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Development of Mg-containing porous β-tricalcium phosphate scaffolds for bone repair

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Abstract

Apatitic tricalcium phosphate (ap-TCP) powders containing Mg were synthesized through wet chemical precipitation method from CaO and H_3PO_4, using MgO as the source of Mg substitution. The influence of small, close to bone-like amounts of Mg on the thermal stability and specific surface area (SSA) of as-synthesized ap-TCP powders was evaluated. The increase in Mg content up to 0.674 ± 0.080 wt% promoted enlarging of SSA of the as-synthesized powders between 91.3 m^2/g and 104.2 m^2/g and the stabilization of the β-tricalcium phosphate (β-TCP) phase up to 1360 °C. The effect of Mg substitution on porosity and microstructure was investigated. Interconnected porous Mg-containing β-TCP scaffolds, with micro-, macroporous structures were developed by an in situ foaming of viscous mass of proper slurry prepared of the as-synthesized ap-TCP powders, using NH_4HCO_3 as a foaming agent, and subsequent sintering at 1150 °C for 2 h. The self-dissolution behavior in vitro was evaluated through soaking of scaffolds in SBF. The release of Ca and Mg ions from the 3D micro-, macroporous β-TCP scaffolds containing various amounts of Mg was determined by a complexometric (EDTA) titration. The samples with increased Mg concentrations showed significantly enhanced Mg-release rate in comparison to the samples with lower Mg substitution level.

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1. Introduction

Calcium phosphates (CaP) constitute inorganic mineral phase in mammalian bones and teeth [1–3]. Therefore it is well recognized by body and biocompatible according to all current standards. CaP ceramics and related compounds represent the most important class of biomaterials for bone regeneration [1–6]. β-Tricalcium phosphate (β-TCP, $Ca_3(PO_4)_2$) is one of calcium phosphate-based bioceramic used as bone replacement material due its close chemical similarity to biological apatite. β-TCP bioceramics as bioresorbable materials have been considered as ideal temporary scaffolds in bone tissue engineering, which would facilitate the initial formation of new bone tissue and finally be replaced by the new bone [2]. Nowadays interest is turning towards modified synthetic CaP involving substitution of ions found in natural bones [3,4]. Incorporation of such ions into the CaP structure is important for a number of reasons; including better understanding of biomineralization processes, increase in bioactivity of the materials, and ion delivery able to act on bone diseases [4]. The substitutions have influence on physical, chemical, and physiological properties of the solid and consequently on mineralization, demineralization, and remineralization process of calcified tissues [5,6]. The effect of bioactive ions (Ca, Mg, Sr, Si etc.) release induces osteogenesis and angiogenesis, i.e. two functions required for effective bone tissue regeneration.

The chemistry of Mg is unique among the cations of biological relevance. Mg is essential for man and is required in relatively large amounts. Mg is needed for bone formation

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and can also be referred to as a natural ‘calcium antagonist’ [7,8]. The importance of Mg in bone is well-known, preventing possible risk factors for osteoporosis in humans [7–10].

Design and development of porous ceramics have attracted much attention in the last years. Successful bone defect repair requires the use of porous scaffolds, which should have the features such as high porosity along with macropores and 3D interconnected pore structures, in order to have permeability and diffusion properties for both cell penetration and proper vascularization of the ingrowths tissue [11–13]. Bone tissues cannot grow into the interior of scaffold when the pore size is less than 100 μm [12–14]. As far, diverse methods have been developed to prepare porous β-TCP materials and a sponge replication [14–16], and a direct foaming of ceramic suspensions [17] were the most common methods. Moreover, Wang et al. reported the fabrication of mesoporous Mg-substituted β-TCP nanospheres by the self-transformation and assembly of metastable precursor nanoparticles under the influence of ethylenediaminetetraacetic acid (EDTA, $C_{10}H_{16}N_2O_8$) [18] and Vecchio et al. described the preparation of porous Mg-substituted β-TCP by hydrothermal conversion of sea urchin spines [19].

The goal of our work was to evaluate the influence of small, close to bone-like amounts of Mg incorporated into β-TCP crystal structure on physicochemical properties and in vitro degradation in simulated body fluid (SBF) of the porous ceramics formed by the ammonium hydrogen carbonate provided viscous slurry foaming.

2. Materials and methods

2.1. Preparation and characterization of Mg-containing ap-TCP powders

The powders of Mg-containing apatitic tricalcium phosphate (ap-TCP) precursors with varying Mg content were prepared through the wet chemical precipitation of calcium oxide (CaO, puriss., *Fluka*), magnesia (MgO, reagent-grade, *ES/Scharlau*) and aqueous solution of orthophosphoric acid (H_3PO_4, puriss., 75%, *Sigma-Aldrich*) according to the scheme shown in Fig. 1. The precipitation reactions were carried out in the laboratory reactor (*Power Control-Visc P7*, IKA EUROSTAR). Dried ap-TCP was milled and used for thermal stability analyses. Furthermore, as-synthesized CaP powders were selectively sieved to obtain powders with particle size less than 100 μm.

Sieved powders were used for analyses of specific surface area (SSA) and porous sample preparation.

Thermal stability from ambient temperature to 1400 °C of Mg-containing ap-TCP was investigated by the differential thermal analyzer (DTA, *DTA703*, BÄHR) at heating rate 10 °C/min. Brunauer–Emmett–Teller (BET) method was used to determine the SSA of as-synthesized, sieved Mg-containing ap-TCP powders by N_2 absorption (*KELVIN* 1042, COSTECH). The samples were degassed at room temperature for 24 h prior the analyses. The values of mean sizes of crystallites of as-synthesized powders were estimated from the N_2 adsorption isotherms using the BET particle diameter (d_{BET}) from the following equation by assuming the primary particles to be spherical:

$$d_{BET} = 6/(q \cdot Sw), \tag{1}$$

where q is theoretical density of β-TCP – 3.07 g/cm^3 (according to ISO 13175-3:2012 Implants for surgery – Calcium phosphates – Part 3: Hydroxyapatite and beta-tricalcium phosphate bone substitutes) and Sw is the SSA.

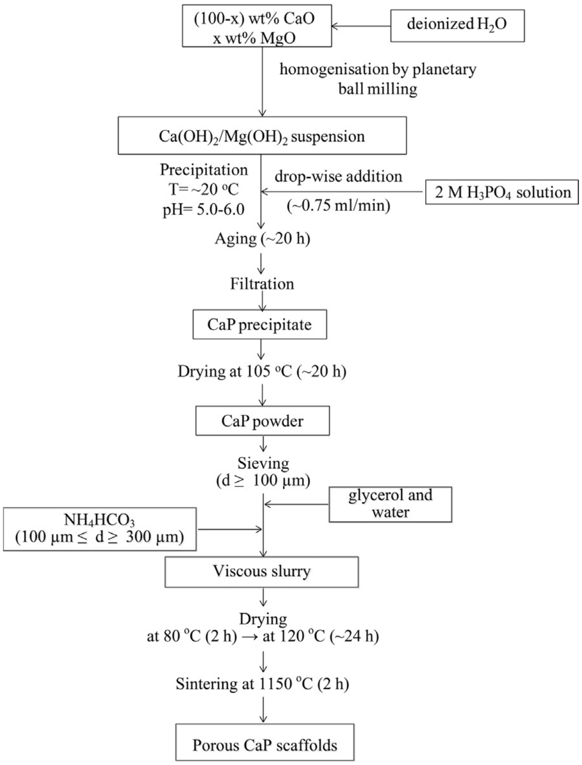

Fig. 1. Schematic representation of the syntheses of ap-TCP powders and preparation of porous β-TCP scaffolds containing various amounts of Mg.

2.2. Preparation and characterization of porous Mg-containing β-TCP scaffolds

In order to prepare porous scaffolds the in situ viscous mass foaming method was used as described elsewhere [20] (Fig. 1). Sieved powders were hand mixed in a pestle with glycerol ($C_3H_5(OH)_3$; purity > 99.8%, molecular weight 92.09 g/mol, *Ltd. BIO-VENTA*) and distilled water in the ratio of weight 1.0: 0.9: 0.1 to obtain highly viscous slurries. At last, ammonium hydrogen carbonate (NH_4HCO_3; purity 99–101%, *Sigma-Aldrich*) with particle size in the range of 100–300 μm was added to the slurries as a foaming agent. For all sample series the amounts of ap-TCP powder, glycerol, NH_4HCO_3 and water were kept constant. Prepared viscous slurries were poured in *Teflon* cylindrical molds with the inner diameter of 10 mm and height of 20 mm. Filled molds were placed in the drying oven to heat up the slurries and initiate foaming (at 80 °C for 2 h) and drying (at 120 °C for ~24 h) processes. Dried samples were removed from the molds and sintered in an air atmosphere at 1150 °C for 2 h to obtain porous Mg-containing β-TCP scaffolds.

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