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Physical and luminescence properties of rare earth doped phosphate glasses for solid state lighting applications

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Abstract

Luminescent glass for use in LED based lighting applications has recently attracted significant research interest. Samples of oxyfluoride phosphate glass doped with Dy_2O_3 and Eu_2O_3 were prepared using the conventional melt quenching process. The samples were studied through analysis of their density, molar volume, refractive index, absorption spectra, emission spectra, excitation spectra, decay time profile and by plotting their characteristics on a CIE diagram. It was found that the density of the glass samples produced increased with the concentration of ions in them. The absorption spectra for glass samples produced using both ions had clear absorption peaks. The excitation spectra that were found for NSDy and NSEu showed the strongest peaks at 350 and 393 nm respectively. It was thought that these peaks corresponded to the transition of $^6H_{15/2}$ and $^7F_0$ to the excited states $^6F_{11/2}$ + $^6H_{9/2}$ and $^5L_6$ respectively. The Judd-Ofelt (JO) theory was used to calculate the JO intensity parameters and radiative properties of the glass samples that were produced. The emission peaks that were observed at 573 nm for NSDy and 612 nm for NSEu show higher intensity than the other peaks. It was thought that these peaks corresponded to the transitions of $^6H_{13/2} \rightarrow {}^4F_{9/2}$ and $^5D_0 \rightarrow {}^7F_3$ respectively. The CIE coordinates showed that the NSDy emits visible light in the white region and the NSEu emits visible light in the Reddish orange regions. It was concluded that both of the glass types produced could be good candidates for applications in the field of solid-state lighting and LEDs.

Keywords: Phosphate glass, refractive index, CIE diagram
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1. Introduction

Luminescent materials doped with Rare Earth (RE) ions have attracted the recent attention of many scholars. This is due to the growth of applications for new tunable solid-state lasers, solar energy converters, white Light Emitting Diodes (w-LEDs), fiber optic communication devices, field emission displays (FEDs), plasma display panels (PDPs), up-converters, stimulated phosphors, energy storage materials, radiation shielding and alternating current driven LEDs (AC-LED) [1].

Among different glass hosts, phosphate glasses have unique properties. They have high thermal stability, high transparency, a low melting point, a high gain density and low dispersion rates [2]. There is a great amount of interest in the study of rare-earths doped phosphate glasses. The addition of modifiers ions such as SrO is also known to increase the stability of the glass against devitrification and corrosion. Glass made with rare earth, RE, ions also has an ever increasing list of potential uses in several technological fields. The addition of Na_2O to the glass mixture improves the RE ion solubility leading to the possibility of using even higher concentrations of ions [2]. Fluorophosphate hosts are suitable for RE^{3+} ions due to their optical properties. Fluorophosphate hosts offer the combined advantages of both fluoride and phosphate glasses. These are high transparency, low phonon energy, good moisture resistance, physical and chemical stability, low nonlinear refractive index and high transparency from near UV to the mid IR spectral range. In addition to this they have a low production cost when produced in a large quantity. Due to their low phonon energies, the host materials, have been effectively used to increase the efficiency of the radiative

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emissions from the ion doped glass [3].

Among rare earth ions that were of interest for this research Dysprosium is perhaps one of the most important. This is because of its compounds having excellent optical, electrical and magnetic properties. The luminescence lines of Dy^{3+} are in the 470-500-nm region due to the $^4F_{9/2}\rightarrow{}^6H_{15/2}$ transition, and in the 570-600-nm region due to the $^4F_{9/2}\rightarrow{}^6H_{13/2}$ transition. If the emission intensity of the two transitions is equal, the color of the luminescence produced is close to white [4]. Triply ionized Europium (Eu^{3+}) ions are also of great interest due to the narrow and monochromatic nature of the $^5D_0\rightarrow{}^7F_2$ transition which produces red light at around 610 nm [5].

In this research Dy_2O_3 and Eu_2O_3 doped phosphate glasses samples were prepared using the melt quenching technique. They were also modified through the addition of Na_2O and SrO in the process. Following this the physical and luminescence properties of the samples were investigated. Density, refractive index, absorption spectra, photo-luminance and decay time were all analysed.

2. Experimental Details

Glass samples were prepared using the melt quenching technique. The chemical composition of he samples was as follows:

NSDy – 10Na_2O: 10SrO: 10GdF_3: 69P_2O_5: 1Dy_2O_3

NSEu – 10Na_2O: 10SrO: 10GdF_3: 69P_2O_5: 1Eu_2O_3.

The terms NSDy and NSEu will be used to refer to these samples for the remainder of this paper.

The compounds Na_2CO_3, $SrCO_3$, GdF_3, $NH_4H_2PO_4$, Dy_2O_3 and Eu_2O_3 were used as raw materials for the production of the glass samples. The compounds were weighed to give the proportions shown above and were mixed thoroughly. Alumina crucibles were used to melt the mixture at 1200 °C in an electric furnace. After being melted for 3 hours in the electric furnace the melt was poured on a stainless-steel mold and allowed to quench. The glass samples were then annealed at 300 °C to remove internal stresses. Finally the glass samples were cut and polished so that they were ready for analysis.

The density of glass samples was measured using the Archimedes' principle. Water was used as the immersion liquid. The weights of the glass samples were measured in water as well as in air. The equation reported in previous literature [6] was then used to calculate the density of each sample using Eq. 1.

$$\rho = \frac{w_{air}}{w_{air} - w_{water}} \times \rho_{water} \quad (\mathrm{g/cm^3}) \tag{1}$$

where w_{air} is the weight of the samples in air and the w_{water} is the weight in water. 1.000 g/cm^3 is the density of the water that was used for the calculation.

The equation used for the calculation of the molar volume was

$$V_{mol} = \frac{M}{\rho} \quad (\mathrm{cm^3/mol}) \tag{2}$$

where V_{mol} is the molar volume, M is the molecular mass and ρ is the density of each sample.

An Abbe refractometer was used to measure the refractive index (n) of each sample. This contained a sodium-vapor lamp as a light source and used mono bromo-naphthalene ($C_{10}H_7Br$) as a contact liquid. The UV-Vis-NIR absorption spectrum for each sample was recorded using a UV-Vis-NIR spectrophotometer (Shimadzu, UV-3600) in the wavelength range from 200 to 2500 nm. Photoluminescence excitation and emission spectra were recorded using a fluorescence spectrophotometer (Agilent Technologies, Cary Eclipse) with a xenon lamp as a light source. Luminescence decay time measurements were carried out using the same device. The CIE 1931 chromaticity diagram was used to characterize the emission color of the samples in the electromagnetic visible spectrum. The quality of the emitting light was evaluated by comparison of the correlated color temperature, or CCT, values.

The f-f transitions that were thought to occur in our glass samples were studied through the use of JO theory. The theory shows that the experimental and the calculated oscillator strengths for observed absorption bands correspond to the transition of an ion from an initial state with wave function ψJ to a final state having wave function $\psi' J'$. These are given by Eq. 3 and Eq. 4 respectively [7].

$$f_{ext} = \frac{3.303mc^2}{\pi e^2 N} \int \varepsilon(v)dv = 4.318 \times 10^{-9} \int \epsilon(v)dv \tag{3}$$

$$f_{cal} = \frac{8\pi^2 mcv}{3h(2j+1)} \frac{(n^2+2)^2}{9n} \sum_{\lambda=2,4,6} \Omega_\lambda (\psi j \parallel U^\lambda \parallel \psi' j')^2 \tag{4}$$

where $\Omega_\lambda (\lambda = 2, 4, 6)$ are known as the JO intensity parameters.

These JO parameters can be determined using the experimental oscillator strengths obtained from Eq. 1 and Eq. 2. The least-squares fitting approximation is then used to obtain JO parameters. Once the JO parameters are obtained they can be used in Eq. 4 to find the calculated oscillator strength.

In Eq. 4 the refractive index of the glass is given by n, J represents the total angular momentum of the ground state, v (cm^{-1}) is the energy of the transition and $\parallel U_\lambda \parallel$ is the squared doubly reduced matrix elements of the unit tensor operator. These are calculated for λ = 2, 4 and 6 from the intermediate coupling approximation for the transition from state with ψj to state with $\psi' j'$at energy v (cm^{-1}). It should be noted

Table 1. Density, molar volume and refractive Index.

Samples	Density (g/cm^3)	Molar volume (cm^3/mol)	Refractive index
Host	2.845±0.003	48.35	1.527
NSDy	2.855±0.001	48.84	1.530
NSEu	2.875±0.005	48.49	1.530

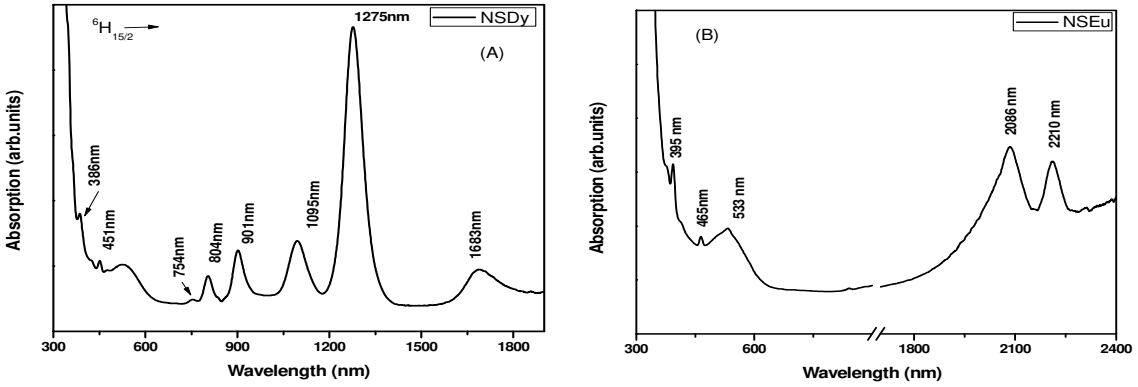

Figure 1: Absorption spectra in the UV-Vis-NIR region for the (A) Dy doped and (B) Eu doped glasses.

that the squared doubly reduced matrix elements are independent of the host [8].

$$A_{R(\psi j,\psi')} = \frac{64\pi^4 v^3}{3h(2j+1)} \frac{n(n^2+2)^2}{9} S_{ed} + \frac{64\pi^4 v^3}{3h(2j+1)} n^3 S_{md} \quad (5)$$

In this equation S_{ed} and S_{md} represent electric-dipole and magnetic-dipole line strengths which are given by the following equations respectively,

$$S_{ed} = e^2 \sum_{\lambda=2,4,6} \Omega_\lambda (\psi j \parallel U^\lambda \parallel \psi' j')^2 \quad (6)$$

$$S_{md} = \frac{e^2 h^2}{16\pi^2 m^2 c^2} \sum_{\lambda=2,4,6} \Omega_\lambda (\psi j \parallel L + 2S \parallel \psi' j')^2 \quad (7)$$

The radiative life time, τ_R, is given by Eq. 8

$$\tau_R = \frac{1}{A_T(\psi j)} \quad (8)$$

where A_T is the total transition probability. A_T is equal to the sum of all energy states at energy lower than $\psi' J'$.

The relative intensities of all of the emission lines originating from a given excited state can be predicted by use of the branching ratio (β_R). The branching ratio of the system can be determined from Eq. 9.

$$\beta_R(\psi J, \psi' J') = \frac{A_R(\psi J, \psi' J')}{A_T(\psi J)} \quad (9)$$

The emission cross section can be calculated using Eq. 10.

$$\sigma(\lambda_p)(\psi J, \psi' J') = \frac{\lambda_p^4}{8\pi c n^{2\Delta\lambda_{eff}}} A(\psi J, \psi' J') \quad (10)$$

In this equation λ_p is the "emission peak wavelength" and $\Delta\lambda_{eff}$ is its "effective linewidth". The effective linewidth can be found by dividing the area of the emission band by its average height.

3. Results and Discussion

3.1. Physical properties

The values for density, molar volume and refractive index that were found for the glass samples that were produced are shown in Table 1. From the table it can be clearly seen that the densities of the glass samples increased with the increasing concentration of RE ions in the samples. The densities of the samples was thought to increase because one of the lower molecular weight components of the glass composition is replaced by a a compound with a higher molecular weight. This has the impact of increasing the average molecular weight of the sample and hence the density. The refractive index of a sample is directly related to its density. the higher density of the glass indicates a more compact structure which slows the movement of light causing a higher refractive index [10].

3.2. Absorption spectra

Figures 1 (A) and (B) show the absorption spectra for the Dy_2O_3 doped and Eu_2O_3 doped samples that were produced. In the NSDy glass absorption spectra eight characteristics peaks were observed. These were

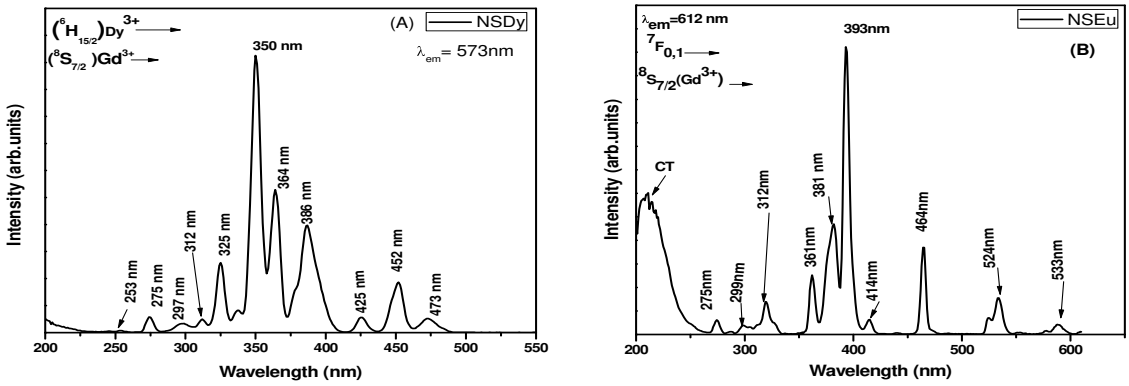

Figure 2: Excitation spectra for the (A) Dy doped and (B) Eu doped glasses.

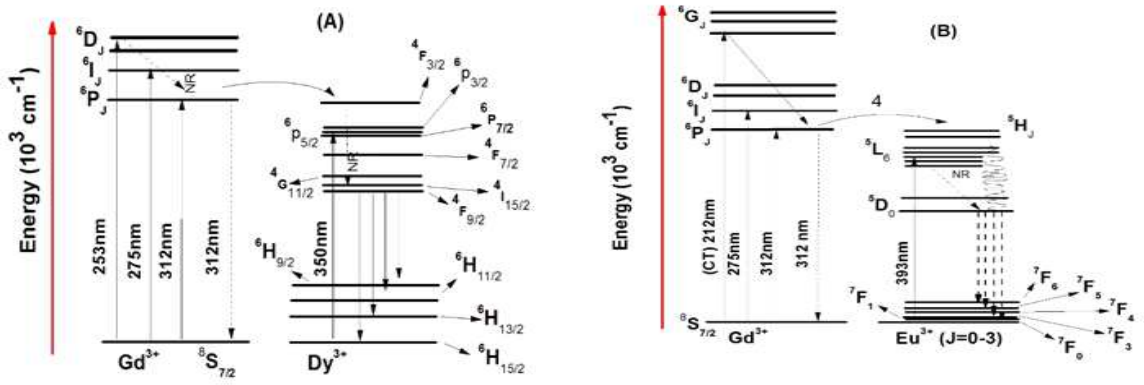

Figure 3: Partial energy level diagram for the (A) Dy doped and (B) Eu doped glasses.

positioned at 386, 451, 802, 901, 1097, 1275 and 1683 nm. These were thought to correspond to the Dy^{3+} transition from the ground state, $^6H_{15/2}$, to the excited states $^4F_{7/2}$, $^4I_{15/2}$, $^6F_{5/2}$, $^6F_{7/2}$, $^6F_{9/2}$, $^6F_{11/2}$ + $^6H_{9/2}$ and $^6H_{11/2}$, respectively [11].

It was thought that the transition at 1275 nm correspond to $^6H_{15/2}\rightarrow{}^6F_{11/2}$ + $^6H_{9/2}$. This was observed as being the hyper sensitive transition and its intensity was seen to be highly affected by the composition of the host glass.

In the NSEu absorption spectra, a total of five characteristics Eu^{3+} ions peaks were observed. These were at 395, 465, 533, 2086 and 2210 nm. It was thought that these corresponded to transitions from the ground state, $^7F_0$, to the excited states of $^5L_6$, $^5D_2$, $^5D_1$ and $^7F_6$ respectively. The peak at 2205 nm appeared to correspond to the transition from the thermally excited state $^7F_1$ to the $^7F_6$ state [12].

The JO intensity parameters were calculated from the refractive index and the bands observed in the absorption spectra. They are listed in Table 2. The Ω_2 corresponded to an asymmetry around the rare ions and bonding nature of rare earth with other ions. Higher values of Ω_2 indicate higher asymmetry around the rare earth ions in glass structures. Higher asymmetry around rare earth ions can result in the efficient emission of white and reddish orange light from Dy and Eu doped glasses. Hence higher values of Ω_2 indicate that the glass samples produced may be potential candidates for solid state lighting applications. Furthermore, the values of Ω_4 and Ω_6 also give information about the bulk structure of glass samples.

3.3. Photoluminescence emission and excitation

Fig. 2 shows the excitation spectra for the glass samples that were produced. The excitation spectrum for the NSDy sample was recorded at 573 nm. Twelve clear peaks were observed. The three peaks positioned at 253, 275 and 312 nm were thought to be due to the transition of Gd^{3+} ions from their ground state, $^8S_{7/2}$, to the excited states $^6D_{9/2}$, $^6I_{9/2}$ and $^6P_{7/2}$. Peaks were observed at 297, 325, 350, 364, 386, 425 452 and 473 nm. These were thought to correspond to transitions from the ground state, $^6H_{15/2}$, to the excited states $^4H_{13/2}$, $^4K_{15/2}$, $^4M_{15/2}$+$^6P_{7/2}$, $^4I_{11/2}$, $^4I_{13/2}$, $^4G_{11/2}$, $^4I_{15/2}$ and $^4F_{9/2}$. The excitation transitions for both types of glass are illustrated in the partial energy level diagram shown in Fig. 3(A).

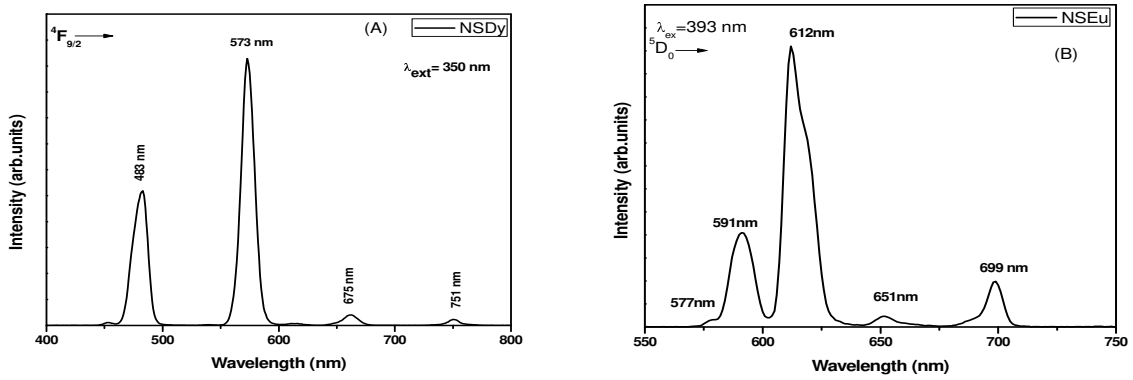

Figure 4: Emission spectra for the (A) Dy doped and (B) Eu doped glasses.

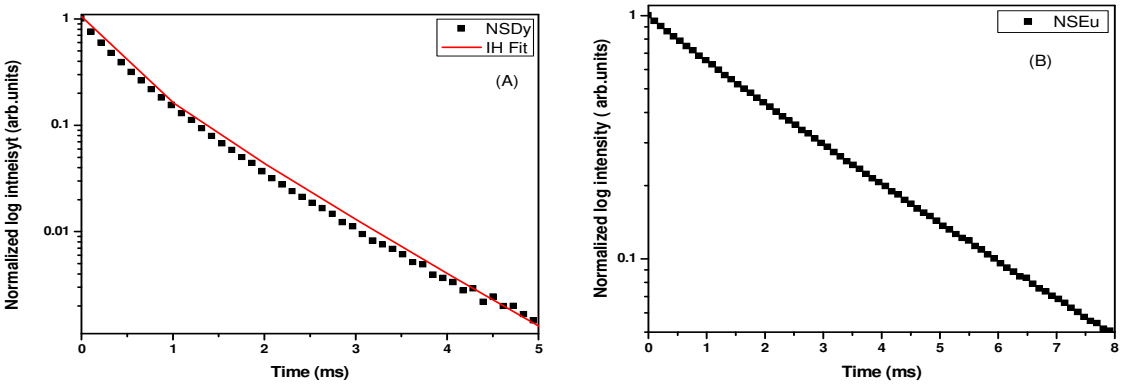

Figure 5: Decay time profile for the (A) Dy doped and (B) Eu doped glasses.

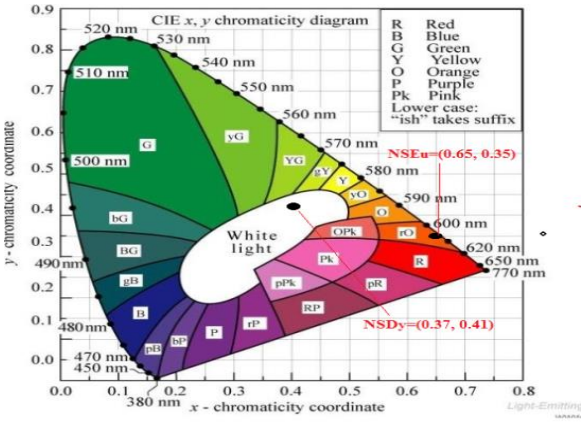

Figure 6: CIE diagram for the NSDy doped and NSEu glasses.

Ten excitation peaks were observed in the analysis of the NSEu sample. The peaks at 275 and 312 nm were thought to corresponding to the Gd^{3+} transitions as mentioned for the NSDy sample. Other peaks were observed at 299, 361, 381, 393, 464, and 524 nm. These were thought to correspond to the transition of Eu^{3+} ions from the ground state, $^7F_0$, to excited states $^5F_1$, $^5D_4$, $^5G_2$, $^5L_6$, $^5D_2$ and $^5D_1$. The two peaks at 414 and 533 nm were thought to correspond to the $^7F_1 \rightarrow {}^5D_3$, and $^7F_1 \rightarrow {}^5D_1$ transitions. A charge transfer band (CT) was also observed in the NSEu sample. This represents a transfer of charge between the metal and ligand ions within the sample. Wavelengths of 350 and 393 nm were used to record the emission spectra for respective samples of NSDy and NSEu because the peaks were the most intense. The excitation transitions for both types of glass are illustrated in the partial energy level diagram shown in Fig. 3(B).

Fig. 4 shows the emission spectra of the NSDy and NSEu glass samples. The characteristics of the Dy^{3+} and Eu^{3+} ion emission peaks were assessed. The emission spectra of the NSDy glass sample was found to have four transition bands at 483, 573, 675 and 751 nm. These were thought to correspond to transitions of the ions from the $^4F_{9/2}$ level to $^6H_{15/2}$, $^6H_{13/2}$, $^6H_{11/2}$ and $^6H_{9/2}$+$^4F_{11/2}$ levels. The NSEu spectra had five emission peaks at 577, 591, 612, 651 and 699 nm. These were thought to correspond to transitions from the 5D0 level to the $^7F_1$, $^7F_2$, $^7F_3$ and $^7F_4$ levels. The peaks at 573 nm for NSDy and 612 nm for NSEu showed the highest intensity. and were therefore used

Table 2. Comparison of J-O intensity parameters with reported studies.

References	Ω_2 (10^{-20} cm^2)	Ω_4 (10^{-20} cm^2)	Ω_6 (10^{-20} cm^2)	Trend
Dy^{3+} Doped glass				
NSDy [present work]	12.38	6.31	3.20	$\Omega_2 > \Omega_4 > \Omega_6$
DyZB10P [13]	3.950	0.915	1.954	$\Omega_2 > \Omega_4 > \Omega_6$
PPBAlDy [14]	8.92	2.55	2.99	$\Omega_2 > \Omega_4 > \Omega_6$
PZSMDy1.0 [15]	11.07	2.8157	2.1841	$\Omega_2 > \Omega_4 > \Omega_6$
CaDy [16]	1.36	0.03	0.56	$\Omega_2 > \Omega_6 > \Omega_4$
1 DZCTFB [17]	9.51	1.8380	3.9577	$\Omega_2 > \Omega_6 > \Omega_4$
LSBP1Dy [18]	5.24	2.32	1.97	$\Omega_2 > \Omega_4 > \Omega_6$
Eu^{3+} doped glass				
NSEu [present work]	15.65	10.00	8.12	$\Omega_6 > \Omega_4 > \Omega_2$
G1 glass [19]	14.83	4.01	2.87	$\Omega_2 > \Omega_4 > \Omega_6$
G2 glass [19]	17.25	4.62	4.6	$\Omega_2 > \Omega_4 > \Omega_6$
PbFPDy10 [20]	7.12	1.59	2.20	$\Omega_2 > \Omega_6 > \Omega_4$
PKMAFDy [21]	7.04	1.73	1.57	$\Omega_2 > \Omega_4 > \Omega_6$
PKAICaFEU10 [22]	7.49	6.30	0.50	$\Omega_2 > \Omega_4 > \Omega_6$

Table 3. The radiative transition probability (Ar, (s^{-1})), stimulated emission cross section (σ,10^{-21} cm^2)) and branching ratio (β_r) of prepared glasses

Transitions	A_R	Σ	$\beta_{R(exp)}$	$\beta_{R(cal)}$
NSDy ($^4F_{9/2}$ →)				
$^6H_{15/2}$	199	0.55	0.37	0.13
$^6H_{13/2}$	978	5.70	0.59	0.63
$^6H_{11/2}$	113	0.97	0.02	0.07
$^6H_{9/2}$	110	1.08	0.01	0.07
NSEu ($^5D_0$ →)				
$^7F_0$	—	—	0.01	—
$^7F_1$	53	2.92	0.12	0.05
$^7F_2$	1131	44.32	0.80	0.75
$^7F_3$	—	—	0.02	—
$^7F_4$	245	30.60	0.06	0.17

for excitation spectra recording. From Fig.4 it can be clearly seen that the glass samples that were produced show efficient luminescence. This is also supported by the high Ω_2 values.

The radiative properties of the glass samples are listed in Table.3. It can be seen that the NSDy sample demonstrated higher values of radiative transition probability, stimulated emission cross section and branching ratio for $^4F_{9/2}$→$^6H_{11/2}$ transitions than the other sample. The NSEu sample showed higher values of these properties for $^5D_0$→$^7F_2$ transition.

3.4. Decay time profile

Fig. 5 show the decay time profile of the glass samples that were produced. The decay time was measured for NSDy at 350 nm excitation wavelength and 573 nm emission wavelength. It was found to be 0.51 ms. The decay profile for the NSDy sample showed non-exponential behavior. This finding fits within the frame work of IH model with S=6[23]. This indicates the dipole-dipole energy transfer between the Dy^{3+}-Dy^{3+} ions and the energy transfer parameter. For the NSDy glass sample it was found to be 0.48. The decay time for the NSEu sample was measured at an excitation wavelength of 393 nm and an emission wavelength of 612 nm. It was found to 2.51 ms. the Energy transfer between the Eu^{3+}-Eu^{3+} ion is not dominant and hence the decay time curve shows single exponential behavior.

3.5. CIE diagram

The emission color of two types of glass was compared using their color coordinates. In 1931; the Commission International de I'Eclairage (CIE) established a universal quantitative model of color spaces, the CIR diagram [24]. Fig. 6 shows the CIE diagram for the two types of glass that were studied.

From the CIE diagram it is clear that the NSDy glass emits visible light in the white region of light spectrum while the NSEu emits visible light in the reddish-orange region. This is the same result as was obtained by visually inspecting the samples. It confirms that the two types of glass may be useful in solid state lighting devices.

4. Conclusions

Dy_2O_3 and Eu_2O_3 doped phosphate glass was prepared using the melt quenching process. The properties the glass samples that were produced were investigated. The absorption spectra for both glass samples had differing characteristics absorption peaks. The excitation spectra for NSDy and NSEu showed the strongest peaks at 350 and 393 nm respectively. The were thought to correspond to the transition from the respective ground states $^6H_{15/2}$ and $^7F_0$ to the excited states $^6F_{11/2}$ + $^6H_{9/2}$ and $^5L_6$. The JO intensity parameter Ω_2 was calculated. It showed relatively high values for the samples that were produced. This reflects the higher asymmetry around rare earth ions in

glass structures. This is thought to result in more efficient radiation emission. This confirms the usefulness of the glass types that were produced in solid state lighting applications. The emission peaks at 573 nm for NSDy and 612 nm for NSEu showed the highest intensity. These peaks were thought to correspond to the transitions $^6H_{13/2} \rightarrow {}^4F_{9/2}$ and $^5D_0 \rightarrow {}^7F_3$. The radiative properties such as radiative transition probability, stimulated emission cross section and branching ratio were calculated for the samples using JO theory. The value that was found for branching ratio was higher than 50%. This indicates that the glass may be suitable for use in lasers. The CIE coordinates were found for the samples. These showed that the NSDy glass emits in the white region and NSEu glass emits in the reddish orange region. From the results that were obtained in this study and those from previous studies, it is concluded that both of the glass systems that were produced would be good candidates for applications in the field of solid-state lighting.

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