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Fabrication of Porous Structure of BCP Sintered Bodies Using Microwave Assisted Synthesized HAp Nano Powder

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Abstract

Using microwave synthesized HAp nano powder and polymethyl methacrylate (PMMA) as a pore-forming agent, the porous biphasic calcium phosphate (BCP) ceramics were fabricated depending on the sintering temperature. The synthesized HAp powders was about 70-90 nm in diameter. In the porous sintered bodies, the pores having 150-180 μm were homogeneously dispersed in the BCP matrix. Some amounts of pores interconnected due the necking of PMMA powders which will increase the osteoconductivity and ingrowth of bone-tissues while using as a bone substrate. As the sintering temperature increased, the relative density increased and showed the maximum value of 79.6%. From the SBF experiment, the maximum resorption of Ca^{2+} ion was observed in the sample sintered at 1000ºC.

Keywords : BCP, Porous, Microwave, PMMA, SBF

1. Introduction

Hydroxyapatite (HAp, $Ca_{10}(PO_4)_6(OH)_2$), tricalcium phosphate (α-/β-TCP, $Ca_3(PO_4)_2$) and biphasic calcium phosphate (BCP) ceramics are widely used as biomaterials for their similar mineral composition with the natural bone and teeth [1]. However, among them, BCP ceramics made with a mixture of HAp and TCP have some advantages to combine the physiochemical properties such as the dissolution of TCP allow to obtain locally supersaturated calcium and phosphate ion to increase degradability as well as the easy nucleation of biological apatites [2]. In general, the common methods have some disadvantages such as low quality control, time-consuming, contamination and micron or submicron powders with needle shape. However, the microwave irradiation method was found to synthesize the high purity nano powders using fast and high energy transformation [3]. Furthermore, to obtain an efficient bone substitute, the biomaterials should be porous structure. It has been reported that the optimum pore size is in a range of 150-250 μm in diameter for the bone ingrowth into the materials, the new cells grow within the biomaterial and progressively degrade them to be replaced by natural bone within several weeks [4].

In this work, using $Ca(OH)_2$ and H_3PO_4 as starting materials, HAp nano powder with spherical shape was synthesized by the microwave assisted synthesis process and its morphology was investigated. Moreover, the porous BCP ceramics was fabricated using PMMA as a pore-forming agent depending on the sintering temperature and SBF experiment was also performed to investigate their biodegrability.

2. Experimental and Results

As starting materials, $Ca(OH)_2$ and H_3PO_4 were used for the synthesis of HAp nano powder. The starting powders were weighed to get the stoichiometric HAp with a molar ratio of Ca/P=1.67. The powders were mixed homogeneously by stirring in de-ionized distilled water for 2hrs. The pH of the starting solution was adjusted at 12 using a pH meter by the addition of ammonia solution. The suspension at pH 12 was kept in a microwave oven which was operated with 2.45 GHz frequency microwave radiation while the operating power was 700W for 25 minutes at 300ºC. After cooling to room temperature, the precipitate was washed with de-ionized water for several times and dried in an oven at 90ºC. Finally, the calcination was carried out at 750ºC in air.

To make the porous BCP ceramics, the HAp and 50 vol% PMMA powders was dry ball milled for 24 hrs by ball mill using Al_2O_3 ball media. The pore-forming agent was burned out at 700ºC in air and then sintering was carried out at different temperatures for 2 hrs in air. SBF experiment was performed using the solution refered by T. Kokubo et al.

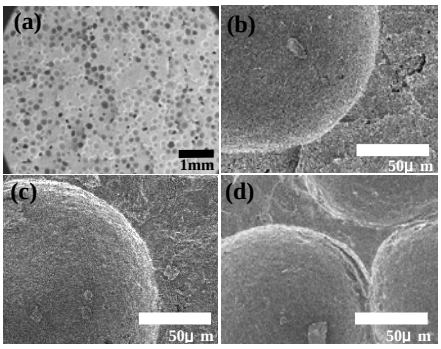

Fig. 1. SEM images of porous BCP bodies; (a) 1000ºC, (b) 1200ºC and (c) 1400ºC

Fig. 1 shows the SEM images of porous BCP bodies depending on the sintering temperature. After burning-out and sintering process, the pore-forming agent was perfectly removed to make porous BCP ceramics. In the low magnification SEM image (a), spherical shape pores having 150-180 μm in diameter were homogeneously dispersed in the BCP matrix. The diameter of the pores was slightly decreased from the PMMA powder as shown in Fig. 1(c), due to the shrinkage of compact bodies during sintering process. However, a few amounts of pores less than 5 μm process were also observed in the enlarged image of (a). At 1400ºC, the BCP body showed high density compared to that of 1000ºC (a, b) because of the high sintering temperature. However, some pores were interconnected due to the necking of PMMA powders, which will increase the osteoconductivity and enhance the ingrowth of bone tissue.

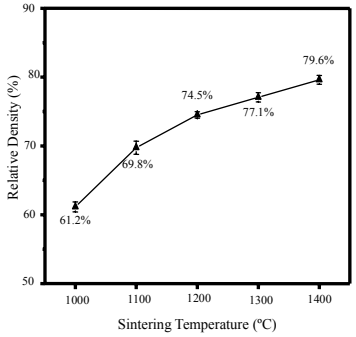

Fig. 2. Dependency of sintering temperature on the relative density of porous BCP bodies.

Fig. 2 shows the dependency of sintering temperature on the relative density of porous BCP bodies. At 1000ºC, the value of relative density was 61.2%, but as increasing the sintering temperature this value was increased. At 1400ºC, the sintered body showed maximum density with about 79.6% due to high densification as shown in Fig. 1(c).

To investigate the stability of porous BCP sintered bodies depending on the sintering temperature, SBF experiment was obtained as shown in Fig. 3. At 1000ºC, Ca^{2+} ion concentration showed maximum value for 12 days, even

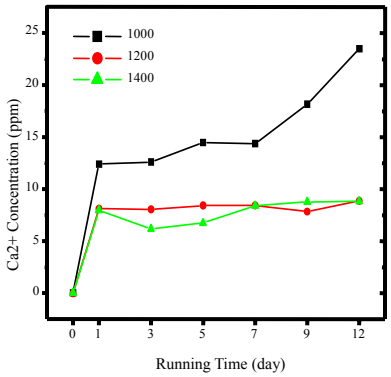

Fig. 3. SBF profile of BCP bodies.

though its composition was mostly HAp phase compared with those of 1200ºC and 1400ºC. In general, it has been reported that HAp phase showed more stability in SBF solution [5]. However, at 1000ºC, the BCP body showed high porosity due to its low sintering temperature and removal of pore-forming agent. But, at higher sintering temperature, Ca^{2+} ion concentration showed low value due to the densification.

3. Summary

The spherical HAp nano powder in a range of 70-90 nm in diameter was synthesized by the microwave assisted process with high energy transformation. Moreover, using PMMA as a pore-forming agent, the porous BCP ceramics with well dispersed pores as a pore-forming agent were fabricated depending on the sintering temperature. The diameter of the pores in the sintered bodies was found with about 150-180 μm with some amounts of interconnected pores which could increase the osteoconductivity while using as bone substitute. At 1000ºC, the sintered porous BCP ceramics showed high Ca^{2+} concentration due the high porosity. However, at 1400ºC, the porous BCP showed low Ca^{2+} concentration in SBF due to the dense matrix structure.

4. Acknowledgement

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5. References

1. K. D. Groot, Biomaterials, **1**, 47 (1980).
2. G. Daculsi, J. M. Bouler and R. Z. LeGeros, Int. Rev. Cytol., **172**,129 (1997).
3. J.K. Han, H.Y. Song, F. Saito and B.T. Lee, Mater. Chem. Phy. (2005), in press.
4. O. Gauthier, J. M. Bouler, E. Aguado, P. Pilet and G. Daculsi: Biomaterials, **19**, 133 (1998).
5. T. Kokubo and H. Takadama, Biomaterials, **27**, 2907 (2006).