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Short Communication

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Thermal Analysis of Two Niobo-Phosphate Glasses

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Abstract

In the present study, two new biocompatible glasses, with the same components, but in different proportions, were produced. The thermal behavior of both glasses was analyzed and the glass transition temperatures (Tg) were determined by differential scanning calorimetry (DSC) for both glasses. The DSC analyses showed that the bioactive glass with Ca/P = 1.68 showed higher Tg than the bioactive glass with Ca/P = 1.33.

Keywords

Niobo-phosphate; Glass; Glass transition; Thermal analysis

Introduction

Studies on the synthesis of phosphate glasses have been reported from different authors, mainly due to their effect on chemical stability [1-3]. Different applications such as in electronic components [4-6], as well as on sealing systems [7], lasers [8] and biomedical materials [9-11] have been reported.

Niobo-phosphate glasses designed to be biocompatible have been reported by several authors [11-13]. In previous studies, a $CaO-P_2O_5-Nb_2O_5-CaF_2$ glass was developed and tested *in vitro* [12,13]. However, transition temperature of this new glass remained undetermined. In the present study, a new biocompatible glass, with the same components, but in different proportions, was produced. The thermal behavior of both bioactive glasses was analyzed and the glass transition temperatures (Tg) were determined by differential scanning calorimetry (DSC) for both glasses.

Experimental

Two niobo-phosphate glasses were produced by mixing different amounts of Nb_2O_5, H_3PO_4, $CaCO_3$ and CaF_2. The mixtures were melted at 1350°C and cooled into ultrapure water. The designed glasses compositions are shown on Table 1.

The produced glasses were milled in a ball mill Marconi MA350/E, using alumina balls. The obtained powder was analyzed by DSC and X-ray Diffraction (XRD). The DSC analyses were performed in a differential scanning calorimeter DSC 404F1 Pegasus (Netzsch) in a heating rate of 20ºC/min from 30ºC to 500ºC, and above 500ºC up to 1000ºC with 5 and 10ºC/min. The analyses were realized with Pt-Rh crucibles and under argon atmosphere. The results were treated with Netzsch Proteus software.

Structural characterization was performed by X-ray diffractometry in a Panalytical X'Pert Pro diffractometer with CoKα at 40 kV, 45 mA with an iron (Fe) filter. The scan was made in 0.02° step from 10° to 100°. The results were treated with the software X'Pert HighScore Plus.

Results and Discussion

The XRD patterns of both glasses are shown in Figure 1. An amorphous band was observed between 20º < 2θ < 40º, confirming that both samples are strongly amorphous. Another band was observed between 40º < 2θ < 60º, indicating the presence of low-range crystallinity.

Figures 2 and 3 show the DSC analysis, for both glasses, with distinct heating rates (5 a 10ºC/min) in interest (5 and 10oC/min). Theses runs were performed in order to confirm if the variation in heat flow between 650ºC and 750ºC was a Tg and exothermic peaks associated to phase transformation at high temperature range. Figure 2 and Table 2 show that Tg = 688.3ºC for BG1 and Tg = 697.4ºC for BG2 at 5°C/min.

It is conventional to adopt the Tg values from DSC analyses with heating rate of 10°C/min. Figure 3 and Table 2 show the DSC results for both glasses under heating rate of 10°C/min. The glass transition temperature for BG1 was found to be Tg = 695.6°C and, for BG2, Tg = 707.0ºC. Glass 1 has higher glass modifier content than glass 2, what explains the Tg reduction. In addition, BG2 has higher Nb_2O_5 content than BG1. Sene et al. reported a rise in the Tg of niobophsophate glasses with increasing Nb_2O_5 content. This was explained in the basis of Nb-O-Nb and Nb-O-P being stronger than P-O-Nb bondings, and thus requiring higher relaxation temperatures.

Above Tg temperature, both glasses exhibit crystalline structure and revealed distinct peaks and phase transformation temperature. Glass 1 DSC curve exhibit four exothermic peaks, where the first two peaks are related with more significative energy associated the peak area. However, Glass 2 DSC curve exhibits only the first two exothermic peaks associated with highest temperature transformation and apparently higher peak area for the first peak, when compared to Glass 1 curve. The probable phases, according to glasses compositions, are calcium phosphates and calcium niobates.

Conclusion

Glass 1 showed lower Tg than glass 2. Additionally, glass 1 shows Ca/P = 1.68. This ratio is very close to that of hydroxyapatite (Ca/P = 1.67), and this is a promising feature regarding bioactivity. Both glasses can be converted to glass ceramics after heat treatments between 750ºC and 900ºC.

References

Table 1: Glasses compositions.

Sample	CaO (mol%)	CaF_2(mol%)	P_2O_5(mol%)	Nb_2O_5(mol%)
BG1	50.1	12.5	18.6	18.8
BG2	20.0	20.0	30.0	30.0

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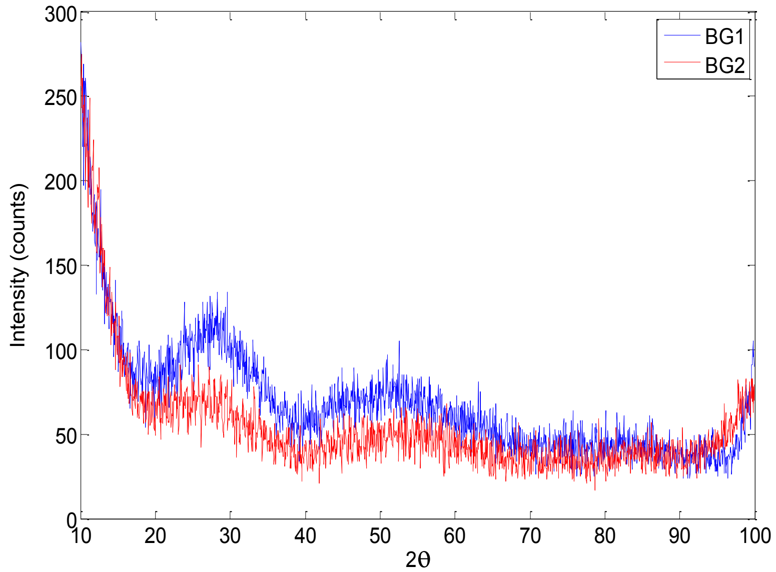

Figure 1: XRD pattern for BG1 (blue) and BG2 (red).

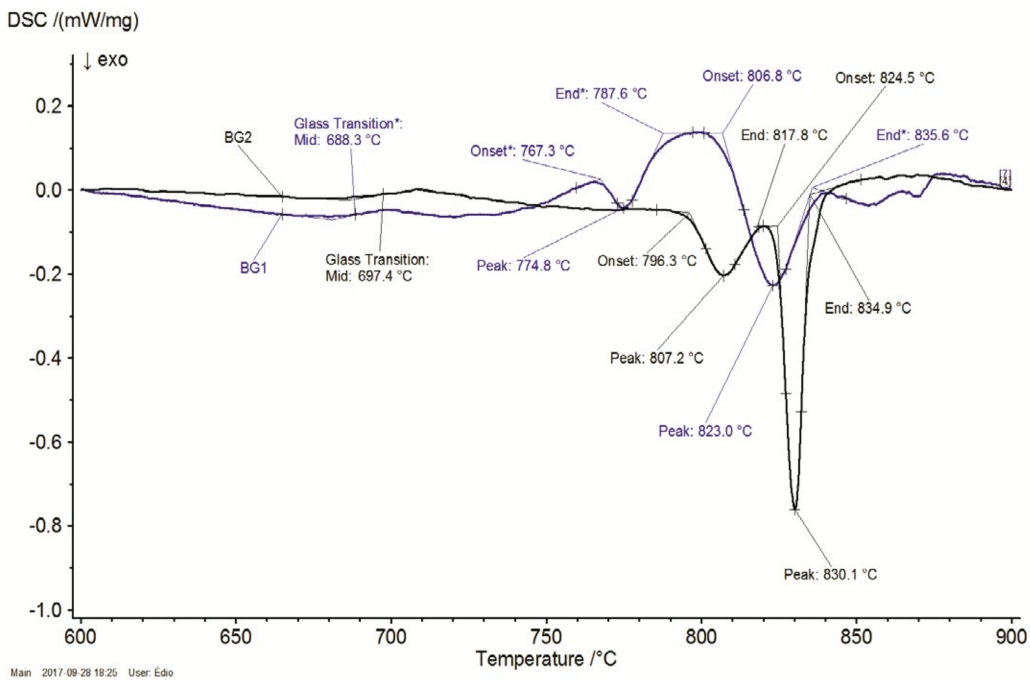

Figure 2: DSC analysis at 5ºC/min.

Table 2: Summary of DSC results for Glasses 1 and 2.

	TG	**Peak1**			**Peak2**		
Sample		**start**	**Peak**	**finish**	**start**	**Peak**	**finish**
Heating rate: 5ºC/min							
BG1	688.3	767.3	774.8	787.6	806.8	823.0	835.6
BG2	697.4	796.3	807.2	817.8	824.5	830.1	834.9
Heating rate: 10ºC/min							
BG1	695.6	777.0	788.1	799.8	819.1	836.5	851.5
BG2	707.0	789.5	825.4	838.6	846.7	854.3	860.9

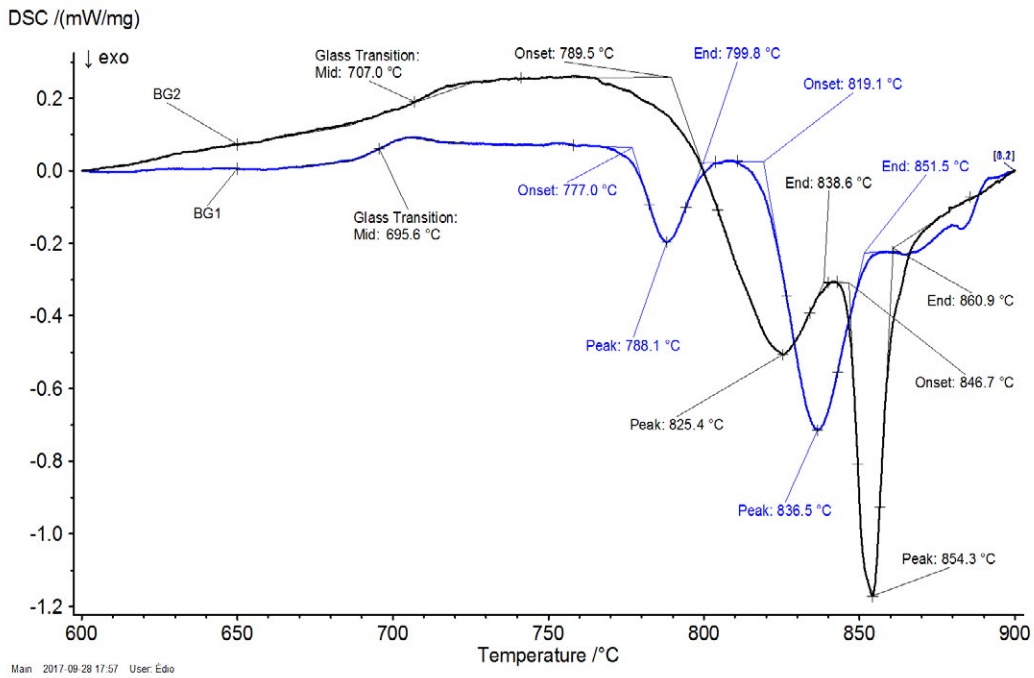

Figure 3: DSC analysis at 10ºC/min.

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Top

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