The Dy$_2$O$_3$ Effect Study on Spectroscopy and Optical Properties of PBiNaGd Glass for Optical Amplification

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Abstract. The medium glass was made from chemical compounds with the compositions (70-x) P$_2$O$_5$ - 10Bi$_2$O$_3$ - 10Na$_2$O - 10Ga$_2$O$_3$ - xDy$_2$O$_3$ (where x = 0; 0.05; 0.1; 0.5; 1.0; 3.0 (mol%)) which was doped by active ion xDy$_2$O$_3$. All of proposed chemical compounds are in the form of powder with the mass of 20 grams of mixture in an alumina crucible and prepared by melt-quenching method. The glass has been obtained with a size (3x10x10) mm$^3$ and smoothed to get a flat surface and high transparency. Optical properties of glass samples Dy:PBiNaGd determined by measuring by absorption spectrum and spectrophotometer at area visible with different concentration. Analysis indicates eleven inhomogeneous displacement bands at various positions and intensities with hypersensitive transitions at 1274 nm ($^6$H$_{15/2} \rightarrow ^{6}$F$_{11/2}$).

Emission spectrum in medium glass Dy:PBiNaGd is supported using excitation wavelength ($\lambda_{eks}$) 350 nm and indicated that the highest intensity is in Dy:PBiNaGd4 where there four transition band emissions are namely $^4$H$_{9/2} \rightarrow ^4$H$_{13/2}$ (483 nm), $^4$H$_{9/2} \rightarrow ^4$H$_{11/2}$ (nm), $^4$H$_{9/2} \rightarrow ^4$H$_{9/2}$ (661 nm) and 4H9/2 → 6H6/2 (750 nm).

1. Introduction

Luminescence materials from rare earth (RE) in recent years have emerged in various fields of research that are interesting because of their characteristics as optical devices. Some of the most popular applications of luminescence materials are solid light lasers, white light-emitting diodes (WLEDs) and optical flat panels, plasma display panels (PDP), etc [1-5]. Glass treated with rare-earth ions can be well developed as luminescent material due to high emission efficiency, according to the 4f-4f and 4f-5d electron transitions in RE$^{3+}$. The 4f-4f transition gives a very sharp fluorescence pattern from ultraviolet to the infrared region, because of the protective effects of the 5s and 5p outer orbitals on 4f electrons. Between the various glass materials doped with dysprosium trivalent (Dy$^{3+}$) ions have played an important role in the telecommunications optical amplifier system [6-9]. Ion Dy$^{3+}$ typically indicates the $^4$F$_{9/2} \rightarrow ^4$H$_{13/2}$ (electric dipole) and $^4$F$_{9/2} \rightarrow ^4$H$_{15/2}$ (magnetic dipole) transitions which correspond to the yellow and blue regions respectively. The $^4$F$_{9/2} \rightarrow ^4$H$_{13/2}$ transition is hypersensitive so the intensity depends on the host, while the $^4$F$_{9/2} \rightarrow ^4$H$_{15/2}$ transition intensity is less...
sensitive to the host. In the right yellow-blue (Y / B) intensity ratio, Dy$^{3+}$ can radiate white light in glass or phosphorus [10-14].

Between the several optical materials of oxide glass, phosphate glass indicate the good nature for use as optical devices such as low melting point, high chemical resistance, good thermal stability, high transparency, low dispersion, ultraviolet (UV) transmission, characteristics of good optical, relatively high refractive index (compared to silica glass) and excellent solubility of rare-earth ions, which allows concentrations of rare earth ions contained in the glass matrix [15-16]. Optical studies reveal that the optical properties of Rare-Earth (RE) in glass depend on the chemical composition of the glass matrix that determines its structure and properties. between the different host materials, bismuth phosphate (BiP) glass is a promising host because this glass benefits the glass of bismuth and phosphate [17]. The composition of Na$_2$O will increase phonon energy from the glass. So the increase in Na$_2$O composition causes a rise in glass transition temperature (T$_g$) so that it is thermally more stable. But the amount of DT is the difference between the crystallization temperature and the glass transition temperature is getting smaller, which illustrates the tendency of the glass to have a larger crystal structure [18-20]. Gd$_2$O$_3$ has become a very attractive material in glass matrices because of the extremely efficient transfer of energy from Gd$^{3+}$ ions to the combined activator, or affordable cost [21].

Research development on phosphate-based glass optical amplifier materials is currently being done, but its development on an industrial scale is currently still limited. Some problems and weaknesses of the optical amplifier medium used today include the structure of the glass composition that is not homogeneous [22]. These conditions cause the light scattering effect produced is also less resistant to high heat. The inhomogeneous structure can cause the light scattering effect on the glass medium thus causing a decrease in the emission intensity of the glass medium itself. This research aims to analyze the effect of variations in the active ion concentration of Dy$^{3+}$ on the spectroscopic properties of the glass medium as an optical amplifier using the Melt-Quenching method with the composition of the Dy:PbiNaGd glass. Also besides, this research also aims to determine the emission spectrophotometer spectrum characterization of glass composition, knowing the effect of composition on the glass refractive index.

### 2. Method

Glass Dy:PbiNaGd made in accordance with the composition (70-x) P$_2$O$_5$ - 10Bi$_2$O$_3$ - 10Na$_2$O - 10Gd$_2$O$_3$ - xDy$_2$O$_3$ (where x = 0; 0.05; 0.1; 0.5; 1.0; 3.0 (mol%)). six samples were used in making this glass, namely PbiNaGd, Dy:PbiNaGd1, Dy:PbiNaGd2, Dy:PbiNaGd3, Dy:PbiNaGd4, and Dy:PbiNaGd5. The process of making glass medium was done by the melt-quenching method with a composition that is used as much as 20 grams and melted at the temperature of 1200°C in an alumina crucible for 3 hours in a furnace. Then poured into stainless steel molds which are heated and annealed at 500°C for 3 hours and the temperature is lowered to room temperature to eliminate thermal tension. After the sample has cooled and formed glass, then the cutting process is carried out with a size of (3x10x10) mm$^3$ and refining to obtain a high level of transparency. To find out the physical properties of glass material Archimedes's principle calculation is used in equation (1)

$$\rho_g = \frac{W_u}{W_u - W_c}\rho_c \quad (1)$$

Where $\rho_g$ = density of glass material, $\rho_c$ = density of the liquid, and $W_u$ and $W_c$ = the weight of objects in air and water, respectively. Other physical properties such as the refractive index are characterized using the Abbe Refractometer (ATAGO). Optical characteristics such as absorption, energy band gap, and emission of each sample were characterized using Shimadzu 3600 UV-VIS-NIR Spectrophotometer and Spektrofluorophotometer. The result sample observation of every tool used
presented in tables and graphs. The tools used in the processing and analyzing data for concluding are Microsoft Excel and Software Origin 8.0.

3. Results and Discussion

3.1 Absorption Spectral

The transition and absorption bands position along with the energy level for all Dy: PBiNaGd glasses were shown in Figure 1.

![Figure 1. Absorption spectra of Dy:PBiNaGd glasses.](image)

The absorption spectra of Dy:PBiNaGd glasses with wavelnegth region of 320–1900 nm are shown in Figure 1. This spectrum shows that the eleven transitions have presented originating from the \( ^6H_{15/2} \) ground state to various excited states \( ^6P_{3/2}, ^4I_{13/2}, ^4G_{11/2}, ^4I_{15/2}, ^4F_{9/2}, ^6F_{3/2}, ^6F_{5/2}, ^6F_{7/2}, ^6F_{9/2}, ^6F_{11/2}, \) and \( ^6H_{11/2} \) with wavelength at 345, 386, 423, 451, 472, 758, 803, 899, 1093, 1093, 1274 and 1678 nm respectively. The absorption spectra is useful to wavelength determining of light that will be used in the excitation of electrons in a glass medium. Based on all the absorption transitions produced by the glass Dy:PBiNaGd there is one most sensitive peak so it is called a hypersensitive transition that is the transition \( ^6H_{15/2} \rightarrow ^6F_{11/2} \) at a wavelength of 1274 nm. The hypersensitive transition occurs at the same position for all Dy:PBiNaGd transitions [23-28].

3.2 Energy Gap

The relationship between the energy bandgap (Eg) with the absorption coefficient for direct and indirect transitions in the sample cup Dy:PBiNaGd can be seen in Figure 2 and Figure 3. The energy value of the bandgap can be determined using the method Tauc plot a graph, where the measurement is done by drawing a line on a graph linear \( hv(\alpha v)^n \) to cut the \( hv \) axis, Optical absorption edge plays an important role in determining the optical bandgap. To determine the optical band gap glass Dy:PbNaGd then use equation 2.

\[
(hv)^n = A(hv - Eg)
\]

Which, \( h \) is Planck's constant, \( v \) is the frequency, \( Eg \) is the bandgap and \( A \) is a constant proportion. The value of \( n = \frac{1}{2} \) for direct transition and \( n = 2, \frac{3}{2} \) for indirect transitions [29]. Where visible on the graph Indirect and direct the sample with the composition Dy:PBiNaGd5 showed the highest bandgap and a decline in Dy:PBiNaGd4.
In general, the optical band gap is nonlinear. Nonlinearity comes from the influence of a mixture of ions of rare-earth (RE) in the glass. Besides the decline Dy$^{3+}$ can grow the donor center that reduces glass. As shown in Figure 2, that the changes in the optical absorption edge and the baseline position of the spectrum can affect the energy bandgap value. From Dy:PbNaGd5 graph shows, it is seen that the edge of the optical absorption edge of the glass medium is more inclined to the right side and the absorption bandwidth is also longer. This causes the gap value of the medium's energy band to transition indirectly is greater than that of other samples which are 3.2 eV.

![Figure 2. Indirect band gap energy of Dy$^{3+}$ doped phosphate glasses](image)

For the direct transition in Figure 3 the Dy:PbNaGd5 medium glass also produces a bandgap of energy that is larger than the other glass which is 3.6 eV. The distribution of Eg for the two types of transitions is the same, but the average Eg for large direct transitions is greater when compared to
indirect transitions. This is due to the lower direct transition spectrum baseline compared to the indirect transition.

### 3.3 Judd-Ofelt analysis and Field Strength

Physical and spectrum data are used to evaluate various parameters in the Judd-Ofelt (JO) theory used to study laser potential. The peak absorption area, glass thickness, and the concentration of Dy$^{3+}$ ions are used to calculate molar absorptivity ($\alpha (\nu)$) from related transitions on energy ($\nu$). Then, $\alpha (\nu)$ is used to evaluate the strength of the experimental oscillator $f_{\text{exp}}$ using a relation[16]:

$$f_{\text{exp}} = 4.318 \times 10^{-9} \int \alpha (\nu) d\nu$$

The energy level for each transition from each glass Dy:PBiNaGd is shown in Table 1. The table shows that most of the same transitions for each glass are in the same position (wavelength). However, this does not apply to all samples, meaning that the same transition is not always at the same wavelength. This difference in wavelength position arises as a result of the partial expansion in the skin $f$ caused by a shift in charge from the ligand to the core of the ion center [30]. This situation is often expressed also as a nephelauxetic effect which can directly affect the molar volume of the glass medium [31].

#### Table 1. Experimental ($f_{\text{exp}}$) and theoretical ($f_{\text{cal}}$) oscillator strength values ($\times 10^6$) for varDy:PBiNaGd glass systems

| Transitions | $\lambda_{\text{exp}}$(nm) | Energy (cm$^{-1}$) | Dy: PBiNaGd1 | Dy: PBiNaGd2 | Dy: PBiNaGd3 | Dy: PBiNaGd4 | Dy: PBiNaGd5 |
|-------------|---------------------------|-------------------|--------------|--------------|--------------|--------------|--------------|
| $^9\text{H}_1/2$ | 345 | 28985 | - | - | - | - | - | 0.17 | 0.0179 |
| $^7\text{P}_2/2$ | 386 | 25906 | - | - | - | - | - | 0.43 | 0.2328 |
| $^7\text{G}_{11/2}$ | 423 | 23640 | - | - | - | 0.61 | 0.04 | 0.12 | 0.01 | 0.04 | 0.0256 |
| $^1\text{S}_{15/2}$ | 451 | 22172 | - | - | - | 0.49 | 0.13 | 0.07 | 0.05 | 0.15 | 0.1364 |
| $^7\text{F}_2/2$ | 472 | 21186 | - | 0.21 | 0.04 | 0.03 | 0.02 | 0.04 | 0.0451 |
| $^7\text{F}_{3/2}$ | 758 | 13192 | - | 0.05 | 0.021 | 0.37 | 0.04 | 0.06 | 0.02 | 0.03 | 0.0483 |
| $^7\text{F}_5/2$ | 803 | 12453 | 0.009 | 0.0009 | 0.79 | 0.112 | 0.44 | 0.20 | 0.11 | 0.08 | 0.33 | 0.2571 |
| $^7\text{F}_{7/2}$ | 899 | 11123 | 0.003 | 0.00045 | 0.72 | 0.450 | 0.63 | 0.54 | 0.23 | 0.21 | 0.61 | 0.5914 |
| $^7\text{F}_{9/2}$ | 1093 | 9149 | 0.010 | 0.00112 | 0.92 | 0.993 | 0.82 | 0.85 | 0.32 | 0.32 | 0.73 | 0.7357 |
| $^7\text{F}_{11/2}$ | 1274 | 7849 | 0.053 | 0.0524 | 3.50 | 3.456 | 3.08 | 3.06 | 1.17 | 1.17 | 2.72 | 2.7160 |
| $^7\text{H}_{11/2}$ | 1678 | 5959 | - | 0.01 | 0.335 | 0.25 | 0.38 | 0.13 | 0.15 | 0.37 | 0.4056 |
| Rms | 0.005 | 0.328 | 0.27 | 0.02 | 0.080 |

Table 1 displays the oscillator force experimentally ($f_{\text{exp}}$) and calculation ($f_{\text{cal}}$) for each type of glass medium Dy:PBiNaGd. The maximum oscillator strength value for each glass medium was obtained in the $^9\text{H}_{11/2}$ transition which explains that the hypersensitive transition was generated at the wavelength region of 1274 nm. Standard deviation ($\Delta f_{\text{rms}}$) the strength of the oscillator shows the best value on the glass medium Dy: PBiNaGd3 which is $\pm 0.275$

The Judd-Ofelt (JO) parameter which states the spectroscopic intensity for the Dy:PBiNaGd sample has been calculated and shown in Table 2. The parameters $\Omega_2$, $\Omega_4$, and $\Omega_6$, which are obtained using the least square fitting method, are used to predict the transition properties of the glass medium. In the previous discussion, it was stated that information related to symmetry and rare-earth ion bonds in the local glass environment can be known from the JO parameters. As shown in Table 2, The value $\Omega_2$ greater than $\Omega_4$ and in line with the results of research $\Omega_4[32-33]$ with the relationship between $\Omega$ produced for the five samples of this glass medium is to follow the pattern $\Omega_2 > \Omega_4 > \Omega_6$. 

\[ \text{Judd-Ofelt (JO) theory used} \]

\[ \text{Experimental (f_{\text{exp}})) and theoretical (f_{\text{cal}})) oscillator strength values (\times 10^6) for varDy:PBiNaGd glass systems} \]

\[ \text{Transitions} \]

\[ \text{\lambda_{\text{exp}}(nm)} \]

\[ \text{Energy (cm^{-1})} \]

\[ \text{Dy: PBiNaGd1} \]

\[ \text{Dy: PBiNaGd2} \]

\[ \text{Dy: PBiNaGd3} \]

\[ \text{Dy: PBiNaGd4} \]

\[ \text{Dy: PBiNaGd5} \]

\[ \text{\Omega_2 > \Omega_4 > \Omega_6} \]
Table 2. Judd-Ofelt parameters ($x10^{-20}$ cm$^2$) and spectroscopic quality factor for various Dy:PBiNaGd glasses.

| Glass      | $\Omega_2$($x10^{-20}$ cm$^2$) | $\Omega_4$($x10^{-20}$ cm$^2$) | $\Omega_6$($x10^{-20}$ cm$^2$) | $\chi(\Omega_2/\Omega_6)$ ($x10^{-20}$) | Ref        |
|------------|--------------------------------|--------------------------------|--------------------------------|------------------------------------------|------------|
| Dy: PBiNaGd1 | 0.057                          | 0.018                          | 0.002                          | 9.000                                    | This work  |
| Dy: PBiNaGd2 | 3.579                          | 1.581                          | 0.263                          | 6.001                                    | This work  |
| Dy: PBiNaGd3 | 3.241                          | 1.049                          | 0.480                          | 2.184                                    | This work  |
| Dy: PBiNaGd4 | 1.226                          | 0.386                          | 0.191                          | 2.026                                    | This work  |
| Dy: PBiNaGd5 | 2.849                          | 0.65303                        | 0.599                          | 1.090                                    | This work  |

3.4 Photoluminescence Spectra

The relationship between excitation and emission is when electrons are fired to the highest peak of energy level, then there is light decay (light rays) at the emission level. For the citation spectrum and emission spectrum shown in Figure 4 and Figure 5.

![Photoluminescence Spectra](image)

Figure 4. The Excitation spectra of Dy:PBiNaGd glasses with $\lambda_{\text{em}} = 574$ nm

The excitation spectra of Dy: PBiNaGd glasses shown in Figure 4. That can be shown that the excitation spectra of Dy: PBiNaGd1 to Dy: PBiNaGd5 from the phosphate glass matrix doped by Dy$^{3+}$ ions. The excitation spectrum consists of seven inhomogeneous excitation peaks which lie at the transition of the ground state $^6H_{15/2}$ to the excited states $^4P_{3/2}$, $^6P_{7/2}$, $^6P_{5/2}$, $^4F_{13/2}$, $^4G_{11/2}$, $^4I_{15/2}$ and $^4F_{9/2}$ with length waves 322 nm, 350 nm, 363 nm, 386 nm, 424 nm, 452 nm and 472 nm. Among these excitation peaks, the excitation peak at 350 nm ($^6H_{15/2} - ^6P_{7/2}$) is relatively high in intensity and this is used for measuring the luminescence spectrum [34-39].
The emission spectrum in medium glass Dy:PBiNaGd is observed using an excitation wavelength (λ_{exc}) 350 nm as shown in figure 6. This material produces four emission band transitions namely \( ^4H_{9/2} \rightarrow ^6H_{13/2} \), \( ^4H_{9/2} \rightarrow ^6H_{11/2} \), \( ^4H_{9/2} \rightarrow ^6H_{9/2} \) and \( ^4H_{9/2} \rightarrow ^6F_{6/2} \) can be consistently produced by each type of glass. Emission spectrum results show that higher intensities are found in Dy:PBiNaGd4 and lower intensity values are found in Dy:PBiNaGd1. The difference in emission intensity increased to the glass Dy: PBiNaGd4 subsequently decreased when the concentration of ion Dy: PBiNaGd5. The difference in the pattern of emission intensity is probably caused by a change in alignment between the glass medium and the optical system in the measuring instrument. But it does not rule out the possibility that this phenomenon occurs because of the decrease in the intensity of the excitation source due to the increase in temperature (thermal effect) around the light source and the resonant energy transfer (RET) [40-42].

3.5 Lifetime
The decay rate curve \( ^4F_{9/2} \rightarrow ^6H_{13/2} \) glass Dy: PBiNaGd was determined at temperatures below excitation 350 nm and at emission wavelength 574 nm. Lifetime aims to see the length of decay (lifetime) the length of light emitted.
The experimental decay time ($\tau_{\text{exp}}$) of Dy$^{3+}$ ion at $^4F_{9/2}$ transition is a single exponential at lower concentrations while non-exponential for ion concentrations Dy$^{3+}$ were higher in glass Dy:PBiNaGd [43]. Figure 6 shows the luminescence decay curve of the level $^4F_{9/2}$ to Dy:PBiNaGd2, Dy:PBiNaGd4, and Dy:PBiNaGd5 which is doped by phosphate glass and lifetime fluorescence ($t_{\text{exp}}$) with excitation $\lambda_{\text{exc}} = 350$ nm and $\lambda_{\text{em}} = 574$ nm.

4. Conclusions

In this research, phosphate glass was doped with various concentrations of dysprosium ions made by the melt-quenching method. Analysis indicates eleven inhomogeneous displacement bands at various positions and intensities with hypersensitive transitions at 1274 nm ($^6H_{15/2} \rightarrow ^6F_{11/2}$). Emission spectrum in medium glass Dy:PBiNaGd is supported using excitation wavelength ($\lambda_{\text{eks}}$) 350 nm and indicated that the highest intensity is in Dy:PBiNaGd4 where there four transition band emission are namely $^4H_{9/2} \rightarrow ^6H_{13/2}$ (482 nm), $^4H_{9/2} \rightarrow ^6H_{11/2}$ (574 nm), $^4H_{9/2} \rightarrow ^6H_{9/2}$ (662 nm) and $^4H_{9/2} \rightarrow ^6H_{0/2}$ (751 nm).

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