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Research Article

Study of the Bioactivity of Various Mineral Compositions of Bioactive Glasses

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Abstract Many types of bone substitutes exist to fill a bony defect. They must be compatible and bond with the host tissue without any formation of fibrous capsule. The direct apposition of bone to bioactive materials, including bioactive glasses and calcium phosphates, has already been demonstrated. In this work, different compositions of bioactive glasses elaborated by melting and rapid quenching were studied. They were based on SiO_2-CaO-Na_2O and P_2O_5. Several physicochemical methods like XRD, FTIR, SEM, and ICP-OES were employed to characterize all bioactive glasses. They were investigated as bony biomaterials using "in vitro" assays. The aim was to evaluate their chemical reactivity and their bioactivity after soaking in simulated body fluid (SBF).

Keywords bioactive glasses; characterization; "in vitro" assays; reactivity; bioactivity

1 Introduction

Synthetic biomaterials used in orthopaedic surgery and jaw bone surgery offer to surgeons many advantages. They can be elaborated with precise mineral composition and without risks of virus transmissions. In this way, some synthetic materials like bioactive glasses [1], calcium phosphate [2,9], calcium carbonate in the aragonite form [7,8] and aluminosilicates, geopolymers [6] were studied for several applications as bony biomaterials in the biomedicine field.

This work concerns particularly the elaboration of bioactive glasses with different mineral compositions and their physicochemical characterization to evaluate their chemical reactivity and their bioactivity after the "in vitro" assays. This kind of biomaterial is able to form a chemical bond directly with natural bone [4,5]. This biomaterial was elaborated by using the melting and rapid quenching.

The used mineral composition was SiO_2 45 wt%, CaO 24.5 wt%, Na_2O 24.5 wt%, and P_2O_5 6% wt%. Several physicochemical methods like thermogravimetric analysis DTA/TG (Labsys 1600), X-ray diffraction (XRD) technique using Philips PW3710 diffractometer with Cu Kα radiation, Fourier-transformed infrared spectroscopy (FT-IR) (BRUKER EQUINOX 55) and scanning electron microscope SEM (JEOL-JSM 6301F) were employed to evaluate the physicochemical behavior of compounds before and after soaking in simulated body fluid (SBF). For estimation of the "in-vitro" chemical reactivity and bioactivity, the compounds were immersed in SBF liquid at different times. In addition, the Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES spectro) was also employed to highlight the ionic exchanges between compounds and SBF solution after soaking versus time. This original method offers a high sensitivity less than 1 μg/g and a good accuracy. It was employed particularly in the goal to evaluate the variations of calcium, phosphorus and silicon concentrations versus soaking time in SBF liquid.

In this work, the elaboration of bioactive glasses, the physicochemical behavior was reported. The chemical reactivity and the bioactivity after the "in vitro" assays were described.

2 Materials and methods

To elaborate our bioactive glasses, the following chemical products were used. Sodium Silicate (Na_2SiO_3), calcium Silicate ($CaSiO_3$) and Sodium Phosphate ($NaPO_3$) were prepared to obtain glasses in the ternary glasses system SiO_2-CaO-Na_2O combined with P_2O_5. They mere synthesizes using melt quenching process. The three melt-derived glasses were denoted:

- E3 (46.5% SiO_2, 23% CaO, 24.5% Na_2O, and 6% P_2O_5),
- E4 (46.5% SiO_2, 28% CaO, 19.5% Na_2O, and 6% P_2O_5),
- E5 (45.5% SiO_2, 10% CaO, 38.5% Na_2O, and 6% P_2O_5).

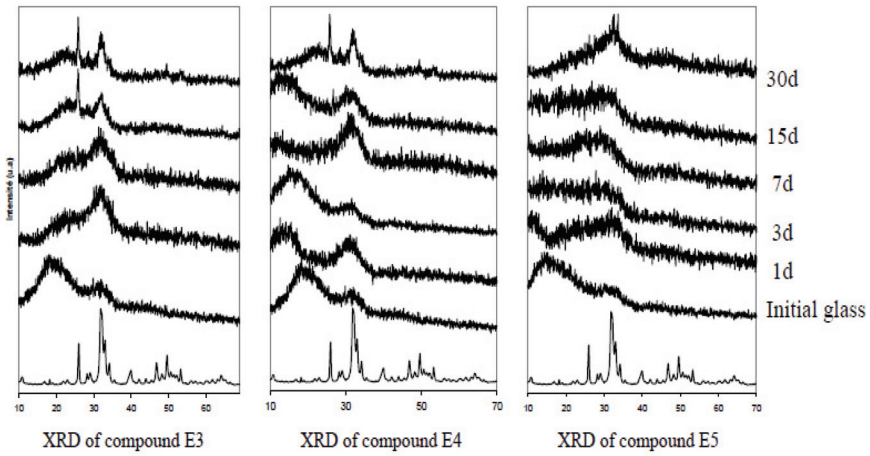

Figure 1: XRD patterns of glasses E3, E4, and E5 versus time of soaking in SBF solution.

Components	(Wt%)
$CaSiO_3$	50
$NaSiO_3$	41.4
$NaPO_3$	8.6

Table 1: Concentrations of chemical products used for glass elaboration.

The weight percentage of all compounds used in glass elaboration are presented in Table 1.

Raw materials were weighed and mixed for one hour in a polyethylene bottle. Premixed products were melted in a covered Pt crucible at the temperature range 1300–1350 °C for two hours. Samples were cast in to a Layton mold to form 8 mm × 13 mm cylinders. Thermogravimetric analysis (TGA) (Labsys TGA-DTA/DSC, Setaram) was performed on the powders calcined to determine the temperature of vitreous transition (T_g). After annealing at $T_g \pm \Delta T_g$ for 8 hr, the cylinders were cut in to disks. Then, those materials were coated in resin in order to detain one surface on air. Polished samples were cleaned in an acetone bath and air-dried [3].

The SBF-K_9 solution [3] was prepared by dissolving the following reagent chemicals in deionizer water: NaCl, $NaHCO_3$, $(CH_2OH)_3CNH_2$, KCl, ($CaCl_2$, $2H_2O$), KH_2PO_4, ($MgCl_2$, $6H_2O$) and 6 HCl to regulate the pH at 7.4. Reagent amounts were added, *in vitro* test samples were performed under static conditions soaking in sealed polyethylene bottles with 8 mL of SBF – K_9 solution at 37°. The solutions were kept at 37 °C in an incubator at static condition for the following time intervals: 1, 4 hr, 1, 3, 7, 15, 20, and 30 days.

The samples were removed from the incubator, rinsed gently, first with pure ethanol and then using deionized water, and left to dry at ambient temperature.

3 Results and discussion

A thermal analysis of all compounds by using Thermogravimetric analysis (TGA) was achieved to determine the temperature of vitreous transition (T_g). It depends widely on chemical composition. The T_g of E3 is at 537 °C and the T_g of E4 is of 550 °C when the T_g of E5 is only of 425 °C.

XRD diagrams show in the three unsoaked samples (Figure 1) an amorphous structure of bioactive glasses E3, E4, and E5. The calcium phosphate layer formed on the E3 surface glass is not crystallized same after 20 days immersion. The kinetics of crystallization of this phase is faster for E4 bioglass. Indeed, after 5 days immersion, the peaks of crystallization relating to the layer of HA formed in the E3 glass surface starts to appear and the intensity increase progressively versus the time of immersion. After 15 days of soaking in SBF, the XRD pattern show rays with maximum at about 32°. These picks correspond respectively to (002), (300), (321), and (004) reticular plan and highlight the apatite-like layer.

The ICP-OES analysis carried out on the SBF solution after each soaking of bioactive glasses highlight different behavior of compounds depending on their chemical composition.

Obtained results (Figure 2) show that the Ca kinetic releasing from bioactive glasses E3 and E4 is different then that from glass E5.

350 hr after soaking of bioactive glass E3 in the SBF solution, the concentration of Ca is of 120 μg/g when this concentration is of 110 μg/g in the SBF after soaking bioactive glass E4. Whereas, this concentration is only of 20 μg/g in the SBF after soaking glass E5.

These results show that glass E5 with its specific mineral composition undergo a high chemical reactivity than that of glasses E3 and E4. It can be considered as a resorbable

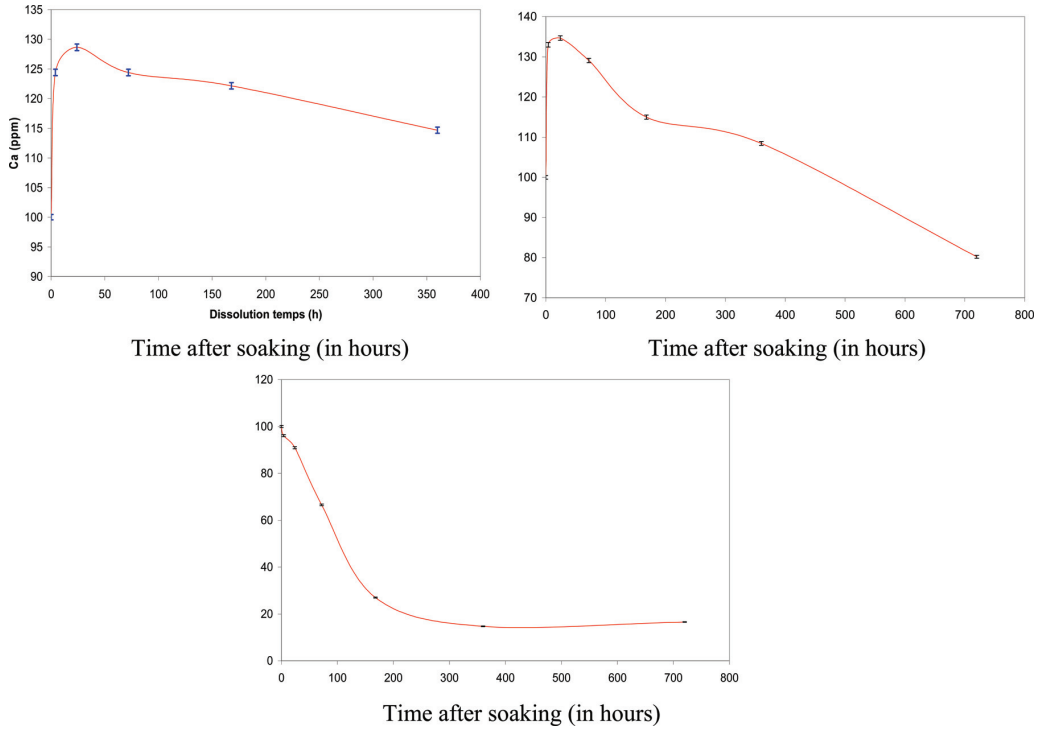

Figure 2: Ca concentrations in SBF solution after soaking of bioactive glasses respectively E3, E4, and E5 in SBF at different times in hours.

biomaterial. The same behavior was observed concerning other chemical elements P and Si.

E3 and E4 bioactive glasses interact with mineral composition of SBF liquid and present a bioactive biomaterials. However, the bioactivities kinetic of E3 mineral composition offer a bioactivity less than that of E4 mineral composition.

Formation of an apatite-like layer on glass surfaces after soaking in SBF solution was evidenced. This study highlighted a bioactive character for the E3 and E4 compositions with different bioactivity kinetic and a resorbable character for the E5 composition. This study was insured by using FTIR and SEM methods.

This result is important because it offers to surgeons more possibilities to apply one of these compositions according to different parameters like the site of implantation, the age and many other factors specific to each patient.

4 Conclusion

This work shows the behavior of various mineral compositions and their bioactivity of bioactive glasses. Obtained results highlight the physicochemical behavior of each bioactive glass. The releasing of calcium from the compounds to the SBF solution with different kinetics show also that the chemical reactivity and the bioactivity depend on the chemical composition of phospholicate compounds.

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