



Compression behaviour of biphasic calcium phosphate and biphasic calcium phosphate–agarose scaffolds for bone regeneration

J.A. Puértolas^{a,b}, J.L. Vadillo^a, S. Sánchez-Salcedo^{c,d}, A. Nieto^{c,d}, E. Gómez-Barrena^e, M. Vallet-Regí^{c,d,*}

^a Department of Materials Science and Technology, Instituto de Investigaciones en Ingeniería de Aragón, I3A, Universidad de Zaragoza, Spain

^b Instituto Ciencia de Materiales de Aragón, ICMA, Universidad de Zaragoza-CSIC, Zaragoza, Spain

^c Dpto. Química Inorgánica y Bioinorgánica, Facultad de Farmacia, Universidad Complutense, Madrid, Spain

^d Networking Research Center on Bioengineering, Biomaterials and Nanomedicine (CIBER-BBN), Madrid, Spain

^e Department of Orthopaedic Surgery, Fundación Jiménez Díaz, Universidad Autónoma, Madrid, Spain

ARTICLE INFO

Article history:

Received 30 April 2010

Received in revised form 1 July 2010

Accepted 25 July 2010

Available online 29 July 2010

Keywords:

Biphasic calcium phosphate

Agarose

Mullins effect

Compression

Bone tissue engineering

ABSTRACT

There is an acknowledged need for shaping 3-D scaffolds with adequate porosity and mechanical properties for biomedical applications. The mechanical properties under static and cyclic compressive testing of dense and designed porous architecture bioceramic scaffolds based on the biphasic calcium phosphate (BCP) systems and BCP–agarose systems have been evaluated. The dense and designed porous architecture scaffolds in BCP systems exhibited a brittle behaviour. Agarose, a biocompatible and biodegradable hydrogel, has been used to shape designed architecture ceramic–agarose scaffolds following a low-temperature shaping method. Agarose conferred toughness, ductility and a rubbery consistency for strains of up to 60% of in ceramic BCP–agarose systems. This combination of ceramic and organic matrix helps to avoid the inherent brittleness of the bioceramic and enhances the compression resistance of hydrogel. The presence of mechanical hysteresis, permanent deformation after the first cycle and recovery of the master monotonous curve indicate a Mullins-like effect such as that observed in carbon-filled rubber systems. We report this type of mechanical behaviour, the Mullins effect, for the first time in bioceramics and bioceramic–agarose systems.

© 2010 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

1. Introduction

The main function of bone in the skeletal system is to provide structural support for the body and its vital organs. The vast majority of bone tissue defects heal spontaneously, but in some cases further treatment is required for compromised healing due to the interposition of soft tissue, improper fracture fixation, loss of bone, metabolic disturbances, impairment of blood supply and infection [1]. For these situations, bioceramics represent an interesting choice as bone filling biomaterials in clinical practice. Biphasic calcium phosphate (BCP) bioceramics are recommended for use as additives, or even as an alternative, to autogenous bone for orthopedic and dental applications due to their excellent bioactivity, biocompatibility and osteoconduction [2,3]. BCPs consist of a mixture of hydroxyapatite (HA) and β -tricalcium phosphate (β -TCP): the higher solubility of the β -TCP component increases the reactivity as the β -TCP/HA ratio increases. Therefore, the bioreactivity of these compounds, and consequently their degradability, can be controlled through the phase composition [4].

A bone graft serving as a bone tissue engineering scaffold should possess a three-dimensional (3-D) structure, as well characteristics such as biodegradability, biocompatibility and biomechanical stability. Furthermore, these materials should possess a highly interconnected porosity with a minimal pore size of 100 μ m as well as enough compressive strength to be comparable to natural bone tissue [5,6]. Nevertheless, bioceramics show high compression strengths and low tensile strengths. In addition, they are stiff materials with a high Young's modulus and are brittle because failure takes place without plastic deformation, making them unsuitable for load-bearing applications [7].

In the manufacture of 3-D bioceramic scaffolds, the use of traditional shaping procedures demands thermal treatment steps to enhance their mechanical resistance. However, a thermal step is not desirable as it leads to an exponential increase in particle size and crystallinity of the ceramic matrix as a result of the decrease in degradability [8]. Several authors have followed an approach based on the combination of bioceramic materials with other components, such as polymers, to produce hybrid structures, thus improving the mechanical and biological properties of the scaffolds [9–11]. Therefore, a good approach is to combine a low-temperature shaping method that offers a combination of the bioactivity and biocompatibility of the bioceramics, together with a biocompatible and biodegradable organic matrix which adds compliance and ductility, acting

* Corresponding author at: Dpto. Química Inorgánica y Bioinorgánica, Facultad de Farmacia, Universidad Complutense, Madrid, Spain. Tel.: +34 91 3941861; fax: 34 91 3941786.

E-mail address: vallet@farm.ucm.es (M. Vallet-Regí).

as a binder for the bioceramic matrix, thus providing compliance and wettability [10]. A suitable candidate is agarose. It is a biocompatible and biodegradable natural hydrogel which acts as gelling agent leading to strong gels, fast room temperature polymerization [12,13] and closely related properties in terms of stiffness and water permeability [14].

Taking into account the interconnected macroporosity as a requisite for successful tissue engineering scaffolds, a combination of the low-temperature shaping method with a technique that provides a controlled and designed porosity such as the solid free form (SFF) fabrication method is needed. In this sense, an epoxy resin generated by stereolithography acts as negative of the scaffold, leading to interconnected macroporosity after its removal.

Considering these points, in the present study we have evaluated the mechanical properties under compressive load of dense and designed porous architecture structures based on BCP bio-ceramics and the BCP–agarose systems. Evaluation of the mechanical properties of the hydrated scaffolds has been carried out to simulate physiological conditions in body fluid.

2. Materials and methods

2.1. Synthesis and fabrication of BCP and BCP–agarose scaffolds

2.1.1. Synthesis of BCP

A controlled crystallization method and subsequent heat treatment at 900 °C for 1 h, yield a biphasic calcium phosphate consisting of 26.9% HA and 73.1% β -TCP, as previously described by Sánchez-Salcedo et al. [15].

2.1.2. Scaffold fabrication by the gel-casting method

The gel-casting method [16] was chosen to manufacture the scaffolds of BCP used as a blank. Dense BCP scaffolds were named D-BCP when only the gel-casting method was used, and designed porous architecture BCP scaffolds (DA-BCP) when the stereolithography [17] was combined with the gel-casting method to produce designed architecture pieces (Scheme 1 g–n). The scaffolds were prepared as previously described [18].

2.1.3. BCP–agarose scaffold fabrication

The mixture of BCP with agarose (for routine use, Sigma–Aldrich) gives a combination of the biocompatibility and bioactivity of the ceramic matrix with the flexibility, wettability and biodegradability of agarose hydrogel.

The BCP–agarose scaffold fabrication was carried out as described in Ref. [11], resulting in non-designed porous architecture disks (D-BCP/AG). In order to obtain designed porous architecture scaffolds, the BCP–agarose suspension was poured into cylindrical moulds filled with an epoxy resin negative previously formed by stereolithography, resulting in DA-BCP/AG scaffolds.

2.1.4. Rehydration of the BCP–agarose scaffolds

The rehydration procedure consisted of the immersion of the dry bioceramic–agarose scaffolds in 2 ml of bidistilled water at 37 °C for 2 h in a closed vessel to avoid water evaporation. Afterwards, the pieces were blotted with absorbent tissue to eliminate any excess of water and were allowed to dry at room temperature for 15 min before starting any mechanical testing. The hydrated pieces of BCP with agarose are termed DA-BCP/AG-H and D-BCP/AG-H scaffolds. The swelling behaviour of the BCP–agarose scaffolds was determined by gravimetric (%W) and volumetric (%V) analysis [18].

2.2. Characterization of bioceramics and bioceramic–agarose scaffolds

Thermogravimetric (TG) analyses were carried out with a Perkin–Elmer Pyris Diamond TG/DTA thermobalance, between 30 and 600 °C in air at a flow rate of 100 ml min⁻¹ and a heating rate of 10 °C min⁻¹.

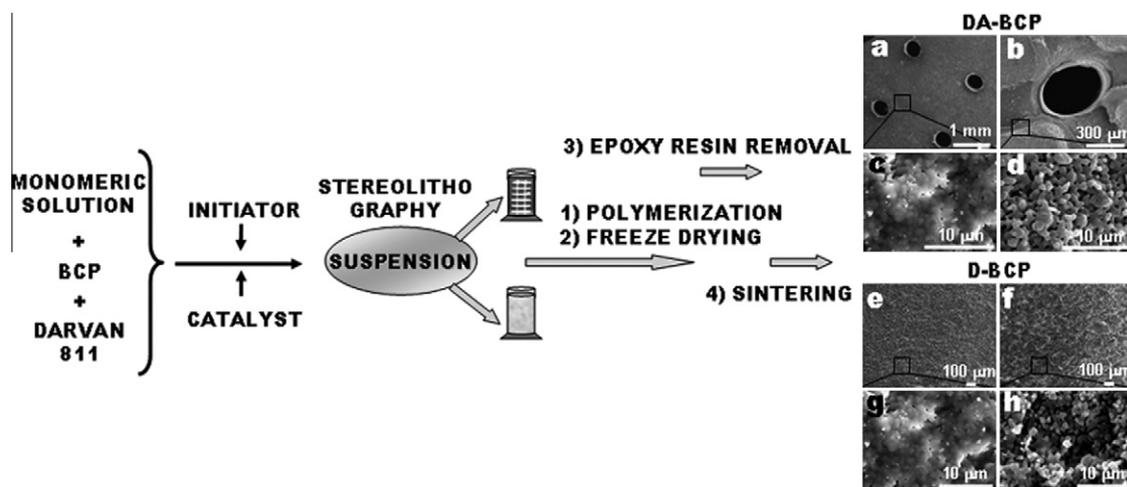
Scanning electron microscopy (SEM) was performed on a JEOL JM-6400 microscope–Oxford Pentafet super ATW system (Electron Microscopy Centre, UCM). Surface and fracture micrographs were obtained.

The porosity percentage and pore size distribution of the materials were determined by Hg intrusion porosimetry using a Micromeritics Autopore III 9410 porosimeter. The different materials were analyzed in the 0.004–412 MPa range, giving the porosity percentage and pore area in the interval of 0.01–300 μ m.

The surface textural features of the MTS in the mesoporous range were measured by nitrogen adsorption/desorption porosimetry at 77 K using an ASAP 2010 analyzer.

2.3. Mechanical tests

Uniaxial and unconfined compression tests were performed in an Instron 5565 static, screw-driven testing machine fitted with a 5 kN load cell. Once manufactured, the pieces were placed between two steel disks. The steel disks are 20 mm in diameter and a crosshead speed of 0.04 mm min⁻¹ was used. The samples were cylinders 4–



Scheme 1. Shaping methods and SEM micrographs of DA-BCP (a–d) and D-BCP (e–h).

ID	Title	Pages
993	Compression behaviour of biphasic calcium phosphate and biphasic calcium phosphate-agarose scaffolds for bone regeneration	7

Download Full-Text Now



<http://fulltext.study/article/993>



-  Categorized Journals
Thousands of scientific journals broken down into different categories to simplify your search
-  Full-Text Access
The full-text version of all the articles are available for you to purchase at the lowest price
-  Free Downloadable Articles
In each journal some of the articles are available to download for free
-  Free PDF Preview
A preview of the first 2 pages of each article is available for you to download for free

<http://FullText.Study>