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Fatigue performance of a high-strength, degradable calcium phosphate bone cement

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ABSTRACT

Calcium phosphate cements (CPCs) are clinically used as injectable materials to fill bone voids and to improve hardware fixation in fracture surgery. *In vivo* they are dynamically loaded; nonetheless little is known about their fatigue properties. The aim of this study was to, for the first time, investigate the fatigue performance of a high-strength, degradable (brushitic) CPC, and also evaluate the effect of cement porosity (by varying the liquid to powder ratio, L/P) and the environment (air at room temperature or in a phosphate buffered saline solution, PBS, at 37 °C) on the fatigue life. At a maximum compressive stress level of 15 MPa, the cements prepared with an L/P-ratio of 0.22 and 0.28 ml/g, corresponding to porosities of approximately 12% and 20%, had a 100% probability of survival until run-out of 5 million cycles, in air. When the maximum stress level, or the L/P-ratio, was increased, the probability of survival decreased. Testing in PBS at 37 °C led to more rapid failure of the specimens. However, the high-strength cement had a 100% probability of survival up to approximately 2.5 million cycles at a maximum compressive stress level of 10 MPa in PBS, which is substantially higher than some *in vivo* stress levels, *e.g.*, those found in the spine. At 5 MPa in PBS, all specimens survived to run-out. The results found herein are important if clinical use of the material is to increase, as characterisation of the fatigue performance of CPCs is largely lacking from the literature.

1. Introduction

Bone loss and fractures, due to, *e.g.*, osteoporosis, may call for the use of bone substituting materials. Calcium phosphate cements (CPCs) are used for this purpose as injectable materials to fill bone voids and to improve hardware fixation in fracture surgery (Larsson and Bauer, 2002; Bajammal, 2008). CPCs are self-setting and form a biomaterial that is chemically similar to the mineral content of human bone (Bohner et al., 2005; Dorozhkin, 2010), possessing biocompatible and osteoconductive properties. Some CPC compositions have shown a fast resorption rate (Apelt et al., 2004; Tamimi et al., 2012), which may be beneficial for the regrowth of new bone tissue, and some compositions have even been shown to stimulate bone tissue formation *in vivo* (Habibovic et al., 2008; Engstrand et al., 2014b, 2015).

A major drawback of CPCs is their brittleness, which limits their use in clinical applications. However, in certain cases, *e.g.*, for confined fractures and where the expected loading scenario is mainly compressive, CPCs may provide adequate support, and be the preferred choice over less biocompatible materials currently used, *e.g.*, acrylic bone cements. In order to evaluate the possible future use of CPCs in such applications, a greater understanding of the materials' fatigue properties is needed.

Mechanical characterisation of CPCs is most commonly done by quasi-static compressive loading (Tamimi et al., 2012; Zhang et al., 2014; Ajaxon and Persson, 2017), which provides a good starting point for evaluation of the material. However, the quasi-static strength alone does not provide enough information on how the material will behave in a clinical application. *In vivo*, repeated loading can be expected and hence the material's resistance to fatigue is very important in order to determine its suitability for clinical use. To aid in the prediction of the cements' behaviour *in vivo*, the fatigue properties of CPCs need to be studied. Unfortunately, the fatigue performance of CPCs alone is rarely reported in the literature: there are only a handful publications on apatite cements (Morgan et al., 1997; Jew et al., 2001; Zhao et al., 2010), one on biphasic CPC (calcium sulphate and brushite (dicalcium phosphate dihydrate)) (Harmata et al., 2015); and only one on pure acidic CPCs (brushite and monetite (dicalcium phosphate anhydrous)) (Ajaxon et al., 2017).

There is a great variation in compressive strength depending on the formulation of the CPC (Tamimi et al., 2012; Zhang et al., 2014; Ajaxon and Persson, 2017). Recent advances have led to the development of a high-strength brushite cement (Unosson and Engqvist, 2014; Engstrand

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et al., 2014a), with an average strength of 74 MPa in compression – at least twice that of human trabecular bone (Kopperdahl and Keaveny, 1998; Perilli et al., 2008), which shows that this material may be promising in certain well-defined load-bearing cases. To further evaluate this high-strength cement, the material needs to be tested under fatigue loading in order to provide more information on the material's suitability for clinical applications.

The porosity of CPCs is an important factor since it influences the degradation rate of the cements and also has a direct negative effect on the mechanical properties (Tamimi et al., 2012; Zhang et al., 2014). Several studies have investigated the influence of porosity on the compressive strength and diametral tensile strength of CPCs (Engstrand et al., 2014a; Zhang et al., 2014). However, none have reported on the influence of porosity or the largest defect (pore) size on the fatigue performance of the cements. Moreover, a wet state has previously been shown to affect the quasi-static strength of both apatite and brushite cements (Pittet and Lemaître, 2000; Gorst et al., 2006; Zhang et al., 2014; Luo et al., 2016), and therefore it can be assumed that the fatigue life of CPCs may be affected if the tests are performed in air or under physiological conditions (wet, 37 °C).

The aims of the present study were to 1) evaluate the compressive fatigue performance of a high-strength brushite cement; 2) evaluate how the fatigue performance is influenced by the porosity and the largest defect size of the cements; and 3) investigate the impact of the environment on the fatigue performance.

2. Materials and methods

2.1. Specimen preparation

All chemicals, including beta-tricalcium phosphate (ß-TCP), disodium dihydrogen pyrophosphate (SPP), citric acid, and phosphate buffered saline (PBS; containing 0.01 M phosphate buffer, 0.0027 M potassium chloride and 0.137 M sodium chloride, pH 7.4) were purchased from Sigma-Aldrich (Sigma-Aldrich, St. Louis, MO, USA), except for monocalcium phosphate monohydrate (MCPM) which was purchased from Scharlau (Scharlau, Sentmenat, Spain).

The self-setting brushite cement was prepared by mixing MCPM (sieved to obtain particle sizes < 75 μm) and ß-TCP in a ratio of 45:55 mol% together with a citric acid solution (0.5 M) for 1 min in a mechanical mixing device (Cap Vibrator Ivoclar Vivadent AG, Schaan, Liechtenstein). SPP (1 wt%), added to the powder phase, acted as a retardant of the setting reaction (Unosson, 2014). Three different liquid to powder (L/P) ratios were used: 0.22 ml/g, 0.28 ml/g and 0.35 ml/g, to achieve cements with different porosities. The cement paste was moulded in cylindrical moulds (6 mm diameter) and specimens were left to set for 24 h in PBS at 37 °C to achieve full setting (Unosson and Engqvist, 2014). The set specimens were wet polished with SiC paper to a final height of 12 mm (specimen dimensions according to the standard ASTM F 451-08 ASTM (2008)).

2.2. Porosity measurements

The total open porosity of the wet cements was evaluated by solvent exchange, which has been previously established as a valid porosity method for wet brushite cements (Ajaxon et al., 2015). Briefly, the specimens were weighed in their wet state and the apparent volumes were determined by Archimedes' principle using a density kit (Mettler Toledo, Greifensee, Switzerland). The specimens were then immersed in isopropanol (10 ml) and left at room temperature (RT) until constant weight was achieved (24 h for L/P-ratios of 0.22 and 0.28 ml/g and 48 h for an L/P-ratio of 0.35 ml/g). Finally the weights of the specimens were recorded. The total open porosity was calculated from the ratio of the weight difference before and after isopropanol immersion and the apparent volume, taking into account the differences in density of water and solvent.

In order to investigate a possible correlation between the largest defect (pore) and the number of cycles to failure, the microstructure of all specimens was studied using micro computed tomography (micro-CT; SkyScan 1172, Bruker microCT, Kontich, Belgium) before testing them under fatigue. The scanner operated at a source voltage of 100 kV and a current of 100 μA, and the specimens were placed on top of each other in a poly(methyl methacrylate) container filled with double distilled water in order to keep them wet throughout the analysis. A Cu-Al filter was used and images were acquired using an isotropic pixel size of 13.9 μm. NRecon (Bruker microCT, Kontich, Belgium) was used to reconstruct the images. Calculations of the largest pore sizes, using a volume-equivalent sphere diameter, and total closed porosity (however, limited by the scanner resolution to pores > 13.9 μm) were performed with CTAn (Bruker, microCT, Kontich, Belgium).

2.3. Quasi-static compressive strength

The quasi-static compressive strength of the cement specimens was assessed by loading them to failure at a speed of 1 mm/min in a universal testing machine (AGS-X, Shimadzu, Kyoto, Japan) equipped with fixed compression platens and a 5 kN load cell. The specimens were kept wet until testing.

2.4. Phase characterisation

After compression testing, the cement specimens were thoroughly ground and homogenized. Six powder specimens were taken at random for analysis with X-ray diffraction (XRD), using a D8 Advance (Bruker, AXS GmbH, Karlsruhe, Germany) in a theta-theta setup with Ni-filtered Cu-Kα irradiation. Diffraction angles of 10–60° (2θ) were analysed in steps of 0.02 degrees with 0.25 s per step, while rotating the sample at a speed of 80 rpm. Rietveld refinement was applied to quantify the phase composition, using Profex (http://profex.doebelin.org) (Doebelin and Kleeberg, 2015) in combination with BGMN (http://www.bgmn.de) (Bergmann et al., 1998; Taut et al., 1998). Crystalline models were taken from PDF# 04–013-3344 (Curry and Jones, 1971) for brushite, PDF# 04–009-3755 (Dickens et al., 1971) for monetite, PDF# 04–008-8714 (Dickens et al., 1974) for β-TCP, and PDF# 04–009-3876 (Boudin et al., 1993) for beta-calcium pyrophosphate (β-CPP; a constituent of the as-received ß-TCP). No other phases were identified in the diffraction patterns. The repeatability of the quantitative phase composition was taken as 2.77 x standard deviation according to ASTM E177-14 ASTM (2013) and Döbelin (2015).

2.5. Fatigue testing

The fatigue tests were performed in two different environments. Tests in air (at RT) were performed for ease of testing and allowed the results to be compared with those of previous studies; tests in PBS at 37 °C were conducted to more closely mimic an *in vivo* situation and to evaluate the influence of the surrounding environment. All tests were performed using a dynamic materials testing system (MTS® Axial 858 Mini Bionix® II, MTS Systems Corp., Eden Prairie, MN, USA) equipped with a 5 kN load cell. An environmental testing chamber (MTS Bionix EnviroBath, MTS Systems Corp., Eden Prairie, MN, USA) with a circulating heat bath (Polystat®, Cole-Parmer, Vernon Hills, IL, USA) was connected to the materials testing system to test specimens in PBS at 37 °C.

Each specimen was subjected to a small preload of 0.5 MPa, followed by a cyclic sinusoidal constant-amplitude compression-compression load (minimum stress level of 0.5 MPa, maximum stress level as described below), at a frequency of 2 or 20 Hz. A frequency of 2 Hz was used as indicated by the standard for fatigue testing of acrylic cements for joint implant fixation ASTM F2118-03 (ASTM, 2009), as there is no standard for CPCs. A frequency of 20 Hz was used to accelerate the test. An increased frequency has previously been shown to negatively

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