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ORIGINAL RESEARCH ARTICLE

Chemical stability and structural changes in xNa$_2$O-(45-x) B$_2$O$_3$-45P$_2$O$_5$-10MnO glasses influenced by Na$_2$O

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ABSTRACT

Borophosphate glasses with the composition xNa2O-45P2O5-(45-x) B2O3-10MnO, where x varies from 5 to 25 mol%, have been synthesised using the conventional melt quenching technique. The prepared glasses were analysed by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and differential scanning calorimetry (DSC) to investigate their structural, thermal and chemical properties. XRD results confirmed the amorphous nature of the glasses, with no crystalline phases detected. Chemical durability studies showed that resistance to chemical attack improved with increasing Na2O content. Glasses with more than 15 mol% Na2O showed exceptional chemical resistance. The relationship between structural variations and composition was investigated by measuring density and glass transition temperature (Tg). The results indicate that higher Na2O concentrations result in increased Tg and improved chemical properties. In addition, the incorporation of Na2O reduces the melting point while increasing the mechanical strength of the glasses. These results highlight the significant influence of sodium oxide on the structural stability and functional performance of borophosphate glasses, underlining their potential for advanced industrial applications.

Keywords: borophosphates; chemical durability; XRD; density; sodium oxide; manganese oxide

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1. Introduction

Most glasses are composed of three different elements, namely lattice formers, modifiers, and intermediates[1,2]. Borophosphate glass consists of P_2O_5 and B_2O_3 as glass-forming groups and is characterized by a very low coefficient of thermal expansion[3], high softening point, chemical resistance, and high thermal shock resistance[4].

Phosphate glasses have gained considerable attention for their exceptional thermal and chemical stability, superior mechanical strength and excellent optical transparency compared to silicate-based materials[5]. These properties make them highly versatile and suitable for applications as diverse as laser glasses[6], radioactive waste containment[7], therapeutic ion delivery, optical amplifiers and lasers, data storage, telecommunications, biosensing, imaging and even as water soluble fertilisers in agriculture[8,9].

P_2O_5 glasses have several advantages over conventional silicate glasses due to their superior physical and chemical properties[10]. However, the poor chemical resistance, high hygroscopicity, and volatility of phosphate glasses have prevented them from replacing

conventional glasses in a wide range of technological applications. In recent years[11], considerable research has been carried out to improve the chemical durability of phosphate glasses by introducing a range of formers and modifying oxides[12,13], alkali halides, and alkali sulfates into the P_2O_5 glass network[14].

The actual role of each oxide in the composition of the glass depends on its concentration and distribution in the glass network[15]. Among the different glass systems, manganese is considered to be the most stable and suitable for the presence of several ions such as Li^+, Na^+, Pb^{2+}, etc[16–18].

The present study aims to investigate the effect of Na_2O on the chemical durability of xNa_2O-$45P_2O_5$-(45-x) B_2O_3-10MnO phosphate glasses with $5 \leq x \leq 25$ mol %. The chemical durability of these glasses was evaluated in three solutions with different initial pH values. The correlation between chemical durability and structural changes was analysed using IR spectroscopy, density measurements, molar volume and glass transition temperature (Tg)[19].

The presence of P-O-Mn and P-O-Na bands at higher concentrations makes the glass more resistant to hydration[15,20]. The addition of metal oxides to the glass leads to the depolymerisation of the network with the breaking of P-O-P bonds and the formation of bridges and non-bridges[21]. The results obtained show that the dissolution rate (DR) decreases with increasing Na_2O concentration.

2. Materials and methods

High purity reagents including H_3BO_3, Na_2CO_3, $MnCO_3$ and $(NH_4)_2HPO_4$ were used to synthesise glasses of the formula xNa_2O-$45P_2O_5$-(45-x) B_2O_3-10MnO ($5 \leq x \leq 25$ mol% Na_2O) by the conventional melt quenching technique. Batches of 4 to 6 g of raw materials were accurately weighed and thoroughly mixed. The mixtures were melted in porcelain crucibles at 950°C for 15 minutes. Details of the glass preparation method have been described in a previous study[22]. The chemical compositions of the samples are given in **Table 1**.

The amorphous nature of the glasses was confirmed by X-ray diffraction (XRD) analysis performed at room temperature using a Siemens D5000 diffractometer with CuKα radiation (λ = 1.5418 Å) [10-60]. Scans were performed within a 2θ range at a speed of 2°/min.

The materials were subjected to FT-IR characterisation using a JAXO FT/IR 4600 spectrometer with a JAXO PRO ONE module.

The sample was scanned in transmission mode at a resolution of 4 cm^{-1} between 1400-400 cm^{-1}.

Diethyl phthalate was used as the immersion liquid during the measurement of the density at room temperature.

The chemical resistance was evaluated by weighing polished glass blocks after 28 days of immersion in three different solutions: deionised water (pH = 7), acidic HCl solution (pH = 4) and basic NH_3 solution (pH = 12). The weight loss of the samples was recorded at regular intervals (24h, 48h, 72h, 96h, 120h, 144h, 288h, 432h, 576h and 672h) using an analytical balance. This method made it possible to evaluate the resistance of the glasses to dissolution under different pH conditions.

The dissolution rate (DR) was estimated using the following equation:

$$DR = \frac{\Delta W}{S \cdot t} \qquad (1)$$

Where ΔW is the weight loss (g), S is the surface area (cm^2) and t is the immersion time (min). The pH of each sample was determined during the dissolution process.

Table 1. Composition (mol %), density ρ (g.cm^{-3}) and glass molar volume Vm (cm^{-3}.mol^{-1}) of the studied glasses**.**

Glass n°	P2O5	B2O3	MnO	Na2O	ρ	Vm
G1	45	40	10	5	2,2239	449,6736
G2	45	35	10	10	2,3469	430,9864
G3	45	30	10	15	2,3614	425,8303
G4	45	25	10	20	2,3755	422,0478
G5	45	20	10	25	2,3843	419,6929

3. Results

3.1. DRX

As shown in **Figure 1**, the X-ray diffraction patterns for all the compositions studied confirm their amorphous nature, as no clear peak is observed in the diffraction pattern.

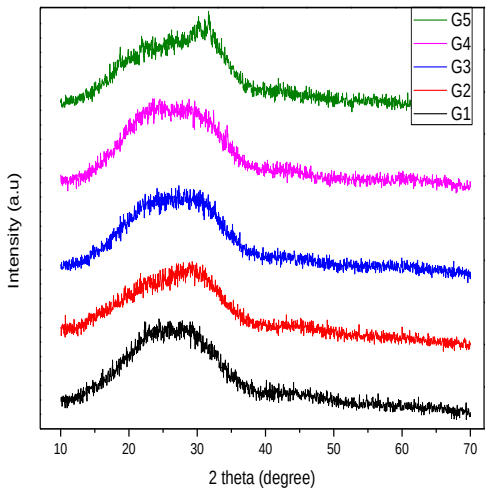


Figure 1. Diffractograms of glasses with formula xNa_2O-45P_2O_5-(45-x) B_2O_3-10MnO.

3.2. IR studies

The infrared (IR) transmittance spectra (**Figure 2**) for the glasses Na_2O-B_2O_3-P_2O_5-MnO show conventional vibrational bands due to phosphate groups in the spectral range 1286-1301 cm^{-1} (antisymmetric PO^{2-} group vibrations/P=O stretching vibrations)[6], 1080-1103 cm^{-1} (a normal vibrational mode of the PO_4^{3-} group from symmetric ν3 stretching)[23], 944-967 cm^{-1} (asymmetric P-O-P bending vibrations/this region may also consist of bands due to $P2O_7^{4-}$ pyrophosphate groups) and another band in the region of 768-787 cm^{-1} due to symmetric P-O-P stretching vibrations[24,25]. These spectra also showed three conventional bands due to borate groups in the spectral regions 1393-1418 cm^{-1}, 944-967 cm^{-1} and 717-732 cm^{-1}from BO_3, BO_4 units and B-O-B bond vibrations respectively[26,27]. The vibrational frequency region of the B-O-B bonds may also consist of bands from the specific vibrations of MnO[28].

The asymmetric vibrations of PO^{2-}/P=O units were identified in the wavenumber region of 1286 to 1298 cm^{-1}[6]. The asymmetric stretching vibrations of $P_2O_7^{4-}$ pyrophosphate units were observed in the wavenumber region of 1036 to 1045 cm^{-1}[26]. The symmetric stretching vibrations of the POP units were observed in the wave number region of 762 to 771 cm^{-1}[29].

The bands of BO_3, BO_4 units and BOB bonds were located in the regions of 1394-1406[23], 926-937 and 692-709 cm^{-1}respectively[30]. However, the BO_4 vibrational structural units had superimposed bands in the

region of PO_4^{-3} fundamental components[6]. The bands due to Mn-O-Mn bonds were predicted to be in the region 694-709 cm^{-1}[26].

Among the various components of the Na_2O-B_2O_3-P_2O_5-MnO glass system, P_2O_5 is a strong glass-forming oxide[31]. It participates in the glass network with the structural clusters PO_4[5]. B_2O_3 is also a strong glass former[27]. When added to phosphate glasses, tetrahedral boron units generally predominate in the phosphate-rich region, forming B-O-P bridges, while trigonal boron units predominate in the borate-rich side[32].

IR spectral studies of these glasses indicated that the bands due to asymmetric vibrations of the phosphate and BO_3 groups should increase at the expense of the symmetric bands of the phosphate and BO_4 groups with increasing MnO content[33]. These variations suggest an increase in the degree of disorder in the glass lattice due to the increasing presence of Mn^{2+} ions acting as modifiers in addition to the Na^+ ion[3].

The observed decrease in the thermal parameters Tg and Tx-Tg (evaluated from the DSC studies) indicates that there is a decrease in the thermal resistance of the glasses to devitrification with the presence of MnO. This trend confirms the decrease in the cross-linking density of the different structural groups in the glass network and the proximity of the package[34].

Similar to manganese ions, sodium ions in the octahedral position depolymerise the glass lattice by increasing the number of bonding defects and non-bridging oxygens (NBO)[35]. The creation of donor centers increases as the Mn^{2+} (Oh) ion concentration in the glass lattice rises[36]. It is anticipated that the empty excited 3d Mn states on the nearby Mn^{3+} sites will begin to overlap with the excited states of the localized electrons initially trapped on the localized Mn^{2+} sites[26]. The impurity or polaron band thus penetrates deeper into the primary band gap[34]. With rising MnO concentration, this new polaron generation may cause the absorption edge's red spectral shift, which ultimately causes a sizable band gap reduction[34].

Up to 15 mol% Na_2O, the observed asymmetric units of phosphate groups and BO_3 units grow at the expense of symmetric units, and the inversion occurs above 15 mol% Na_2O.

IR spectral observations of P_2O_5-MnO-Na_2O-B_2O_3 glasses showed that the intensity of the bands due to asymmetric vibrations of the phosphate and BO_3 groups and the symmetric bands of the phosphate and BO_4 groups increase with Na_2O concentration up to 15 mol%.

These variations suggest that the increase in sodium ions in the octahedral sites up to 15 mol% Na_2O is associated with an increase in the degree of disorder and concentration of NBO's in the glass lattice. From this observation, it can be concluded that the coordination of phosphorus with different ligands increases with the concentration of Na_2O up to 15 mol% and the inversion can be stimulated beyond 15 mol% Na_2O.

As shown in **Figure 2**, the IR spectra of P_2O_5-MnO-Na_2O-B_2O_3 glasses show the traditional vibrational bands of borate groups, phosphate groups, sodium and MnO.

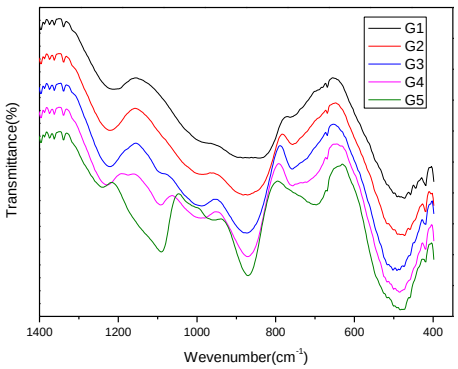


Figure 2. IR spectra of Na_2O-P_2O_5-B_2O_3-MnO glasses recorded at room temperature.

3.3. Density and molar volume

The densities ρ and molecular weights M of the glasses P_2O_5-MnO-Na_2O-B_2O_3 were used to compute their molar volumes (Vm = M/ρ). The results are graphically displayed in **Figure 3** and summarized in **Table 1**.

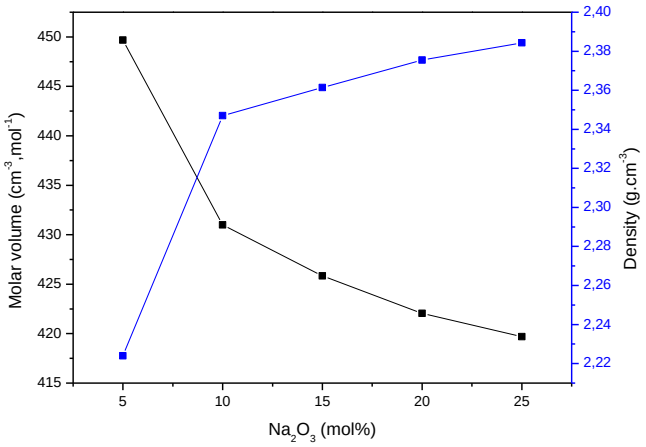


Figure 3. Evolution of density and molar volume as a function of Na_2O content for glasses xNa_2O-45P_2O_5-(45-x) B_2O_3-10MnO.

Increasing the Na_2O content leads to an increase in density, indicating that the Na_2O ions are cross-linking the glass network. To confirm this result, we looked at the evolution of the molar volume in the glass series as a function of Na_2O (**Figure 3**). As the Na_2O content increases, the molar volume decreases, confirming that the oxide is involved in the cross-linking of the glass network. Indeed, these results attribute the decrease in molar volume Vm with increasing density ρ and Na_2O content to the formation of Na-O bonds at the expense of P-O bonds, which cross-link the phosphate network and lead to the narrow structure of the glasses. Thus, if the glassy network is assimilated to a stack of O^{2-} ions with the other ions inserted, the density should normally increase with the addition of MnO in the glassy matrix. However, in **Figure 2** we see that it increases. This would indicate that the sodium ions are cross-linking the glass network. To confirm this result, we have plotted the evolution of the molar volume of the glasses as a function of the sodium oxide content (**Figure 3**).

3.4. Chemical durability

Previous results, such as density, molar volume, and glass transition temperature, have shown that the addition of Na_2O to the P_2O_5-B_2O_3-MnO glass significantly increases the density and Tg.

We will present the alteration of glasses under different conditions. Several parameters influencing the dissolution of glasses have been studied. These are the composition of the glasses, the pH, and the immersion time.

All of the leaching experiments in our study were conducted in a closed, non-agitated environment. This involves placing glass blocks in an aggressive solution in a beaker so that all surfaces of the sample are in contact with the solution. After each immersion time, the glass samples are then removed from the leaching solution, dried, and weighed. The pH of the leachate is then measured. The experimental design can be summarized as follows:

Choice of temperatures

Only one temperature was chosen for our experiments: room temperature.

Choice of alteration solutions

We chose 3 solutions of different pH:

- a slightly acidic solution of hydrogen chloride HCl at pH=4 was chosen to simulate the attack of acid rain;

- a demineralized and distilled water solution of pH=7

- a basic solution of ammonia NH_3 at pH=12 was chosen to model the attack in cementitious matrices.

The distilled water, HCl, and NH_3 solutions were chosen to avoid adding ions already present in the glasses and to avoid complexing them.

$$\text{Total mass loss} = \frac{\Delta m}{S} \quad (2)$$

With:

Δm: mass loss of the glass sample (g)

S: surface area of the glass (cm^2)

The weight loss as a function of immersion time in deionised water, HCl solutions and NH_3 solutions for the glasses labelled G1, G2, G3, G4 and G5 are shown in **Figures 4a**, **5a** and **6a** respectively. Meanwhile, the variation of the pH values over the immersion time is shown in **Figures 4b**, **5b** and **6b**.

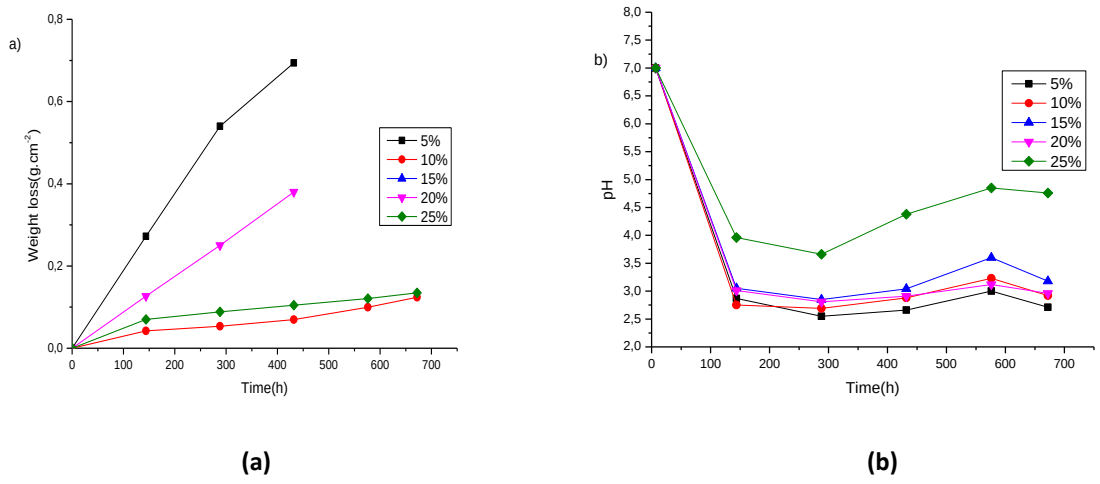
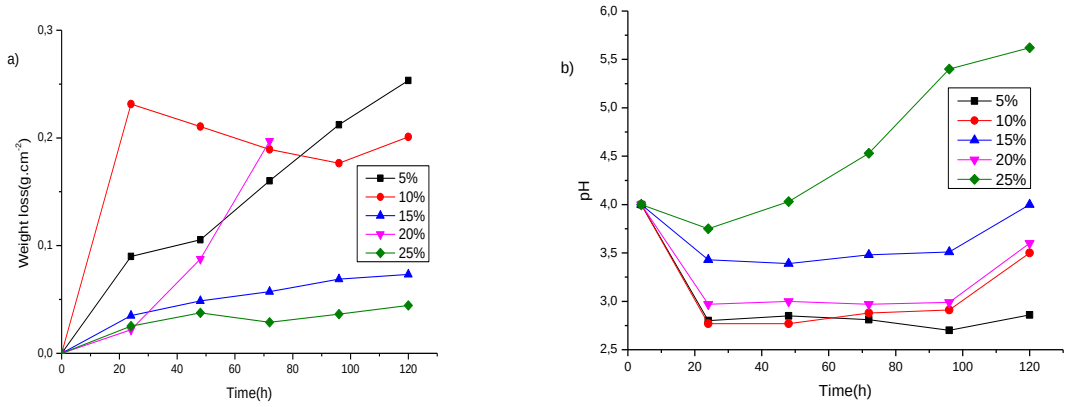


Figure 4. Weight loss (a) and pH variation (b) as a function of immersion time in deionised water of xNa_2O-$45P_2O_5$-$(45-x)$ B_2O_3-10MnO glasses with 5<x<25 mol%.

In deionised water (**Figure 4a**), the dissolution rate showed a slight increase for immersion times up to 150 hours, after which it became moderate and approximately linear for G2 and G5. The increase in weight loss with increasing Na_2O content observed after immersion times of more than 150 hours was accompanied by a rapid decrease in solution pH for the samples, with only a minimal decrease observed for G4 and G5 (**Figure 4b**).

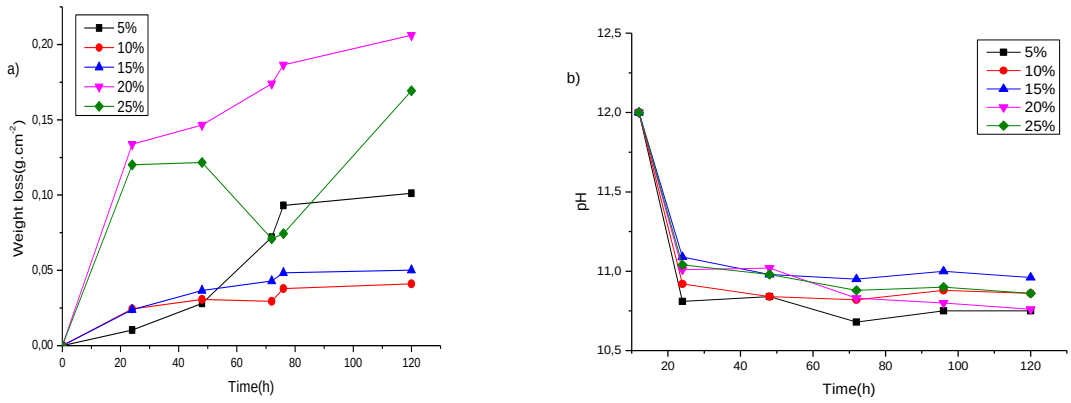
After 24 hours of immersion in the HCl solution, the samples showed a rapid increase in weight loss (**Figure 5a**). Beyond this point, the weight loss continued to increase slightly, especially for samples G1, G3 and G5. As shown in **Figure 5b**, this weight loss is associated with a slight increase in the pH within the acidic range of the HCl solution.



(a) **(b)**

Figure 5. Weight loss (a) and pH variation (b) as a function of immersion time in HCl solution of xNa_2O-$45P_2O_5$-$(45-x)$ B_2O_3-10MnO glasses.

In the NH_3 solution, the weight loss of samples G1, G2 and G3 increases steadily with the immersion time, whereas it increases rapidly for G4 and G5 (**Figure 6a**). The pH values show a slight, approximately linear decrease (**Figure 6b**).



(a) **(b)**

Figure 6. Loss of weight (a) and variation of the pH of the solution (b) as a function of the time of immersion in an NH_3 solution of xNa_2O-$45P_2O_5$-$(45-x)$ B_2O_3-10MnO.

Figure 7(a-c) shows the influence of Na_2O content on the dissolution rate (DR) of glasses exposed to three solutions: deionised water (pH = 7), HCl (pH = 4) and NH_3 (pH = 12). In deionised water (**Figure 7a**), the DR decreased slightly with increasing Na_2O content, although G1 and G2 dissociated completely after 432 hours. For samples immersed in HCl solution (**Figure 7b**), the DR decreased steadily up to 72 hours of immersion, after which G4 dissociated completely, while the DR of the other samples continued to decrease. In NH_3 solution (**Figure 7c**) the DR showed a continuous decrease, with G3 and G4 showing a linear decrease.

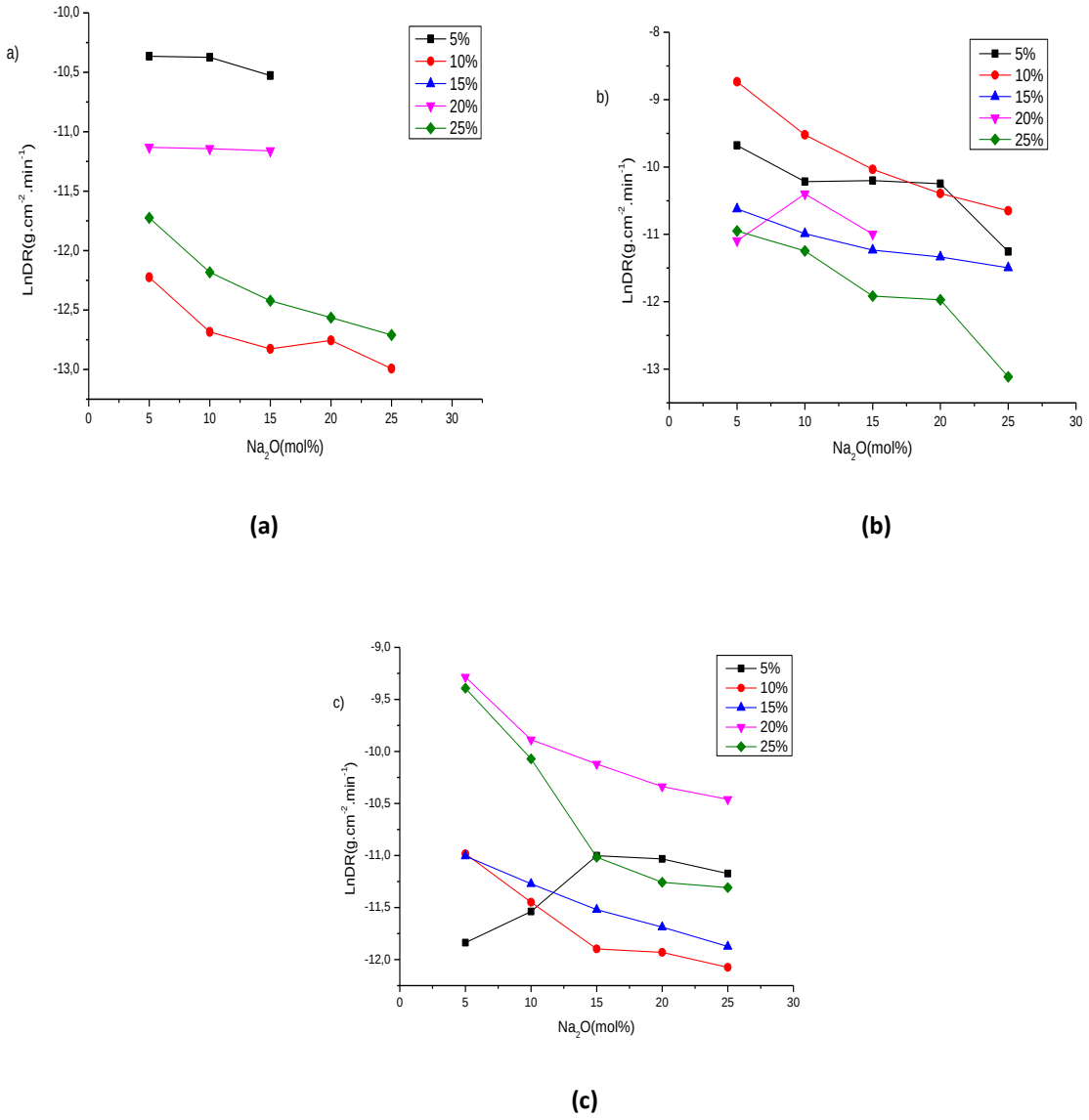


Figure 7. Dependence of dissolution rates (DR) on composition of xNa_2O-$45P_2O_5$-$(45-x)$ B_2O_3-10MnO glasses ($5<x<25$ mol%) after their immersion in deionized water (a); HCl solution (b); NH_3 solution (c) at 25°C for 6, 12, 18, 24 and 28 days.

The dissolution rate, DR ($g.cm^{-2}.min^{-1}$), varies with the sodium oxide content, as shown in **Figure 7**. There is a slight reduction in DR between 20 and 30 mol% Na_2O, but for additions above 15 mol% DR decreases sharply with increased Na_2O content. For Na_2O contents below 20 mol%, an increase in the dissolution rate was attributed to the release of metaphosphate chains into solution. In the present investigation, at these levels Na_2O has little effect on the structural features of the glasses dominated by continuous tetrahedral PO_4^3 chains. This behavior is very consistent with the small addition of ZnO to $NaPO_3$ glass[37].

With the increase of Na_2O content, the metaphosphate chains break into the smaller phosphate units like $P_4O_{13}^{6-}$, $P_3O_{10}^{5-}$ and $P_2O_7^{4-}$, which form P-O-Na bonds with sodium[38]. This is explained by the fact that sodium oxide promotes increased cross-linking between phosphate units and demonstrates its cross-linking effect[21,39]. The dissolution of phosphate glasses is initiated by hydration, a process involving the diffusion of water onto the glass surface and the migration of phosphate chains into solution. The presence of Mn^{2+} ions increase the cross-linking within the phosphate network through the formation of P-O-Na bonds, which are more resistant to hydration than P-O-K and P-O-P bonds, hence increasing the chemical resistance of the glasses.

Increased durability of the borophosphate glasses can be ascribed to replacement of easily hydrated P-O-P bond with more corrosion resistant Na-O-P bond. As increasing the content of Na_2O, the number of Na-O-P.

bonds increases in a directly proportional way[40,41]. Sample G4 among all the samples showed the highest chemical resistance. The pH evolution with the content of Na_2O displays a continuous decline in less than 12 hours of testing and, after that, a slowing down of the rate of this decline[42]. Tests showed that from 2.5 pH of distilled water, the pH value of the solution increased with constant growth with immersion time up to 11.09.

Glasses with >15 mol% Na_2O presented higher resistance and, consequently, a slight decrease of pH from the initial value, while in the solutions where the glasses with <15 mol% Na_2O were immersed, a much more marked decrease of pH was observed. This decrease in pH is in agreement with the higher DR measured. This basification of the solution at pH = 6 can be attributed to an ion exchange process whereby protons in the solution exchange with network-modifying elements. Such ion exchange will increase residual OH^- ions in the solution, leading to an increase in pH observed[13].

4. Conclusion

In this work, the addition of Na_2O in the xNa_2O_3-$45P_2O_5$-(45-x) B_2O_3-10MnO glass compositions has been studied. Results show that the increase in sodium oxide in phosphate glasses increases density and Tg. This behavior is ascribed to the cross-linking of the phosphate network through P-O-Na bonds, which developed a more compact glass structure. IR spectra confirm the depolymerization of phosphate chains with the increase in Na_2O content, together with the formation of P-O-Na bonds and the incorporation of sodium into the glass matrix. Improved chemical resistance for higher content of Na_2O relates to the replacement of easily hydrolysed P-O-P bonds by more chemically stable P-O-Na bonds.

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Conflict of interest

All authors declare that they have no conflict of interests.

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