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Fabrication and compressive strength of porous hydroxyapatite scaffolds with a functionally graded core/shell structure

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Abstract

A novel type of porous hydroxyapatite (HA) scaffolds with a functionally graded core/shell structure was fabricated by freeze casting HA/camphene slurries with various HA contents into fugitive molds containing a graphite template with three-dimensionally interconnected pores for the creation of a highly porous core. All the fabricated samples had functionally graded core/shell structures with 3-D periodic pore networks in a core surrounded by a relatively dense shell. The overall porosity of the sample decreased from 60 to 38 vol% with increasing HA content in the HA/camphene slurry from 20 to 36 vol% due to a decrease in porosity in both the core and shell regions. In addition, the compressive strength was improved remarkably from 12 ± 1.1 to 32 ± 3.0 MPa. The *in vitro* cell test using a pre-osteoblast cell line showed that the samples had good biocompatibility.
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1. Introduction

Functionally graded materials (FGMs) with a gradient of properties have attracted recent interest, owing to their superior mechanical and biological properties to conventional porous materials.[1–9] For example, it was reported that hydroxyapatite (HA) scaffolds with a porosity gradient mimicking the architecture of natural bone could induce rapid bone ingrowth into the high-porosity portion and withstand physiological mechanical stress through the low-porosity portion, when implanted.[1] Thus far, a variety of manufacturing methods for the production of FGMs have been developed, including the utilizations of multiple and differentiated impregnations,[1] multiple tape casting,[3] modified sponge replication method,[5] injected molding,[7] and freeze casting.[8,9] However, the development of new manufacturing methods that can tightly control the gradient of properties in a cost-effective manner is still a challenge.

This paper demonstrates the utility of the camphene-based freeze casting for the production of porous hydroxyapatite (HA) scaffolds with a functionally graded core/shell structure. To accomplish this, a fugitive mold consisting of a graphite template with three-dimensionally interconnected pores and a square mold was prepared, as illustrated in Fig. 1(A). Subsequently, HA/camphene slurries with various HA contents, ranging from 20 to 36 vol%, were freeze-cast into the fugitive molds at room-temperature, followed by freeze drying and sintering at 1250 °C for 3 h. This simple method allowed the formation of a highly porous core with a three-dimensional periodic pore network, surrounded by a relatively dense shell, as shown in Fig. 1(B). In addition, a porosity gradient could be created by controlling the HA content in the HA/camphene slurry. The fabricated samples were characterized by considering their porous structures, such as the overall porosity, pores formed both in the core and shell regions, as well as the densification of the HA walls. The crystalline phases of the samples were examined by X-ray diffraction (XRD). The compressive strength of the samples was also assessed to determine their structural integrity. The preliminary osteoblastic activity of the samples was also evaluated using *in vitro* tests to determine their biocompatibility.

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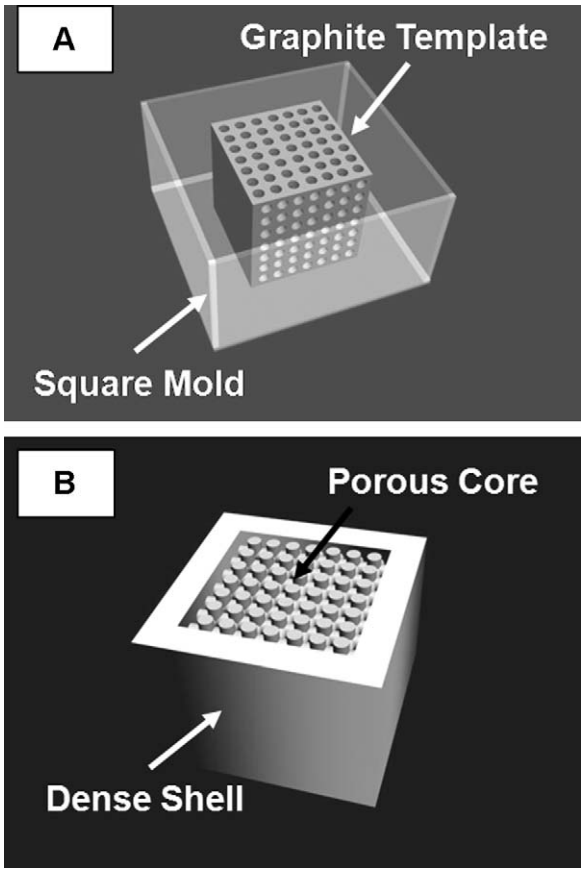

Fig. 1. Schematic diagrams showing (A) the fugitive mold used to produce the porous HA scaffolds with a functionally graded core/shell structure and (B) the resulting HA scaffold with a functionally graded core/shell structure.

2. Experimental procedure

Porous hydroxyapatite (HA) scaffolds with a functionally graded core/shell structure were produced by freeze casting HA/camphene slurries into fugitive molds. First, a graphite template was prepared by machining solid graphite, 13 mm × 13 mm × 13 mm in size, using a mini-CNC machine according to a predetermined design. The graphite template contained tightly controlled pores, 1 mm in diameter, with a fixed distance between the 0.7 mm pores. A fugitive mold was then prepared by assembling the graphite template with a square polymeric mold (see Fig. 1(A)).

Commercially available HA powder ($Ca_{10}(PO_4)_6(OH)_2$; Alfa Aesar Co., Milwaukee, WI, USA) and camphene ($C_{10}H_{16}$; Alfa Aesar/Avocado Organics, Ward Hill, MA, USA) were used as the ceramic powder and freezing vehicle, respectively. In particular, the as-received HA powders were calcined at 1000 °C for 1 h to reduce the specific surface area, which would be expected to improve the rheological properties of the HA/camphene slurry.[10] The HA/camphene slurries with various HA contents (20, 25, 30, and 36 vol%) were prepared by dispersing the HA powders in molten camphene by ball-milling at 60 °C for 24 h using an oligomeric polyester (Hypermer KD-4; UniQema, Everburg, Belgium) as a dispersant at 3 wt%. Subsequently, the warm slurries prepared were poured into the fugitive molds at room-temperature and then kept at this temperature for 1 h to allow complete solidification. After demolding, the green bodies, ~20 mm × 20 mm × 13 mm in size, were freeze dried to remove the solid camphene. The green samples were then heated at 900 °C for 3 h to remove the graphite template and dispersant completely, followed by subsequent heat-treatment at 1250 °C for 3 h to sinter the HA walls.

The porous structures (e.g., overall porosity, pores formed both in the core and shell regions, and densifications of the HA walls) of the fabricated samples were observed by scanning electron microscopy (FE-SEM, JSM-6330F, JEOL Techniques, Tokyo, Japan). The crystalline phases of the samples were characterized by X-ray diffraction (XRD, M18XHF-SRA, MacScience Co., Yokohama, Japan). For the compressive strength tests, the samples with dimensions of ~16 mm × 16 mm × 10 mm, which had been prepared by slightly grinding their top and bottom surfaces were loaded at a crosshead speed of 5 mm/min using a screw driven load frame (Instron 5565, Instron Corp., Canton, MA, USA). The stress and strain responses of the samples were monitored during the compressive strength tests. Five samples were tested to obtain an average value and standard deviation.

The *in vitro* cell tests of the samples were performed using a pre-osteoblast cell line (MC3T3-E1; ATCC, CRL-2593, USA). The cells were plated at a density of 5×10^4 cells/mL and cultured in a humidified incubator in an atmosphere containing 5% CO_2 at 37 °C. Minimum essential medium (α-MEM; Welgene Co., Ltd., Seoul, Korea) supplemented with 10% fetal bovine serum (FBS; Life Technologies, Inc., USA) and 1% penicillin–streptomycin was used as the culturing medium. The cell attachment was observed by SEM after culturing for 1 day.

3. Results and discussion

The freeze casting of HA/camphene slurries into fugitive molds was used to produce porous hydroxyapatite (HA) scaffolds with a functionally graded core/shell structure. In particular, a highly porous core with a three-dimensional periodic pore network was achieved using a graphite template that could be removed by thermal oxidation.[11] In addition, it was possible to create porosity gradients in the samples, in which the microstructures and porosity of the shells were controlled by adjusting the initial HA content in the HA/camphene slurry, ranging from 20 to 36 vol%, which would enable tailoring of their mechanical properties. In other words, compressive strength would increase with increasing HA content due to the lower porosity of the shell.[12,13]

Fig. 2(A) shows an optical micrograph of the samples produced with an initial HA content of 30 vol% before and after sintering at 1250 °C for 3 h. Regardless of the initial HA content, it was possible to infiltrate the HA/camphene slurries into the graphite template with three-dimensionally interconnected pores. This consequently endowed the samples with a tightly controlled pore structure after sintering. However, a HA content >36 vol% hindered the complete infiltration of the slurry into the pores formed in the graphite template owing to its high viscosity. All the fabricated samples showed similar linear shrinkage

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