# Borosilicate Phase-Separated Glasses for GICs: Ion-Release under Acid Condition

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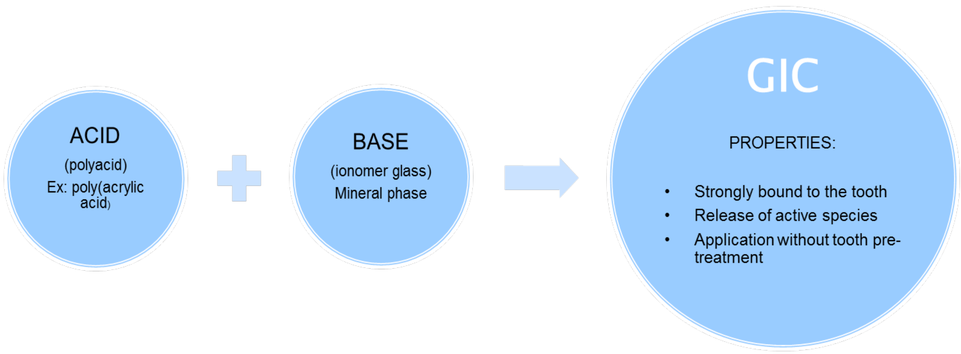
## INTRODUCTION AND OBJECTIVES

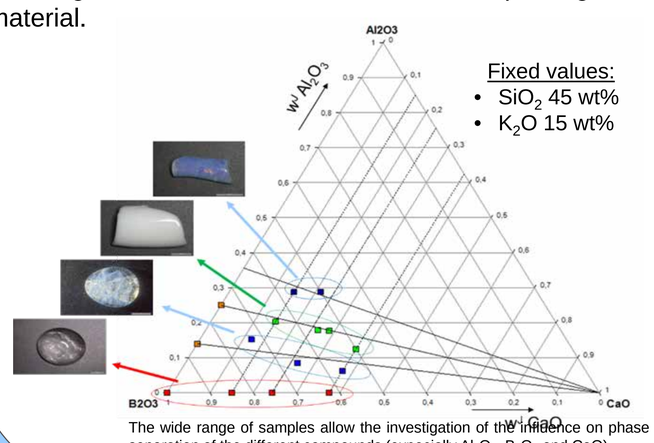
Glass-ionomer cement (GIC) were designed nearly five decades ago by Wilson and Kent in 1969. Currently, they are used in a variety of clinical dental situations as restorative, lining, luting and sealing materials; no other restorative material has such wide applications[1]. Since the requirements are day by day more demanding, the needs of restorations with good manipulative properties and adequate working time together with the ability to set and harden rapidly once placed in position, are extremely important.



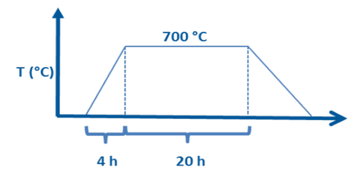

The aim of this work is to develop and characterize a novel powder in the borosilicate glass system establishing the relationship between the glass structure and the cement forming ability. The study is focus on the ions leaching that will influence the working and setting time as well as the biological properties.

## DEVELOPMENT OF THE NOVEL BOROSILICATE GLASS BY MELT QUENCHING TECHNIQUE

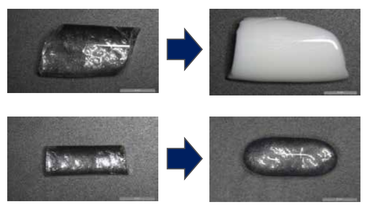
The novel glass powder compositions reported here, based on $SiO_2$-$B_2O_3$-$K_2O$-CaO-$Al_2O_3$, is inspired by the Vycor-type glasses[2]. The borosilicate system exhibits a strong tendency to phase separate after a thermal heat treatment[3]; one of the phases created is more reactive and susceptible to acid attack and will be leached out from the glass earlier. The GIC setting time is thus related to the acid susceptibility of the glass and it is proposed that a controlled phase separated glass can improve the workability and the final properties of the cement thanks to the controlled formation of the acid susceptible phase. The other phase in the glass can remain in the cement improving the mechanical properties of the dental restorative material.




The wide range of samples allow the investigation of the influence on phase separation of the different compounds (expecially $Al_2O_3$, $B_2O_3$ and CaO)

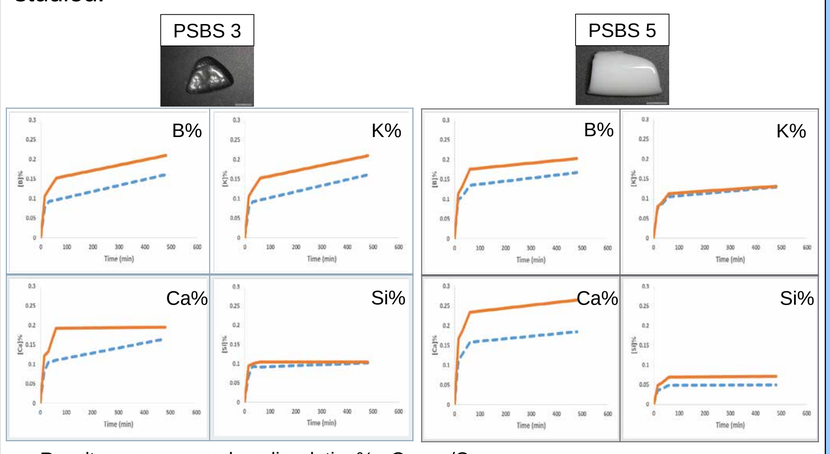



Depending on the composition, after the heat treatment some of the samples becomes opalescent and others remain transparents.



## ICP: EFFECT OF HEAT TREATMENT ON IONS LEACHING

The borosilicate glass particle size: D90:<40µm.
Ions concentration measured in a $HNO_3$ solution (0,003M) at 37ºC for different time-points before and after the heat-treatment.
PSBS3 and PSBS5 respectively transparent and opalescent are studied.

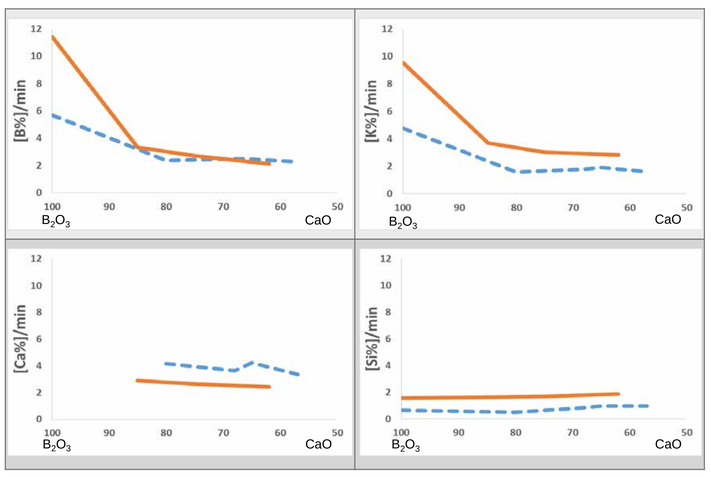



Results are expressed as dissolution%= $C_{solution}/C_{initial}$.

For both types of glasses the heat treatment **enhance** the ions leaching. It is supposed to be due to the phase separation of the borosilicate system that cause a changing in the microstructure of the glasses.

## ICP: EFFECT OF THE GLASS COMPOSITION ON IONS LEACHING

Kinetic study: comparison between opalescent and transparent glasses.



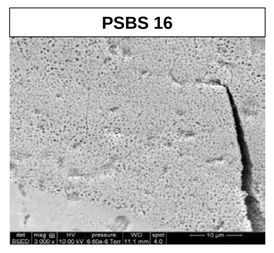

Opalescent glasses tend to release faster calcium ions into the acid solution, while in transparent glasses the potassium and the silicon are leached out at higher rates. The dissolution process thus depends on solution conditions (pH, temperature, etc.) as well as on **composition and microstructure** of the glass. The latter one changes after the heat treatment.

## SURFACE MICROSTRUCTURAL STUDIES

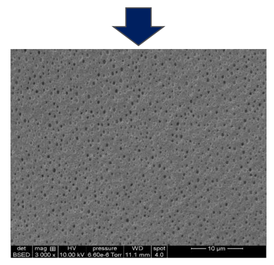
One opalescent (PSBS16) and one transparent (PSBS8) glasses are compared in this study with scanning electron microscopy.

**NORMAL CONDITION**: Immersion of bulk glasses in $HNO_3$ (1M) at 37ºC for 48h.

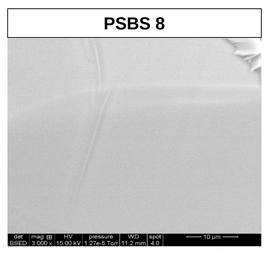
**HARD CONDITION**: Immersion of bulk glasses in $HNO_3$ (1M) at 90ºC for 48h.



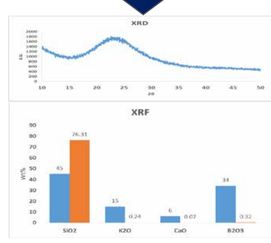

Porous structure detected for the opalescent sample.



Porous structure detected for the opalescent sample. Demonstrate high glass durability for this composition.



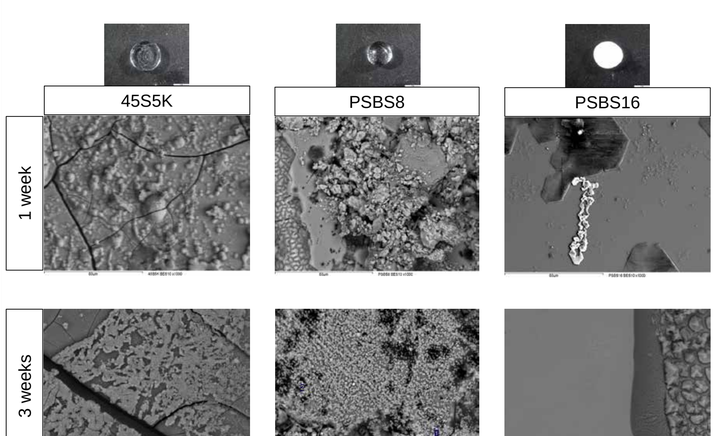

Homogeneous surface detected for the transparent sample.




Pure amorphous silica powder remain, the boron-rich phase is dissolved.

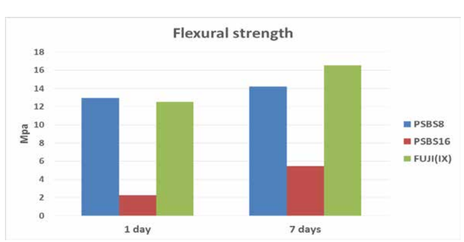
## BIOACTIVITY & MECHANICAL TEST

Bioactivity measured directly on bulk glasses following the ISO 23317 standard.
Immersion in SBF and measuring HA formation at different time-points. 45S5K glass, with $K_2O$ instead of $Na_2O$, was used as a control.

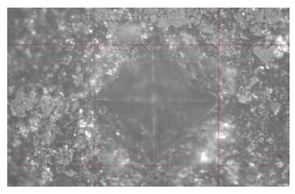



SEM confirm the formation of HA on the surface of 45S5K and PSBS8 with the tendency to expand on the surface during long periods. On PSBS16 surface are detected salts depositions and initial formation of calcium agglomerates.

Flexural strength and Vicker's microhardness measured on the cement formed with PSBS glass powders mixed with GC Fuji IX GP® liquid with a 1:2 ratio.




PSBS8 shows values comparable with the FUJI (IX)® GIC. The fact that is leaching large amount of boron could be a determining factor.



Microhardness tests are ongoing. PSBS8 shows HV=43.5 after 45 days of maturation. The value is comparable with others GICs.

## CONCLUSION & FUTURE PERSPECTIVES

- All the samples were characterized and proved to be 100% amorphous also after the thermal treatment
- The heat treatment affect the ions leaching enhancing the glass dissolution
- $Al_2O_3$ affects the glass resistance increasing the network connectivity and durability stabilizing the glass structure
- High $B_2O_3$/CaO ratio slightly enhances calcium and potassium leaching
- SEM studies shows different surface microstructures between the transparent and the opalescent samples. Phase separated structures can be hypothesized
- PSBS8 shows HA formation and flexural strength values closed to the commercial product used as a control. It can be considerate as a good candidate for further studies. The high boron release could be a key point for increasing bioactivity and mechanical properties.

## BIBLIOGRAPHIC REFERENCES

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