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Production of porous scaffolds from Bioglass 45S5-derived glasses

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Abstract

Bioglass 45S5 has been successfully implemented in clinical applications, because of its capability of developing a strong bond with the bone tissue. However, its low mechanical strength does not allow applying it in places that support load. In addition to the high reactivity, an important requirement for its application is that the material must have a high porosity with pore sizes greater than 100 microns, one of the key features tissue engineering materials. In a previous work, glass compositions derived from Bioglass 45S5 were designed by means of natural minerals such as feldspar and quartz. Besides, leucite was developed as a reinforcing phase after thermal treatment, getting good bioactivity results. The objective of this study is to evaluate the possibility of producing porous scaffolds from such glasses. The foam replication technique is used to manufacture the scaffolds. Thermal evolutions of glasses are studied, so as to choose the best heat treatment schedule for the production of porous scaffolds. Textural characteristics are evaluated.

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1. Introduction

Bioactive glasses and glass-ceramics are ceramic materials characterized by a functionality that allows them to bond to bone tissue through a hydroxyapatite layer that forms on its surface, similar to bone mineral phase, getting a very resistant tissue material interface bonding (Hench (1991)).

Within this range of materials, Bioglass 45S5 is one of the bioceramic of faster developing hydroxyapatite layer on its surface (Tancred et al. (2001)). In addition, its functionality is so high that it can also bond to soft tissue, such as cartilage and muscles, and it may even induce the formation of new tissue. Its main disadvantage lies on its low mechanical strength, which makes only to be applicable to body areas where the material does not suffer tensions that can lead to breakage.

A way to improve the mechanical properties of Bioglass is through heat treatment, allowing the glass to crystallize into a glass-ceramic. At the same time, crystallization does not significantly modify bioactivity (Peitl et al. (2001)).

On the other hand, there are other bioceramics, such as apatite-wollastonite glass-ceramics, which are bioactive and have good mechanical strength; but the formation of hydroxyapatite layer takes longer than in the case of Bioglass 45S5.

Porous scaffolds are designed in such a way to obtain a high porosity material (80-90%). In addition, the pores must be interconnected, allowing the circulation of fluids and the growth of tissue within them (Hench (1991)). By means of this type of structures it seeks to provide a temporary structural support to the affected area, which, in turn, allows the formation of new tissue. By the type of structure having porous scaffolds, it is necessary that the material used to manufacture them have good mechanical strength.

In recent years, efforts have been focused on materials with very good bioactivity, but at the same time having good mechanical properties, making it useful for applying into different body areas.

In order to make textural comparisons, porous scaffolds were produced in the present work, through the foam replication method (Chen et al. (2006)) from two glasses derived from Bioglass 45S5 composition, with different aggregates of K_2O and Al_2O_3 that developed as leucite additional reinforcement phase.

2. Experimental

Raw materials used to produce glasses were analytical grade sodium carbonate, calcium carbonate, potassium carbonate, monobasic ammonium phosphate; and high purity potassium feldspar and natural quartz, provided by a national mining industry. The nominal compositions of glasses were calculated in such a way to have different theoretical weight ratios of leucite ($KAlSi_2O_6$) and Bioglass 45S5 (45% SiO_2, 24.5% CaO, 24.5% Na_2O, 6% P_2O_5). Thus there were two compositions, L25Bg75 glasses (25 wt. % of leucite and 75 wt. % of Bioglass) and L30Bg70 (30 wt. % of leucite and 70 wt. % of Bioglass).

Mixtures were fused at 1350°C for 1 hour in platinum crucible and cast into distilled water to obtain glass frits. Glasses were milled into porcelain mortar, passed through a 100 mesh sieve, followed up by an intense grinding in a high-speed planetary ball mill Fritsch Pulverisette 7 to obtain a particle size less than 10 μm.

Differential thermal analyses (DTA) were conducted on a Netzsch STA409 equipment. Glasses transformations were analyzed up to 1050°C with a heating rate of 10°C/min into air flow rate of 50 cm^3/min, using α-Al_2O_3 as reference substance.

Glasses and glass-ceramics produced by heat treatment of both glasses at 925°C were characterized by a Philips 3010 X ray diffractometer using Cu Kα radiation (λ = 1.5405 A) at 40 kV and 35 mA and Ni filter, with a step of 0.04° (2θ) and 2 seconds per step.

Porous scaffolds were produced by means of a 40 wt. % glass suspension in a solution of poly-vinyl alcohol (PVA). Small pieces of polyurethane sponge of a size of approximately 1x1x1.5 cm^3 were used. The procedure consisted in dipping and pressing a piece of sponge inside the suspension to fill all their pores and generate a coating of suspension in the channels. Coating is left to air dry for a day prior to the different thermal treatments carried out. Four final temperatures of sintering were selected on the basis of the results of differential thermal analysis, these were 925, 950, 1000 and 1050°C.

3. Results and discussion

3.1. Differential thermal analyses

Differential thermal analyses of glasses L25Bg75 and L30Bg70 are shown in Figs. 1 and 2, respectively.

Between 700 and 950°C, there is a more pronounced exothermic peak, which corresponds to crystallization. A shoulder is visible in the range 750-775°C, which would be an indication that the full peak in both cases is the sum of the contributions of two crystallization peaks, which would correspond to the formation of the two major crystalline phases. The first phase that crystallizes would be in a lower proportion than the second, since the second exothermic effect is larger. There is a change of concavity followed by an endothermic effect between 950 and 1000°C, which would be indicating that in this range of temperatures the material begins to melt.

Figure 2, corresponding to the sample L30Bg70, shows two exothermic effects at the beginning, similar to those described above. The exothermic peak of crystallization appears between 725 and 975°C. In this case, the shoulder is not so noticeable, but a change in concavity can still be seen around 775°C, indicating also that there are two major crystalline phases developed. In the range 975-1025°C, an endothermic process can be distinguished, that indicates the beginning of the melting of the material.

Different final heat treatment temperatures were selected for glass sintering, based on the range of temperatures where the beginning of fusion in both materials became evident.

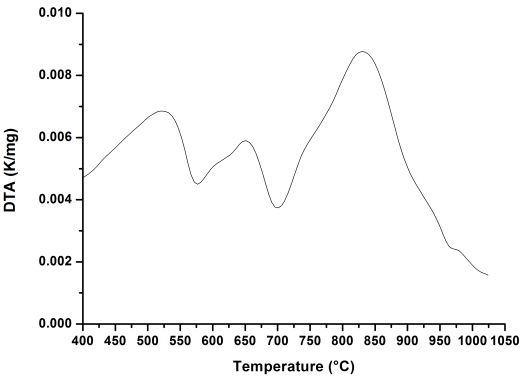

Fig. 1. Differential thermal analysis of glass L25Bg75.

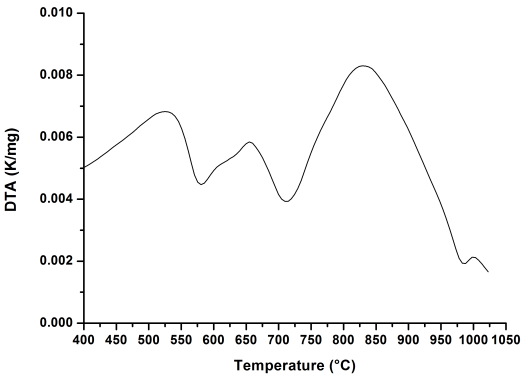

Fig. 2. Differential thermal analysis of glass L30Bg70.

3.2. X-Ray diffraction

The diffractogram of L25Bg75 glass can be seen in bottom part of Fig. 3, in which there is a characteristic band of materials that lack of long-range order. Upper part of the figure shows the diffractogram of the crystallized glass (glass-ceramic) above 900 °C, where two crystalline phases, leucite and sodium calcium silicate, could be identified.

Glass L30Bg70 (Fig. 4) has the same amorphous feature than the one described above. The crystalline phases found in the crystallized glass were the same, although in different proportions. Relative intensities of sodium calcium silicate diffraction peaks decreased, while those of the leucite increased. This is in accordance with the change of composition of 25 wt. % of leucite and 75wt. % of Bioglass to 30 wt. % of leucite and 70wt. % of Bioglass.

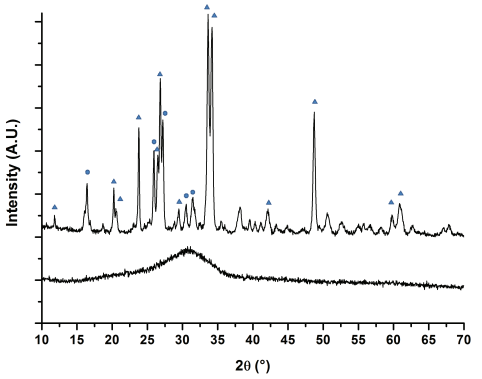

Fig. 3. Diffractograms of L25Bg75 glass and glass-ceramics. ▲Sodium calcium silicate, ● Leucite.

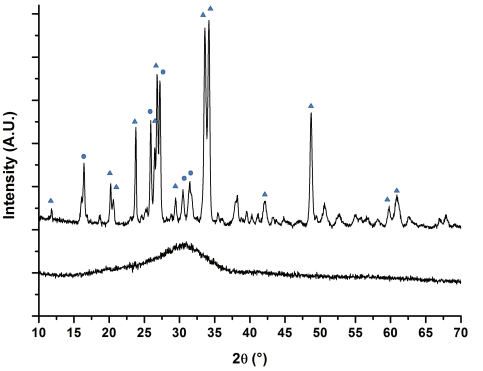

Fig. 4. Diffractograms of L30Bg70 glass and glass-ceramics. ▲Sodium calcium silicate, ● Leucite.

3.3. Porous scaffolds production

Porous scaffolds obtained from L25Bg75 and L30Bg70 glasses at different temperatures are shown in Figs. 5 and 6, respectively.

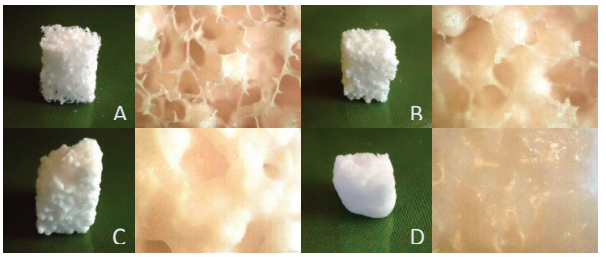

Fig. 5. Porous scaffolds produced from L25Bg75 glass obtained by means of 925 (A), 950 (B), 1000 (C), and 1050°C (D) heat treatments for 1 hour.

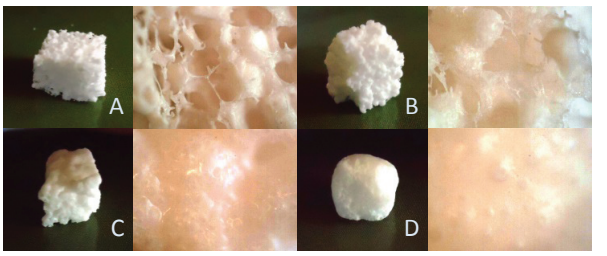

Fig. 6. Porous scaffolds produced from L30Bg70 glass obtained by means of 925 (A), 950 (B), 1000 (C), and 1050°C (D) heat treatments for 1 hour.

Glass L25Bg75 with a final treatment of 925ºC (Fig. 5A) produced a resistant scaffold, with well interconnected pores. One can observe the well clear sponge struts, and in turn will note that they remain well separated, which makes the pores quite open. The scaffold resisted manipulation in an adequate manner.

In the case of the 950°C treatment (Fig. 5B), scaffold sustained an appreciably porosity, but lower than the previous case. Although the channels of the sponge are noticed; the pores had collapsed and were no more interconnected. Scaffold sintered at 1000°C (Fig. 5C) retained a relief of the original format, but the pores were completely closed. In the 1050°C treated scaffold (Fig. 5D), the relief caused by the presence of pores was practically negligible, and it showed a smooth surface.

The appearance of the scaffold produced with L30Bg70 glass (Fig. 6 A, B, C and D) as a function of temperature was similar to the one described for those produced with the glass L25Bg75. It can be seen that the scaffold with a 925°C heat treatment (Fig. 6A) has quite defined pores and well appreciated sponge struts, although the interconnection is not as good as it is the case of the glass L25Bg75 at the same temperature.

4. Conclusion

By means of the foam replication method, it was possible to get scaffold with high porosity that could be visually characterized.

Thermal treatment on scaffolds originated a more compact system, getting increasingly closed pores up to the point to obtain a total absence of porosity.

The two scaffolds made at 925°C were those who retained a more porous structure; although the appearance of the scaffold through the glass L25Bg75 was much better than in the case of L30Bg70, in which there was a greater struts thickness. It is likely to be required a lower sintering temperature in the case of the L30Bg70 glass.

Finally, it is necessary to make more careful studies to find sintering optimal times and temperatures for each composition.

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