

Study of Chromium-Lead-Phosphate Glasses by XRD, IR, Density and Chemical Durability

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Abstract

Glasses in the ternary system $\text{Cr}_2\text{O}_3\text{-PbO-P}_2\text{O}_5$ were prepared by direct melting of the mixture with stoichiometric proportions of the reagents Cr_2O_3 , PbO and $(\text{NH}_4)_2\text{HPO}_4$ at 1080°C . The glasses obtained are transparent in colour and have a non-hygroscopic appearance. The study of the dissolution rate was carried out on ternary glasses $x\text{Cr}_2\text{O}_3\text{-(45-x)PbO-55P}_2\text{O}_5$ with ($1 \leq x \leq 4$; mol%), immersed in distilled water at 90°C for 24 days, indicating a maximum of chemical durability when the level of chromium oxide passed through 2 mol%. Both, IR spectra and X-ray diffraction have indicated the predominance of metaphosphate or cyclic metaphosphate groups with some traces of isolated orthophosphate groups when the Cr_2O_3 content is equal to $x = 2$. Analysis of the density values also, has showed a maximum density for $x = 2$ mol%. The covalent radius values of oxygen have indicated that the minimum value $r_{\text{cal}}(\text{O}^{2-})$ is observed for $x = 2$ mol% and therefore a relatively high reinforcement of the metal-oxygen-phosphorus (Cr-O-P) bonds. SEM Micrographs have exhibited two phases, a vitreous phase and a crystalline phase. The radical change in the structure from ultraphosphate Q^3 groups to ring metaphosphate Q^2 and orthophosphate groups Q^0 seems to be the cause of the formation of crystallites. Beyond 2 mol% of Cr_2O_3 , the structure of the glass changed relatively and the orthophosphate phases increased to the detriment of the metaphosphate phases. We observed a decrease in chemical durability. However, it was confirmed that the dissolution rate (D_R) of the S_2 analysed compound is comparable to the values of borosilicate glasses which are used as alternative materials for the immobilisation of nuclear waste substances.

Keywords

Chemical Durability, Phosphate Glasses, Chromium Oxide, IR, XRD, SEM,

1. Introduction

The phosphate-based oxide glasses P_2O_5 , compared to their silicate homologous, have important properties due to their low preparation temperatures. These properties, such as a low melting point, high coefficient of thermal expansion and optical properties, make these glasses potential candidates for many technological applications such as: medical field (biomaterials), solid electrolytes, vitrification of nuclear waste, etc. [1]-[10]. The lead iron phosphate glasses used for the disposal of nuclear waste were produced in 1984 [9]. The combination of chromium-doped phosphate glasses with different types of nuclear waste has shown that it is possible to have a waste form with a corrosion rate comparable to that of borosilicate glasses [2] [5] [7]. The previous work performed by our group demonstrated that the substitution of Na_2O with lead oxide by more than 28 mol%, with the presence of 2 mol% of Cr_2O_3 in the vitreous lattice appears to be an unfavourable factor for chemical durability. The phenomenon has been explained by the approach of the boundary zone between the crystal and the glass by the continuous formation of groups of isolated phosphate PO_4^{3-} [5] [6] [7] [11]. Hence, the crystallites exceed a certain limit, and the equilibrium between the glass bath and these crystallites are not longer maintained; we notice, once, a few decrease in the chemical durability. The purpose of this work is to study the evolution of the dissolution rate as a function of the chromium oxide content in distilled water at $90^\circ C$ for the studied glasses of the series $xCr_2O_3-(45-x)PbO-55P_2O_5$ with ($1 \leq x \leq 4$; mol%). The study of the dissolution rate, carried out on these glasses, reveals an important chemical durability for the low contents of chromium oxides (2 mol%).

2. Experimental Section

The synthesis of chromium lead phosphate glasses of composition $xCr_2O_3-(45-x)PbO-55P_2O_5$ avec ($1 \leq x \leq 4$; mol%) was carried out by the direct fusion of mixtures of $(NH_4)_2HPO_4$, $Pb(NO_3)_2$ and Cr_2O_3 in proper proportions. The reagents are finely crushed and then introduced into a porcelain crucible. They are heated to $300^\circ C$ for 1 h in the first instance and then to $500^\circ C$ for 1 h to complete their decomposition. The reaction mixture is then carried to $1050^\circ C \pm 10^\circ C$ for 15 min. The liquid obtained is homogeneous. It is then poured on to an aluminium plate previously heated to $200^\circ C$ to avoid thermal shocks. The vitreous state was first evidenced from the shiny and transparency aspect, which was then confirmed from the X-ray diffraction (XRD) patterns. Samples S_1 , S_2 and S_4 were respectively annealed at $560^\circ C$ and $580^\circ C$, for 72 hours. The chemical durability of these glasses was evaluated by the weight loss of the sample. The samples were polished by glass paper of silica carbon (CSI), cleaned with acetone

and immersed in beakers of Pyrex containing 100 ml of distilled water and brought to 90°C. The surface of the sample must be constantly immersed in the distilled water for 24 days. The density of the glass has been measured at ambient temperature using the Archimedes method. The glass is immersed in a solution of diethyl orthophthalate density, depending on the temperature, is known. The precision is 0.05 g/cm³. The density of the glass is given by the following equation:

$$\rho = \frac{m_{\text{glass}}}{\left[m_{\text{glass}} + (m_{\text{ortho}} - m_{\text{ortho+glass}}) \right]} \rho_{\text{ortho}}$$

with:

ρ = Density

m_{air} = Weight of glass measured in air

m_{ortho} = Weight of diethyl orthophthalate only

$m_{\text{ortho+glass}}$ = Weight of glass immersed in diethyl orthophthalate

$\rho_{\text{ortho}} = 1.11422 \text{ g/cm}^3$

The infrared spectra of the phosphate glasses studied have been determined in the region between 1600 and 400 cm⁻¹ with a resolution of 2 cm⁻¹. The samples were finely ground and mixed with KBr (potassium bromide), which is transparent in the infrared, and whose role was to serve as a matrix.

Chemical composition of the departure mixture and some characteristics of the quaternary glasses, are summarized in **Table 1**.

3. Results and Discussion

3.1. Analysis of Chemical Durability of Series

$x\text{Cr}_2\text{O}_3-(45-x)\text{PbO}-55\text{P}_2\text{O}_5$

The chemical durability (D_R) of the glass is strongly dependent on its composition, in the case of the glass of the $x\text{Cr}_2\text{O}_3-(45-x)\text{PbO}-55\text{P}_2\text{O}_5$ composition series, the approximate durability analysis is carried out by measuring the dissolution rate (D_R), which is defined as the weight loss of the glasses in g·cm⁻²·min⁻¹. The D_R values shown in **Table 1** show a very low dissolution rate for low levels of chromium oxide (2 mol% of Cr_2O_3) introduced into the phosphate network to the detriment of PbO. The substitution of PbO by Cr_2O_3 for more than 2 mol% becomes an unfavourable factor for D_R . **Figure 1** shows the variation in the

Table 1. Compositions, density and chemical durability of $x\text{Cr}_2\text{O}_3-(45-x)\text{PbO}-55\text{P}_2\text{O}_5$ with ($1 \leq x \leq 4$; mol%).

Glass sample	Starting glass composition (mol%)			O/P ratio	(D_R) (g·cm ⁻² ·min ⁻¹) 24 days	ρ (g/cm ³)
	Cr_2O_3	PbO	P_2O_5			
S ₁	1	44	55	2.92	$(1.07 \pm 0.20) \times 10^{-6}$	4.175
S ₂	2	43	55	2.94	$(5.79 \pm 0.20) \times 10^{-8}$	4.238
S ₃	3	42	55	2.96	$(2.90 \pm 0.20) \times 10^{-7}$	4.134
S ₄	4	41	55	2.98	$(2.02 \pm 0.20) \times 10^{-7}$	4.136

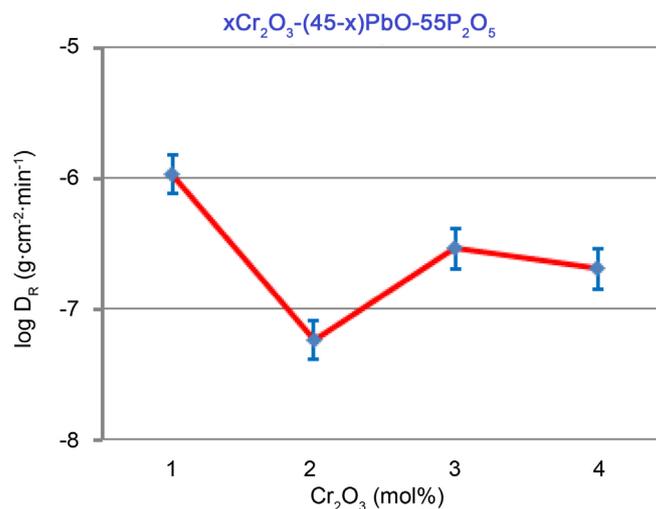


Figure 1. Chemical durability versus Cr₂O₃ for the series of composition xCr₂O₃-(45-x)PbO-55P₂O₅.

dissolution rate of the glasses immersed in distilled water at 900°C for 24 days [6] [8] [11]. A progressive improvement of D_R from 1.07×10^{-6} to 5.80×10^{-8} (g·cm²·min) was noted when the Cr₂O₃ content varied from 1 to 2 mol%, respectively. However, we noted an increase in the dissolution rate (D_R) from 5.80×10^{-8} to 2×10^{-7} (g·cm⁻²·min⁻¹) when the Cr₂O₃ content varies from 2 to 4 mol%, respectively.

3.2. Density and Molar Volumes

Density measurements allowed us to follow the evolution of the molar volume depending on the composition of the system xCr₂O₃-(45-x)PbO-55P₂O₅. The density measurements were completed at room temperature. As can be observed from the **Figure 2**, the variation in density versus Cr₂O₃ content (mol%) indicates a maximum value of x = 2. The molar volume of oxygen (V_{OM}) and the radius of anions of oxygen $r_{cal}(O^{2-})$ in the glass have been determined from Equations (1) and (2), respectively

$$V_{OM} = M / \rho N_A N_0^* \quad (1)$$

$$r_{cal}(O^{2-}) = \frac{\sqrt[3]{V_{OM}}}{2} \quad (2)$$

With M = molar mass, ρ = density, N_A = Avogadro number; N_0^* = number of oxygen atoms in the molecular formula. The value of the molar volume and the oxygen radius were calculated from the approximate hypothesis of close packing of oxygen anions O²⁻, having $r(O^{2-})$ recapitulated for each composition in **Table 2** [12] [13] [14] [15]. A detailed analysis of the data in **Table 2** shows that the molar volume passes through a minimum when the Cr₂O₃ content reaches 2 mol%. However, the covalent radius value of the oxygen atom (O²⁻), calculated by the molar volume using the Equation (2) for each composition, seems to decrease slightly.

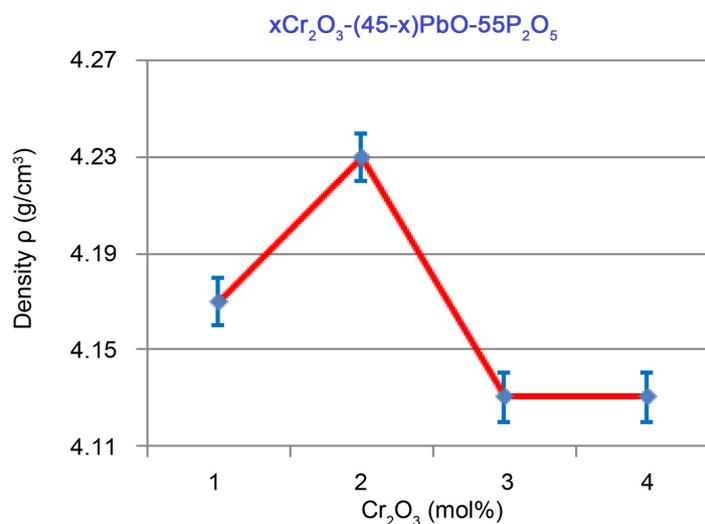


Figure 2. Variation of density versus Cr_2O_3 for the series of composition $x\text{Cr}_2\text{O}_3-(45-x)\text{PbO}-55\text{P}_2\text{O}_5, \text{Cr}_2\text{O}_3$ (mol%).

Table 2. Density and molar volume of system $x\text{Cr}_2\text{O}_3-(45-x)\text{PbO}-55\text{P}_2\text{O}_5$.

Samples	Molar formula Oxygen/Mol (N_O)	Molar mass (g/mol)	ρ (g/cm ³)	Molar volume (nm) ³ $V_{OM} = M/\rho N_A N_O^*$	Calculated oxygen radius (nm) $r_{cal}(\text{O}^{2-})$
S1	$1\text{Cr}_2\text{O}_3-44\text{PbO}-55\text{P}_2\text{O}_5$ (322)	17,782.8	4.175	0.0219	0.140
S2	$2\text{Cr}_2\text{O}_3-43\text{PbO}-55\text{P}_2\text{O}_5$ (324)	17,711.6	4.238	0.0214	0.138
S3	$3\text{Cr}_2\text{O}_3-42\text{PbO}-55\text{P}_2\text{O}_5$ (326)	17,640.4	4.134	0.0217	0.139
S4	$4\text{Cr}_2\text{O}_3-41\text{PbO}-55\text{P}_2\text{O}_5$ (328)	17,569.2	4.136	0.0215	0.139

3.3. Structural Approach by Infrared Spectroscopy

The infrared spectra for the $x\text{Cr}_2\text{O}_3-(45-x)\text{PbO}-55\text{P}_2\text{O}_5$ glasses series (with $1 \leq x \leq 4$) are shown in **Figure 3**. As can be seen from this figure, all the phosphate vibration bands of the treated sample are presented in the frequency range between 1600 and 399 cm^{-1} [16] [17]. The band of approximately $1244 - 1228 \text{ cm}^{-1}$ is attributed to the asymmetric vibration modes $\nu_{as}(\text{PO}_2)$ or the two non-bridging oxygen atoms linked to a phosphorus atom in the phosphate tetrahedron Q^2 [17]. The vibrations of the bands around $1070 - 1047 \text{ cm}^{-1}$ are characteristic of the stretching vibrations $\nu_{asy}(\text{PO}_3)$ and the terminal groups $\nu_s(\text{PO}_2)$ [15] [16] [18]. The band about $912 - 894 \text{ cm}^{-1}$ is attributed to the ν_{asy} P-O-P stretching vibrations [16] [17] [18] [19] [20], while the band at $779 - 771$

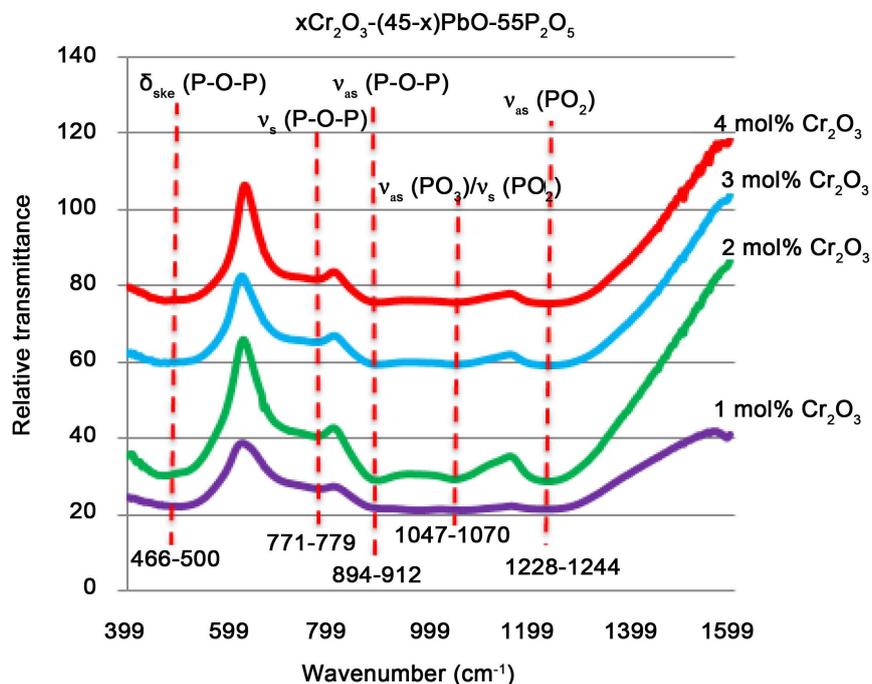


Figure 3. IR spectra of phosphate glasses of composition $x\text{Cr}_2\text{O}_3-(45-x)\text{PbO}-55\text{P}_2\text{O}_5$, with ($1 \leq x \leq 4$; mol%).

cm^{-1} is assigned to the stretching vibration ν_{sy} P-O-P band of the transition oxygen atoms of phosphorus to a phosphate tetrahedron Q^1 [16] [19] [20] [21]. Bands that appear between 500 and 466 cm^{-1} are attributed to the P-O-P vibration modes of the skeleton [2] [16] [17] [19]. All characteristics of the phosphate vibrations show that the phosphate glasses of compositions $x\text{Cr}_2\text{O}_3-(45-x)\text{PbO}-55\text{P}_2\text{O}_5$ (with $1 \leq x \leq 4$) may have chains or rings of the metaphosphate groups, with some traces of pyrophosphate groups. In fact, when the Cr_2O_3 content (mol%) is equal to $x = 2$, the vibration bonds $\nu_{\text{as}}(\text{PO}_2)$ assigned to metaphosphates groups tend to be the dominant characteristic of the spectrum. As shown in **Figure 3**, the band becomes more intense.

3.4. X-Ray Diffraction

The XRD pattern shown in the **Figure 4** indicates that the local structure of chromium-lead phosphate glasses, evolved from ultraphosphate ($\text{O/P} = 2.93$) to nearby chains (cyclic metaphosphate $\text{O/P} = 3$, isolated orthophosphates $\text{O/P} = 4$) structures $\text{Cr}(\text{PO}_3)_3$, $\text{Cr}_2(\text{PO}_3)_6$, $\text{Pb}(\text{PO}_3)_2$, $\text{Pb}_2(\text{PO}_3)_4$, $\text{Pb}_3(\text{PO}_4)_2$, $\text{Pb}_9(\text{PO}_4)_6$, $\text{Pb}_3\text{Cr}(\text{PO}_4)_3$ and some trace of $\text{Pb}_2\text{P}_2\text{O}_7$. When the sample S_1 was thermally treated at 560°C , the amorphous phase partially disappeared and major $\text{Cr}(\text{PO}_3)_3$ [JCDSD File N°: 01-077-0672], $\text{Pb}(\text{PO}_3)_2$ [JCDSD. File N°: 00-043-0335], $\text{Pb}_2(\text{PO}_3)_4$ [JCDSD File N°: 01-086-21] Phases occurred in the sample, with minor $\text{Pb}_3(\text{PO}_4)_2$ [JCDSD File N°01-070-1790] and $\text{Pb}_9(\text{PO}_4)_6$ [JCDSD file N°: 00033-0768] Phases. When the Cr_2O_3 content increased in the glass (S_2), the heat treatment caused an increase in crystallization temperature at 580°C , which

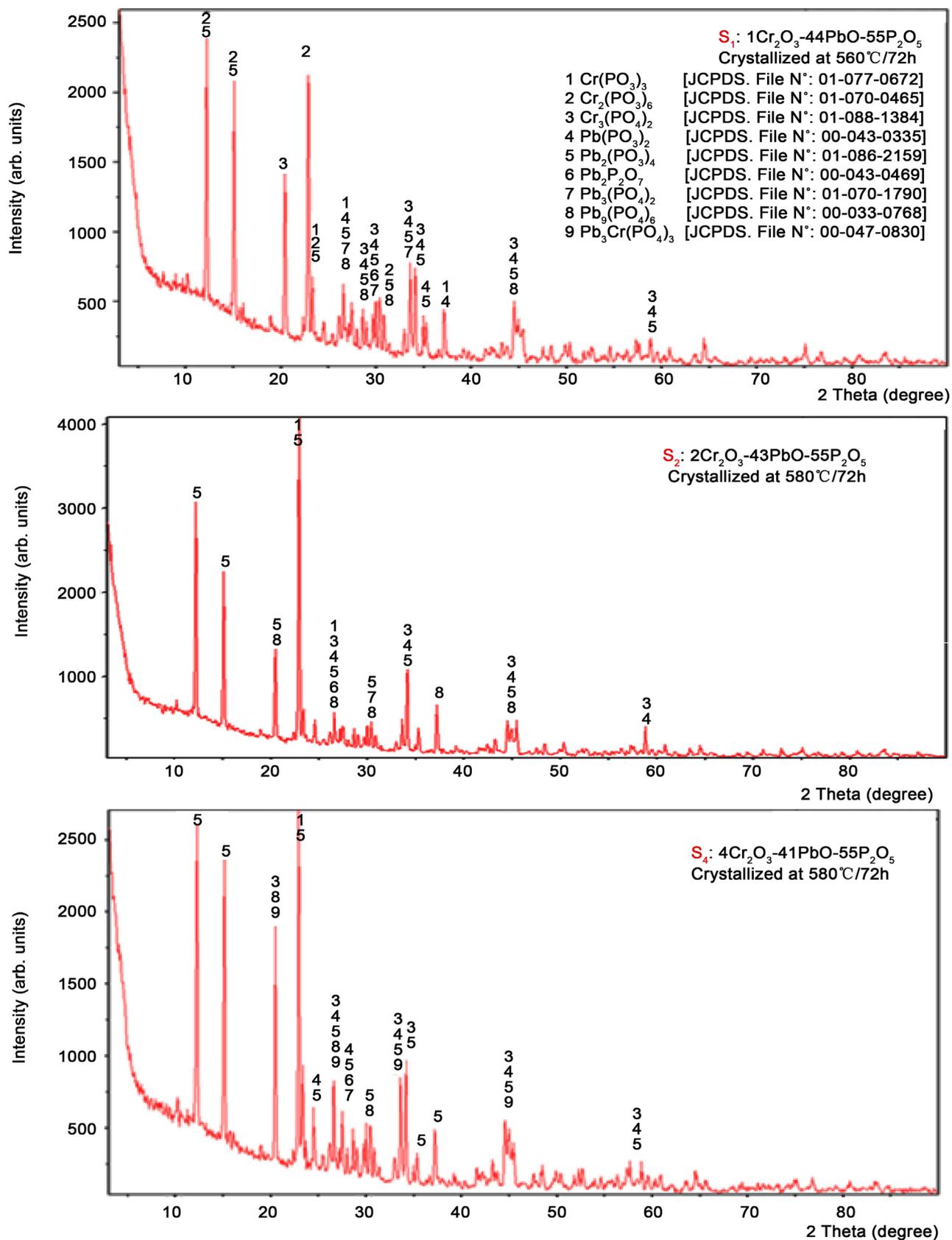
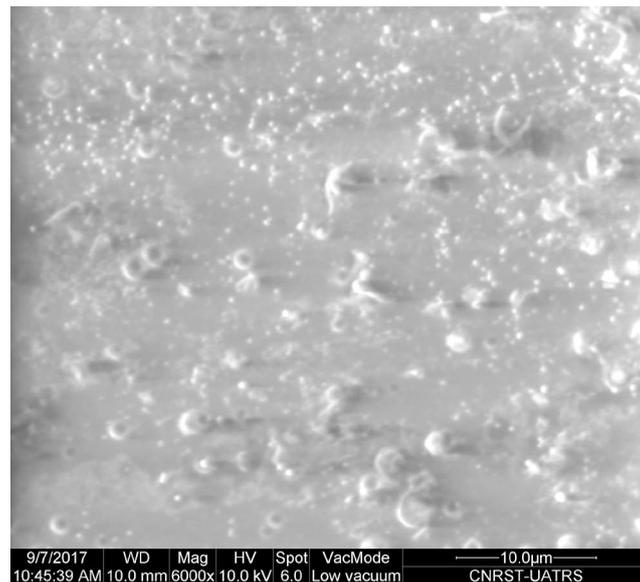


Figure 4. X-ray spectrum of the glasses of compositions S₁, S₂ and S₄ devitrified respectively at 560°C, 580°C and 56°C for 72 hours.

resulted in the disappearance of isolated orthophosphate phase and the formation of CrPO_3 , $\text{Pb}_2(\text{PO}_3)_4$ and $\text{Pb}(\text{PO}_3)_2$. However, the S_4 sample thermally treated at 560°C , indicate the formation of metaphosphate and/or rings of metaphosphate phase $\text{Cr}(\text{PO}_3)_3$, $\text{Pb}_2(\text{PO}_3)_4$ with the appearance of isolated orthophosphate phase $\text{Pb}_9(\text{PO}_4)_6$ and $\text{Pb}_3\text{Cr}(\text{PO}_4)_3$ [JCDDS File N°: 00-047-0830] at the expense of metaphosphate phases [6] [22] [23].

3.5. SEM Micrograph

SEM images in **Figure 5** illustrate the morphology of the glasses considered in



(a)



(b)

Figure 5. SEM micrograph showing the visual structure of the samples glasses S_2 (a) and S_4 (b).

this work. The glass form of S_2 , shown in **Figure 5(a)**, exhibits two phases, a vitreous phase and a crystalline phase. This last one indicates the formation of crystalline agglomerates particle having ring form that indicate, the crystallisation tendency is enhanced and major $\text{Cr}(\text{PO}_3)_3$ and $\text{Pb}_2(\text{PO}_3)_4$ phases are crystallised in these glasses. The presence of the crystalline phase seems to explain the increase in chemical durability [14] [15] [16]. The SEM micrograph for the sample S_4 indicates a radical change in the structure. There is formation of crystalline phase agglomerates of various sizes, ranging from some microns. This probably explains the structural change towards more short isolated orthophosphate chains as the Cr_2O_3 content increases in the glass network.

4. Discussion

The structure and the chemical durability of the glasses series $x\text{Cr}_2\text{O}_3-(45-x)\text{PbO}-55\text{P}_2\text{O}_5$ (with $1 \leq x \leq 4$; mol%) have been investigated using various techniques such as density, X-Ray diffraction and IR. The measured properties indicate that the glasses series chromium-lead-phosphorus-oxygen network become stronger for $x = 2$. X-ray diffraction indicates that the samples S_1 , S_2 and S_4 , respectively annealed at 560°C and 580°C for 72 h, contain metaphosphates, orthophosphates and certain traces of pyrophosphate phases in all the glasses. When the Cr_2O_3 content is equal to 2 mol%, X ray diffraction and IR spectra, both, confirmed the predominance of metaphosphate groups most probably cyclic. The bands at $1244 - 1228 \text{ cm}^{-1}$, attributed to the asymmetric vibration modes $\nu_{\text{as}}(\text{PO}_2)$, become more intense. The predominance of cyclic metaphosphate chains as $\text{Cr}(\text{PO}_3)_3$ and $\text{Pb}_2(\text{PO}_3)_4$ have led to maximum chemical durability. Analysis of the density values also showed a maximum density for $x = 2$ mol%. The covalent radius values of oxygen calculated from Equation (2) indicate that the minimum value $r_{\text{cal}}(\text{O}^{2-})$ is observed for $x = 2$ mol% and therefore a relatively high reinforcement of the metal-oxygen-phosphorus (Cr-O-P) bond. Additionally, because of the big stability of the energy of field of ligand of d^3 systems in octahedral symmetry, the ions Cr^{3+} occupy almost exclusively the sites having this symmetry type [23] [24], while the Pb is estimated to be in the tetrahedral site forming PbO_4 pyramids which are connected in phosphate tetrahedron by covalent links P-O-Pb [25] [26]. Above 2 mol% Cr_2O_3 , the glass structure changes relatively, it is found that the DRX spectra intensity of the ring metaphosphate phases decreases, whereas the appearance of the isolated orthophosphate phase becomes important when the Cr_2O_3 content reaches 4 mol%. The substitution of PbO with chromium oxide by more than 2 mol% in the vitreous lattice appears to be an unfavorable factor for chemical durability. The origin of this phenomenon is explained almost probably by the approach of the boundary zone between the crystal and the glass by the continuous formation of groups of isolated phosphate PO_4^{3-} [6] [19]. The decrease of chemical durability observed, can be also explained by the existence of the critical concentrations between the cation ions beyond which each ion seeks, by competition, to

have a site that is appropriate to it by moving away from the other. This behavior leads to an increase in the glass volume and a relaxation of the structure [27] [28] and consequently a decrease in density and chemical durability. On the other hand, the PbO oxide have a low melting temperature and can participated with P₂O₅ Oxyde, basis glass matrix, in the extend of the area glass. That explains probably the non evidence of the attributed band of orthophosphate isolated units in the IR spectra beside the X-ray diffraction spectra. Added, the elaboration method of the glasses (melting temperature, tempering speed, etc.) remains a significant factor in the deep understanding of the phenomenon [29].

5. Conclusion

The structure and the chemical durability of chromium lead phosphate glasses of composition xCr₂O₃-(45-x)PbO-55P₂O₅ avec (1 ≤ x ≤ 4; mol%) have been investigated using various techniques such IR, X-ray diffraction, density and SEM micrograph. The study of the dissolution rate carried out on the glasses, immersed in distilled water at 90°C for 24 days, indicates a maximum chemical durability when the level of chromium oxide passes through 2 mol%. The analysis of the values of the density also showed a maximum density for x = 2 mol%. The covalent radius values of the oxygen indicate that the minimum values are observed for x = 2 mol%, and therefore, a relatively high reinforcement of the metal-oxygen-phosphorus (Cr-O-P) bonds. However, the increase in the Cr₂O₃ content in the vitreous network to the detriment of PbO beyond 2 mol%, is an unfavourable factor for both chemical durability and density. The increase in the Cr + Pb/P ratio leads to an increase in the number of metal-O-P bonds which cause a high tendency for crystallisation and confirms that the dissolution rate (D_R) of the analyzed compounds is comparable to the values of borosilicate glasses and 40 times less than BaBa glasses which are used as alternative materials for the immobilisation of nuclear waste substances.

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