellow (Y) emission transition (4F_{9/2}→6H_{13/2}) at 575 nm were having high intensity than the other red emission transition (4F_{9/2}→6H_{11/2}) at 667 nm wavelength.

Table 6 Emission properties of Dy³⁺ doped different fluoroPhosphate glasses for observed transitions (peak emission wavelength, λ_p (nm), effective bandwidths, $\Delta\lambda_{eff}$ (cm⁻¹), stimulated emission cross sections, σ_e (10⁻²²cm²), and optical gain bandwidths, $\sigma_e \times \Delta\lambda_{eff}$ (10⁻²¹cm³).

Transition→	4F _{9/2} →6H _{15/2}				4F _{9/2} →6H _{13/2}				4F _{9/2} →6H _{11/2}			
	Glass	λ_p	$\Delta\lambda_{eff}$	σ_e	$\sigma_e \times \Delta\lambda_{eff}$	λ_p	$\Delta\lambda_{eff}$	σ_e	$\sigma_e \times \Delta\lambda_{eff}$	λ_p	$\Delta\lambda_{eff}$	σ_e
LFPD	485	13.7	1.39	19.0	575	11.3	16.46	185.9	667	14.5	3.75	54.4
NFPD	485	13.7	18.5	253.4	575	11.2	137.4	1539	667	14.9	29.1	433
CFPD	485	13.3	6.15	81.8	575	11.5	131.6	1513	667	14.5	37.2	539
SFPD	485	13.7	6.92	94.8	575	11.4	130.6	1489	667	15.0	37.3	559
AFPD	485	13.6	8.52	115.9	575	11.4	135.6	1546	667	15.2	28.9	439

The blue emission transition (4F_{9/2}→6H_{15/2}) was due to magnetic dipole (MD) transition, whose intensity typically does not depend on the short crystal field environment of the prepared glass matrices [55-57]. It is worth noting that the yellow emission transition (4F_{9/2}→6H_{13/2}) was an electric dipole (ED) transition ($\Delta L=2, \Delta J=2$). This transition is a hypersensitive emission transition. The intensity of this transition depends on local environment.

In the present work, for 0.5 mol% of Dy³⁺ doped fluoro phosphate glasses, the emission intensity of the peaks increased with the increase of ionic state. The luminescence properties such as peak emission wavelengths (λ_p), effective bandwidths ($\Delta\lambda_{eff}$), branching ratios (β) (experimental and calculated) and peak emission cross-sections (σ_p) for the three emission transitions 4F_{9/2}→6H_{15/2}, 4F_{9/2}→6H_{13/2}, and 4F_{9/2}→6H_{11/2} were calculated and were presented in Table 5. The stimulated emission cross sections which decide the threshold and efficiency for a given transition in a laser material were calculated. The highest value obtained is 16.96x10⁻²⁴cm² for AFPD glass composition. The branching ratio values of any transition are measure of lasing action. It was observed from the table that the branching ratio (β) of the emission transition 4F_{9/2}→6H_{13/2} was high. It was also observed from the table that, the experimental and calculated β values were in close agreement for 4F_{9/2}→6H_{13/2} which happened to be Electric dipole transition. The stimulated emission cross-section (σ_p) is an important measurement used to identify a medium as laser active with directly proportional relation. In the current endeavour, the peak emission cross-section (σ_p) was found higher for the transition 4F_{9/2}→6H_{13/2} than the other two emission transitions 4F_{9/2}→6H_{11/2} and 4F_{9/2}→6H_{15/2}. As seen from Table 5, the value of emission cross-section has increased up to 168.43x10⁻²⁴cm² with the variation of Li, Na, Ca, Sr, and Al. Hence, AFPD glass matrix can be used as viable alternative lasing material.

CIE coordinates and generation of yellowish green light

The colour of any light source can best be described and classified by three variables $\bar{X}(\lambda)$, $\bar{Y}(\lambda)$ and $\bar{Z}(\lambda)$ which are dimensionless quantities. To estimate the emission colour of the glass matrices, the Commission International de l'Eclairage (CIE) chromaticity coordinates are determined from the tri stimulus values using the following standard relations,

$$x = \frac{\bar{X}}{\bar{X} + \bar{Y} + \bar{Z}} \quad y = \frac{\bar{Y}}{\bar{X} + \bar{Y} + \bar{Z}}$$

and $\bar{Z}(\lambda)$ representing the relative brightness. The locus of all monochromatic colour coordinates makes the perimeter of CIE 1931 chromaticity diagram. Colour coordinates of Dy³⁺ doped fluoro-phosphate glass matrix with different metal cations are shown in Table.

Fig 8 represents the colour chromaticity diagram of Dy³⁺ doped Fluoro-phosphate glasses. In the present study, x and y colour co-ordinates (0.47, 0.48) are located in the yellowish green region for all the glass matrices

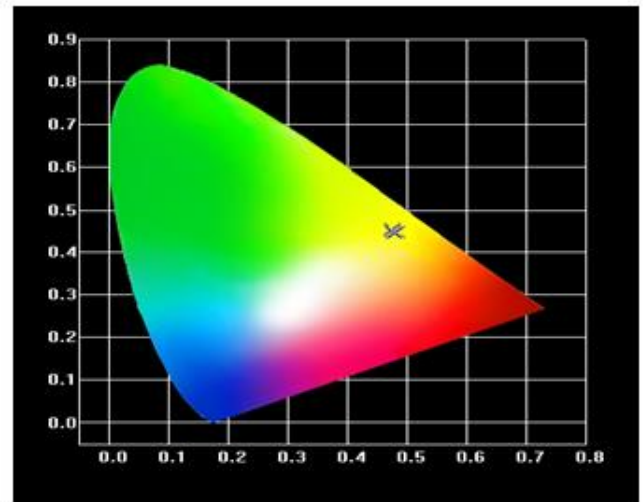


Fig 8 CIE chromaticity diagram for Dy³⁺ doped different fluorophosphate glasses

From the emission spectra of Dy³⁺ doped FP glasses, yellow to blue transitions ratio (Y/B) values were calculated and were presented in Table 6. These values were 1.91, 1.80, 1.82, 1.78 and 1.76 for LFPD, NFPD, CFPD, SFPD and AFPD glasses respectively. Higher the value of Y/B ratio indicates higher degree of covalency. From the values of Y/B ratios, it was noticed that LFPD glass matrix has higher degree of covalency [58-59].

Table 7 The calculated chromaticity colour co-ordinates (x, y), CCT and Y/B ratio values of Dy³⁺ ions activated FP glasses excited at a wavelength of 353 nm.

Sample	Color coordinates (x, y)	CCT (k)	Y/B
LFPD	(0.486, 0.457)	2702	1.91
NFPD	(0.473, 0.456)	2855	1.80
CFPD	(0.472, 0.451)	2834	1.82
SFPD	(0.466, 0.446)	3012	1.78
AFPD	(0.465, 0.456)	2960	1.76

CONCLUSIONS

Different alkali alkaline and aluminium fluorophosphate glasses with 0.5mol% Dy³⁺ doping, were produced by melt quenching process. These glass samples were characterized by XRD, FT-IR, FT-RAMAN, 31P MAS NMR, Optical absorption, luminescence, and emission measurements. The XRD has reaffirmed the amorphous nature of prepared glass samples. The various structural groups present in the prepared glass samples were identified by FTIR and FT Raman. From 31P MAS NMR spectra, it was detected the presence of meta

phosphate units, and pyrophosphate structures in the sample. J-O intensity $\Omega\lambda$ ($\lambda=2, 4$ and 6) parameters and radiative parameters (AT, τ_R , β and Σ) were calculated for prepared fluorophosphate glasses. Among the three intensity parameters, LFPD glass matrix had higher magnitude (18.72×10^{-20} cm²) of Ω_2 parameter which could be attributed to higher covalency and higher asymmetry of ligand field around the Dy³⁺ ion. AFPD glass has exhibited lower Ω_2 value (12.04×10^{-20} cm²). From Li to Al the Ω_2 values have decreased indicating decrease in covalency nature and increase in ionic nature. The Y/B values corroborate this conclusion. This could be due to increase in the charge of cation that replaced earlier one. Ω_4 and Ω_6 which reflect the bulk properties such as viscosity and rigidity of the glass matrix have scattered values. Emission properties of all the excited levels of Dy³⁺ were studied. Branching ratio values which play an important role in attaining stimulated emission were found to be higher for the transition $4F_9/2 \rightarrow 6H_{13/2}$ of LFPD glass matrix (83.5%). So, it is useful for laser applications. From the colour chromaticity diagram, it was concluded that these glasses are quite suitable for greenish yellow emission.

Declaration of Competing Interests

Title of Manuscript: "Delving into the Spectroscopic properties of Dy³⁺ in different fluorophosphate glasses for multitude of applications

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The authors declare that, they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. We have authority over manuscript preparation and decisions to submit the manuscript for publication.

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