



Novel production and characterization of porous calcium phosphate suitable for bone tissue engineering applications

F. Stergioudi^a, A. Choleridis^a, E. Paulidou^b, E. Smyrniaios^a, N. Michailidis^{a,*}

^aPhysical Metallurgy Laboratory (PML), Mechanical Engineering Department, Aristotle University of Thessaloniki, 54124 Thessaloniki, Greece

^bSolid State Section, Department of Physics, Aristotle University of Thessaloniki, 54124 Thessaloniki, Greece

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Abstract

Porous calcium phosphate materials have found enormous use in biomedical applications including hard tissue regeneration. In this study, tailored made open-cell porous calcium phosphate samples were produced by employing a novel dissolution sintering process, using crystalline raw cane sugar as a leachable pore former material. The process parameters of the production stages were properly adjusted to optimise the quality and structure of open-cell porous calcium phosphatesamples. The influence of sintering conditions on the macro and micro-structure of the resultant porous product is investigated with a view to produce high-quality and strong porous calcium phosphate samples. Preliminary in-vitro biodegradation results indicate formation of a bone-like apatite on the porous samples surface. Mechanical performance of the produced porous calcium phosphate samples is interesting and dictates their potential use as low load-bearing implants for hard tissue replacement or as filling materials and scaffolds for cancellous bone defects repairs.

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1. Introduction

The development of advanced materials suitable for hard tissue engineering applications, at the same time possessing mechanical properties and biological behavior comparable to those of the natural teeth or bones is among the most challenging problems facing modern materials science and engineering [1–4].

A number of techniques have been developed for producing porous bioceramics, which can be divided into those using natural resources (like coral or natural bone) [1,5–8] and those employing synthetic processing routes [1,7–9]. The bone grafts implants of natural origin (coral for example) present certain drawbacks such as supply difficulty, biological variability and viral or bacterial contamination risks and therefore their

utilization is limited [3,4,8]. On the other hand synthetic bioceramics offer an attractive alternative to natural bone grafts and have been widely used as bone substitutes [3,9–12].

Among them, calcium phosphate ceramic materials are used extensively in bone tissue repair, due to their inherent biological properties such as biocompatibility and osteoconduction. The greatest potential for bone substitution is shown by materials based on hydroxyapatite (HAp), $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$, tricalcium phosphate (TCP), $\text{Ca}_3(\text{PO}_4)_2$, and biphasic calcium phosphates (BCP, a mixture of HAp and TCP). Hydroxyapatite (HAp), is considered as low biodegradable ceramic, whilst tricalcium phosphate (TCP) exhibits considerably higher resorption ability in physiological fluids [11,13,14]. Attempts to gain improved biological response and mechanical behavior of these bioceramics have led to investigations on various chemical compositions, architectures and microstructures [3,4,8,11,12,15].

Several synthetic techniques have been developed for the fabrication of porous calcium phosphate bioceramics. Among them, replication of polymer foams [8], freeze casting [8,16],

*Correspondence to: Department of Mechanical Engineering, Physical Metallurgy Laboratory, Aristotle University of Thessaloniki, Thessaloniki 54124, Greece. Tel.: +30 2310 995891; fax: +30 2310 996069.

E-mail address: nmichail@eng.auth.gr (N. Michailidis).

gel-casting foaming [8,12,17], foaming with employment of several pore-creating additives [8,18,19]. Various combinations or alterations of these methods are the most widely employed techniques for producing porous hydroxyapatite-based materials.

An ideal bone graft should be osteoconductive, osteoinductive, resorbable, easy to shape and handle, having adequate mechanical strength [1,2]. Nevertheless, the development and optimization of new techniques are necessary to implement calcium phosphate biomaterials according to the clinical requirements and further improve their structural architecture, biological performance and mechanical response.

In order to optimize manufacturing processes, enhanced understanding of the underlying processing-structure-property relationships of these materials is necessary. Correlations for essential mechanical and biological properties such as elastic moduli, compressive strength and biodegradation rates are also imperative, so as to obtain specific structural properties and especially a controlled open macroporous internal architecture for bioceramic implants [8,17].

In this study, a novel space-holder method for manufacturing open-cell porous calcium phosphate using crystalline raw cane sugar as a leachable pattern, is reported. Crystalline raw cane sugar was successfully used in the past as a pore former material for manufacturing Al-foams and ceramic alumina-vanadia foams by a dissolution-sintering process [20,21]. This method is economically attractive and environmentally friendly, insofar as it does not lead to any hazardous by-products or emissions, while no toxic, flammable or explosive materials are utilized, and the use of chemicals is not required at any stage of the manufacturing process. The space holder material is not only cheap and commercially available, but also chemically stable in contact with the parent metal material. Additionally, the space holder material can be easily removed from the green product prior to sintering, by a simple water leaching process at room temperature, assuring both minimum contamination of the final product and overall structural and morphological integrity of the porous sample. The flexibility and considerable control with regard to pore size, shape, and density of the produced porous samples is an important advantage of this process. The final porous sample exhibit homogeneously distributed open pores with uniform size, ensuring high structural quality and enhanced stiffness and strength. The reproducibility of the structure and properties of the porous sample within a narrow scattering range is among the advantages of the proposed method. It is also possible to obtain a purposely tailored distribution of pore size in the final sample with different particle size ranges.

In the space holder-sintering technique, the crucial step is the removal of the space holder from the green compacts. Dissolution techniques and pyrolysis processes are used for the elimination of the space-holders from the green product so as to generate the final porous structure. Sodium chloride, carbonates, and several polymers have been used as space holder materials for manufacturing porous ceramics, whilst organic binders such as PVA, cellulose based binders etc were necessary during the compaction stage for creating a stable and easy to handle green compact. Nevertheless, during the thermal

decomposition of such carbonates and polymeric materials, environmentally harmful gases are released. Additionally, most of the space holders used, possess low melting and decomposition temperatures in comparison with the high melting point of ceramic materials, thus making imperative the employment of complex, multi-step thermal cycles using extremely slow heating rates so as to prevent the collapse of the porous ceramic structure during the decomposing process. This, in turn, leads to time consuming sintering processes and increased energy costs during the production of porous ceramics. Furthermore, due to the gas evolution resulting from decomposition of the binder and the mismatch between thermal coefficients of the binder and ceramic powder during heating, fabrication defects like longitudinal cracks can form during the thermal cycle [22,23].

Concerning sodium chloride, although it can be leached in water, it is highly possible to leave residues that cannot be removed completely during sintering thus contaminating the final porous product.

The proposed method is particularly attractive given that the use of water as leaching agent eliminates the environmental drawbacks related to the removal of organic space holders. Since the whole process does not involve the use of organic or polymeric binders the anisotropic thermal expansion associated with typical organic substances that can lead to fabrication defects is thereof not an issue. As an additional benefit, the equipment necessary for water leaching is very simple whilst the need for complex, multi-step and time consuming thermal cycles is minimized.

The present paper aims to determine the influence of the proposed production process conditions on both the macrostructure morphology, as well as the microstructure of the resultant porous bioceramic, with a view to produce high-quality porous calcium phosphate. Among the scopes of the present study is to obtain preliminary results concerning both the mechanical strength and the biodegradation behaviour of the resulting porous bioceramic, to evaluate their potential use in medical and clinical applications for hard tissue replacements.

2. Experimental procedures

2.1. Production method

The raw materials used for manufacturing the porous calcium phosphate are hydroxylapatite powder ($\text{Ca}_5\text{HO}_{13}\text{P}_3$, purum) supplied by Fluka Chemika company and a crystalline raw cane sugar, in powder form, consisting of cube like crystals with smooth surfaces and relatively even size. In order to control the pore size of the final porous bioceramic within the desired range, the crystalline raw cane sugar powder was divided by a series of sieves into two size groups: 0.5–1.0 mm and 0.125–0.5 mm, with nominal mean sizes of 0.70 and 0.35 mm respectively. The HAp powder and the raw cane sugar with a chosen size range were weighed using an electronic balance with an accuracy of 0.001 g. The volume fraction of each constitutive element in the powder mixture can

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