



Influences the Addition of Neodymium Oxide on Structural and Optical Properties of Oxyfluoride Lead Borate Glass

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Abstract

The rare-earth-doped glasses have a great significance for promise applications in optical devices and laser technology. The glass samples prepared by melt quenching technique. XRD, FTIR, UV/VIS/NIR spectrophotometer and photoluminesces used to study the structure and properties of the glass samples. And found that the glass samples in the amorphous, the lead oxide enters the glass network as a former, analysis of splitting of Nd³⁺ in glasses, UV—Vis—IR spectra exhibit ten prominent bands centered at 428, 457, 471, 511, 524, 582, 624, 679, 744, 803 and 875 nm correspond to the transition from the ground state ⁴I_{9/2} to ²P_{1/2}, ⁴G_{11/2}, ²D_{3/2+2}G_{9/2+2}P_{3/2}, ⁴G_{9/2}, ²K_{13/2+4}G_{7/2}, ⁴G_{5/2+2}G_{7/2}, 2H_{11/2}, ⁴F_{9/2}, ⁴S_{3/2+4}F_{7/2}, ⁴F_{5/2+2}H_{9/2}, and ⁴F_{3/2} respectively.

The Judd–Ofelt intensity parameters Ω_λ (λ=2, 4, 6) determined from the absorption bands intensities of as Ω₆>Ω₄>Ω₂. The emission peak intensity at 625 nm decreases with the Nd³⁺ concentrations increase due to the quenching effect.

Keywords: Lead Oxyfluoride borate glass; XRD technique; DTA technique; FTIR spectroscopy; Density; Optical properties absorption and emission

1. Introduction

The glass system PbO-B₂O₃ display a low rate of crystallization, it is humidity resistant, stable, transparent, and has the highest refractive index [1]. Glass containing rare earth exhibit broad absorption and emission bands owing to the heterogeneous crystal field inherent to the amorphous structure [2]. Oxide and oxyfluoride lead borate glass system containing Rare earth ions belong to heavy metal, glass family that used for optical applications) [3-5]. The luminescence of alkaline-earth Fluoro halides containing Pb²⁺ studied and found that this emission originates from two excited state. Next, to a normal ultraviolet emission (³P_{0,1}→₁S₀) a visible emission found and ascribed to a transition from a higher level, in all probability of charge-transfer nature (D transition) [6]. Joanna Pisarska studied the laser properties of lead borate glass containing Nd³⁺ ions and found Luminescence in the visible and near-infrared region represent ⁴F_{3/2}—⁴I_{9/2}, ⁴F_{3/2}—⁴I_{11/2}, and ⁴F_{3/2}—⁴I_{13/2} transitions of Nd³⁺ [7].

J. Suresh Kumar [5] prepared neodymium doped calcium fluoroborate glasses. The optical absorption, NIR fluorescence and visible upconversion of the

samples investigated. The Radiative parameters and the experimental lifetime determined.

In our investigated, we tend to demonstrate the recent results obtained lead borate glasses, wherever PbO and PbF₂ are in equal concentration with different concentration of rare earth and therefore the PbO completely substituted by PbF₂.

2. Experimental Details

Glass composition (70 B₂O₃ - 15 PbO - 15 PbF₂) doped with x Nd₂O₃, x = 0, 1, 3, 5 and 10 mol % and sample (70 B₂O₃ - [30-y] PbO - y PbF₂) where y=0 and 30 mol % created by the melt quenching technique.

The samples melted at 1150 °C for 1 hour in an electric muffle furnace (LENTON). A pair of copper blocks used to quench the glass samples. The crystalline and amorphous of glass samples examined using Philips analytical X-Ray diffraction. X-ray unit kept constant at 40 KV with a current of 30 mA. The samples pounded into fine powder and then mixed with potassium bromide and measured using an infrared spectrometer (type JASCO FT/ IR-4100).

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The optical absorption of glass samples examined using a spectrophotometer (type JASCO, V-570). The luminescence bands of samples tested using (JASCO-FP-6300) Spectrofluorometer.

3. Results and Discussion

From the measured sample at XRD, it can be observed that the prepared glass samples in amorphous nature as shown in figure 1.

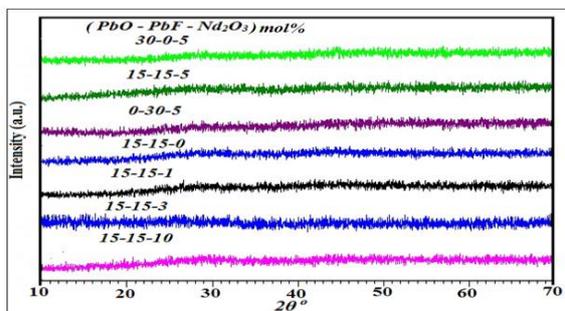


Fig. 1. XRD of lead borate and lead fulgurate glass samples containing neodymium oxide.

Figure (2 and 3) show the infrared spectra of lead oxyfluoride borate glass containing various neodymium oxide content and other samples containing different content of PbO and PbF₂. The vibration types of the borate glass network comprise of three critical band regions [8].

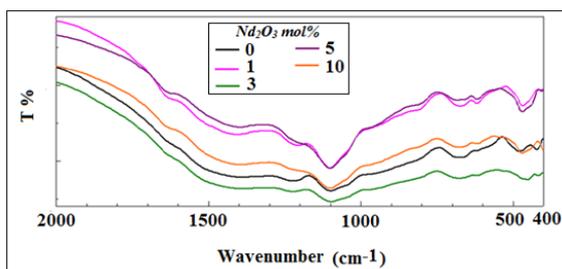


Fig. 2. FTIR spectra of lead borate and lead fluoride glass samples containing neodymium oxide.

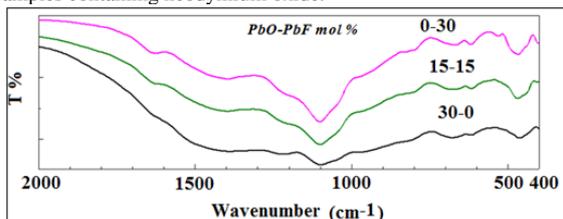


Fig. 3. Effect of the substitution of PbO by PbF₂ on FTIR spectra of glass samples containing neodymium oxide.

In this work, the band observed around a wavenumber 1620 cm⁻¹ is owing to the H₂O [9]. The bands that represent the attendance of lead oxide as (PbO₄) indicated at 460 cm⁻¹ and 470 cm⁻¹, 670 cm⁻¹ and at 1100 cm⁻¹ and these bands overlap with the other forms of borate bands [[10], [11]]. The band appears at (400–650cm⁻¹) indicate the B—O—B and Pb—O—B bending vibration in borate ring deformation [12]. The 1400 cm⁻¹ band is due to B—O

stretching vibrations of trigonal BO₃ units [10]. The band at 1203 cm⁻¹ may attribute to the asymmetric stretching vibration of B—O bonds from orthoborate groups [10,13]. The band formed in the range between 844 and 1120 cm⁻¹ denotes that numerous possible structural groups (di-, tri-, tetra- or Penta borate units) in the [BO₄] vibration group units. The two bands at 671 and 617 cm⁻¹ due to the B—O—B bond bending vibrations from pentaborate groups as the attendance of lead as lead oxide and lead fluoride (the fluorine and oxygen similar to electronegativity, mass and ionic radius) [13]. Therefore, is precipitation within the network not expected to show by deep changes in the mid-IR spectra.

Usually, per Funck the vibrations of B—F bonds presence at 1454 and 1075 cm⁻¹ for BF₃ and BF₄ species higher frequencies than those of B—O bonds that saw at 1400 and 1030 cm⁻¹. Thus, although no definite bands owing to B—F stretching vibrations are present in the IR spectra of fluorinated glasses, the observed trends are indicative of fluorine participation within the network. This affected mainly by four-coordinating atoms and to a lesser extent by three-coordinating boron (BO₂F) units [14].

The effect of fluorine on the lead borate glass appeared from the deconvolution of the glass prepared from the concentration of 30% PbO and glass prepare from 30 mol% PbF₂ in figure 4.

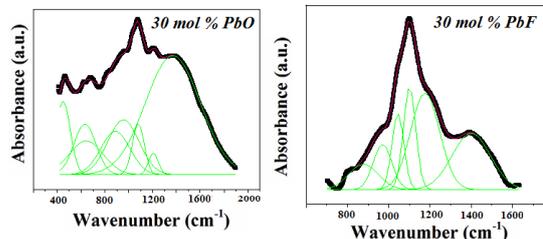


Fig. 4. Deconvolution of the glass samples containing 30% PbO and 30 mol% PbF₂.

From the deconvolution, it often calculated the $N_4 = BO_4/(BO_4+BO_3)$ that equal 0.368 for 30% PbO glass and 0.75 for 30% PbF₂ that the addition of fluorine increase the N_4 value that represents the increase the BO₄ that certain the above result.

From the deconvolution, it can be calculated the $N_4 = BO_4/(BO_4+BO_3)$ that equal 0.368 for 30% PbO glass and 0.75 for 30% PbF that the addition of fluorine increase the N_4 value that represents the increase the BO₄ that sure the above result.

Optical absorption spectra of Nd³⁺ doped in lead borate glasses are recorded at room temperature in the wavelength range from 300 nm to 1000 nm and are shown in Fig. 5. Figure 5 shows 11 noticeable well-resolved absorption bands nearly at 428, 457, 471, 511, 524, 582, 624, 679, 744, 803 and 875 nm and these are nominated as ²P_{1/2}, ⁴G_{11/2}, ²D_{3/2}+²G_{9/2}+²P_{3/2}, ⁴G_{9/2}, ²K_{13/2}+⁴G_{7/2}, ⁴G_{5/2}+²G_{7/2}, ²H_{11/2}, ⁴F_{9/2}, ⁴S_{3/2}+⁴F_{7/2}, ⁴F_{5/2}+²H_{9/2} and ⁴F_{3/2} respectively.

From table 1 and 2 observed the RMS is small. Figure 6 and 7 shows the change of J-O parameters in glass samples under study.

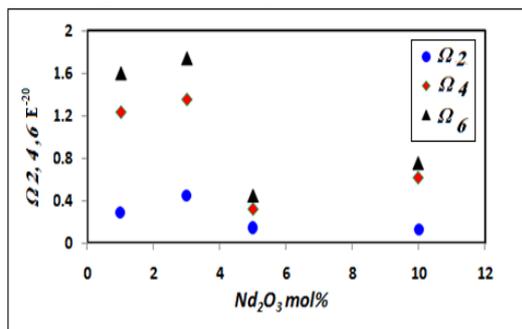


Fig. 6. J-O parameters lead borate glass containing lead oxide and lead fluoride containing different content of neodymium oxide.

The strength of three parameters, Ω_2 , Ω_4 and Ω_6 , give knowledge about the homogeneity and construction of the RE³⁺ ion site, the strength of the electron-phonon coupling and the degree of covalence RE-O [17]. From figure 6 can observe that the value of Ω_2 , Ω_4 , and Ω_6 increase up to 3mol% Nd³⁺ and then decreases. Also, the results validate that the glass sample containing 3 mol% Nd₂O₃ matrix has a larger Ω_2 and Ω_6 parameter value in glass matrices, which indicates the higher covalency of the Nd-O bond in this sample, symmetry of ligand field around Nd³⁺ site, additionally has higher rigidity and strong interaction between 4f and 5d orbits than other samples of the glass matrices [[18], [17], [19], [20], [21], [22], [23]].

The increase of Ω_2 indicates that the effects of short-range structural disturbance and relatively high symmetry in areas surrounding Nd³⁺ ions are increasing and the covalency effects in the Nd-O bonds increases, which provides a rise in Ω_6 values. Figure 7 illustrates the substitution of PbO by PbF₂ on the strength of three parameters, Ω_2 , Ω_4 , and Ω_6 . From this figure can observe that the glass sample containing 30 mol% PbF₂ matrixes has a higher Ω_2 and Ω_6 parameter value in glass matrices that indicates that this sample has higher covalency and higher rigidity than other samples of the glass matrices. And also the glass sample containing PbO and PbF₂ matrix has a lower Ω_2 and Ω_6 parameter value in glass matrices. From the parameters Ω_4 and Ω_6 can calculate the spectroscopic quality factor $Q = \Omega_4/\Omega_6$ [[24]]. When its value is less than 1, there is the probability of spontaneous emission of laser transition for Nd³⁺ (⁴F_{3/2} → ⁴I_{11/2}). The spectroscopic quality factor calculations of all samples under studied smaller than one and the strength of the ⁴F_{3/2} → ⁴I_{11/2} transition are stronger [25].

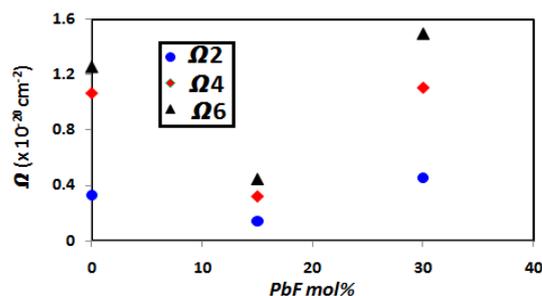


Fig. 7. The substitution of PbO by PbF₂ on the intensity of three parameters, Ω_2 , Ω_4 , and Ω_6 .

The quality of the association is defined as whether it is a covalence or ionic from the calculation of the bonding parameter. If it is positive, the link is covalence and vice versa, if it is negative, it is ionic. The bonding parameter calculated using the Nephelauxetic ratio. Which obtained by $\beta = \nu_c/\nu_a$ where ν_c is a wave number of a transition for an ion in the investigation host and ν_a is the wave number of the same transition for the aqua ion. The value of δ from the average value of β (β') using $\delta = 1 - \beta'/\beta$. The values of β and δ for the present glasses equal 1.008 and -0.00815 respectively. From the β value observed that all prepared glasses have the same Nephelauxetic effect that represents that the glasses have the same 4f-4f transition. The values of δ of glass samples under study are negative, which clear that the nature of Nd³⁺ ligand bond is an ionic. And this result is sure from results obtained from the Judd-Ofelt parameter that has the smallest values of Ω_2 [18,26].

Figure 8 shows the room temperature up-conversion emission spectra under the figure 8 shows the luminescence spectra of samples excited with a wavelength of 314 nm. From figure 8, can be observed one emission band at 625 nm, because of the transitions [⁴H_{11/2} → ⁴I_{9/2}], [⁴G_{5/2} → ⁴I_{11/2}], [²G_{7/2} → ⁴I_{11/2}], [²G_{7/2} → ⁴I_{13/2}] or [²G_{9/2} → ⁴I_{13/2}]. This agrees with Silva et al. [27] and Balda et al. results [28]. The emission intensity reduces with the Neodymium content expanded by a factor to five for Nd³⁺ up to 10 mol%. The quenching in the luminescence strength attributed to the distance between Nd³⁺ and Pb²⁺ reduce [29]. The emission strength reduces with the increase of Nd³⁺ content because of concentration quenching at higher doping levels, that generally observed in RE³⁺ doped glasses/crystals [30-32]. Two important effects were seen in lead borate systems doped with (Pr³⁺ or Eu³⁺ or Er³⁺ or Dy³⁺).

First, the infrared bands shifted to higher wavelengths (lower frequency), once PbO was completely replaced by PbF₂. Second, the intensity of the band 850-1050 cm⁻¹ decreases. This indicates that the number of BO₄ units that were reduced by replacing PbO with PbF₂ and supposed BO₄ → BO₃ back change appears [33,34].

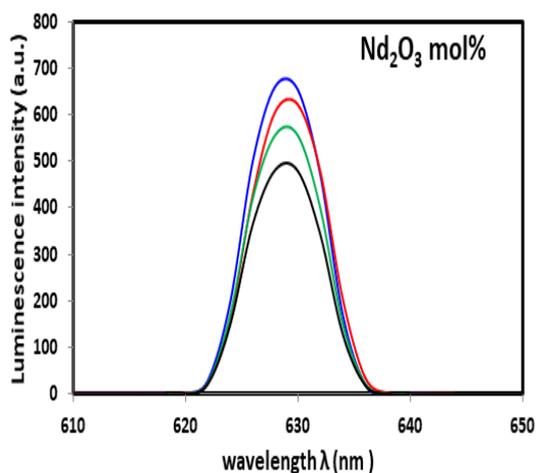


Fig. 8. the luminescence spectra of samples excited with a wavelength of 314 nm. — 1 — 3 — 5 — 10 mol % Nd_2O_3 .

In the present work, when PbO completely supplanted by PbF_2 in lead borate glass containing Nd observed the opposite behavior. From infrared results, the quantity of BO_4 units increased by the exchange of PbO by PbF_2 , then the $\text{BO}_3 \rightarrow \text{BO}_4$ change appears. Effect of PbF_2 on the thermal behavior of lead borate glasses containing neodymium studied using differential scanning calorimetry (DSC).

The glass transition temperature for lead borate glasses containing (0 -30), (15, 15) and (30, 0) mol % ($\text{PbF}_2 - \text{PbO}$) equal 552, 556 and 550 °C respectively. It is clear that the glass transition temperature of the sample containing equal share from PbF_2 and PbO has a higher T_g than other samples containing PbF_2 or PbO alone. Which indicate that the value of T_g strongly depends on the O/F ratio in the glass composition.

Previous researchers [35,36] found that replacing the oxide element in the fluoride matrix leads to an increase in glass transition temperatures, due to the formation of stronger chemical bonds. We found the reverse behavior in our samples.

First, if PbF_2 dissolves within the glass structure and acts as a network modifier, its increase leads to a significant decrease in the value of the glass transition temperatures. Secondly, the bond between Pb - O is more covalent than the bond between Pb - F. This illustrates the effect of the Pb-F bond on the elements surrounding Nd^{3+} ion and increases it by increasing PbF_2 . Therefore, the bond between Nd-O / F is less covalent.

Therefore, according to the value of T_g , the addition of fluoride ions in the glass structure decreases the strength of the average chemical bond that reduces the bond links within the glass network.

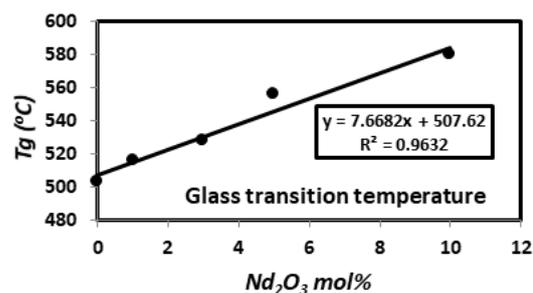


Figure 9. The glass transition temperature of glass samples containing neodymium oxide.

Figure 9. displays the glass transition temperature increase with the increasing of neodymium content. Figure 10 shows the luminescence spectra of lead borate glass samples. From it can be observe that the glass samples provide a broad emission band at 630 nm with excitation of 314 nm. And also the PbO substitution by PbF_2 caused the decrease in the emission intensity.

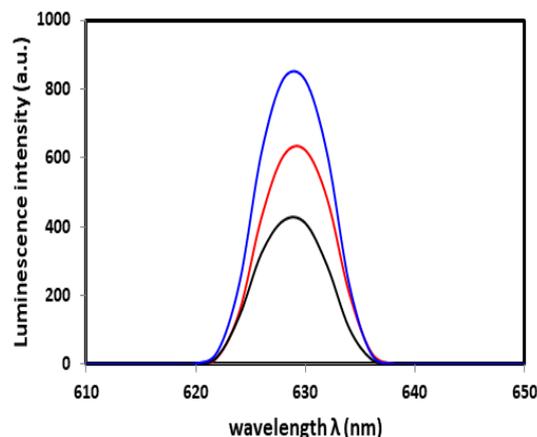


Fig. 10. The emission spectra of lead borate glass containing lead oxide and lead fluoride. — 30 — 15 — 0 mol % PbF_2 .

4. Conclusions

Glass samples of composition (70 B_2O_3 -15 PbO -15 PbF_2) doped with $x\text{Nd}_2\text{O}_3$, $x = 0, 1, 3, 5$ and 10 mol% and sample (70 B_2O_3 - [30 - y] PbO - y PbF_2 - 5 Nd_2O_3) where $y=0$ and 30 mol % prepared. JO parameters obtained from JO theory revealed a trend of $\Omega_6 > \Omega_4 > \Omega_2$ for prepared glass samples.

Glass sample containing 30 mol% PbF_2 matrixes has a higher Ω_2 and Ω_6 parameter value indicates the glass sample possess a highly covalent character. And also has higher rigidity than other samples of the glass matrixes. And the glass sample containing mixed of PbO and PbF_2 has a lower Ω_2 and Ω_6 parameter value. The emission strength decrease as the Nd^{3+} concentration increased due to the quenching effect.

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