Synthesis and Luminescence properties of Lithium Aluminium Phosphate Glasses Doped with Nd$^{3+}$ Ion for Laser Medium

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Abstract. Nd$^{3+}$ ion doped lithium aluminium phosphate glasses were synthesized and studied their properties for laser medium application. The density and the refractive index of the glass samples were carried out at the room temperature. The optical and luminescence properties were studied by investigating absorption and NIR emission spectra of the glass samples. The glasses absorbed photons in ultraviolet (UV), visible light (VIS) and near-infrared (NIR) regions are clearly observed from absorption spectra. For the photoluminescence properties, the glass samples showed the strongest emission at 1063 nm when it were excited by 581 nm which assigned to the energy transitions of Nd$^{3+}$. The three phenomenological Judd-Ofelt parameters ($\Omega_2$, $\Omega_4$, $\Omega_6$) were determined from the spectral intensities of absorption band in order to calculate the radiative transition possibility ($A_\text{R}$), stimulated emission cross section ($\sigma(\lambda_p)$) and branching ratio ($\beta_\text{R}$) of the $^{4}F_{3/2} \rightarrow ^{4}I_{9/2}$, $^{4}I_{11/2}$ and $^{4}I_{13/2}$ transitions. From the obtained results the conclusions made about the possibility of using these glasses as laser material.

1. Introduction

Trivalent rare earth (RE$^{3+}$) ion show interesting optical and luminescence properties, such properties play an important role in the high technological applications of RE$^{3+}$ doped materials like lighting devices [1], optical fiber amplifiers [2], optical displays [3], fiber communications [4] and lasers [5]. Because of the f-f and f-d electronic transitions of RE ion afford stable optical and luminescence. Among them, trivalent neodymium (Nd$^{3+}$) ion is one of the most significant activator ion due to its abundant lasing emissions in the visible and infrared regions. In the near-infrared (NIR) region, their brings about three important emission bands at around 905 ($^{4}F_{3/2} \rightarrow ^{4}I_{9/2}$), 1063 ($^{4}F_{3/2} \rightarrow ^{4}I_{11/2}$) and 1334 ($^{4}F_{3/2} \rightarrow ^{4}I_{13/2}$) nm which finds potential applications in the fields of near-infrared laser, optical amplifiers and telecommunication [6]. During the last decade, many studies [6,7,8] have been focused on Nd$^{3+}$ ion doped into glass materials for laser and infrared optical communications because of their possess high NIR luminescence efficiencies. In general, the spectroscopic properties of Nd$^{3+}$ ion in optical glasses are closely related to the host matrix. Therefore, the development of the potential optical function of glasses requires optimum host material.
Phosphate glasses have received a great deal of attention for the development of compact solid-state lasers due to their promising properties such as low phonon energy, high quantum efficiency, easy incorporation of RE$^{3+}$ ion, high thermal expansion coefficients, high optical transparency and low preparation temperature [3,9,10,11]. However, it is well known that a pure P$_2$O$_5$ is very hygroscopic and not very stable, hence the addition of glass modifiers such as Li$_2$O in phosphate glasses breaks the P–O–P bonds and created non–bridging oxygens in glass networks significantly enhance the chemical durability of pure phosphate glass [9,12,13]. Moreover, the addition of Al$_2$O$_3$ the chemical, thermal stability, transparency and homogeneity of phosphate glasses will be further improved [14].

In recent years several research groups were carried out on Nd$^{3+}$ ion doped glasses for the development of laser applications. Babu et al. [6] investigated the thermal, structural and optical properties of Nd$^{3+}$ ion doped alkali niobium zinc tellurite glasses for lasing action at 1.06 mm. Nanda et al. [8] presented the effect of Nd$^{3+}$ ion on physical, structural and optical properties of barium tellurite borate glasses. Rao et al. [15] reported the spectroscopic properties of Nd$^{3+}$ ion doped fluoro containing zinc-alumino phosphate glasses to understand the effect of network modifiers (alkali and alkaline earth) on radiative process for laser material. Ratnakaram et al. [16] depicted the optical and luminescence studies of Nd$^{3+}$ doped alkali borate glasses useful for laser excitation.

In this work, reports the systematic analysis of physical properties, optical absorption, NIR emission and lasing potential (Judd-Ofelt(JO) analysis) of Nd$^{3+}$ doped lithium aluminium phosphate glasses with different concentrations of Nd$^{3+}$ ion to assess their possibility for laser applications.

2. Experimental detail

2.1 Sample compositions and preparation

Nd$^{3+}$ion doped lithium aluminium phosphate (LAP:Nd) glasses were synthesized by melt quenching technique with composition 15Li$_2$O : 20Al$_2$O$_3$ : (65-x)P$_2$O$_5$ : xNd$_2$O$_3$ (where x = 0.00, 0.05, 0.10, 0.50, 1.00 and 2.00 mol%). The chemical, lithium carbonate (LiCO$_3$), aluminium oxide (Al$_2$O$_3$), ammonium dihydrogen orthophosphate (NH$_4$H$_2$PO$_4$) and neodymium oxide (Nd$_2$O$_3$) were mixed and melted at 1200 °C for 3 h and then annealed at 500 °C for 3 h. The glass samples were cut and polished for optical and spectroscopic measurements. The photograph of the glass samples are shown in Figure 1.

2.2 Measurements of physical and spectroscopic properties

The density ($\rho$) of samples was measured by Archimedes' method using water as an immersion liquid, weighted samples in air and water via a 4-digit sensitive microbalance (AND, HR 200). The refractive index (RI) were recorded by Abbe refractometer (ATAGO) with a sodium vapor lamp as the light source (589.3 nm) and using mono-bromonaphthalene (C$_{10}$H$_7$Br) as an adhesive coating. The absorption spectra were measured by UV-Vis-NIR spectrophotometer (Shimadzu, UV-3600) in the range of ultraviolet, visible and near-infrared region. The emission spectra were measured by phosphorescence/fluorescence spectrophorometer (QuantaMaster300, PhotonTechnology International) using xenon lamp as an excitation source.

3. Judd-Ofelt (JO) analysis

3.1 Oscillator strengths and Judd-Ofelt (JO) parameters

JO analysis has been used to study the laser potential and its influence from the RE$^{3+}$ environment. In the first step, the area absorption spectrum was taken to evaluate the experimental oscillator strength ($f_{\text{exp}}$) by using the following formula[17,18]

$$f_{\text{exp}} = 4.318 \times 10^{-9} \int (\nu) d\nu$$  (1)
where $\varepsilon(\nu)$ is the molar excitation coefficient at average energy, $\nu \text{ cm}^{-1}$. Then find the calculated oscillator strength ($f_{\text{cal}}$) for an induced transition from the ground state ($\psi J$) to upper state ($\psi' J'$) and $\Omega_s$ using the relation Eq. (2).

$$f_{\text{cal}} = \frac{8\pi^3 mc
u}{3h(2f+1)} \times \left( \frac{n^2 + 2}{9n} \right) \sum_{\lambda=2,4,6} \Omega_s \left[ \left( \psi J \right| U \left| \psi' J' \right) \right]^2$$

(2)

where $m$ is the mass of the electron, $c$ is velocity of light, $\nu$ is the energy of the transition, $h$ is Planck’s constant, $n$ is the refractive index, $\Omega_s (\lambda = 2,4,6)$ are the JO parameters and $\left| \left( \psi J \right| U \left| \psi' J' \right) \right|^2$ is the doubly reduced matrix element of the unit tensor operator.

3.2 Radiative properties

The second process of JO analysis is calculating the radiative parameters. $\Omega_s$ value and area emission spectrum were brought to find the radiative transition probability ($A_R$) of each emission from relation [17,19]

$$A_R(\psi J, \psi' J') = \frac{64\pi^4 \nu^2 e^2}{3h(2f+1)} \left( \frac{n^2 + 2}{9n} \right) \sum_{\lambda=2,4,6} \Omega_s \left[ \left( \psi J \right| U \left| \psi' J' \right) \right]^2$$

(3)

the term $\left( \frac{n^2 + 2}{9n} \right)$ is the local field correction for the electric dipole transitions. Total radiative transition probability ($A_T$) of each excited state is the sum of the $A_R(\psi J, \psi' J')$ terms calculated over all the terminal states

$$A_T(\psi' J') = \sum_{\psi J} A_R(\psi J, \psi' J')$$

(4)

Obtain $A_T$ value was used to find branching ratio ($\beta_{\text{cal}}$) from relations (5), while experimental branching ratio ($\beta_{\text{exp}}$) can be evaluated from ratio between area of each emission peak.

$$\beta_{\text{cal}}(\psi J, \psi' J') = \frac{A_R(\psi J, \psi' J')}{A_T(\psi' J')}$$

(5)

Finally, emission peak area and $n$ value were used to calculate stimulated emission cross-section $\sigma(\lambda_p)$ for each peak wavelength ($\lambda_p$) via Eq. (6) [19].

$$\sigma(\lambda_p) = \frac{\lambda_p^4}{8\pi n^2 \Delta \lambda_{\text{eff}}} A_T(\psi J, \psi' J')$$

(6)

where $\Delta \lambda_{\text{eff}}$ is the effective bandwidth of each peak, derived by dividing the peak area by height.

![Figure 1. Photograph of lithium aluminium phosphate glasses.](image-url)

4. Results and discussion

4.1 Density and refractive index

The density and the refractive index of lithium aluminium phosphate glasses are shown in Figure 2. As seen from Figure 2, it is found that both the density and the refractive index increases with increase in Nd$_2$O$_3$ content, reflecting the denser nature of LAP: Nd glasses. This variation is a result from the
addition of heavier Nd$_2$O$_3$ in glass composition. Moreover, the greater atomic weight of Nd$_2$O$_3$ (336.478 g/mol) than P$_2$O$_5$ (141.994 g/mol) imparts more density to the glasses [8,20]. While the increase of the refractive index due to the denser nature of the glasses increases the refractive index of the medium with Nd$_2$O$_3$ concentration.

Figure 2. The density and the refractive index of LAP:Nd glasses.

4.2 Optical absorption and emission spectra
The absorption spectra of the Nd$^{3+}$ doped lithium aluminium phosphate glasses were recorded at room temperature in the wavelength range of 300-1000 nm is shown in Figure 3. The peaks shown in the absorption bands corresponding to the transitions originated from $^4$I$_{9/2}$ state to the excited state, i.e., $^4$D$_{1/2}$ (351 nm), $^2$P$_{1/2}$ (431 nm), $^4$G$_{11/2}$ (463 nm), $^4$G$_{9/2}$ (474), $^4$G$_{5/2}$ (513 nm), $^4$G$_{7/2}$ (526 nm), $^4$G$_{9/2}$ (581 nm), $^2$H$_{11/2}$ (627 nm), $^4$F$_{9/2}$ (683 nm), $^4$F$_{7/2}$ (745 nm), $^4$F$_{5/2}$ (803 nm) and $^4$F$_{3/2}$ (873 nm) [21]. These spectra are similar to those found for other Nd$^{3+}$ doped glasses [6,7,20,22]. The strongest absorption band at 581 nm is due to the $^4$I$_{9/2}$→$^4$G$_{5/2}$ transition of Nd$^{3+}$ and has higher absorption coefficient compared with other transitions. Therefore, this wavelength was then used to investigate the NIR emission spectra of glass samples as shown in Figure 4. Moreover, it is observed that the energy level intensities of transitions vary with the concentration Nd$^{3+}$ as absorption intensity of bands increases with the increase of Nd$_2$O$_3$ concentration.

Figure 3. Optical absorption spectra of LAP:Nd glasses.
The NIR emission spectra of the LAP:Nd glasses under 581 nm excitation wavelength were recorded and shown in Figure 4. It was found three emission bands at 908, 1063 and 1334 nm, these peaks were assigned as $^4F_{3/2} \rightarrow ^4I_{9/2}$, $^4F_{3/2} \rightarrow ^4I_{11/2}$ and $^4F_{3/2} \rightarrow ^4I_{13/2}$, respectively. These emission spectra were assigned by comparing the band positions in the emission spectra with those reported in the literature [6,22]. Among them, the emission band at 1063 nm corresponds to $^4F_{3/2} \rightarrow ^4I_{11/2}$ transition is the most intense and sharp. Moreover, it can be observed that the intensity of emission increases with increasing of Nd$^{3+}$ concentration until 1.00 mol%, after that intensity suddenly decreased at higher concentrations (>1.00 mol%) due to concentration quenching effect (inset picture in Figure 4).

Figure 4. NIR emission spectra of LAP: Nd glasses under 581 nm excitation wavelength.

4.3 JO analysis
LAP: Nd1.00 glass was used to investigate the laser potential by using JO analysis. The experimental ($f_{exp}$) and calculated ($f_{cal}$) oscillator strengths of the $^4I_{9/2} \rightarrow ^4G_{5/2}$ transition are the highest values due to the best absorption at 581 nm in VIS region as seen in Table 1. This transition is referred as the hypersensitive transition and it follows the selection rule $|\Delta S| = 0, |\Delta L| \leq 2$ and $|\Delta J| \leq 2$ [6,7,22]. So the $^4I_{9/2} \rightarrow ^4G_{5/2}$ transition is very sensitive to the environment around Nd$^{3+}$ ion that made this transition found to be more intense than the other transitions. The small $\sigma_{rms}$ value (1.6×10$^{-6}$) indicating good correlation of the experimental results with theoretical JO calculations.

Table 1. Experimental ($f_{exp}$) and calculated ($f_{cal}$) oscillator strengths and JO parameters of LAP: Nd1.00 glass.

| Transition $^4I_{9/2}$ | Wavelength (nm) | Energy (cm$^{-1}$) | Oscillator strengths ($\times 10^{-6}$) |
|-----------------------|-----------------|---------------------|---------------------------------------|
| $^4D_{1/2}$           | 351             | 28490               | 7.07                                  |
| $^2P_{1/2}$           | 431             | 23202               | 1.32                                  |
| $^4G_{11/2}$          | 463             | 21598               | 1.09                                  |
| $^2G_{9/2}$           | 474             | 21142               | 1.39                                  |
| $^4G_{9/2}$           | 513             | 19493               | 2.26                                  |
| $^4G_{7/2}$           | 526             | 19011               | 3.93                                  |
| $^4G_{5/2}$           | 581             | 17182               | 14.10                                 |
| $^2H_{11/2}$          | 627             | 15949               | 0.18                                  |
| $^4F_{9/2}$           | 683             | 14641               | 0.35                                  |
| $^4F_{7/2}$           | 745             | 13423               | 4.22                                  |

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The highest magnitudes of the JO parameters, their trend and spectroscopic quality factor of the LAP:Nd1.00 glass are compared in Table 2. Basically, the values $\Omega_{3}$ parameter depends on the site symmetry and the covalent nature between Nd-O bonds, whereas the values of $\Omega_{4}$ and $\Omega_{6}$ are related to the bulk properties like viscosity and stiffness of the host glass [22, 23]. From Table 2, the magnitudes of the three JO parameters of LAP:Nd1.00 glass increase in the order $\Omega_{6} > \Omega_{4} > \Omega_{4}$. It can explain that LAP:Nd1.00 glass shows higher viscosity nature between Nd$^{3+}$ ion and ligand anions. The ratio of $\Omega_{3}/\Omega_{4}$ is known as spectroscopic quality factor ($\chi$) [22], which decide the luminescence efficiency. The increase in the value of $\chi$ factor usually implies an increase in luminescence efficiency of the $^{4}F_{3/2} \rightarrow ^{4}I_{11/2}$ transition. Hence, the $\chi$ value of LAP:Nd1.00 glass is found to be 0.72 suggests the possibility of laser action at 1063 nm.

### Table 2. JO parameters, spectroscopic quality factor ($\chi=\Omega_{3}/\Omega_{4}$) and their trend for Nd$^{3+}$ doped glasses.

| Glass         | $\Omega_{3}$ | $\Omega_{4}$ | $\Omega_{6}$ | $\chi$ | Trend            | Reference   |
|---------------|--------------|--------------|--------------|--------|-----------------|-------------|
| LAPNd1.00     | 4.19         | 3.90         | 5.41         | 0.72   | $\Omega_{6} > \Omega_{4} > \Omega_{4}$ | Present work|
| TZN LN        | 2.13         | 3.29         | 3.83         | 0.86   | $\Omega_{4} > \Omega_{2}$                   | [6]         |
| TZN LNd       | 3.91         | 2.65         | 2.92         | 0.91   | $\Omega_{4} > \Omega_{2}$                   | [7]         |
| PNb KANd1.0   | 9.83         | 4.98         | 4.52         | 1.10   | $\Omega_{3} > \Omega_{2}$                   | [20]        |
| BS KNLd       | 9.93         | 8.05         | 8.35         | 0.96   | $\Omega_{4} > \Omega_{2}$                   | [22]        |
| PZN TB        | 5.73         | 1.59         | 4.95         | 0.32   | $\Omega_{4} > \Omega_{2}$                   | [22]        |
| LCZS FB       | 6.11         | 9.29         | 10.14        | 0.92   | $\Omega_{4} > \Omega_{2}$                   | [23]        |
| Te W Nd       | 4.71         | 4.06         | 3.89         | 1.04   | $\Omega_{2} > \Omega_{6}$                   | [24]        |
| Cdo-P 2 O5    | 4.8          | 6.18         | 7.14         | 0.86   | $\Omega_{4} > \Omega_{2}$                   | [25]        |
| Fluorophosphates | 2.90       | 5.00         | 7.27         | 0.69   | $\Omega_{6} > \Omega_{4}$                   | [25]        |
| Nd Na ZnP     | 4.28         | 3.89         | 5.19         | 0.75   | $\Omega_{4} > \Omega_{2}$                   | [26]        |

The radiative parameters such as emission peak position ($\lambda_p$), radiative transition possibility ($A_R$), stimulated emission cross-section ($\sigma(\lambda_p)$) and branching ratio ($\beta_R$) from emission spectrum of LAP:Nd1.00 glass are calculated by JO analysis and are presented in Table 3. The highest magnitudes of stimulated emission cross-section (1.28x10$^{-22}$ cm$^2$) and branching ratio indicate that the emission spectrum at 1063 nm has been considered to a good potential lasing transition, that mean the LAP:Nd1.00 glass in this work are useful for laser application.

### Table 3. Emission peak position ($\lambda_p$), radiative transition probabilities ($A_R$), stimulated emission cross-sections ($\sigma(\lambda_p)$), experimental branching ratios ($\beta_{exp}$) and calculated branching ratios ($\beta_{cal}$) of LAP:Nd1.00 glass.

| Level $^{4}F_{3/2} \rightarrow ^{4}I_{p}$ | $\lambda_p$ (nm) | $A_R$ | $\sigma(\lambda_p)$, ($\times 10^{-22}$ cm$^2$) | $\beta_{exp}$ | $\beta_{cal}$ |
|------------------------------------------|------------------|-------|-----------------------------------------------|----------------|---------------|
| $^{4}I_{9/2}$                            | 908              | 28.52 | 0.02                                          | 0.02           | 0.01          |
| $^{4}I_{11/2}$                           | 1063             | 1209.91| 1.28                                         | 0.61           | 0.56          |
| $^{4}I_{13/2}$                           | 1334             | 918.14| 1.20                                          | 0.37           | 0.43          |

$^\sigma$ refers r.m.s. deviation between experimental and calculated.
5. Conclusion
In this work, Nd\textsuperscript{3+} ion doped lithium aluminium phosphate glasses were synthesized by melt-quenching technique and studied physical, optical and luminescence properties to determine the potential for using as laser medium application. Both the density and the refractive index of glasses increase with increasing of Nd\textsubscript{2}O\textsubscript{3} content, showing the denser nature of the glass structure. The glasses absorbed photons in UV-VIS-NIR regions are clearly observed from absorption spectra, assigned to the transition from $^4$I\textsubscript{9/2} ground state of Nd\textsuperscript{3+}. The emission spectra, excited with 581 nm excitation wavelength show three emission peaks corresponding to the $^4$F\textsubscript{3/2} $\rightarrow$ $^4$I\textsubscript{9/2} (908 nm), $^4$F\textsubscript{3/2} $\rightarrow$ $^4$I\textsubscript{11/2} (1063 nm) and $^4$F\textsubscript{3/2} $\rightarrow$ $^4$I\textsubscript{13/2} (1334 nm) transitions. From the JO analysis found that The higher magnitudes of branching ratios and stimulated emission cross-sections for the $^4$F\textsubscript{3/2} $\rightarrow$ $^4$I\textsubscript{11/2} transition of LAP:Nd glass suggests that this transition is suitable for the potential laser emission at 1.063 µm.

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