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Fabrication of octacalcium phosphate foam through phase conversion and its histological evaluation

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ABSTRACT

Octacalcium phosphate (OCP) foam with an interconnected porous structure was fabricated through phase conversion *via* a dissolution–precipitation reaction using calcium sulfate hemihydrate (CSH: $CaSO_4 \cdot 1/2H_2O$) granules as precursors in a sodium dihydrogen phosphate (NaDP: NaH_2PO_4) solution. The diametral tensile strength and porosity of the OCP foam were 0.15 ± 0.04 MPa and 69.4% ± 0.04%, respectively. When the OCP foam was implanted into bone defects in a rabbit femur, the OCP foam showed an excellent tissue response, and the bone penetrated into the porous structure. The osteoconductivity and bone-replacement rate were significantly higher than those of an OCP compact.

1. Introduction

Attaching an interconnected porous structure (i.e., a foam) to a scaffold is an effective way to enhance tissue response because the interconnected pores allow cells and tissues to penetrate into the interior of the scaffold, thereby facilitating the supply of vital nutrients by vascular ingrowth [1–3]. Furthermore, foams accelerate the replacement of the scaffold to bone because this process occurs only on the surface of the scaffold.

Octacalcium phosphate (OCP: $Ca_8(HPO_4)_2(PO_4)_4 \cdot 5H_2O$) is a metastable phase of calcium phosphate that can be replaced by a new bone through bone remodeling [4–9]. For this reason, OCP has attracted attention as a bone substitute. However, in previous studies, only sub-mm or micron-sized OCP granules, which are too small to fully reconstruct bone defects were obtained [10,11].

We previously demonstrated that the fabrication method of compact OCP shows excellent tissue response, in particular, for osteoconductivity and bone replacement [12]. The osteoconductivity and bone-replacement rate of the OCP scaffold are expected to be increased by imparting the scaffold with a foam structure. Therefore, in this study, feasibility to fabricate OCP foam scaffold was evaluated along with initial histological evaluation using experimental animals to determine the tissue response to the OCP foam.

2. Materials and methods

2.1. Preparation of OCP foam from precursor granules

All reagents were purchased from Wako Pure Inc., Japan. $CaSO_4 \cdot 1/2H_2O$ (CSH; 9 g) and NaH_2PO_4 (NaDP; 6 g) were mixed with 1.8 mL H_2O by using a motor and pestle and then dried. The CSH–NaDP mixture was crushed and sieved to 200–300 μm.

Approximately 0.1 g of the sieved CSH–NaDP granules was placed into a separated-type mold ranging φ6 mm and 3 mm. Subsequently, 0.1 mL of 70% ethanol saturated CSH was dropped onto the sieved CSH–NaDP granules to fabricate CSH–NaDP foam through partial surface melting. After dropping, the CSH–NaDP granules were left at room temperature to dry.

Nine set CSH–NaDP foams were immersed into 15 mL of 1 mol/L Na_2HPO_4 solution at 80 °C for 24 h. The treated CSH–NaDP foams were washed by distilled water and 99.5% ethanol and then dried.

2.2. Characterization

The treated and set CSH–NaDP granules were characterized by X-ray diffraction (XRD; D08 ADVANCE, Bruker AXS Co., Japan; 40 kV and 40 mA). The inner structures of the specimens were determined by micro-computed tomography (microCT; SkyScan1085, Toyo Technica Co., Japan; 60 kV and 160 μA) and scanning electron microscopy (SEM; S-3400 N, Hitachi Hightech Co., Japan; 5 kV).

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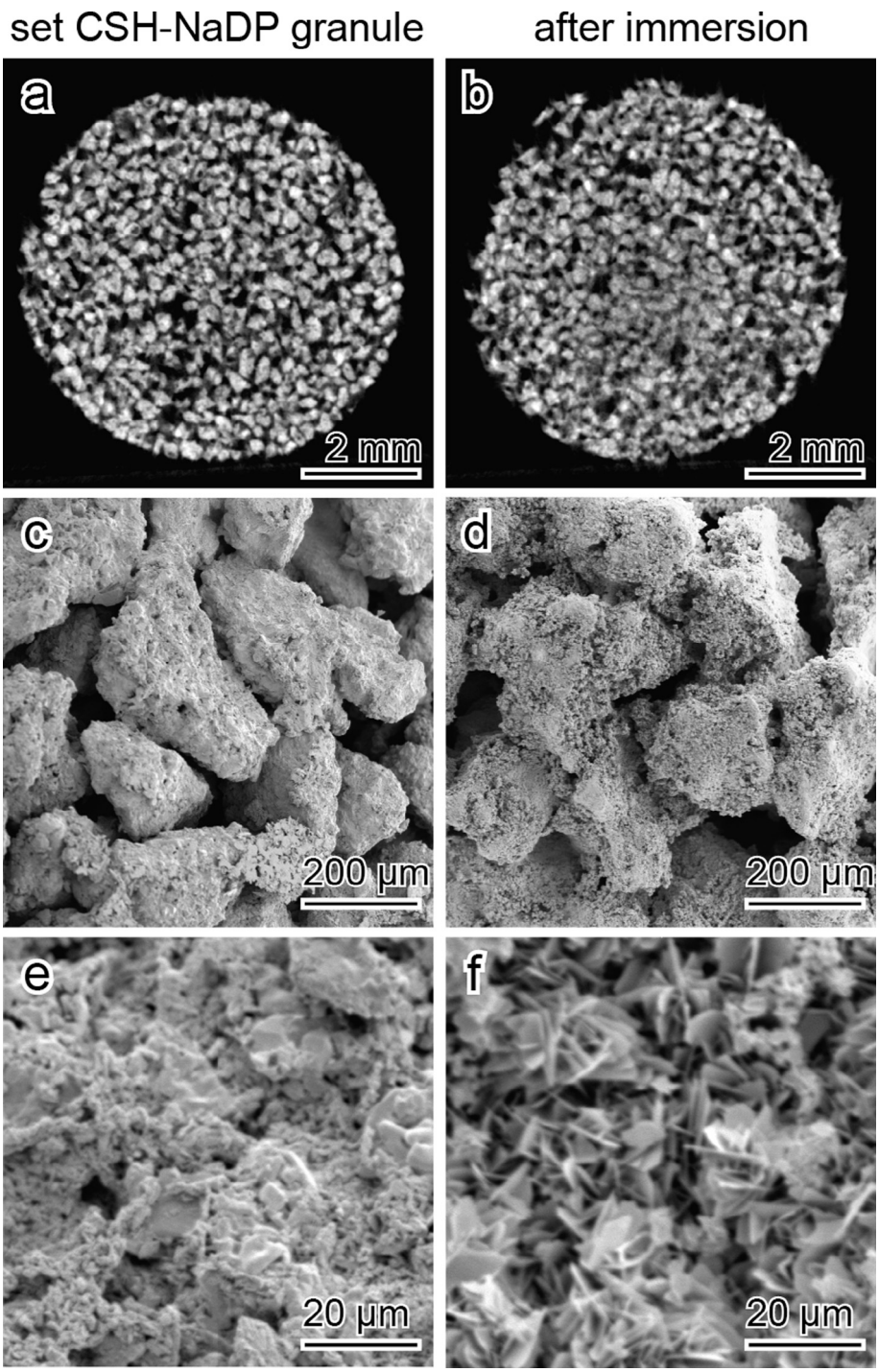

Fig. 1. The macroscopic structure of the set CSH–NaDP granules (a) after immersion (b) shown by microCT. SEM micrographs and the high magnified images of the set CSH–NaDP granules (c, e) after immersion (d, f).

Diametral tensile strength (DTS) was measured using an autograph (AGS-J, Shimadu Co., Japan) with a cross-head speed of 1 mm/min.

2.3. Histological evaluation of OCP foam

All animal experiments were conducted with the approval of the ethical committee of animal experimentation of Kyushu University (approval number: A27-270-0). Four male Japanese white rabbits (18 weeks old, each weighing between 3.0 and 3.5 kg) were used in this study. Under systemic anesthesia, the femurs of both legs were carefully exposed by exfoliation. After dissecting the periosteum, artificial bone defects of $\varphi 6.25 \times 3$ mm were prepared in the cancellous bones of both femurs by drilling with a trephine-bur ($\varphi 6.25$ mm) attached to a dental hand piece. The bone defect was reconstructed up to the level of the previous bone surface with OCP foam or OCP compact (as a reference) that had been sterilized at 80 °C for 8 h.

The skin flap was then closed with suturing. After the surgery, buprenorphine hydrochloride (LepetanVR, Otsuka Pharmaceutical Co., Japan) was injected as an analgesic, and gentamicin sulfate