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Hierarchically porous hydroxyapatite ceramics prepared with wheat flour and their microstructural characterization via mercury porosimetry, image analysis and tomography

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Hydroxyapatite (HA) is the main inorganic component of human bone and therefore HA ceramics are widely used as artificial replacements for natural biomaterials, both for implant applications and bone tissue engineering scaffolds [1-3]. In order to ensure not only biocompatibility (bioactivity), but also osteoconductive and osteoinductive properties enabling bone cell ingrowth, as well as sufficient delivery of nutrients, drugs and / or growth factors, the microstructure of these artificial biomaterials has to be hierarchically porous, i.e. similar to the microstructures of natural cortical or trabecular bone. In the present work hierarchically porous HA ceramics, i.e. ceramics with a wide range of pore sizes, has been prepared by a new ceramic shaping and foaming technique using starch and wheat flour [4,5]. Aqueous HA suspensions have been prepared with commercially available HA powder (tribasic calcium phosphate with 30–40% Ca, Alfa Aesar, Germany) in concentration 36 wt.% with 0.7 wt.% dispersant (Dolapix CE64, Zschimmer & Schwarz, Germany), 20 vol.% wheat flour and 5 vol.% corn starch. The wheat flour acts as a foaming agent (foaming being achieved by ball milling in polyethylene bottles with alumina balls for 6 h), while the corn starch acts as a foam stabilizer (stabilization being achieved via starch gelatinization during heating at 80 ℃ for 6 h), improves dimensional accuracy and increases the strength of the materials before firing. After drying (105 ℃) the samples were fired to 850 and 1200 ℃ (heating rate 2℃/min, dwell time 2 h) and characterized by X-ray diffraction, where HA has been confirmed as the only phase in both cases. Mercury porosimetry confirmed open porosities of 83 and 66 % as well as trimodal and bimodal pore size distributions after firing at 850 and 1200 ℃, respectively (with modes at around 100 μm and 1 μm in both cases and an additional pore size mode below 100 nm for the material sintered at 850 ℃). Based on scanning electron microscopic images of polished sections of samples immersed in and infiltrated with transparent epoxy resin (Araldite 2020, Ciba, Germany), the complete set of global microstructural descriptors has been determined via image analysis (porosity, surface density / mean chord length and mean curvature integral density / generalized Jeffries size) [6,7]. Additionally, based on spatial images obtained via X-ray microtomography, the total curvature integral density / 3D Euler characteristic has been determined. Finally, size distributions of the large pores (i.e. the foam bubbles representing approximately 50 ± 2 % porosity in both cases) have been determined, corrected for the random section problem [8], transformed to volume-weighted distributions and compared to the pore size distribution determined directly from tomography (using the Hildebrand-Rüegsegger thickness measure) and the pore throat size distributions determined via mercury porosimetry.

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