



## Effect of thermal treatment on the structure of Nd<sub>2</sub>O<sub>3</sub> barium borophosphate glasses

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### Abstract

Borate glasses of different Nd<sub>2</sub>O<sub>3</sub> concentrations were prepared by a quenched method. The vitreous samples were all obtained in an amorphous form. The effect of the addition of small amounts from Nd<sub>2</sub>O<sub>3</sub> on the short, intermediate, and long-range ordered structure of xNd<sub>2</sub>O<sub>3</sub> - (40-x) B<sub>2</sub>O<sub>3</sub> - 30 BaO - 25 Na<sub>2</sub>O - 5 P<sub>2</sub>O<sub>5</sub> (0 ≤ x ≤ 7 mol%) has been investigated using different experimental tools. Thermal heat treatment transforms the glass to glass ceramics as evidenced by XRD patterns and TEM-EDP micrographs. It has been investigated how Nd<sub>2</sub>O<sub>3</sub> affects NMR characteristics by altering the coordination of both boron and neodymium. Through the application of <sup>11</sup>B NMR spectroscopy, the quantitative proportion of boron atoms with four-coordinated neighbors (N<sub>4</sub>) has been readily evaluated. The influence of Nd<sub>2</sub>O<sub>3</sub> on the NMR parameters has been examined by considering the changing coordination environments of both boron and neodymium atoms. As the Nd<sub>2</sub>O<sub>3</sub> content increases, the ability of neodymium oxide to act as a network modifier enhances, which is substantiated by the declining fraction of boron tetrahedral units (N<sub>4</sub>) and the chemical shift of boron nuclei (δ). These observations corroborate the anticipated role of Nd<sub>2</sub>O<sub>3</sub> in modifying the network structure.

*Keywords:* NMR Spectroscopy; Neodymium Oxide; Barium Borate glasses; Glass-Ceramics.

### 1. Introduction

Sodium barium borophosphate glasses are an important class of multi-component inorganic materials that possess attributes like good thermal stability, high visible transparency, high rare earth solubility, and the ability for rare earth ion inclusion without clustering [1-4]. This enables their development for various photonic, optical, and magneto-optic applications when doped with rare earth oxides like Nd<sub>2</sub>O<sub>3</sub> [5].

The parent sodium barium borophosphate glass matrix without rare earth additions possesses a set of properties making it well-suited as an excellent host material [6]. It exhibits a wide glass formation region, good energy transfer efficiency to rare earth ions, high transparency exceeding 80% in the visible spectrum, refractive indices tunable between 1.5-1.6, moderately high density approaching 2.8 g/cc, and

thermal stability with glass transition temperatures over 350°C [7].

The sodium barium borophosphate glass system with high rare earth oxide solubility without clustering exhibits a versatile combination of thermal, optical, and spectroscopic properties that enable usage in high-power NIR lasers around 1060 nm, Optical amplifiers, Integrated optical devices, high-density data storage media, and magneto-optic memory, Solid-state lighting when doped with other Lanthanides, optical sensors, and bio-imaging platforms and photovoltaic upconversion for enhanced solar harvesting [8, 9].

Borate glasses are increasingly popular due to their appealing properties [10-12], although silica glasses fulfill most requirements for various applications. Compared to silica glasses, borate glasses offer advantages such as lower transition and

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melting temperatures, superior thermal stability, and excellent transparency [13, 14]. Nonetheless, borate glasses in their unmodified form often lack the desirable characteristics required for practical applications, owing to their susceptibility to chemical degradation and a pronounced tendency to absorb moisture from the environment.

Borate glasses doped with rare earth ions ( $RE^{3+}$ ) have attracted significant research attention due to their suitability for this application [15, 16]. Extensive efforts have focused on understanding how rare earth dopants influence the structural properties of borate glasses. Pure borate glasses typically exhibit poor chemical durability, so they are often combined with other oxides to improve this characteristic. Some of the most common rare earth elements impart unique functionalities like antibacterial or bioimaging properties. Glasses containing rare earth show promise for biomedical uses such as brachytherapy, luminescence imaging, MRI contrast, and more, owing to the electronic configurations of the rare earth ions. Despite the increasing study, the potential of rare earth-doped bioactive glasses as biomaterials remains underexplored [17].

Studying the addition of neodymium oxide ( $Nd_2O_3$ ) in borophosphate glass systems reveals network modification as  $Nd_2O_3$  acts as a network modifier and influencing the structural arrangement of the boron and phosphate units. The presence of  $Nd_2O_3$ , even in trace amounts, has been observed to promote the so-called "borate back conversion" process. This phenomenon involves the partial reversal of the conversion of  $BO_4$  tetrahedral units to  $BO_3$  trigonal units, which can significantly impact the glass properties. Investigating this behavior is crucial for tailoring the glasses for specific applications. Moreover, the broadening of NMR spectra observed with increasing  $Nd_2O_3$  content suggests changes in the structural dynamics of the glass network [18, 19]. Understanding these changes can provide valuable insights into the glass formation mechanisms and the role of rare earth modifiers in influencing the overall glass structure. Borophosphate glasses are often doped with rare earth ions for applications such as solid-state lasers, optical fibers, and luminescent materials. Considerate the role of  $Nd_2O_3$  in these glass systems can aid in optimizing the incorporation and performance of rare earth dopants, leading to enhanced functionality [16, 18].

The current study aims to enhance the crystallinity of base glass compositions by doping with select elements intermixed into the core glass materials. Given the bioactive nature of certain rare earth elements, this work explores incorporating a small quantity of neodymium oxide ( $Nd_2O_3$ ) into a glass

matrix to improve both crystallinity and biocompatibility. Incorporating rare earth oxides helps satisfy the preferred octahedral coordination environments for the rare earth ions, increasing non-bridging oxygens in the glass network structure. The observed depolymerization of the glass network resulting from rare earth doping may promote improved bioactivity [18].

In this study, we concentrate our research on specific types of borate glasses containing  $Nd_2O_3$ . This is because this type can reveal a high surface hardness with a low thermal expansion coefficient. Such characteristics made the glasses to be more resistant to fatigue stresses and temperature changes. It has been observed that adding a network modifier to  $B_2O_3$  causes the B-O bonds to break and  $BO_3$  triangles to become  $BO_4$  tetrahedral units [19]. The main aim of this paper is to investigate the effect of  $Nd_2O_3$  on the structure  $xNd_2O_3 - (40-x) B_2O_3 - 30 BaO - 25 Na_2O - 5 P_2O_5$  ( $0 \leq x \leq 7$  mol%) using different techniques such as XRD, NMR, and FTIR to reveal the correlations between structure and properties of glass doped with rare earth.

## 2. Experimental Methods

### 2.1. Glass Preparation

The nominal  $xNd_2O_3 - (40-x) B_2O_3 - 30 BaO - 25 Na_2O - 5 P_2O_5$  ( $0 \leq x \leq 7$  mol%) glass composition was prepared utilizing melt-quenching process using a porcelain crucible.  $Nd_2O_3$ ,  $H_3BO_3$ ,  $CaCO_3$ ,  $NaCO_3$ , and  $NH_4H_2PO_4$  (chemical precursors) were used as a starting material. The required amount of the last precursor was heated at 300 °C for about 90 min to exclude water and ammonia. Then, the required amounts of the other precursors were well-mixed added to the porcelain crucible, and melted at 1000–1200 °C. The melt was frequently swirled to ensure homogeneity and then poured between two stainless-steel plates. The glass materials were kept in a dissector until the characterization process.

### 2.2. Characterization techniques

X-ray diffraction (XRD) measurements were performed using Shimadzu X-ray diffractometer type Dx-30. Present phases can be identified by comparing intense peak position with the patterns present in the international powder diffraction file (PDF) database of the Joint Committee for powder diffraction standards (JCPDS).  $^{11}B$  and  $^{31}P$  NMR spectra were captured using a high-resolution solid-state MAS NMR spectrometer, JEOL RESONANCE GSX-500. At a frequency of 160.4 MHz and a high external magnetic field of 11.747T. An accumulation of 200–300 scans was recorded for each glass sample scanned with a single pulse length of 0.5–1.0 ms and a pulse delay of 2.5 s. Fourier transform Infrared Spectroscopy (FTIR) spectra were collected from

Bruker spectrometer FTIR at room temperature in the wavenumber range of 4000 to 400 cm<sup>-1</sup> at 2.0 cm<sup>-1</sup> resolution. Data was recorded in absorption mode for pulverized powder for at least three samples of each glass composition. Then the obtained spectra were deconvoluted to enable shedding further light on the structural changes of BO<sub>3</sub> triangles and BO<sub>4</sub> tetrahedra, as described elsewhere.

### 3. Results and Discussion

#### 3.1. Glass Preparation

In the studied system, the resultant structural configuration should be analyzed using XRD diffraction regularly to differentiate between crystalline and amorphous phases upon increasing Nd<sub>2</sub>O<sub>3</sub>. The information gathered verified that glass production is possible in every mixture under investigation. This indicates that all of the glasses under study have an amorphous structure, as seen in Figure 1.

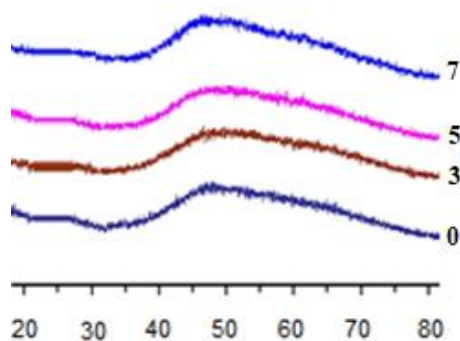


Figure 1. XRD patterns for glasses of different Nd<sub>2</sub>O<sub>3</sub> contents.

The advantage of the present investigated glasses is that they could contain nano-sized crystalline species that can be separated from amorphous glasses through heat treatment processes just above T<sub>g</sub>. The nano-crystalline species are usually indicated by the presence of sharp XRD peaks which confirm that the short-range ordered structure is transformed to the intermediate and long-ordered structure by the effect of thermal heat treatment, Figures 2 and 3 respectively.

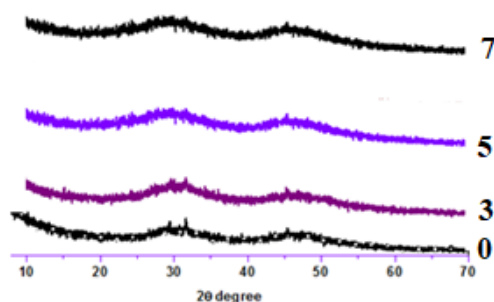


Figure 2, XRD patterns for glass treated at 400 °C

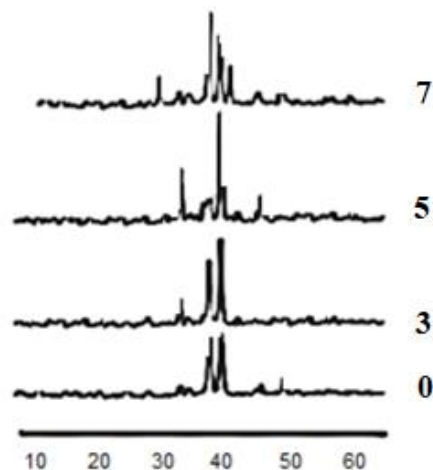


Figure 3. XRD spectra for glasses containing 0, 3, 5, and 7 mol% Nd<sub>2</sub>O<sub>3</sub> treated at 580°C for 8 hours

Micrographs based on TEM –and its electron diffraction patterns (Figure 4) confirm the features based on XRD spectra. Upon the heat treatment, the accumulated crystalline species could be changed and the main crystalline apatite Ba<sub>3</sub>(BO<sub>3</sub>)<sub>2</sub> is dominant.

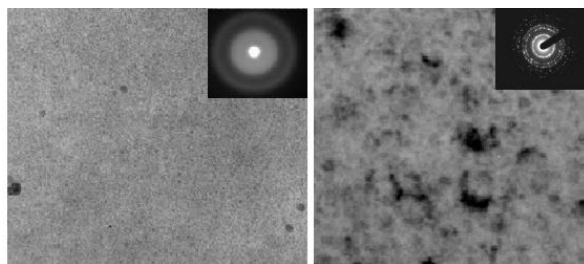


Figure 4 TEM micrograph for samples treated at 400 °C and 580 °C for 8 hours

#### 3.2. <sup>11</sup>B MAS NMR Spectra

Figure 5 shows the <sup>11</sup>B MAS NMR spectra of xNd<sub>2</sub>O<sub>3</sub> - (40-x) B<sub>2</sub>O<sub>3</sub> -30 BaO - 25 Na<sub>2</sub>O - 5 P<sub>2</sub>O<sub>5</sub>, x = 0, 3 and 5 and 7 mol% mol% Nd<sub>2</sub>O<sub>3</sub>. In the spectra of base glass (at x = 0), there are two main peaks at a chemical shift of about 0 and 10 ppm attributed to BO<sub>4</sub> and BO<sub>3</sub>, respectively [20]. With the addition of Nd<sub>2</sub>O<sub>3</sub> the intensity of the peak assigned to BO<sub>4</sub>

tetrahedral groups decreases, while that of  $\text{BO}_3$  triangle groups increases. This indicates qualitatively that the relative concentration of  $\text{BO}_3$  units increases while that of  $\text{BO}_4$  decreases.

This strongly suggests that  $\text{Nd}^{3+}$  delays the transformation of  $\text{BO}_3$  into  $\text{BO}_4$ . It was thus considered that  $\text{Nd}^{3+}$  is sufficiently larger than that of  $\text{Na}^+$  and  $\text{Ba}^{2+}$  to result in the formation of  $\text{BO}_4$  [21]. It therefore can suggest that the Nd/B ratio is important for the nature of the  $\text{BO}_3/\text{BO}_4$  ratio because  $\text{BO}_4$  decreases with being replaced by  $\text{Nd}_2\text{O}_3$  sites. Therefore, considering the above data and suggestion, it is assumed that the amount of  $\text{BO}_4$  is greatly changed concerning increasing  $\text{Nd}_2\text{O}_3$  concentration.

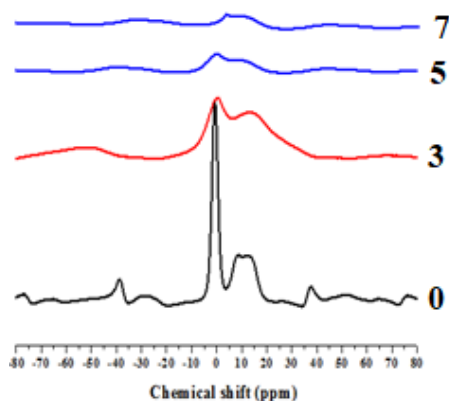


Figure 5  $^{11}\text{B}$  MAS NMR spectra of  $x\text{Nd}_2\text{O}_3\text{-(}46\text{-}x\text{)B}_2\text{O}_3\text{-}27\text{ BaO-}24.4\text{Na}_2\text{O-}2.6\text{P}_2\text{O}_5$  at  $x = 0, 3$  and  $5$  and  $7$  mol% mol%  $\text{Nd}_2\text{O}_3$

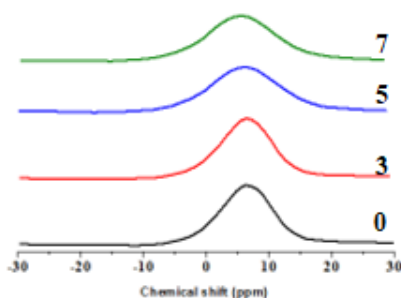
The decrease in the concentration of  $\text{BO}_4$  indicates that some of the network modifiers are withdrawn to convert  $\text{BO}_4 \rightarrow \text{BO}_3$ . This is a surprising behavior, which is unexpected at this lower concentration of  $\text{Nd}_2\text{O}_3$  compared to that of  $\text{B}_2\text{O}_3$ . Indeed, the participation of 5 and 7 mol% of  $\text{Nd}_2\text{O}_3$  in the glass network formation is assumed to be limited since its low concentration is not accessible to withdraw some modifier from the borate network. This indicates that  $\text{Nd}_2\text{O}_3$  does not affect directly the  $\text{BO}_4 \rightarrow \text{BO}_3$  transformation. The so-called back conversion effect has been documented in rare earth-doped borate glasses that have a high concentration of lead oxide as a constituent [22]. In other words, there is another indirect role played by  $\text{Nd}_2\text{O}_3$  which has a main responsibility for back conversion of boron transformation.  $^{11}\text{B}$  NMR for 0 Nd glass should contain a higher  $\text{BO}_4$  fraction. The average full width at half maximum FWHM of  $^{11}\text{B}$  NMR spectra is found to increase. The fast increase in FWHM should be referred to as increasing asymmetric  $\text{BO}_3$  units through the transformation of  $\text{BO}_4$  to asymmetric  $\text{BO}_3$ . The smaller value of FWHM in the base glass leads that only symmetric borate groups are dominant. On the other hand, the higher value of FWHM in glasses containing rare earth support the

latter played the role of back conversion agent.  $\text{BO}_4$  in such a case is highly transformed to asymmetric  $\text{BO}_3$  units which in turn reduce  $\text{BO}_4$  fraction. The formation of B-O-Nd bonds increases the cross-linked bonds in the glass network which leads to increased  $T_g$ , particularly in samples of  $> 0.5$  mol%  $\text{Nd}_2\text{O}_3$ .

### 3.3. $^{31}\text{P}$ MAS NMR Spectra

Direct information on the different  $\text{Q}^n$  species present in the glass network is provided by  $^{31}\text{P}$  MAS NMR measurements. The  $\text{Q}^n$  nomenclature is used to categorize the phosphate tetrahedral, where  $n$  is the number of BOs in each tetrahedral group. Phosphate glass has four distinct units ( $\text{Q}^3$  to  $\text{Q}^0$ ) that may be identified by the nomenclature since  $\text{Q}^4$  cannot be created because of the  $\text{P}=\text{O}$  bond.  $\text{P}_2\text{O}_5$  is present in the glass matrix at a concentration of roughly 5%. Thus, orthophosphate ( $\text{Q}^0$ ) is the dominant structural species that forms the phosphate network, and there is no extra  $\text{Q}^n$  type because the modifier contents are very high exceeding several times of phosphate concentration. Therefore, this composition is corresponding to ( $\text{Q}^0$ ). This has been demonstrated by the recorded  $^{31}\text{P}$  NMR spectra in Figure 6. The dominant peaks are located in around 7 ppm range which correspond to the  $\text{Q}^0$  phosphate units ( $\text{PO}_4$ ) $^{3-}$ . Adding mol%  $\text{Nd}_2\text{O}_3$  does not affect the  $^{31}\text{P}$ -NMR spectra since a similar value of chemical shift is found. It can be observed that the glass free from  $\text{Nd}_2\text{O}_3$  is as well as glasses containing 3, 5, and 7 mol%. Monophosphate ( $\text{PO}_4$ ) $^{3-}$  units are represented by a single, symmetric Gaussian peak. It is believed that the frequency peaks match orthophosphate  $\text{Na}_3\text{PO}_4$ . Spectra from pure crystalline substances are used to confirm the assignments [22]. Furthermore,  $^{31}\text{P}$  NMR spectra of glass containing 7 mol%  $\text{Nd}_2\text{O}_3$  are broader than that of glasses of lower  $\text{Nd}_2\text{O}_3$  contents, the chemical shift is changed toward lower values. This could be explained by the development of certain ordered apatite phases that serve as charge compensators by holding Nd ions. Furthermore, the slight alteration in the phosphate network's NMR chemical shift with the addition of  $\text{Nd}_2\text{O}_3$  may also be the result of  $\text{Nd}_2\text{O}_3$ 's minor phosphate network modification. This indicates that the majority of  $\text{Nd}_2\text{O}_3$  is solely used to alter the borate network, which creates a significant decrease in the fraction of boron tetrahedral units ( $\text{N}_4$ ). The chemical shift in  $^{31}\text{P}$  NMR towards lower values and the slight increase in its FWHM (Table 1), with  $\text{Nd}_2\text{O}_3$  content is following the literature [23, 24].  $\text{Q}^1$  phosphate units ( $\text{P}_2\text{O}_7$ ) $^{4-}$  which vary from -7.0 to -8.7 ppm—also make a little contribution. Their presence in the structures

of the examined glasses alters the evidence of the proximity of phosphorus as the concentration of Nd<sub>2</sub>O<sub>3</sub> increases



**Figure 6:** <sup>31</sup>P NMR spectra of borate glass Nd free and glasses containing respectively 0, 3, 5, and 7 mol% Nd<sub>2</sub>O<sub>3</sub>.

**Table 1: NMR observed absorption peak positions.**

Content of Nd <sub>2</sub> O <sub>3</sub>	(ppm)	FWHM / ppm	Intensity/ %
0 mol % Nd <sub>2</sub> O <sub>3</sub>	2.1	8.2	97
3 mol % Nd <sub>2</sub> O <sub>3</sub>	1.9	8.5	97
5 mol % Nd <sub>2</sub> O <sub>3</sub>	1.7	11	77
7 mol % Nd <sub>2</sub> O <sub>3</sub>	1.4	14	71

#### 4. Conclusions

In binary borate glass systems, Nd<sub>2</sub>O<sub>3</sub> plays a crucial role as a glass former, existing in the form of Nd<sub>2</sub>O<sub>3</sub> units, as demonstrated by NMR spectroscopy data. The NMR spectrum exhibits significant broadening, and the formation of ordered Nd-B-Nd linkages hinders the conversion of trigonal BO<sub>3</sub> units into tetrahedral BO<sub>4</sub> units. A higher concentration of Nd<sub>2</sub>O<sub>3</sub> is strongly correlated with a reduction in the overall fraction of four-coordinated boron units (N<sub>4</sub>), leading to the development of more ordered structures upon thermal treatment. As the Nd<sub>2</sub>O<sub>3</sub> content increases from 2 to 7 mol%, the chemical shifts of <sup>11</sup>B nuclei transition from 12 ppm to 0 ppm. This shift is attributed to the generation of more shielded borate units facilitated by the formation of B-O-Nd bonds. In the second coordination sphere, each boron atom can be coordinated with neodymium atoms. The broadening of the observed NMR spectra is primarily attributed to the presence of neodymium ions. It is evident that in a glass matrix containing Nd<sub>2</sub>O<sub>3</sub>, the relative intensity of the peak associated with BO<sub>4</sub> units decreases compared to the intensity of peaks corresponding to BO<sub>3</sub> units. This observation indicates that the modifier oxide is stimulated to be more active in facilitating the borate back conversion process and promoting the formation of asymmetric units, even in the presence of trace amounts of rare earth oxides (≤ 5 mol%), represented by neodymium ions. The addition of a small quantity of Nd<sub>2</sub>O<sub>3</sub> to the

borate glasses partially reverses the conversion of BO<sub>4</sub> groups to BO<sub>3</sub> groups.

#### Conflicts of interest

The authors declared that there is no conflict of interest

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#### 6. References

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