A novel method to fabricate unidirectional porous hydroxyapatite body using ethanol bubble in a viscous slurry

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Introduction: Calcium phosphate based bioceramics are widely used as bone substitutes. Porous bioceramic should be produced in such a way that it yields controlled pore size, regular shape and orientation in a reliable and economical way. The few of the processing methods of porous bioceramics include incorporation of volatile organic particles, microwave processing of porous hydroxyapatite (HAp)/ tricalcium (TCP), replication of a polymer sponge, salt leaching method and extrusion process. But most of these methods do not result in a regular porous structure with high compression compressive strength. In this work, a novel fabrication method has been developed to produce unidirectional porous HAp body. It has been shown that a unidirectional porous HAp body can be produced easily by forming ethanol bubbles in a viscous slurry of methylcellulose. The microstructure was investigated using SEM (JSM-635, JEOL, Tokyo, Japan) and phase analysis was carried out by X-ray diffraction (XRD, D/MAX-250, Rigaku, Tokyo, Japan). Pore size, pore distribution and the total porosity (PoreMasterTM, Quantachrome Instruments, FL, USA) were also reported.

Methods: As starting materials, commercial HAp (Sulzer Metco Europe GmbH, Germany), methylcellulose (3000-5500 CPS, Yakuri Pure Chemicals Co. Ltd, Kyoto, Japan), ethanol (Samchun Pure Chemicals Co. Ltd, Korea) and water were used in this experiment. First of all 17 gm of HAp powder and 8.5 gm (120 vol% of HAp) of methylcellulose powder were mixed in a liquid media of ethanol (350 ml) and distill water(50 ml). The mixer was then ball-milled for nearly 4 h using alumina ball to produce a viscous slurry. The as prepared slurry was then simultaneously stirred and heated to 70-80°C nearly for 2 h. While ethanol and water was evaporating out from the slurry at this temperature, unidirectional porous HAp started to form at the bottom of the beaker. After complete evaporation of ethanol and water, the porous body was taken out of the beaker and dried in the oven at 90°C for 6 h to remove the trace ethanol and water. Then it was calcined at 600°C for 20 min in presence of air to remove methylcellulose. After that, sintering was carried out at 1200°C for 3 hrs in presence of air. Compressive strength was measured by a Universal Testing Machine (UnitechTM, R&B, Daejeon, Korea) with a cross head speed 0.5 mm/min.

Results/Discussion: Figure 1 shows the SEM images of the unidirectional porous body. The unidirectional pores are through pore and are homogeneously distributed throughout the body as can be seen in Fig. 1(a). The pores here represent the bubble formation sites i.e. the places from where the bubble came out continuously and left void spaces at the end of the operation. Longitudinal image in Fig. 1(b) confirmed that the pores were unidirectional. The fracture surface image in Fig. 1(c) shows the pore and frame region. The frame part was highly porous. This is due to the large size of HAp granules which caused incomplete sintering thus resulting porosity in the frame region. The average pore-frame thickness was about 50 µm. The pore and frame region shows also a very rough surface which is beneficial for biodegradability, cell proliferation and mechanical interlocking of cell.

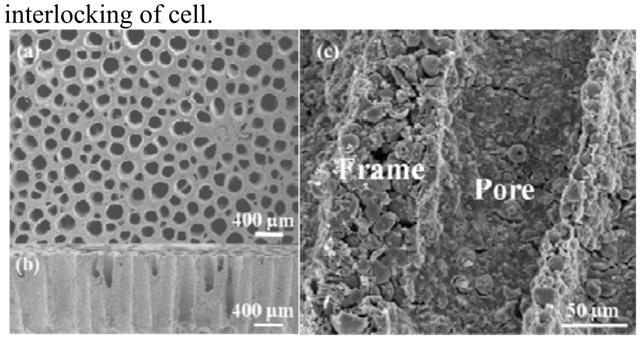

Fig.2. SEM images of the sintered unidirectional porous body: (a) cross-sectional, (b) longitudinal and (c) fracture surface.

XRD results confirmed that there were no phase change and processing residue after sintering the green body at 1200°CThe unidirectional pores were in the range of 10-244 µm which had a total pore volume of 51%. It was reported that a pore size of 100-400 µm is required for osteoconduction.[1] The total porosity of the sample was 70% and the largest pore had a diameter of 244 µm. The compressive strength obtained for the porous body was 10 MPa while the required strength for human cancellous bone is 2-12MPa[2].

Conclusion: A novel and easy method have been developed for the fabrication of unidirectional porous HAp body using ethanol bubble as a pore former. The compressive strength of the unidirectional porous body was 10 MPa which is comparable to that of cancellous bone. 50% of the total pore volume was occupied by the pores that ranges from 10 to 244 µm in diameter. The total porosity of the porous body was 70%. XRD profiles showed that there were no phase occured after high-temperature sintering. Pore sizes directly depend on the size of the ethanol bubbles which can be altered by changing the viscosity of the slurry.

References: (1. Liu. DM, Ceramics Int. 1997;23[2]135-9, 2. Hench LL. J. Am. Ceram. Soc. 1991;74[7]:1487-1510)