

## Bioactivity Evaluation of Glasses and Glass-Ceramics by Electron Microscopy

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For some years the bioactivity of different special ceramic materials has been studied (glasses, glassceramics and ceramics), as well as its capacity of bonding with soft and hard tissues, to determine its possible use like bone substitution in humans. Hench and coworkers discovered that it is possible the chemical bonding between bones and glasses of certain compositions, which denominated bioactive glasses, finding numerous applications in the repair and reconstruction of diseased and damaged tissues, especially hard tissues (bones). These materials have the advantage of not being rejected by the organism due to their compositional similarity with regard to the human bone, but they have as disadvantage their fragility. Contrary to glass, glassceramics generally have better mechanical properties. Boron oxide ( $B_2O_3$ ) and alumina ( $Al_2O_3$ ) are used to modify the dissolution surface, the durability and the coalition and formability characteristics of bioactive glasses. Both oxides have opposite effects on the bioactive material – bone bonding development. Therefore, it is critical to control these oxides ratio in the glass composition.

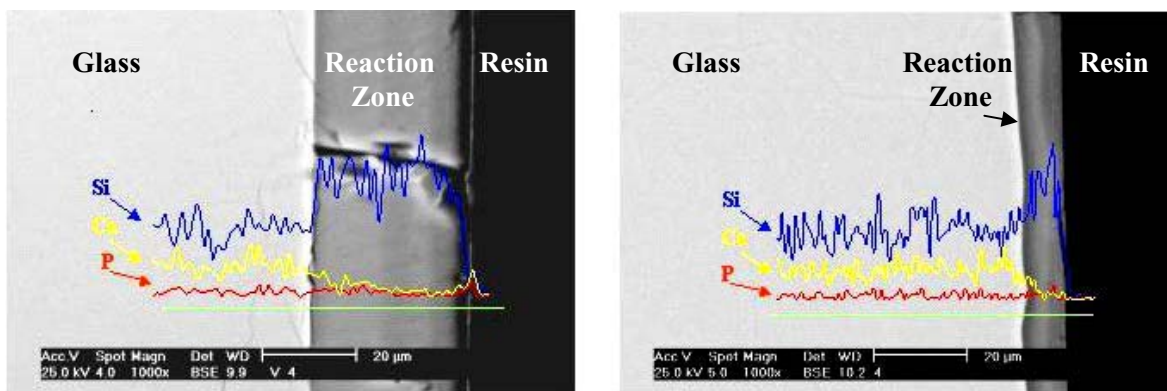
The objective of this work was to study the influence of  $B_2O_3$  and  $Al_2O_3$  additions in different proportions on glasses and glass-ceramics of the  $Na_2O$ ,  $K_2O$ ,  $CaO$ ,  $MgO$ ,  $SiO_2$ ,  $P_2O_5$  system (Table 1) on the bioactivity, analyzing the apatite layer formation in vitro through two methods, one of them with SBF and the other with osteoblastic living cells. Glass-ceramics were obtained from glasses submitted to three different crystallization treatments, using  $550^\circ C$  and  $750^\circ C$  as nucleation ( $T_n$ ) and growth ( $T_g$ ) temperatures, respectively and, varying the holding time at these temperatures with a heating and cooling rate of  $5^\circ C/min$ , (Table 2). The bioactivity test with SBF consisted on immersing the samples in this fluid whose ionic concentration is similar to that of the human blood plasma and the other method is through osteoblastic cell mineralization, both tests had duration of 4 weeks. After that, the samples were embedded in a polyester resin; their cross-sections were polished with  $1\mu m$  diamond paste, coated with a carbon film to characterize the reaction surface using Electron Microscopy, analyzing the layer deposited through EDX compositional profiles. These profiles show the elemental distribution (Ca, P and Si) of the layer formed on the surface which allows determining the development of a rich layer in Si and other rich in Ca and P, being the latter identified in previous works as apatite, characteristic of bioactive glasses. It is observed that glasses of composition 4 (1% of  $Al_2O_3$ ) develop a silicon rich layer in both tests (figures 1A – 1B), which appoints them as biocompatibles. Glassceramics of composition 6 (0,5% $B_2O_3$ ) submitted to the second crystallization treatment showed a bioactive behavior with the development of a rich calcium and phosphorous layer for both tests (figures 2A and 2B).

**Table 1. Compositions of studied glasses (%)**

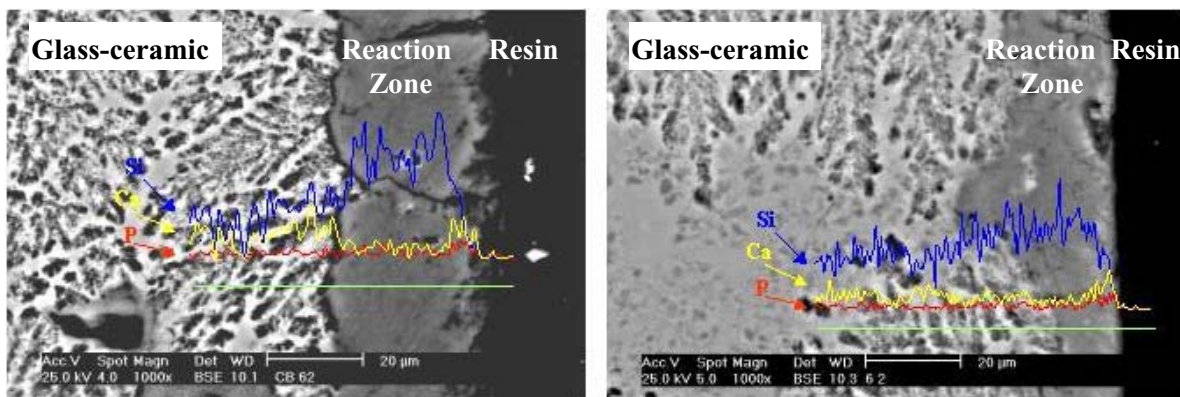
Compositions	SiO <sub>2</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	CaO	MgO	P <sub>2</sub> O <sub>5</sub>	B <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>
1	50.5	20	4.8	20	4.7	0	0	0	50.5
2	49.5	20	4.3	20	4.2	2	0	0	49.5
3	49	20	4.3	20	4.2	2	0	0.5	49
4	49	20	4	20	4	2	0	1	49
5	48.5	20	4	20	4	2	0	1.5	48.5
6	49	20	4.3	20	4.2	2	0.5	0	49
7	48.5	20	4.3	20	4.2	2	1	0	48.5
8	48.5	20	4	20	4	2	1.5	0	48.5
9	48	20	4	20	3.5	2	1	1.5	48
10	49	20	4	20	3.6	2	0.5	0.9	49

**Table 2. Crystallization cycles**

Cycle	Nucleation time (min)	Growth time (min)
1	30	60
2	30	120
3	60	30



**Figure 1.** Glass 4, SEM and compositional profile: A) SBF test, B) osteoblastic cells test.



**Figure 2.** Glassceramic 6, SEM and compositional profile A) SBF test, B) osteoblastic cells test. Si (blue), Ca (yellow), P (red)

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