



## 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 Nd<sup>3+</sup> 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 <sup>4</sup>I<sub>9/2</sub> to <sup>2</sup>P<sub>1/2</sub>, <sup>4</sup>G<sub>11/2</sub>, <sup>2</sup>D<sub>3/2+2</sub>G<sub>9/2+2</sub>P<sub>3/2</sub>, <sup>4</sup>G<sub>9/2</sub>, <sup>2</sup>K<sub>13/2+4</sub>G<sub>7/2</sub>, <sup>4</sup>G<sub>5/2+2</sub>G<sub>7/2</sub>, 2H<sub>11/2</sub>, <sup>4</sup>F<sub>9/2</sub>, <sup>4</sup>S<sub>3/2+4</sub>F<sub>7/2</sub>, <sup>4</sup>F<sub>5/2+2</sub>H<sub>9/2</sub>, and <sup>4</sup>F<sub>3/2</sub> respectively.

The Judd–Ofelt intensity parameters Ω<sub>λ</sub> (λ=2, 4, 6) determined from the absorption bands intensities of as Ω<sub>6</sub>>Ω<sub>4</sub>>Ω<sub>2</sub>. The emission peak intensity at 625 nm decreases with the Nd<sup>3+</sup> 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-B<sub>2</sub>O<sub>3</sub> 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 Pb<sup>2+</sup> studied and found that this emission originates from two excited state. Next, to a normal ultraviolet emission (<sup>3</sup>P<sub>0,1</sub>→<sub>1</sub>S<sub>0</sub>) 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 Nd<sup>3+</sup> ions and found Luminescence in the visible and near-infrared region represent <sup>4</sup>F<sub>3/2</sub>—<sup>4</sup>I<sub>9/2</sub>, <sup>4</sup>F<sub>3/2</sub>—<sup>4</sup>I<sub>11/2</sub>, and <sup>4</sup>F<sub>3/2</sub>—<sup>4</sup>I<sub>13/2</sub> transitions of Nd<sup>3+</sup> [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 PbF<sub>2</sub> are in equal concentration with different concentration of rare earth and therefore the PbO completely substituted by PbF<sub>2</sub>.

### 2. Experimental Details

Glass composition (70 B<sub>2</sub>O<sub>3</sub> - 15 PbO - 15 PbF<sub>2</sub>) doped with x Nd<sub>2</sub>O<sub>3</sub>, x = 0, 1, 3, 5 and 10 mol % and sample (70 B<sub>2</sub>O<sub>3</sub> - [30-y] PbO - y PbF<sub>2</sub>) 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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Receive Date: 30 December 2020, Accept Date: 15 January 2021

DOI: 10.21608/EJCHEM.2021.55860.3183

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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.

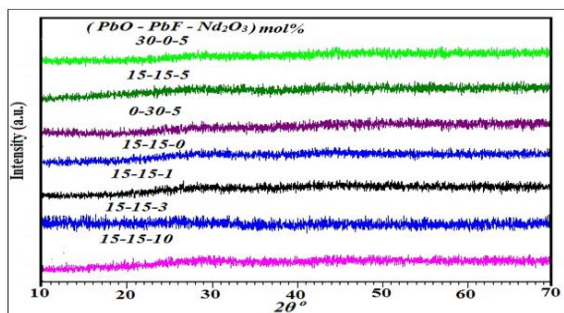


Fig. 1. XRD of lead borate and lead fulgurate glass samples containing neodymium oxide.

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 PbF<sub>2</sub>. The vibration types of the borate glass network comprise of three critical band regions [8].

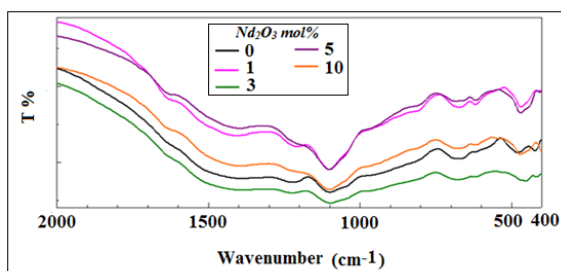


Fig. 2. FTIR spectra of lead borate and lead fluoride glass samples containing neodymium oxide.

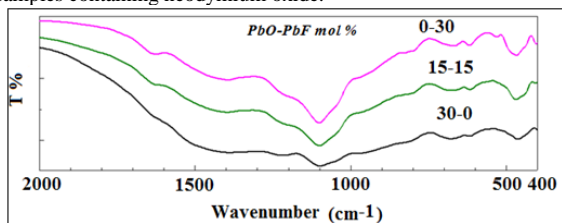


Fig. 3. Effect of the substitution of PbO by PbF<sub>2</sub> on FTIR spectra of glass samples containing neodymium oxide.

In this work, the band observed around a wavenumber 1620 cm<sup>-1</sup> is owing to the H<sub>2</sub>O [9]. The bands that represent the attendance of lead oxide as (PbO<sub>4</sub>) indicated at 460 cm<sup>-1</sup> and 470 cm<sup>-1</sup>, 670 cm<sup>-1</sup> and at 1100 cm<sup>-1</sup> and these bands overlap with the other forms of borate bands [[10], [11]]. The band appears at (400–650cm<sup>-1</sup>) indicate the B—O—B and Pb—O—B bending vibration in borate ring deformation [12]. The 1400 cm<sup>-1</sup> band is due to B—O

stretching vibrations of trigonal BO<sub>3</sub> units [10]. The band at 1203 cm<sup>-1</sup> 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<sup>-1</sup> denotes that numerous possible structural groups (di-, tri-, tetra- or Penta borate units) in the [BO<sub>4</sub>] vibration group units. The two bands at 671 and 617 cm<sup>-1</sup> 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<sup>-1</sup> for BF<sub>3</sub> and BF<sub>4</sub> species higher frequencies than those of B—O bonds that saw at 1400 and 1030 cm<sup>-1</sup>. 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 (BO<sub>2</sub>F) 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% PbF<sub>2</sub> in figure 4.

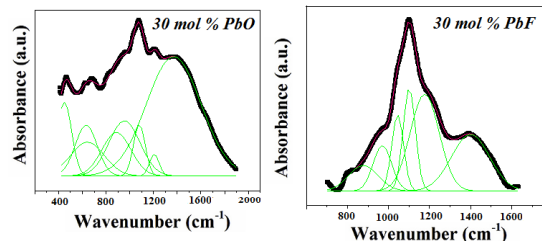


Fig. 4. Deconvolution of the glass samples containing 30% PbO and 30 mol% PbF<sub>2</sub>.

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% PbF<sub>2</sub> that the addition of fluorine increase the  $N_4$  value that represents the increase the BO<sub>4</sub> that certain the above result.

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Optical absorption spectra of Nd<sup>3+</sup> 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 <sup>2</sup>P<sub>1/2</sub>, <sup>4</sup>G<sub>11/2</sub>, <sup>2</sup>D<sub>3/2</sub>+<sup>2</sup>G<sub>9/2</sub>+<sup>2</sup>P<sub>3/2</sub>, <sup>4</sup>G<sub>9/2</sub>, <sup>2</sup>K<sub>13/2</sub>+<sup>4</sup>G<sub>7/2</sub>, <sup>4</sup>G<sub>5/2</sub>+<sup>2</sup>G<sub>7/2</sub>, <sup>2</sup>H<sub>11/2</sub>, <sup>4</sup>F<sub>9/2</sub>, <sup>4</sup>S<sub>3/2</sub>+<sup>4</sup>F<sub>7/2</sub>, <sup>4</sup>F<sub>5/2</sub>+<sup>2</sup>H<sub>9/2</sub> and <sup>4</sup>F<sub>3/2</sub> respectively.

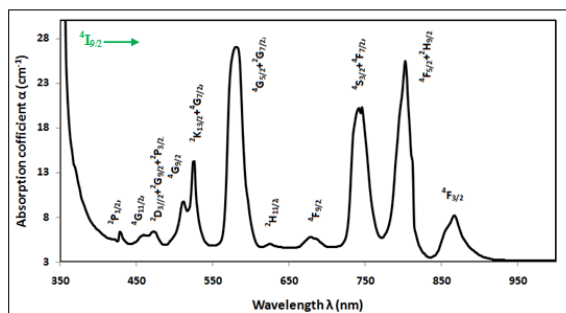


Fig. 5. Optical absorption spectra of  $\text{Nd}^{3+}$  doped in lead borate glasses.

By using the Judd–Ofelt theory the experimental and theoretical spectral intensities of various bands of

$\text{Nd}^{3+}$  in lead borate glasses obtained [15,16]. The oscillator strengths for an electric dipole transition within the 4f $n$  configurations between two multiples separated are calculated at the interval the framework of the Judd–Ofelt theory.

The deviation between the experimental and calculated oscillator strengths (RMS) determined using [15]:

$$\text{rms}\Delta f = \sqrt{\frac{\sum_{i=1}^L (f_{\text{exp}} - f_{\text{cal}})^2}{L-3}}$$

Where  $L$  is the number of absorption bands. The consequence of substitution  $\text{PbO}$  by  $\text{PbF}_2$  and the supplement of the various content of  $\text{Nd}_2\text{O}_3$  on the values of  $f_{\text{exp}}$ ,  $f_{\text{cal}}$  and rms tabulated in table 1 and 2.

Table 1. The effect of the addition of the various content of  $\text{Nd}_2\text{O}_3$  on the values of  $f_{\text{exp}}$ ,  $f_{\text{cal}}$  and rms

$\lambda$ (nm)	PbO-PbF-Nd <sub>2</sub> O <sub>3</sub>					
	15-15-5		30-0-5		0-30-5	
	$f_{\text{exp}}$	$f_{\text{cal}}$	$f_{\text{exp}}$	$f_{\text{cal}}$	$f_{\text{exp}}$	$f_{\text{cal}}$
868	0.203	0.211	0.706	0.667	0.735	0.725
802	0.801	0.727	2.28	2.13	2.67	2.44
746	0.680	0.782	1.94	2.21	2.30	2.61
680	0.048	0.0601	0.137	0.172	0.160	0.201
582	1.11	1.15	3.18	3.34	3.70	3.86
524	0.0979	0.350	0.282	1.06	0.334	1.18
510	0.028	0.141	0.0762	0.431	0.0884	0.478
472	0.0573	0.100	0.160	0.308	0.178	0.341
428	0.0106	0.0555	0.0276	0.184	0.0364	0.192
Rms	0.26		0.86		0.9	

Table 2. The effect of substitution  $\text{PbO}$  by  $\text{PbF}_2$  on the values of  $f_{\text{exp}}$ ,  $f_{\text{cal}}$  and rms

$\lambda$ (nm)	Nd <sub>2</sub> O <sub>3</sub> mol%							
	1		3		5		10	
	$f_{\text{exp}}$	$f_{\text{cal}}$	$f_{\text{exp}}$	$f_{\text{cal}}$	$f_{\text{exp}}$	$f_{\text{cal}}$	$f_{\text{exp}}$	$f_{\text{cal}}$
868	0.512	0.765	0.853	0.836	0.203	0.211	0.436	0.395
802	3.23	2.51	3.19	2.74	0.801	0.727	1.34	1.27
746	2.17	2.66	2.64	2.88	0.680	0.782	1.20	1.32
680	0.113	0.205	0.181	0.224	0.048	0.0601	0.0822	0.103
582	3.48	3.44	4.35	4.17	1.11	1.15	1.69	1.74
524	0.308	1.20	0.384	1.33	0.0979	0.350	0.173	0.610
510	0.100	0.496	0.111	0.543	0.028	0.141	0.0512	0.253
472	0.148	0.355	0.210	0.387	0.0573	0.100	0.0946	0.181
428	0.032	0.205	0.0361	0.224	0.0106	0.0555	0.0201	0.107
Rms	0.712		0.627		0.26		0.461	

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.

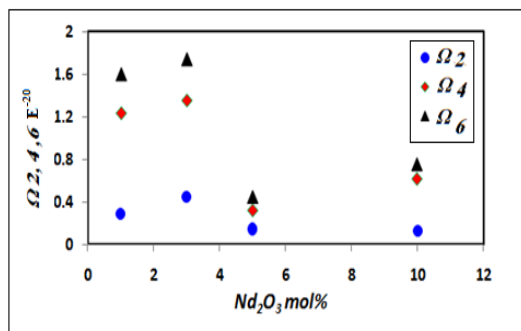


Fig. 6. J-O parameters lead borate glass containing lead oxide and lead fluoride containing different content of neodymium oxide.

The strength of three parameters,  $\Omega_2$ ,  $\Omega_4$  and  $\Omega_6$ , give knowledge about the homogeneity and construction of the RE<sup>3+</sup> 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  $\Omega_2$ ,  $\Omega_4$ , and  $\Omega_6$  increase up to 3mol% Nd<sup>3+</sup> and then decreases. Also, the results validate that the glass sample containing 3 mol% Nd<sub>2</sub>O<sub>3</sub> matrix has a larger  $\Omega_2$  and  $\Omega_6$  parameter value in glass matrices, which indicates the higher covalency of the Nd-O bond in this sample, symmetry of ligand field around Nd<sup>3+</sup> 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  $\Omega_2$  indicates that the effects of short-range structural disturbance and relatively high symmetry in areas surrounding Nd<sup>3+</sup> ions are increasing and the covalency effects in the Nd-O bonds increases, which provides a rise in  $\Omega_6$  values. Figure 7 illustrates the substitution of PbO by PbF<sub>2</sub> on the strength of three parameters,  $\Omega_2$ ,  $\Omega_4$ , and  $\Omega_6$ . From this figure can observe that the glass sample containing 30 mol% PbF<sub>2</sub> matrixes has a higher  $\Omega_2$  and  $\Omega_6$  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 PbF<sub>2</sub> matrix has a lower  $\Omega_2$  and  $\Omega_6$  parameter value in glass matrices. From the parameters  $\Omega_4$  and  $\Omega_6$  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 Nd<sup>3+</sup> (<sup>4</sup>F<sub>3/2</sub> → <sup>4</sup>I<sub>11/2</sub>). The spectroscopic quality factor calculations of all samples under studied smaller than one and the strength of the <sup>4</sup>F<sub>3/2</sub> → <sup>4</sup>I<sub>11/2</sub> transition are stronger [25].

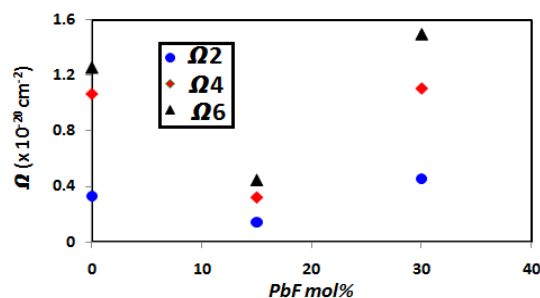


Fig. 7. The substitution of PbO by PbF<sub>2</sub> on the intensity of three parameters,  $\Omega_2$ ,  $\Omega_4$ , and  $\Omega_6$ .

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  $\nu_c$  is a wave number of a transition for an ion in the investigation host and  $\nu_a$  is the wave number of the same transition for the aqua ion. The value of  $\delta$  from the average value of  $\beta$  ( $\beta'$ ) using  $\delta = 1 - \beta'/\beta$ . The values of  $\beta$  and  $\delta$  for the present glasses equal 1.008 and -0.00815 respectively. From the  $\beta$  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  $\delta$  of glass samples under study are negative, which clear that the nature of Nd<sup>3+</sup> ligand bond is an ionic. And this result is sure from results obtained from the Judd-Ofelt parameter that has the smallest values of  $\Omega_2$  [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 [<sup>4</sup>H<sub>11/2</sub> → <sup>4</sup>I<sub>9/2</sub>], [<sup>4</sup>G<sub>5/2</sub> → <sup>4</sup>I<sub>11/2</sub>], [<sup>2</sup>G<sub>7/2</sub> → <sup>4</sup>I<sub>11/2</sub>], [<sup>2</sup>G<sub>7/2</sub> → <sup>4</sup>I<sub>13/2</sub>] or [<sup>2</sup>G<sub>9/2</sub> → <sup>4</sup>I<sub>13/2</sub>]. 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 Nd<sup>3+</sup> up to 10 mol%. The quenching in the luminescence strength attributed to the distance between Nd<sup>3+</sup> and Pb<sup>2+</sup> reduce [29]. The emission strength reduces with the increase of Nd<sup>3+</sup> content because of concentration quenching at higher doping levels, that generally observed in RE<sup>3+</sup> doped glasses/crystals [30-32]. Two important effects were seen in lead borate systems doped with (Pr<sup>3+</sup> or Eu<sup>3+</sup> or Er<sup>3+</sup> or Dy<sup>3+</sup>).

First, the infrared bands shifted to higher wavelengths (lower frequency), once PbO was completely replaced by PbF<sub>2</sub>. Second, the intensity of the band 850-1050 cm<sup>-1</sup> decreases. This indicates that the number of BO<sub>4</sub> units that were reduced by replacing PbO with PbF<sub>2</sub> and supposed BO<sub>4</sub> → BO<sub>3</sub> back change appears [33,34].

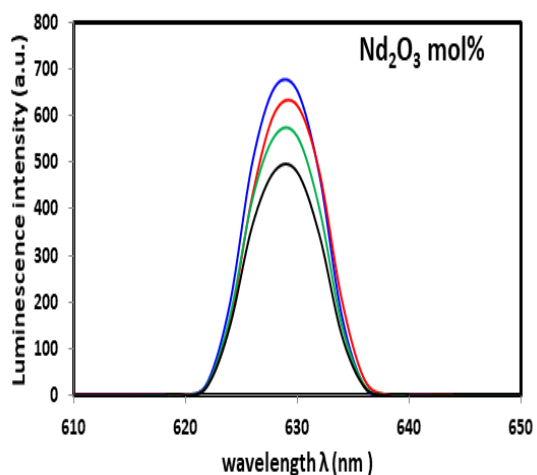


Fig. 8. the luminescence spectra of samples excited with a wavelength of 314 nm. — 1 — 3 — 5 — 10 mol %  $\text{Nd}_2\text{O}_3$ .

In the present work, when PbO completely supplanted by  $\text{PbF}_2$  in lead borate glass containing Nd observed the opposite behavior. From infrared results, the quantity of  $\text{BO}_4$  units increased by the exchange of PbO by  $\text{PbF}_2$ , then the  $\text{BO}_3 \rightarrow \text{BO}_4$  change appears. Effect of  $\text{PbF}_2$  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  $\text{PbF}_2$  and PbO has a higher  $T_g$  than other samples containing  $\text{PbF}_2$  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  $\text{PbF}_2$  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  $\text{Nd}^{3+}$  ion and increases it by increasing  $\text{PbF}_2$ . 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.

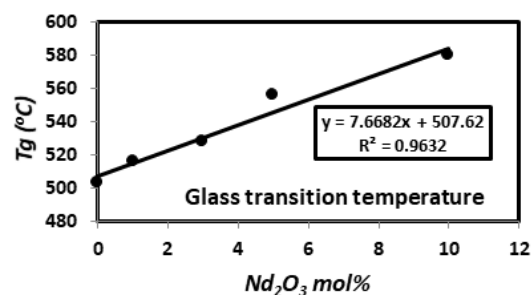


Figure 9. The glass transition temperature of glass samples containing neodymium oxide.

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  $\text{PbF}_2$  caused the decrease in the emission intensity.

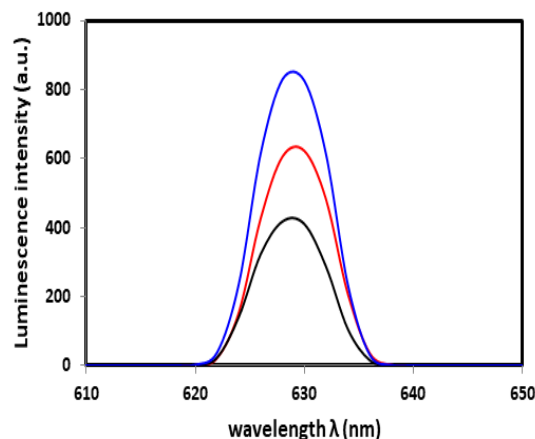


Fig. 10. The emission spectra of lead borate glass containing lead oxide and lead fluoride. — 30 — 15 — 0 mol %  $\text{PbF}_2$ .

#### 4. Conclusions

Glass samples of composition (70  $\text{B}_2\text{O}_3$ -15 $\text{PbO}$ -15  $\text{PbF}_2$ ) doped with  $x\text{Nd}_2\text{O}_3$ ,  $x = 0, 1, 3, 5$  and 10 mol% and sample (70  $\text{B}_2\text{O}_3$  - [30 -  $y$ ]  $\text{PbO}$  -  $y$   $\text{PbF}_2$  - 5  $\text{Nd}_2\text{O}_3$ ) 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%  $\text{PbF}_2$  matrixes has a higher  $\Omega_2$  and  $\Omega_6$  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  $\text{PbF}_2$  has a lower  $\Omega_2$  and  $\Omega_6$  parameter value. The emission strength decrease as the  $\text{Nd}^{3+}$  concentration increased due to the quenching effect.

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