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Effect of Heat-Treatment Temperature on the Osteoconductivity of the Apatite Derived from Bovine Bone

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Abstract. Effect of heat-treatment temperature on the osteoconductivity of the apatite derived from bovine trabecular bone was investigated. Three different heat-treatment temperatures (600, 800 and 1000 °C) were adopted in the experiment and their effects on the physical properties of apatite granules, which could affect on the osteoconductivity, were evaluated. The content of carbonate ions in the apatite structure was assessed by FT-IR and its crystallinity was evaluated by X-ray diffractometry. The microstructure was assessed by field emission electron microscopy. Apatite granules heat-treated at 600 °C and 1000 °C were implanted into the calvaria of New Zealand White rabbit for 4 weeks, respectively, and the undecalcified ground histologic specimens stained with multiple staining method was observed. As increasing the heat-treatment temperature, the crystal size and crystallinity of the apatite increased while the content of carbonate ions decreased. The apatite granules heat-treated at 600 °C showed much better osteoconductivity comparing to that heat-treated at 1000 °C. The results were explained in terms of the physical properties of apatite which could affect to the osteoconductivity.

Introduction

Apatite is the most prevalently used material for the application as bone grafting materials because it exists in natural bone and is known to have good osteoconductivity. Thus, many researches have already been made to synthesize it artificially [1]. The carbonate ions in the structure of the apatite and its low crystallinity are known as the critical factors affecting to the bone-bonding ability and biodegradability *in vivo* [2]. However, it can hardly achieve those properties via synthetic processing method because the crystallinity increases and the carbonate ions escape from the sample by CO_2 gas during the sintering process at high temperature. Thus, several approaches have been made to solve this problem but it has not been completely solved, yet.

The apatite derived from bovine bone is prevalently being used as a bone filling material in dental field such as periodontal and maxillofacial surgery [3]. Its advantages comparing to the synthetic one are it can easily achieve low crystallinity, high content of carbonate ions, and porous structure because it does not need a sintering process at high temperature. However, there have been no reports on the relationship between heat-treatment temperature and osteoconductivity, yet. In the present investigation, the effect of heat-treatment temperature on the osteoconductivity of the apatite granules which derived from bovine trabecular bone was examined with the main focus on the content of carbonate ions, crystallinity, and crystal size of apatite.

Materials and Methods

Bovine trabecular bone was supplied from NIBEC Inc. (Korea) who produces the OCS-B®, the xenograft bone filler. It was defatted, deproteinized and then heat-treated at temperature 600 °C, 800 °C, and 1000 °C for 3 hours, respectively.

The content of carbonate ions in the apatite structure was assessed by Fourier transformed infrared spectroscopy (FT-IR; Nexus, Thermo-Nicolet, U.S.A.) and its crystallinity was evaluated by X-ray diffractometry (XRD; D8 Advance, Bruker, Germany). The microstructure was assessed by field emission electron microscopy (FE-SEM; S-4700, Hitachi, Japan).

The heat-treated apatite granules which had the size range from 212 to 425 μm were used for the osteoconductivity testing. Apatite granules heat-treated at 600 °C and 1000 °C were implanted into the calvaria of New Zealand White rabbits, respectively. Two full-thickness holes, each 10 mm in diameter, were trephined in the vault of the skull on either side of the sagittal suture of the calvarium. Two groups of 5 animals were sacrificed at 4 weeks after implantation. Undecalcified ground histologic specimens were prepared in the routine manner at 40 μm thickness and stained with multiple staining.

Results and Discussion

Fig. 1 shows the FT-IR results after the heat-treatment at 600, 800, and 1000 °C for 3 hours, respectively. After the heat-treatment at 600 °C, the bending (ν_4) and symmetric stretching (ν_1) modes of PO_4 ions were detected at around 604 and 964 cm^{-1}, respectively. The asymmetric mode of PO_4 ion (ν_3) was detected at around 1119 cm^{-1}. The bands at 1627 and 3415 cm^{-1} were assigned to the stretching and bending modes of OH bond, respectively, coming from the adsorbed water. Furthermore, an out-of-plane (ν_2) and two stretching (ν_3) modes of CO_3 ion were also observed at around 874, 1418 and 1471 cm^{-1}, respectively. It means that the PO_4 sites of the apatite structure (B-site) were partly substituted by carbonate ions [4]. The noted decrease of peak height of carbonate ions after the heat-treatment at 800 °C was observed and they were not observed at all after the heat-treatment at 1000 °C for 3 hours. The liberation and stretching modes of OH ion were newly detected at around 630 and 3572 cm^{-1} after the heat-treatment at above 800 °C. It means that the carbonate apatite was converted to hydroxyapatite after the heat-treatment at above 800 °C for 3 hours.

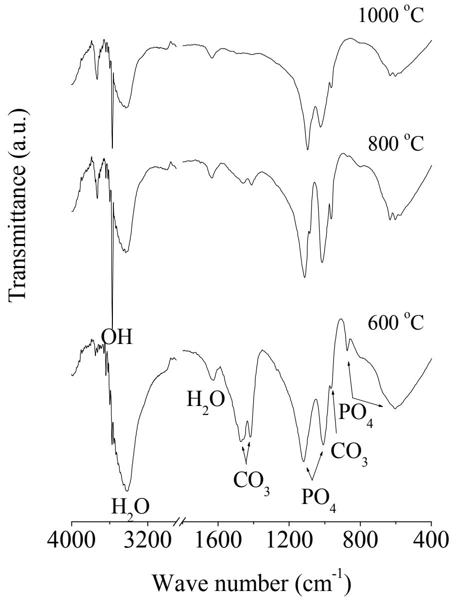

Fig. 1. FT-IR patterns of the apatite granules heat-treated at 600, 800, 1000 °C for 3 hours.

Fig. 2 shows the XRD patterns of the apatite granules after the heat-treatment at 600, 800, and 1000 °C for 3 hours, respectively. The results showed that they were apatite single phase. The crystallinity of apatite granules converted from low to high after the heat-treatment at above 800 °C.

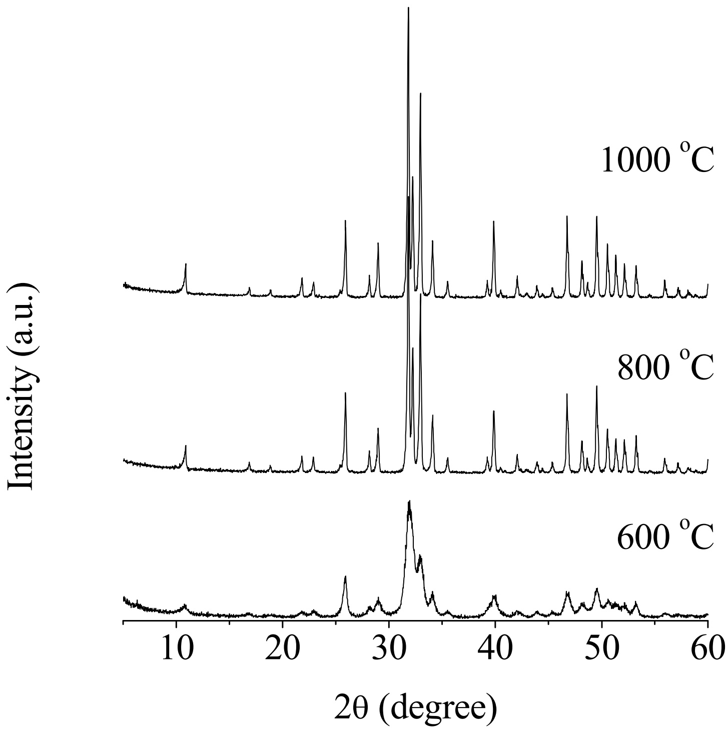

Fig. 2. XRD patterns of the apatite granules after the heat-treatment at 600, 800, and 1000 °C for 3 hours, respectively.

Fig. 3 shows the FE-SEM photographs of the apatite granules after the heat-treatment at 600 °C and 1000 °C for 3 hours, respectively. The grain size of apatite found in the specimen heat-treated at 600 °C was about 50 nm while those found in the specimen heat-treated at 1000 °C was about 1 μm, respectively. The grain size of the apatite granules heat-treated at 1000 °C was about 20 times bigger than those heat-treated at 600 °C. From the results, it can be known that the apatite granules heat-treated at 600 °C almost keep the original physical properties of the apatite found in natural bone.

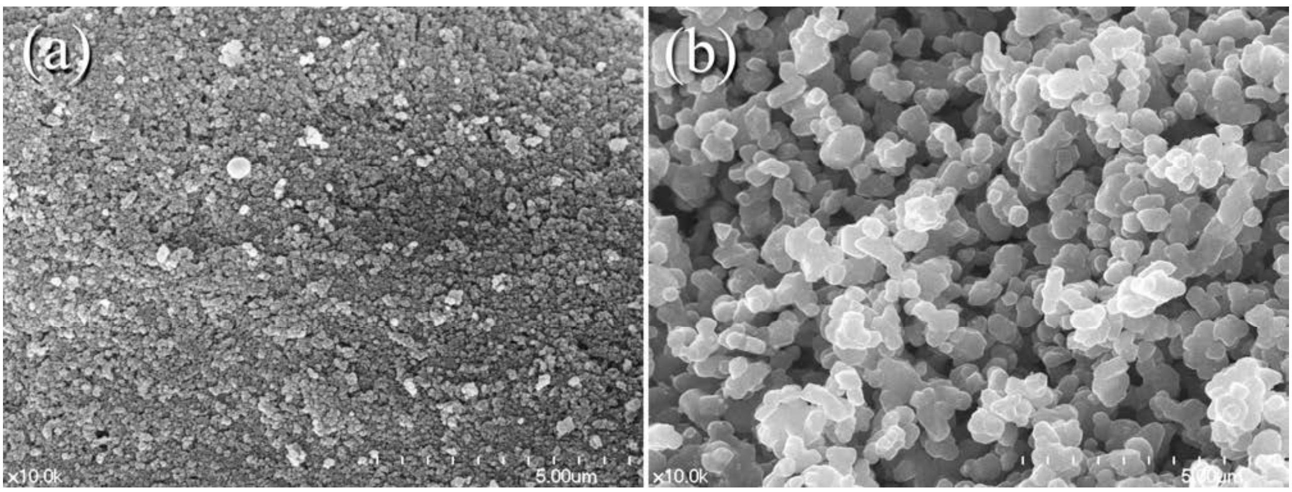

Fig. 3. FE-SEM photographs of the apatite granules heat-treated at (a) 600 °C and (b) 1000 °C for 3 hours.

Fig. 4 shows the histologic view of the white rabbit calvarial defect filled with the apatite granules heat-treated at (a) 600 °C and (b) 1000 °C, respectively, after 4 weeks implantation. The formation of new bones on the apatite granules without intervening fibrous tissue was observed in the sample heat-treated at 600 °C while fibrous tissues which partly covered the apatite granule surfaces were observed in the sample heat-treated at 1000 °C.

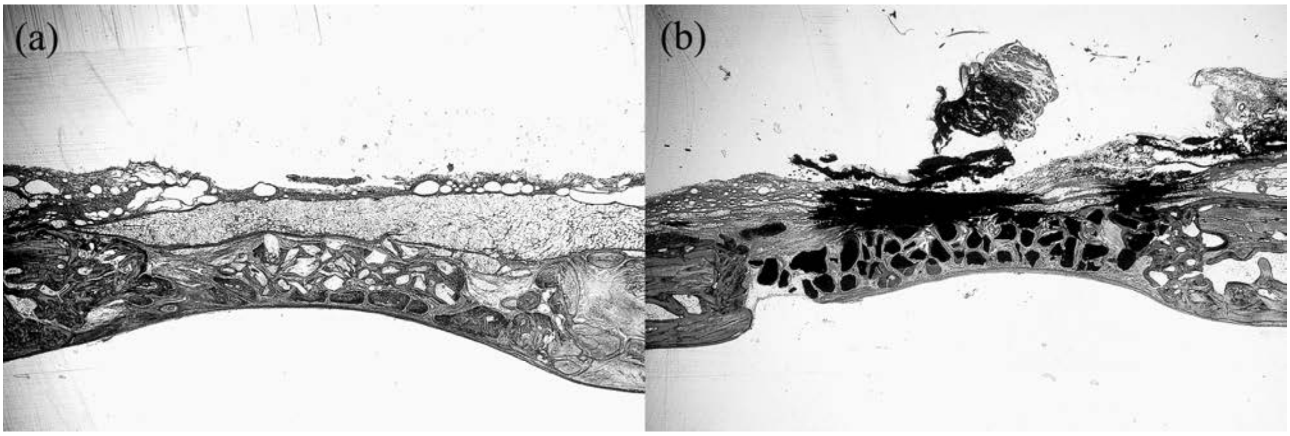

Fig. 4. Histologic view of the white rabbit calvarial defect filled with the apatite granules heat-treated at (a) 600 °C and (b) 1000 °C for 3 hours (Multiple staining, original magnification, ×10).

From the results, it can be concluded that the apatite granules heat-treated at 600 °C had much better osteoconductivity than those heat-treated at 1000 °C. The different osteoconductivities between two apatite granules were likely to originate from the different physical properties such as the content of carbonate ions, crystallinity, and the size of apatite crystals. Therefore, one of the factors to increase the osteoconductivity of the apatite granules must mimic the natural bone itself.

Conclusion

The effect of heat-treatment temperature on the osteoconductivity of apatite granules derived from bovine bone was investigated. It was defatted, deprotenized, and then heat-treated at 600 °C, 800 °C, and 1000 °C, respectively. As increasing the heat-treatment temperature, the crystal size and crystallinity of the apatite increased while the content of carbonate ions decreased. The apatite granules heat-treated at 600 °C showed better osteoconductivity than those heat-treated at 1000 °C. The similar physical properties of apatite granules comparing to those of natural bone was likely the main reason for good osteoconductivity.

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