

Eutectic Bioceramics

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Eutectic bioceramics [1, 2] have been developed from glasses [3] in the CaO-P₂O₅-SiO₂ system in order to replace bone tissue. These materials have two phases, one of them is bioactive (calcium silicate) and the other is bioabsorbible (calcium phosphate) [4]. When they are in contact with the body fluids, bioabsorbible phase is dissolves, producing porous materials similar to the bone mineral structure, including its mechanical properties. In this work a technique for the synthesis of these materials was developed, and the materials obtained were characterized by X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), and Energy dispersive Spectroscopy (EDS)

The raw materials were quartz (SiO₂), calcium carbonate Ca(CO)₃ and tricalcium phosphate (β-TCP). Calcium was calcined to obtain calcium oxide which was mixed in 1:1 molar rate with quartz, placed in a Pt-Rh crucible and synthesised in solid state reaction. The resulting material was mixed 1:3 molar with β-TCP, placed again in the crucible, and was fused above the eutectic point in 1402 °C. Liquid material was quenching on steel plate to room temperature (25 °C). The glass was crushed until a 60 μm-particle size powder was obtained. The powder was placed on a sheet platinum crucible and heated up 1500°C. Later, a slow cooling process (0.5 °C/h) was applied until 1390 °C. Cooling speed was varied to obtain different samples, that were studied by XRD, SEM and EDS.

The XRD analysis shows that the sample whose cooling rate is 3.5 °C/min (sample 1), was composed by crystalline phase mixture: calcium phosphate (hydroxyapatite HA), calcium silicate and oxide calcium silicate. When the cooling rate increased to 5 °C/min (sample 2) phosphate phase changed to HA and tricalcium phosphate (Ca₃(PO₄)₂), whereas silicate phase became undetectable. Besides, at 10 °C/min cooling rate (sample 3), diffraction showed only HA.

The SEM analysis presented that samples obtained were constituted by two different phases, whose main elements, according to EDS were Ca, P, O for one of the phases and Ca, Si, O for the other. Nevertheless the microstructure of each one was different (FIG. 1). Sample 1 displayed two different zones, white and gray, with lamellae structure in different directions; this sample presented fractures in white zone, indicating a fragile material (a). The sample 2, was composed by two zones, which were not distinguished as easily as in previous case; it was possible to observe that small aligned grains from guides in different directions constituting the clearest zone (b). In the sample 3 could be observed a dendritic structure ordered in different directions (c). The etched with diluted HCl revealed different eutectic structures. It was in the sample 3 where irregular lamellae structures could be observed (FIG. 2). This morphology is very similar to bone structure.

According to XRD analysis it is possible to conclude that calcium phosphate was the first phase formed, because was presented in all samples. Nevertheless calcium phosphate phase was not always the same one, for example, in the sample 2 this phase was $\text{Ca}_3(\text{PO}_4)_2$, whereas in the other two samples it was HA. This is the reason why the change depends on the cooling rate.

The SEM analysis demonstrated that all samples had two phases, one of them was calcium silicate (bioactive) and the other was calcium phosphate (bioabsorbible) Nevertheless, the HCl etching showed for the sample, whose sample 3; it was very similar to the bone structure.

References

- [1] Tejeda-Cruz Adriana. Master Thesis. UNAM C.U. 2003.
- [2] De Aza P. Et al. *Biomaterials*. 18 (1997) 1285 – 1291.
- [3] Clément J. Et al. *Bioceramics* 11 (1998) 375 – 378.
- [4] Hench L. J. *Am. Ceram. Soc.* 7 1991 1847-1510.
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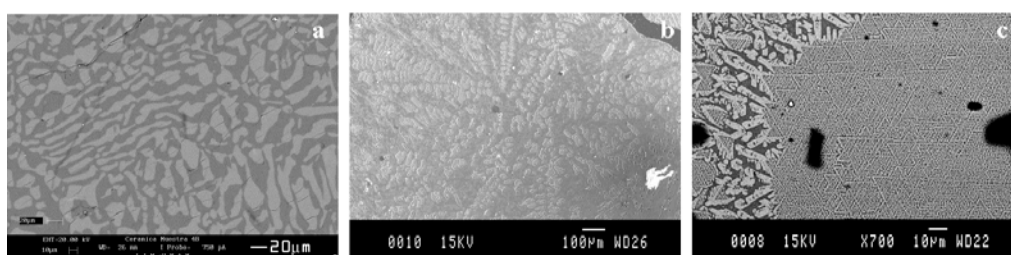


FIG. 1. SEM analysis for samples with different cooling rate. (a) Sample 1, 3.5 °C/min, (b) sample 2, 5 °C/min and (c) sample 3, 10 °C/min.

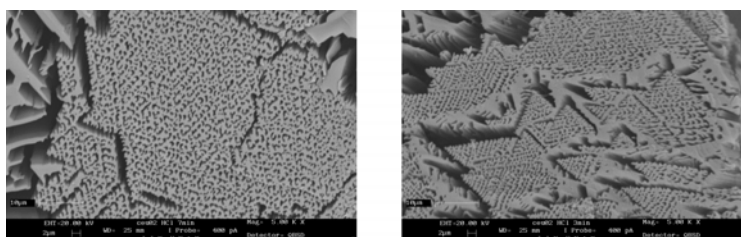


FIG. 2. The etched with diluted HCl revealed different eutectic structures It was in the sample 3 where the morphology is very similar to bone structure.