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On the mechanical characteristics of a self-setting calcium phosphate cement

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ABSTRACT

Objective: To perform a mechanical characterization of a self-setting calcium phosphate cement in function of the immersion time in Ringer solution.
Materials and methods: Specimens of self-setting calcium phosphate cement were prepared from pure α-TCP powder. The residual strains developed during hardening stage were monitored using an embedded fiber Bragg grating sensor. Additionally, the evolution of the elastic modulus was obtained for the same time period by conducting low-load indentation tests. Micro-computed tomography as well as microscope-assisted inspections were employed to evaluate the porosity in the specimens. Moreover, diametral compression tests were conducted in wet and dried specimens to characterize the material strength.
Results: The volume of the estimated porosity and absorbed fluid mass, during the first few minutes of the material's exposure in a wet environment, coincide. The immersion in Ringer solution lead to a noticeable increase in the moduli values. The critical value of stresses obtained from the diametral compression tests were combined with the data from uniaxial compression tests, to suggest a Mohr-Coulomb failure criterion.
Conclusions: This study presents different techniques to characterize a self-setting calcium phosphate cement and provides experimental data on porosity, mechanical properties and failure. The investigated material possessed an open porosity at its dried state with negligible residual strains and its Young's modulus, obtained from micro-indentation tests, increased with hardening time. The failure loci may be described by a Mohr-Coulomb criterion, characteristic of soil and rock materials.

1. Introduction

Calcium phosphate cements (CPCs), discovered in the 1980s, are produced by mixing water or aqueous phosphate solution with a calcium phosphate compound. The mixture results in a malleable paste that progressively solidifies and then hardens, either by sintering it in high temperature (Andriotis et al., 2005) or through hydrolysis by immersing the cement in a hardening liquid (Zhang et al., 2014). The main interest in this class of materials relies on their potential use as a solution in human bone defects due to their excellent biocompatibility, bioactivity, osteoconduction and osteogenesis. Moreover, CPCs are more biocompatible with the human bone than many other ceramic and inorganic nanoparticles. Also, their variable stoichiometry, functionality, and dissolution properties (Bose and Tarafder, 2012) combined with the exhibited intrinsic porosity enables the potential of using this material as a drug eluting carrier.

Despite CPCs' numerous advantages, a number of limitations have to be addressed in order to satisfy the clinical requirements. Apart from poor injectability (Bohner and Baroud, 2005; Habib et al., 2008) and weak cohesion of CPC pastes (Khairoun et al., 1999), they also exhibit low mechanical properties (Zhang et al., 2014), thus their use is limited to low-load bearing applications (Zhang et al., 2014; Sakka et al., 2013). Several published works have focused on dealing with the aforementioned challenges by adding various additives either to the powder or to the liquid compound of CPCs (Cherng et al., 1997; Liu et al., 2013; Saint-Jean et al., 2005; Burguera et al., 2006; Hofmann et al., 2009). In these works, an improvement in handling properties, desired porosity or bioactivity was achieved, but a decrease in the mechanical strength was also reported. Moreover, the category of self-setting CPCs, like α-TCP, present a unique feature: their mechanical properties can be improved when are immersed in a liquid of similar composition of human body fluids because of occurred microstructural changes (Zhang et al., 2014; Ginebra et al., 1997), hence are considered as promising CPCs to overcome current mechanical challenges.

Most of the studies that deal with CPCs measure their strength by conducting only compression tests. However, compression strength

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cannot be the only criterion in order to assess the mechanical performance of a CPC, since they are meant to be subjected to complex stress fields developed in human bones (Burstein et al., 1972). Therefore, a failure surface based on experimental data could provide a baseline information to better assess the structural integrity for future applications. Moreover, porosity is not always evaluated despite the fact that is the most detrimental factor to CPC mechanical properties (Zhang et al., 2014). Last but not least, little information exist in the literature about the induced strains during self-setting cements' hardening period. This parameter should be considered of significant importance since any strain fields developed during this period can affect not only the way CPCs fail under loading but also the material's dimensional stability in wet environment.

The main aim of this work is to perform a characterization on a self-setting CPC, fabricated by using pure α–TCP powder and hardened with hydrolysis reaction. The obtained information is important for the development of reliable future clinical applications in bone defects. The material's porosity was investigated by means of micro-computed tomography (μ-CT) imaging to examine the homogeneity of the microstructure and microscopy images for quantitative measurements of the porosity. The evolution of the effective Young modulus during cement's hardening was measured by indentation tests. At the same period, the induced strains were measured with the use of a fiber Bragg grating sensor embedded in the bone cement, through which material's residual strains development was investigated. Finally, diametral compression tests were conducted in the material, in wet and dry state after the completion of the hardening period. These results combined with the ones on uniaxial compression strength for the same material, reported in (Bimis and Karalekas, 2015), are used to propose a Mohr-Coulomb failure criterion.

2. Materials and methods

2.1. Sample preparation

The material used throughout this study is α-TCP, in powder form, with particle diameter raging between 2 and 7 μm, evaluated by scanning electron microscopy images. The α-TCP powder and aqueous solution 4% Na_2HPO_4 were initially stored for a few days and subsequently mixed manually (applied ratio: 1 ml: 2.06 g), in a controlled temperature room at 23 °C. The resulting paste was casted in three different, custom made molds. The first one was made from silicon, with cuboid cavities of 6×6×12 mm (width×height×length), to easily extract the specimens. The second one was made from aluminum, having a cylindrical cavity of 12 mm diameter and 40 mm length. It was specially designed in order to allow the proper placement of a 0.125 mm diameter optical fiber, along the specimens' longitudinal axis. The third one was built by a 3-D printer, and consisted of 2 parts that could be assembled and create a cylindrical cavity of the same dimensions as the previous one.

Overall, nine cuboid specimens were fabricated as well as three cylindrical ones, of which one contained an embedded optical fiber with a centrally located fiber Bragg grating (FBG) sensor. Upon their solidification, the specimens were hardened by immersion in Ringer solution (provided by Laboratorium Dr. G. Bichsel AG, Switzerland, with a composition of Na 155.2 mmol/l, K 4.0 mmol/l, Ca 2.7 mmol/l, Cl 163.4 mmol/l and HCO3 1.2 mmol/l). The cylindrical sample with the embedded optical fiber was submerged in hardening liquid having a sample/ liquid volume ratio equal to 1.29×10^{-2}, while the other two cylindrical specimens were submerged in a bath with sample/liquid volume of 1.50×10^{-2}. Regarding the cuboid specimens, they were immersed collectively in a single Ringer solution bath with sample/liquid volume ratio of 0.85×10^{-2}. The solidification and hardening of samples lasted 4 and 14 days, respectively, while both stages took place in a controlled environment, at room temperature (23 °C). The time interval of hardening period is considered long enough (Ginebra et al., 1995; Hurle et al., 2015), to safely assume that hydrolysis of α-TCP was completed.

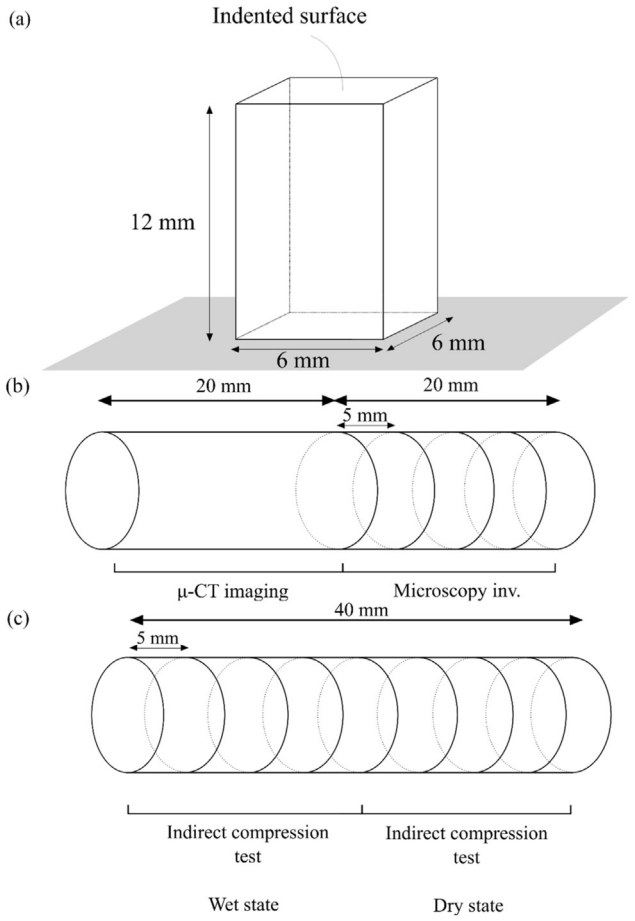

Fig. 1. Schematic of the (a) cuboid specimens for indentation and (b),(c) cylindrical specimens employed for the investigation of the porosity and the indirect compression tests, respectively.

Every 2 days during hardening period (i.e. 2, 4, 6, 8, 10, 12, 14 days) a different cuboid specimen (Fig. 1a) was recovered and subjected to indentation testing. One of them was indented unhardened and the results were used as a reference value. The strains during solidification and hardening were monitored via the reflected wavelength of the embedded FBG sensor in the cylindrical specimen. Additional specimens were cut from cylindrical blocks without embedded FBG using a diamond wire saw at the end of the hardening period of 14 days. The cuts were performed perpendicular to the longitudinal axis of the cylinder. As a result, two cylindrical specimens, 20 mm long (Fig. 1b) as well as 8 disks of 5 mm thickness (Fig. 1c) were produced for porosity investigation (cylindrical specimens) and diametral compression tests (disk specimens).

2.2. Porosity investigation

One of the two cylindrical specimens, 20 mm in length, was dried and used for micro-computed tomography (μ-CT) imaging (Bruker microCT, Kontich, Belgium). The highest resolution was applied which corresponds to 9 μm voxel size. The resulting images were software processed (CTAN image analysis) to construct the 3-D image of the specimen and calculate its porosity following an experimental procedure similar to the technique previously employed to study bone microstructures (Bimis et al., 2016). The calculation of the material's porosity is based on voxel's brightness: low brightness voxels represent low density areas (pores) while high brightness voxels represent bulk material (Bouxsein et al., 2010; Christiansen, 2016). Then porosity is

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