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OBTAINING POROUS STRUCTURES FROM BIOGLASS AND TRICALCIUM PHOSPHATE THROUGH THE REPLICATION METHOD

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Abstract. The aim of this work was to make scaffolds based on β-tricalcium phosphate (TCP) and bioglass (BG) by the method of replicating a polymeric sponge. Initially, the ceramic powders were obtained by co-precipitation and the bioglass by the sol-gel method. Subsequently, the powders were used to obtain porous structures. Different compositions were obtained by varying the TCP/BG ratio. Finally, the scaffolds were subjected to a thermal process to burn the sponge and the second step implied sintering the ceramic structure. The scaffolds were tested to evaluate the morphology, mechanical and bioactive properties and were also biologically characterized by in vitro tests.
Keywords: Bioglass; Tricalcium phosphate; Scaffold; Replication method

Rezumat. Scopul lucrări a fost de a realiza scaffold-uri pe bază de β-fosfat tricalcic (TCP) și biosticlă (BG) prin metoda replicării unui burete polimeric. Inițial, au fost obținute pulberile ceramice prin metoda co-precipitării și biosticla prin metoda sol-gel. Ulterior, pulberile au fost utilizate pentru obținerea structurilor poroase. Au fost obținute diferite compoziții prin varierea raportului TCP/BG. În final, scaffold-urile au fost supuse unui proces termic în vederea arderii buretelui și sinterizării structurii ceramice. Scaffold-urile au fost teste în vederea evaluării morfologiei, proprietăților mecanice și bioactive și au fost, de asemenea, caracterizate din punct de vedere biologic prin teste *in vitro*.

Introduction

Bone defects are characterized by a lack of bone tissue, which can be caused by various pathological processes, such as: osteoporosis, osteoarthritis, osteomyelitis, cancer or various traumas [1]. The main bone defect caused by pathological processes, especially osteoporosis, is represented by fractures [2]. Osteoporosis is the most common bone disease in humans, representing a major public health problem and being characterized by low bone density and changes in bone micro-architecture, which can compromise bone strength and increase the risk of fractures [3]. Although bone tissue has the capacity to regenerate, this capacity is sufficient only for the healing of small bone defects, such as cracks or fractures up to about 2 cm, any type of larger defect requiring specialized treatment. The standard treatment at this moment, for large bone defects, may involve: the use of a metal device for bone fixation, the use of autografts or allografts. The risks associated with the standard treatment of large bone defects have led to the development of tissue engineering. At this time, there are new opportunities for the development of high-performance materials for the treatment of bone defects because great progress has been made in the synthesis and processing of materials, and bone structure is increasingly better understood [4].

Materials and Methods used to obtain scaffolds

Tricalcium phosphate powder was obtained by co-precipitation method. Calcium nitrate tetrahydrate and diammonium acid phosphate were used as precursors, and the required amounts were set so that the Ca/P molar ratio to be 1.5.

The bioglass powder was obtained by the sol-gel method, using tetraethylorthosilicate, triethylphosphate, calcium nitrate and sodium nitrate as precursors. The required quantities were calculated to obtain a 45S5 bioglass.

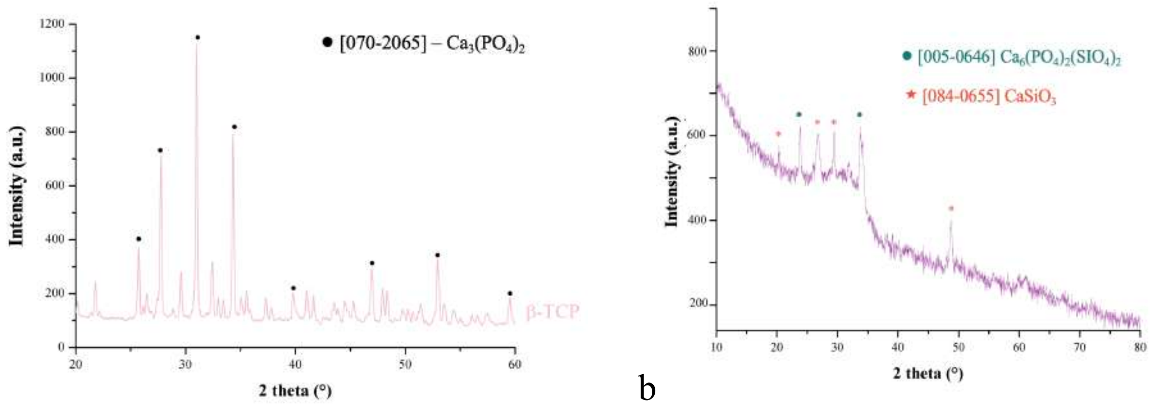
The scaffolds were obtained by the replication method. A suspension was obtained from a mixture of ceramic powder and bioglass powder, in different mass ratios, and a 4% polyvinyl alcohol (PVA) solution. The concentration of the powder in relation to the PVA solution was 50%, and its homogenization was achieved under magnetic stirring. Polyurethane sponges, which were 1x1x1 cm in size, were later immersed in the suspension. The samples were subsequently dried at room temperature for 24 h and placed in an oven to perform the heat treatment.

Results

X-ray diffraction (XRD) was performed at room temperature using a Shimadzu XRD 6000 diffractometer (Shimadzu, Kyoto, Japan). Ni-filtered Cu Kα radiation (α=1.5406 Å) from a Cu X-ray tube was used. The powders, which were previously mortared, were scanned in the Bragg angle, the 2θ range being 10-70°, with a scanning rate of 2°/min.

In Figure 1.a. is presented the diffractogram of the ceramic powder, which shows that the material is crystalline as the diffraction maxima show a high intensity. The mineralogical phase that stands out, according to ASTM sheet no. 070-2065, is β-$Ca_3(PO_4)_2$.

In Fig. 1.b. are presented the diffractogram of the bioglass powder and the mineralogical phases that stand out, according to ASTM sheets no. 084-0655 and no. 005-0646, namely $CaSiO_3$, respectively $Ca_6(PO_4)_2(SiO_4)_2$.



a b

Fig. 1. Diffractogram of the powder: a. tricalcium phosphate, b. bioglass

Scanning electron microscopy (SEM) was used to determine the morphology of the scaffolds and to show the compatibility between the phases. SEM micrographs were taken with a HITACHI S2600N equipment with an EDAX probe. All samples were pre-coated with a gold layer.

In Figure 2 are presented both the morphology of the control scaffold made only of TCP, and the morphology of the composite ceramic scaffolds, at both sintering temperatures. From these microscopy images, it is evident that all samples exhibit open porosity with interconnected pores and a pore size ranging from 140 µm to 600 µm. The size of the pores and their interconnected structure can provide a favorable environment for cell penetration and proliferation and can also stimulate the development of new bone tissue throughout the three-dimensional matrix of the structures.

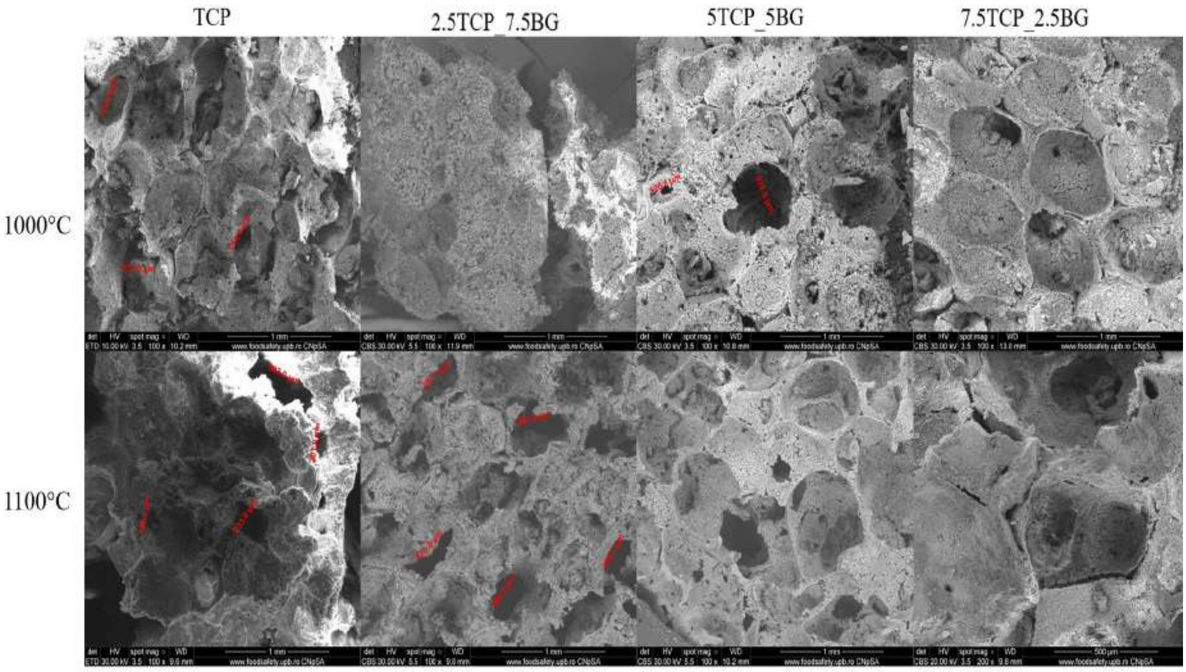


Fig. 2. SEM micrographs of the scaffolds

Conclusions

In this work, scaffolds based on tricalcium phosphate and bioglass - 45S5 composite ceramics were successfully obtained, by the method of replicating a polymer sponge. Several tests were made to the samples in order to investigate the microstructure and morphology. The morphology was observed by means of scanning electron microscopy, and all samples were observed to exhibit open porosity with interconnected pores, which could favor the penetration of cells into the three-dimensional structure.

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