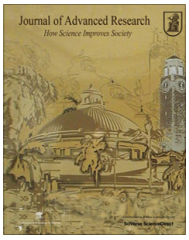
Journal of Advanced Research (2016) 7, 297–304

Cairo University

Journal of Advanced Research



ORIGINAL ARTICLE

Porous bodies of hydroxyapatite produced by a combination of the gel-casting and polymer sponge methods

Jazmín I. González Ocampo *, Diana M. Escobar Sierra, Claudia P. Ossa Orozco

Biomaterials Research Group, Bioengineering Program, University of Antioquia, Street 70 # 52 – 21, Medellin 1226, Colombia

ARTICLE INFO

Article history:
Received 11 March 2015
Received in revised form 19 June 2015
Accepted 26 June 2015
Available online 3 July 2015

Keywords:
Gel-casting
Hydroxyapatite
Polymer sponge
Porous body

ABSTRACT

A combination of gel-casting and polymeric foam infiltration methods is used in this study to prepare porous bodies of hydroxyapatite (HA), to provide a better control over the microstructures of samples. These scaffolds were prepared by impregnating a body of porous polyurethane foam with slurry containing HA powder, and using a percentage of solids between 40% and 50% w/v, and three different types of monomers to provide a better performance. X-Ray Diffraction (XRD), and Fourier Transformed Infrared (FTIR) and Scanning Electron Microscopy (SEM) were employed to evaluate both the powder hydroxyapatite and the scaffolds obtained. In addition, porosity and interconnectivity measurements were taken in accordance with the international norm. Bioactivity was checked using immersion tests in Simulated Body Fluids (SBF). After the sintering process of the porous bodies, the XRD results showed peaks characteristic of a pure and crystalline HA (JCPDS 9-432) as a single phase. SEM images indicate open and interconnected pores inside the material, with pore sizes between 50 and 600 μm. Also, SEM images demonstrate the relatively good bioactivity of the HA scaffolds after immersion in SBF. All results for the porous HA bodies suggest that these materials have great potential for use in tissue engineering.

Introduction

Hydroxyapatite (HA), with a chemical composition of $Ca_{10}(PO_4)_6(OH)_2$, is a biocompatible and bioactive material with a similar crystal structure to the biological apatite that can be found in hard tissues such as teeth and bones [1]. It has been widely used in orthopedics and dentistry because of its close biocompatibility with the human body and its good integration with bones. Additionally, it offers diverse conformation possibilities, given that it is possible to manufacture

* Corresponding author. Tel.: +57 011 574 2198589.
E-mail address: jazmin.gonzalez@udea.edu.co (J.I. González Ocampo).

Peer review under responsibility of Cairo University.

http://dx.doi.org/10.1016/j.jare.2015.06.006

powder, coatings, dense bodies, and porous bodies [2–4]. As a result, it has been suggested that HA is the best substitute for bone.

HA is a ceramic material that exhibits low mechanical properties, in particular low tensile strength and fracture toughness. Its application is limited to human body parts subject to either reduced mechanical strain or compressive stress only [5]. Consequently, several material properties need to be modified, i.e. mechanical strength, solubility and sintering processes. This is achieved by controlling composition, morphology, and particle size [6,7].

In spite of the limitations listed above, the use of porous hydroxyapatite bodies to repair bone defects is now a common practice in tissue engineering. The porous bodies provide the basis for new tissue growth and features such as biocompatibility, biodegradability and bioactivity. Also, the presence of interconnected pores makes nutrient diffusion and vascular growth possible, as well as providing mechanical strength, migration, cell proliferation, and growth [8,9].

The formation of new bone depends greatly on pore characteristics such as porosity percentage, pore size, pore size distribution and pore shape. Such factors must be controlled to establish the relationship between key structural features (pore size, pore size distribution and interconnectivity) and the mechanical performance of these materials.

The aim of this work was to manufacture HA porous bodies employing the gel-casting technique combined with polymeric foam infiltration. This method has the advantage of allowing a high level of interconnectivity, and so provides a uniform distribution of porosity.

In relation to the formation mechanism of the ceramic porous body is given by the replication of the polymeric foam structure used as template once the ceramic slurry gets inside its porosity. Besides, the use of monomers in gel-casting technique generates a 3D network that provides a temporary support to the HA particles. Both polymeric elements -monomers and polymeric foam- are completely burned when the bodies are sintered, providing cavities or porosities to the new only-ceramic structure granting not only an open and interconnected porosity, but also a uniform particle distribution [6,10,11]. Three different monomers and 40% and 50% w/v of HA solids were used to achieve the optimum values for some of the properties required in tissue engineering, such as morphology, pore size, bioactivity, percentage of porosity and interconnectivity. This report is the first to evaluate the behavior of porous bodies when varying the type of monomer and percentage of solids used.

Material and methods

Characterization of hydroxyapatite powder

The hydroxyapatite powder used as a raw material was evaluated by Fourier Transform Infrared Spectroscopy – FTIR – (Perkin Elmer Spectrometer – model Spectrum One detector DTGS). A wave range number of 4000–400 cm^{-1} was used. X-Ray Diffraction analysis was performed with a diffractometer (Brand Rigaku) and a copper (Cu) target as follows: λ = 1.5818 Å; angle 2θ; angle range of 0–60°.

Table 1 Sample nomenclature.

Sample	Nomenclature
40% of solids and Methacrylamide	40HAM
40% of solids and Acrylamide	40HAA
40% of solids and N-methylolacrylamide	40HAN
50% of solids and Methacrylamide	50HAM
50% of solids and Acrylamide	50HAA
50% of solids and N-methylolacrylamide	50HAN

Manufacture of porous bodies

Commercial hydroxyapatite powder from Strem Chemicals was used for the manufacturing process. The average particle size was 12.9 μm. The porous bodies were made according to the gel-casting technique combined with polymeric foam infiltration. Methacrylamide, Acrylamide, and N-methylolacrylamide were employed as functional monomers and the HA percentages were 40% and 50% w/v. The nomenclature of the samples employed is shown in Table 1.

Initially, a Velp Scientifica Arex magnetic agitator was used to mix the following substances for 3 min until a homogeneous solution was achieved: distilled water, the functional monomer, bisacrylamide (crosslinker), polyvinyl alcohol (binder), and methacrylic acid (dispersant). Afterward, the solution was mixed with hydroxyapatite powder in a Kika Labortechnik RW mechanical mixer for 15 min to break the agglomerates present in the powder. Next, the existing bubbles were removed from the mixture in a vacuum chamber. The catalyst and the initiator were then added and the homogenization was continued for another 5 min. Once the mixture was ready, infiltration inside the suspension of polyurethane foams was performed. The foams were left inside the chamber for 30 min so the polymerization process could be completed. Thermal treatment was then carried out, beginning with drying at room temperature for 24 h in order to eliminate the excess water. Samples were then dried in a Blinder model 53 ED drying oven at 70 °C for 15 h, thus producing a mechanic strength that enabled the samples to be handled. Finally, the samples were sintered at 1200 °C for 3 h.

Polyurethane foam was selected in accordance with preliminary tests and commercial grade was chosen to make it affordable. The selected foam presented a pore size average of 500 μm, wall thickness of approximately 150 μm, and interconnectivity, as shown in Fig. 1.

Characterization of porous bodies

For the characterization of the porous bodies, several tests were carried out using Scanning Electron Microscopy (SEM) with a JEOL microscope (model JSM-6490LV), and X-Ray Diffraction (XRD) using a Rigaku diffractometer with a copper (Cu) source (λ = 1.5818 Å, at an angle of 2θ and in a range of 0–60°). Bioactivity essays were then undertaken using immersion tests in simulated body fluid (SBF), in accordance with the procedure used by Kokubo and Takadama [12]. To verify the formation of the apatite layer formed on the surface, Ca/P of this was evaluated by energy dispersive spectroscopy (EDS) in JEOL microscope (model JSM-6490LV). In