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# The Effect of Sodium Fluoride in Lithium Fluorophosphate (LFP) Glasses Doped with $Nd_2O_3$ Ion

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**ABSTRACT**
The lithium fluorophosphate glasses doped with neodymium oxide were synthesized by melt quenching technique with chemical composition $20Li_2O–10AlF_3–69P_2O_5–1Nd_2O_3$ and $20Li_2O–10AlF_3–10NaF–59P_2O_5–1Nd_2O_3$. The glasses after synthesizing were investigated through physical, FTIR and absorption spectra. The density and refractive index decrease with addition of sodium fluoride content inside the glass sample. The FTIR spectra of the glasses showed mainly of $[PO_3]$ and $[PO_4]$ structural units. The absorption spectra were investigated in the UV–vis–NIR region from 300–2000 nm. The optical bandgap energy ($E_{opt}$) was observed to decrease with addition of sodium fluoride content in glass. The near-infrared spectra are investigated using NIR luminescence spectroscopy.

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Fluorophosphate glass; neodymium oxide; physical; FTIR; optical properties; NIR luminescence

## 1. Introduction

The research focuses on fluorophosphate glass system because of their potential as laser host matrix [1]. Fluorophosphate glasses are attractive due to their promising properties such as low phonon energy [2], low nonlinear refractive index [3], good transparency in UV–vis–NIR [4], and high emission cross-section [5]. Phosphate glasses based on the alkali metaphosphates are hygroscopic [6], but the addition of fluorides increases the resistance to water [1]. Lithium oxide ($Li_2O$) is added to modify the glass structure, which improve chemicals stability [7] and reduce melting point [8], metal fluorides ($AlF_3$) also improves the IR cutoff edge toward longer wavelengths [8, 9]. The phosphate glasses with fluorine find different applications in high power laser, vitrification of radioactive waste, fast ion conductor and glass to metal seals [7]. Phosphate glass doped with neodymium ($Nd^{3+}$) ion show potential use as laser glasses [2, 10–13]. The $Nd^{3+}$ doped with phosphate glass is promising in application such as the optical communication at infrared (IR) wavelength range with energy transitions $^4F_{3/2} \rightarrow {}^4I_{11/2}$ [12, 14]. The concentration quenching in near-infrared luminescence of $Nd^{3+}$ doped glass at

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1.00 mol% had reported many researchers [10, 12, 15–19]. Then 1.0 mol% of $Nd_2O_3$ concentration has been chosen in this research to study the present Nd-doped glass.

The aim of the present work is to study the effect of sodium fluoride (NaF) to $Nd^{3+}$ doped lithium fluorophosphate glass. The physical, FTIR, optical and luminescence properties of the glass samples were studied to determine the effect of the addition of NaF. From this study, we expect that the addition of NaF to $Nd^{3+}$ doped lithium fluorophosphate glass matrix has an effect in enhancing its function as a laser medium.

## 2. Experiment

In the present work, added alkali fluoride glasses were prepared using standard melt quenching technique. The glass compositions investigated in this work are as follows: $20Li_2O-10AlF_3-69P_2O_5-1Nd_2O_3$ and $20Li_2O-10AlF_3-10NaF-59P_2O_5-1Nd_2O_3$. The oxide mixtures with 15 g batch composition were transferred into porcelain crucible and melted at 1100 °C for 2 h, then the melted sample was poured into preheated graphite steel molds. The glass sample was annealed at 350 °C for 3 h to remove the thermal stresses produced during the quenching. After that, transparent glass samples were cut to the same size and optically polished to maintain a smooth surface. The result can be seen in Figure 1. Density of the glasses was established through Archimedes's principle. The refractive index was measured by Abbe refractometer at sodium wavelength (589 nm) and using mono bromonaphthalene ($C_{10}H_7Br$) as a contact liquid. FTIR spectra were recorded using Cary 630 FTIR Agilent technologies. The absorption spectra were measured with UV–vis–NIR spectrophotometer (Shimadzu, UV-3600) in the range 300–2000 nm. Near-infrared luminescence spectra were recorded at room temperature using a spectrophotometer under the excitation wavelength at 581 nm.

## 3. Results and discussion

### 3.1. Physical properties

The physical properties used in this work were explained in literature [20, 21]. From Table 1, the density and molar volume of the glass decrease with add sodium fluoride to $Nd^{3+}$ doped glass. This can be explained on the basis that the molecular weight of NaF is 41.98 g/mol which is low as compared without NaF. This reduction may be attributed to the creation of non-bridging oxygens and breaking of covalent bond between phosphorus and oxygen atom at a concentration of 1.00 mol% $Nd_2O_3$. Polaron


Figure 1. $Nd^{3+}$ doped lithium fluorophosphate glasses system.

Table 1. Physical properties of $Nd^{3+}$ doped lithium fluorophosphate.

| Physical properties | Glass samples | |
|---|---|---|
| | Without NaF | With NaF |
| Molecular weight, $M$ (g) | 115.6833 | 105.6875 |
| Density, $\rho$ (g/cm$^3$) | 2.5564 | 2.5478 |
| Molar volume, $M_V$ (cm$^3$/mol) | 45.2524 | 41.4819 |
| Thickness (cm) | 0.30 | 0.33 |
| Ion concentration, $N$ ($\times 10^{22}$ ion/cm$^3$) | 1.3308 | 1.4518 |
| Polaron radius, $r_i$ (Å) $\times 10^{-8}$ | 1.7003 | 1.6517 |
| Inter ionic distance, $r_p$ (Å) $\times 10^{-8}$ | 4.2198 | 4.0992 |
| Field strength, $F$ ($\times 10^{17}$ cm$^{-2}$) | 1.2546 | 1.3377 |
| Refractive index, $n$ | 1.5258 | 1.5231 |
| Molar refractivity, $R_m$ (cm$^{-3}$) | 13.8857 | 12.6746 |
| Electronic polarizability, $\alpha_e$ ($\times 10^{-24}$ cm$^3$) | 5.5076 | 5.0272 |
| Metallization criterion (M) | 0.6932 | 0.6945 |
| Dielectric constant, $\varepsilon$ | 2.3281 | 2.3199 |
| Reflection losses, R (%) | 4.3335 | 4.2988 |

radius is observed to decrease with the addition of NaF and results in high field strength (F) around $Nd^{3+}$ ion. This is an effect of an increase in ion concentration ($N$) on the addition of NaF. The decrease in inter ionic distance $r_i$ with addition of NaF may be suggesting that the atom in the present investigated with NaF glass is packed more tightly with each other. While the decreasing of the refractive index can be explained by the classic dielectric theory, the refractive index of glass decreases with decreasing of its density. According to [18], the refractive index and molar refraction have a closed relationship with electronic polarizability. The electronic polarizability reduces with addition of NaF since it is inversely proportional to the number of oxide ions, suggesting that the investigated NaF glass is more stable. The metallization criterion increase for the addition of NaF, if the value is more than 0.5 thereby suggesting their behavior lies between insulator and conductor [19]. Other physical properties such as dielectric constant and reflection loss also decrease with add of NaF, which can be seen in Table 1.

### 3.2. FTIR spectra

Figure 2 shows Fourier transform infrared (FTIR) spectra of $Nd^{3+}$ doped lithium fluorophosphate glasses. The band assignments of studied glass are presented in Table 2. The vibrational intensity was correlated based on the results obtained by the spectra which were compared with the reported literature [2, 5, 13, 24–27]. The strong intensity vibrational lines are observed between 895 and 1262 cm$^{-1}$, as we can see that the addition of NaF shows higher intensity than the glass sample without NaF. The effect comes from the addition of fluoride content in the glass sample [21], hence $(P–O)^-$ ionic vibration is visible at 1152 cm$^{-1}$, this vibration is intense in the addition of NaF than without NaF. The wavenumber at 2000–3500 cm$^{-1}$ is arising due to hydroxyl vibrations of OH groups, the addition of NaF makes low intense bands. The addition of alkali oxide such as lithium oxide to phosphate glass degrades the structure, such linear chain degradation in their structure results in the creation of non- bridging oxygen's (NBO's) [21].

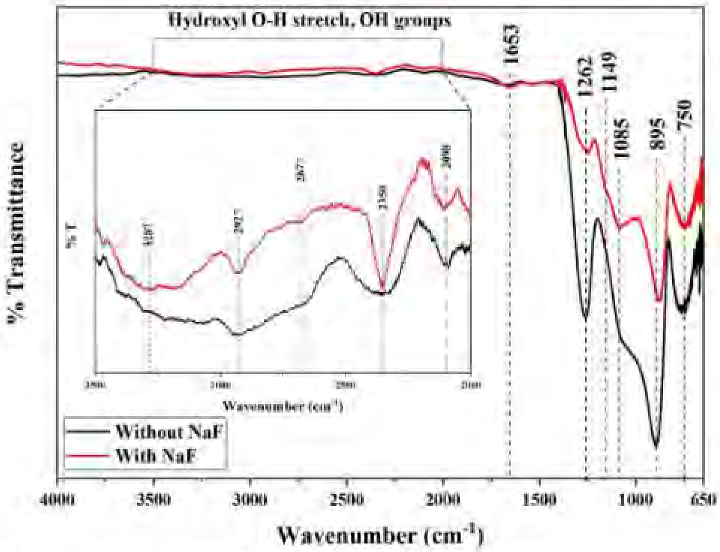


**Figure 2.** FTIR of $Nd^{3+}$ doped lithium fluorophosphate glasses system.

**Table 2.** FTIR of $Nd^{3+}$ doped lithium fluorophosphate.

| | | Wavenumber ($cm^{-1}$) | |
|---|---|---|---|
| Band assignment | Range ($cm^{-1}$) | Without NaF | With NaF |
| Symmetric stretching vibration mode of P–O–P bridging oxygen linked pyrophosphate $(P_2O_7)^{4-}$ and $P_2(O,F)_7$ [22] | 750–780 | 755 | 750 |
| Asymmetric stretching vibration of P–O–P linked with linear meta-phosphate chain and P-F groups [2, 5] | 830–940 | 895 | 880 |
| Symmetric stretching vibration mode of non-bridging oxygen $[P–O]^-$ group of $PO_4$ tetrahedra [11] | 1030–1090 | 1085 | 1085 |
| Symmetric stretching vibration modes of P = O and $(P–O)^{(-)}$ ionic vibration [21] | 1233–1270 | 1262 | 1244, 1154 (less intense) |
| Bending vibrations of H–O–H and P–OH in the network [20] | 1600–1660 | 1669 | 1653 |
| Hydroxyl O–H stretch, OH groups [23] | 2000–3500 | 2098, 2350, 2676, 2927, 3287 | |

### 3.3. Absorption spectra and optical bandgap

The absorption spectra of lanthanide ions in the visible and near-infrared region are known to arise due to 4f–4f transition. Twelve absorption bands have been observed as shown in Figure 3 with the addition of NaF in $Nd^{3+}$ doped lithium fluorophosphate glass. The assignment of these bands from ground state $^4I_{9/2}$ to the various excited state, i.e. $^4D_{1/2}$ (352 nm), $^2P_{1/2}$ (431 nm), $^4G_{11/2}$ (461 nm), $^2G_{9/2}$ (475 nm), $^4G_{9/2}$ (512 nm), $^4G_{7/2}$ (525 nm), $^4G_{5/2}$ (581 nm), $^2H_{11/2}$ (628 nm), $^4F_{9/2}$ (683 nm), $^4F_{7/2}$ (747 nm), $^4F_{5/2}$ (805 nm) and $^4F_{3/2}$ (875 nm) [2, 19, 28, 29]. From Figure 3, the spectra reveal strong absorption bands in the visible region at 581 nm ($^4I_{9/2} \rightarrow {}^4G_{5/2}$) which is known as a hypersensitive transition, because of its strong dependency on the environment of the

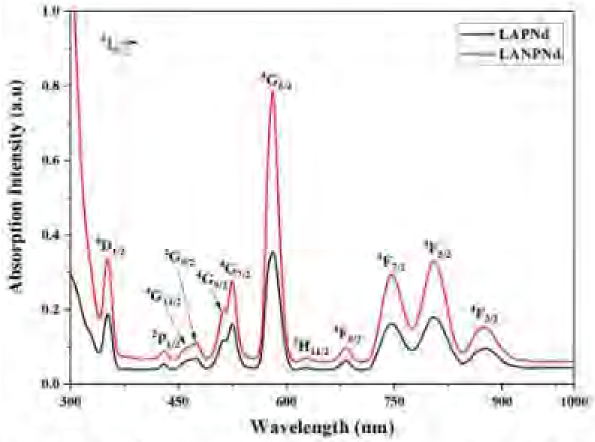


Figure 3. Absorption spectra of $Nd^{3+}$ doped lithium fluorophosphate glasses system.

neodymium ions [26]. Furthermore, when adding NaF to $Nd^{3+}$ doped with lithium fluorophosphate glass which increases the intensity of absorption. To analyze the optical transition and the electronic band structure of the crystalline and amorphous materials, we use the fundamental absorption edge of the absorption spectra. The optical bandgap in this work were also applied and explained in literature [30–32]. Figure 4 shows the tauc's plot for the indirect allowed transition of the $Nd^{3+}$ doped lithium fluorophosphate glasses (the inset shows the dependence of $(\alpha h\nu)^2$ on photon energy ($h\nu$) for the direct transition). The direct bandgap and indirect bandgap values without NaF are found in the range of 3.79 and 3.57. The direct bandgap and indirect bandgap values with NaF are found in the range of 3.75 and 3.52. From these values can be confirmed, the optical bandgap decreases with the addition of NaF to $Nd^{3+}$ doped lithium fluorophosphate.

### 3.4. Nephelauxetic effect

To understand the covalent or ionic ligand bonding, bonding parameter ($\delta$) and nephelauxetic ratio ($\beta$) it is evaluated quantitatively by using the equation

$$\beta = \frac{\nu_c}{\nu_a} \tag{1}$$

Here, $\nu_c$ is wavenumber of particular transition under investigation, $\nu_a$ is wavenumber of same transition of an aqua ion [28].

$$\delta = \frac{(1-\overline{\beta})}{\overline{\beta}} \tag{2}$$

The $\delta$ parameter can be negative or positive indicating ionic or covalent bonding between Nd-O bonds which strongly depends upon the ligand field environment around the $Nd^{3+}$ ions. From Table 3, it is found that the '$\delta$' value is positive and confirms the $Nd^{3+}$ doped lithium fluorophosphate has a covalent bonding nature [29].

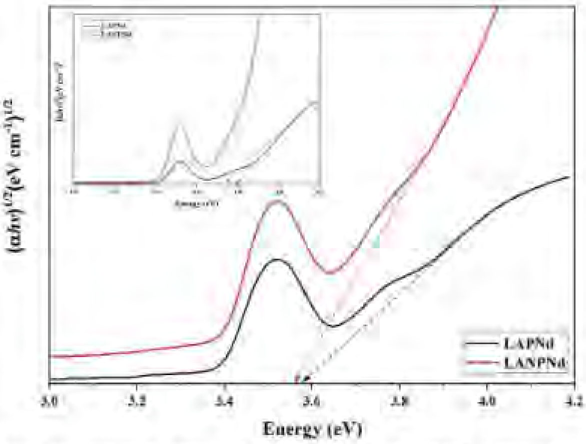


**Figure 4.** Tauc's plot for the indirect allowed transition of the $Nd^{3+}$ doped lithium fluorophosphate glasses. (The inset shows the dependence of $(\alpha h\nu)^2$ on photon energy ($h\nu$) for the direct transition.)

**Table 3.** Judd–Ofelt parameters, experimental ($f_{exp} \times 10^{-6}$) and calculated ($f_{cal} \times 10^{-6}$) oscillator strength for $Nd^{3+}$ doped lithium fluorophosphate glass sample.

| | | | Without NaF | | With NaF | |
|---|---|---|---|---|---|---|
| Transitions $^4I_{15/2}\rightarrow$ | $\lambda_{abs}$ (nm) | Energy ($cm^{-1}$) | $f_{exp}$ | $f_{cal}$ | $f_{exp}$ | $f_{cal}$ |
| $^4D_{1/2}$ | 352 | 28409.09 | 6.6501 | 4.6787 | 6.6706 | 4.9408 |
| $^2P_{1/2}$ | 431 | 23201.86 | 0.5534 | 0.5412 | 0.5991 | 0.5715 |
| $^4G_{11/2}$ | 461 | 21691.97 | 0.9674 | 0.2447 | 1.1732 | 0.3475 |
| $^2G_{9/2}$ | 475 | 21052.63 | 1.2932 | 0.4991 | 1.3907 | 0.3372 |
| $^4G_{9/2}$ | 512 | 19531.25 | 2.4451 | 1.5886 | 2.4977 | 2.1287 |
| $^4G_{7/2}$ | 525 | 19047.62 | 3.9146 | 3.5219 | 5.8030 | 4.7732 |
| $^4G_{5/2}$ | 581 | 17211.70 | 13.6134 | 13.6379 | 22.2354 | 22.2919 |
| $^2H_{11/2}$ | 628 | 15923.57 | 0.1438 | 0.192 | 0.2815 | 0.2885 |
| $^4F_{9/2}$ | 683 | 14641.29 | 0.5114 | 0.6967 | 0.7705 | 1.0556 |
| $^4F_{7/2}$ | 747 | 13386.88 | 4.8982 | 3.1368 | 6.9321 | 4.9288 |
| $^4F_{5/2}$ | 805 | 12422.36 | 5.7241 | 6.7558 | 8.4796 | 9.6718 |
| $^4F_{3/2}$ | 875 | 11428.57 | 2.0621 | 2.2861 | 2.6068 | 2.7362 |
| | $\Delta_{rms}$ | | ±0.92 | | ±0.99 | |
| | $\Omega_2$ ($\times 10^{-20}$ $cm^2$) | | 3.74 | | 7.27 | |
| | $\Omega_4$ ($\times 10^{-20}$ $cm^2$) | | 4.29 | | 4.54 | |
| | $\Omega_6$ ($\times 10^{-20}$ $cm^2$) | | 6.68 | | 10.55 | |
| | $\beta$ | | | 0.9992 | | |
| | $\delta$ | | | 0.0008 | | |

### 3.5. Near infrared emission (NIR) spectra

The NIR emission spectra for $Nd^{3+}$ doped lithium fluorophosphate are shown in Figure 5 in the wavelength range 1000–1500 nm under the excitation at 581 nm. The peaks corresponding to $^4F_{3/2} \rightarrow {}^4I_{9/2}$, $^4F_{3/2} \rightarrow {}^4I_{11/2}$, and $^4F_{3/2} \rightarrow {}^4I_{13/2}$ are observed at 903, 1062, and 1330 nm, respectively. The NIR emission spectra shows a faint peak at a wavelength of 903 nm appear in the sample without NaF glass. Meanwhile, [12] the wavelength at transition $^4F_{3/2} \rightarrow {}^4I_{9/2}$ is very low but in many references [2, 30] it does not show any peak at this transition $^4F_{3/2} \rightarrow {}^4I_{9/2}$. This situation occurs due to the effect of adding fluoride to the glass sample which affects and enhance this transition at $^4F_{3/2} \rightarrow {}^4I_{9/2}$ to appear and sometimes undetectable based on host matrix. The dominant laser emission peak corresponds to $^4F_{3/2} \rightarrow {}^4I_{11/2}$ (1062 nm), due to high intensity than other bands. It can be seen from Figure 5, when the addition of NaF inside the glass, the luminescence

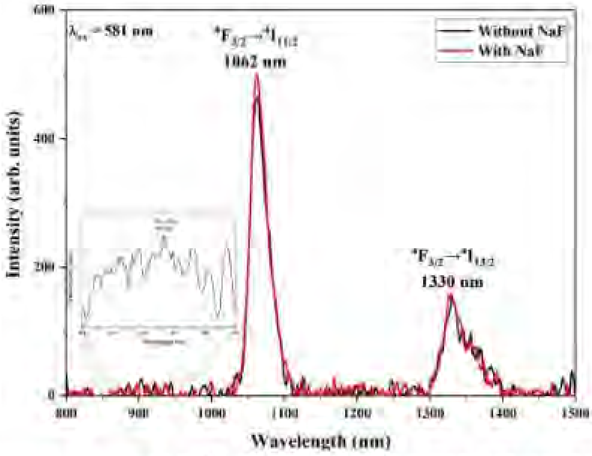


Figure 5. NIR luminescence spectra of $Nd^{3+}$ doped lithium fluorophosphate glasses system.

is higher. This is due to effect of adding more fluoride content in the host matrix, as we have known that addition of fluoride reduces phonon energy in the host glass.

### 3.6. Judd–Ofelt parameter and radiative properties

The best set of Judd–Ofelt (JO) intensity parameters ($\Omega_2$, $\Omega_4$, and $\Omega_6$) is obtained using the following formula in Ref. [31] and is presented in Table 3 for both the glass sample. The JO parameters for sample without NaF glass are found to be $\Omega_2 = 3.74$, $\Omega_4 = 4.29$, $\Omega_6 = 6.68$, and follows the trend $\Omega_6 > \Omega_4 > \Omega_2$. The JO parameters for sample with NaF glass are found to be $\Omega_2 = 7.27$, $\Omega_4 = 4.54$, $\Omega_6 = 10.55$, and follows the trend $\Omega_6 > \Omega_2 > \Omega_4$. The value indicates viscosity and rigidity of glass are higher [32]. The result is well-related to the value of bonding parameter ($\delta$) and nephelauxetic ratio ($\beta$) which is discussed that the $Nd^{3+}$ doped lithium fluorophosphate has covalent bonding. As we can see the sample with NaF glass has more covalent bonding nature than in without NaF glass. The JO values are evaluated and compared with other reported $Nd^{3+}$ doped glasses shown in Table 4. As can be referenced from the table, some of these parameters fall within the range of values commonly reported for the $Nd^{3+}$ doped glass system. Some of the values shown for other reported $Nd^{3+}$ doped glasses are comparable and the present fluorophosphate glass shows higher than the reported glass which is presented in Table 4.

The JO parameters are used to determine the radiative properties such as transition probabilities ($A_R$), branching ratios ($\beta_R$) and stimulated emission cross-sections ($\sigma_e$) were presented in Table 5. It was found that different emission levels, among them the $^4I_{11/2}$ level exhibits higher radiative transition probabilities, branching ratio and emission cross-sections. From Table 5, it can be seen that stimulated emission cross-section is found to be higher when adding NaF inside the glass. The strong laser NIR emission at $\approx 1.06\,\mu m$ is considered to be a potential use for optical system.

## 4. Conclusions

To examine the effect of sodium fluoride in lithium fluorophosphate glasses doped with $Nd_2O_3$ were prepared and characterized for their physical properties, FTIR, absorption

**Table 4.** Judd–Ofelt parameters ($\times 10^{-20}$ $cm^2$), spectroscopic quality factor ($\chi = \Omega_4/\Omega_6$) of $Nd^{3+}$ doped lithium fluorophosphate and other reported $Nd^{3+}$ doped glasses.

| Label glass | $\Omega_2$ | $\Omega_4$ | $\Omega_6$ | $\chi$ | Trend | Reference |
|---|---|---|---|---|---|---|
| Without NaF | 3.74 | 4.29 | 6.68 | 0.64 | $\Omega_6 > \Omega_4 > \Omega_2$ | Present work |
| With NaF | 7.27 | 4.54 | 10.55 | 0.43 | $\Omega_6 > \Omega_2 > \Omega_4$ | Present work |
| CaNd4 | 1.09 | 1.97 | 3.37 | 0.58 | $\Omega_6 > \Omega_4 > \Omega_2$ | [14] |
| PANCaFN2 (1.0$Nd^{3+}$) | 7.35 | 6.89 | 9.70 | | $\Omega_6 > \Omega_2 > \Omega_4$ | [12] |
| ZANP10 | 6.13 | 4.35 | 6.99 | 0.62 | $\Omega_6 > \Omega_2 > \Omega_4$ | [20] |
| $Nd^{3+}$-doped $TeO_2$–$PbF_2$–$AlF_3$ | 4.21 | 5.97 | 5.45 | – | $\Omega_4 > \Omega_6 > \Omega_2$ | [33] |
| $Nd^{3+}$ doped $Li_2O$ fluoro-phosphate | 8.04 | 1.47 | 7.67 | – | $\Omega_2 > \Omega_6 > \Omega_4$ | [2] |
| $Nd^{3+}$ doped $Na_2O$ fluoro-phosphate | 6.44 | 3.62 | 6.13 | – | $\Omega_2 > \Omega_6 > \Omega_4$ | [2] |
| 75$KPO_3$-24$MoO_3$-1$Nd^{3+}$ | 2.3 | 1.4 | 2.0 | 0.70 | $\Omega_2 > \Omega_6 > \Omega_4$ | [34] |
| BBFBNd1 | 2.45 | 1.07 | 1.35 | 0.79 | $\Omega_2 > \Omega_6 > \Omega_4$ | [35] |
| Li | 4.32 | 3.66 | 6.00 | – | $\Omega_2 > \Omega_6 > \Omega_4$ | [36] |
| Li-Na | 4.01 | 3.69 | 5.92 | – | $\Omega_2 > \Omega_6 > \Omega_4$ | [36] |
| TAKLNP20 | 5.47 | 3.79 | 4.83 | – | $\Omega_2 > \Omega_6 > \Omega_4$ | [37] |
| $NdPb_{10}$ | 6.34 | 3.69 | 5.23 | – | $\Omega_2 > \Omega_6 > \Omega_4$ | [38] |

**Table 5.** Radiative properties of $Nd^{3+}$ doped lithium fluorophosphate.

| Transition | $\lambda_p$ (nm) | $\Delta\lambda_{eff}$ (nm) | $A_R$ ($s^{-1}$) | $\sigma_e$ ($\lambda_p$) ($\times 10^{-20}$) ($cm^2$) | $\beta_R$ | |
|---|---|---|---|---|---|---|
| | | | | | Exp | Cal |
| $^4F_{3/2}$ → Without NaF | | | | | | |
| $^4I_{11/2}$ | 1062 | 24.97 | 1674.07 | 4.37 | 0.7182 | 0.8896 |
| $^4I_{13/2}$ | 1330 | 26.31 | 354.68 | 2.16 | 0.2818 | 0.1104 |
| $^4F_{3/2}$ → With NaF | | | | | | |
| $^4I_{11/2}$ | 1062 | 24.93 | 2468.39 | 6.45 | 0.7049 | 0.8749 |
| $^4I_{13/2}$ | 1330 | 29.92 | 556.67 | 2.98 | 0.2951 | 0.1251 |

spectra, NIR luminescence and JO parameter. The density, molar volume and refractive index decreases with addition of fluoride in lithium fluorophosphate glass doped with $Nd_2O_3$. The strong intensity vibrational lines are observed between 895 and 1262 $cm^{-1}$, as we can see that addition of fluoride content in the glass decreases the intensity. At wavenumber 2000–3500 $cm^{-1}$, the vibration of OH group show low intense band when adding more fluoride content in the glass. These spectra reveal strong absorption bands in the visible region at 581 nm ($^4I_{9/2} \rightarrow {}^4G_{5/2}$). The sample with NaF glass show absorption at 581 nm than other glass. The optical bandgap energy ($E_{opt}$) was observed to decrease with addition of sodium fluoride content in the glass. The NIR luminescence spectra reveal strong intensity at 1062 nm ($^4F_{3/2} \rightarrow {}^4I_{11/2}$). The addition of fluoride content enhances the NIR luminescence intensity, because the fluoride content reduces the phonon energy of the host glass. The JO parameter indicates the glass show more viscosity and rigidity which is due to more covalency nature confirmed by nephelauxetic ratio of the glass. The stimulated emission cross-section is found to be higher with addition of NaF inside the glass. Hence, it is concluded that these parameters suggests that the present glass act as a potential candidate for the solid-state lighting applications.

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