MicroCT and preparation of β-TCP granular material by the polyurethane foam method

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Introduction
Commercial β-tricalcium phosphate (β-TCP) is commercially available in granules manufactured by sintering of powders. β-TCP has been recognized as a suitable ceramic material with bioactive properties since several decades [1]. TCP is a highly resorbable material that can be prepared in various conditions. Macroporous blocks can be used in orthopedics and granules are preferred in dentistry and maxillofacial surgery for filling small bone defects such as alveolar sockets after tooth extraction or for sinus lift elevation. A large number of papers have been published concerning the fabrication of TCP scaffolds or granules. An interesting method is the use of polyurethane foam to prepare 3D scaffolds that are sintered and crushed in a second time. Several commercial processes are available but the different steps of the full process have not been fully analyzed. The method was originally proposed by Schwartzwalder and Somers and has been used in a number of patents for preparing different types of porous biomaterials [2].

Method
Briefly, a polymer foam is used as a template and filled with a ceramic slurry; the composite adhere onto the surface of the foam and this creates macroporosity. The composite is then submitted to heat-treatment at an elevated temperature to remove the polymeric template and to sinter the ceramic coating layer. The polymer foam is thus destroyed and leaves small internal voids (microporosity). We have evaluated the different steps of the manufacturing process of β-TCP ceramics granules prepared from blocks obtained with the polyurethane foam technology. 3 types of slurry were prepared with 10, 15 and 25g of β-TCP per gram of polyurethane foam [3]. Analysis was done by scanning electron microscopy, EDX, Raman spectroscopy and microcomputed tomography combined with image analysis. A special algorithm was use to identify the internal microporosity (created by the calcination of the foam) from the internal macroporosity due to the spatial repartition of the material.

Results and Conclusions
The low β-TCP dosages readily infiltrated the foam and the slurry was deposited along the polymer rods. On the contrary, the highest concentration produced inhomogeneous infiltrated blocks and foam cavities appeared completely filled in some areas. 2D microcomputed sections and reconstructed 3D models evidenced this phenomenon and the frequency distribution of the thickness and separation of material trabeculae confirmed the heterogeneity of the distribution. When crushed, blocks prepared with the 25g slurry provided the largest and irregular granulates.

References
Figure 1. Micro computed tomography (microCT), 3D models of: A) the polyurethane foam; B) the sintered material prepared with the 10g slurry; C) with 15g; D) with 25g. In each case, the internal porosity due to pyrolysis of polyurethane has been reconstructed in a colored model; the $\beta$-TCP material in a semi-transparent blue model.

Figure 2. Histogram of frequency distribution of the thickness of the $\beta$-TCP material prepared with a 10 (black symbols), 15 (medium grey) and 25g (light grey) slurry. With 25g, compact and dense granules are obtained.