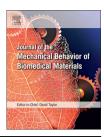


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#### Research Paper

## Compressive, diametral tensile and biaxial flexural strength of cutting-edge calcium phosphate cements



Jun Luo<sup>a,1</sup>, Ingrid Ajaxon<sup>a,1</sup>, Maria Pau Ginebra<sup>b</sup>, Håkan Engqvist<sup>a</sup>, Cecilia Persson<sup>a,\*</sup>

<sup>a</sup>Materials in Medicine, Division of Applied Materials Sciences, Department of Engineering Sciences, Uppsala University, Box 534, 751 21 Uppsala, Sweden

<sup>b</sup>Research Centre in Biomedical Engineering, Biomaterials Division, Department of Materials Science and Metallurgy, Technical University of Catalonia (UPC), Av. Diagonal 647, E08028 Barcelona, Spain

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#### ABSTRACT

Calcium phosphate cements (CPCs) are widely used in bone repair. Currently there are two main types of CPCs, brushite and apatite. The aim of this project was to evaluate the mechanical properties of particularly promising experimental brushite and apatite formulations in comparison to commercially available brushite- and apatite-based cements (chronOS™ Inject and Norian® SRS®, respectively), and in particular evaluate the diametral tensile strength and biaxial flexural strength of these cements in both wet and dry conditions for the first time. The cements' porosity and their compressive, diametral tensile and biaxial flexural strength were tested in wet (or moist) and dry conditions. The surface morphology was characterized by scanning electron microscopy. Phase composition was assessed with X-ray diffraction. It was found that the novel experimental cements showed better mechanical properties than the commercially available cements, in all loading scenarios. The highest compressive strength (57.2 $\pm$ 6.5 MPa before drying and  $69.5\pm6.0$  MPa after drying) was found for the experimental brushite cement. This cement also showed the highest wet diametral tensile strength (10.0  $\pm$  0.8 MPa) and wet biaxial flexural strength (30.7  $\pm$  1.8 MPa). It was also the cement that presented the lowest porosity (approx. 12%). The influence of water content was found to depend on cement type, with some cements showing higher mechanical properties after drying and some no difference after drying.

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<sup>\*</sup>Correspondence to: Division of Applied Materials Science, Department of Engineering Sciences, The Ångström Laboratory, Uppsala University, Uppsala, Sweden.

E-mail address: cecilia.persson@angstrom.uu.se (C. Persson).

<sup>&</sup>lt;sup>1</sup>These authors contributed equally to this work.

#### 1. Introduction

Calcium phosphate cements (CPCs) are clinically used as bone void fillers and as complements to hardware in fracture fixation (Larsson and Bauer, 2002). They are produced by mixing one or more calcium phosphate based powders with a liquid phase to form a paste that sets into a hard cement in a restricted period of time. Different types of CPCs can be obtained depending on the pH of the chemical reaction: when the pH is higher than 4.2, the end product is basic CPC, apatite (such as hydroxyapatite (HA), calcium-deficient hydroxyapatite (CDHA), carbonated apatite); and when the pH is lower than 4.2, the product is acidic CPC, brushite (dicalcium phosphate dihydrate, DCPD) or monetite (dicalcium phosphate anhydrous, DCPA). The precipitation kinetics of monetite are slower than brushite, so brushite is generally formed as the main product when pH is lower than 4.2 (Bohner, 2007 and 2000). A main, clinically relevant difference between brushite and apatite is the solubility. Brushite is metastable under physiological conditions, and could be resorbed faster than apatite (Vereecke and Lemaître, 1990; Gisep et al., 2003). However, transformation of brushite to apatite may occur in vivo (Bohner, 2007).

Since the first commercial CPC products were introduced two decades ago, many more have become available and have showed promising results in terms of bone regeneration, but some issues remain to be solved (Bohner et al., 2005; Bohner, 2010). Ideally, a bone substitute material should have mechanical properties similar to the host bone. The mechanical properties of CPCs are however generally poor compared to the surrounding bone, in loading scenarios other than compressive. In fact, CPCs are only approved for use in nonload bearing applications or are not used alone in loadbearing applications. Therefore, knowledge of the mechanical properties is important for decisions regarding possible use in certain, well-defined load-bearing scenarios. However, there is a general lack of knowledge on mechanical properties other than compressive strength (CS) for CPCs (Zhang et al., 2014). In particular, there is no data on the biaxial flexural properties of commercially available CPCs.

Apatite is the most investigated CPC type, as it has traditionally shown a higher mechanical strength than brushite. However, brushite cements have attracted an increasing interest as they have shown faster setting and resorption than apatite cements in vivo (Bohner et al., 2005). Also, brushite cements with a strength matching that of apatite cements have recently been reported (Unosson and Engqvist, 2014; Engstrand et al., 2014), with a maximum wet CS of 91.8 MPa after setting for 24 h. On the other hand, a fast setting apatite cement with good mechanical strength (wet CS around 40 MPa) has also been developed recently, where almost full strength could be achieved in 24 h (Ginebra et al., 2004).

The aim of this study was to evaluate and compare the mechanical properties of the above recently developed cutting-edge experimental CPCs, i.e. the strong brushite cement and the fast setting apatite cement, with two commercially available brushite and apatite based cements,  $\operatorname{chronOS}^{\text{\tiny TM}}$  Inject and  $\operatorname{Norian}^{\text{\tiny ®}}$  SRS<sup>®</sup>, respectively.  $\operatorname{chronOS}^{\text{\tiny TM}}$ 

Inject is a brushite based bone mineral substitute with low mechanical strength, similar to the lower range of cancellous bone (Donaldson and Wright, 2011). Norian<sup>®</sup> SRS<sup>®</sup> is a fast-setting apatite bone mineral substitute. It forms a low crystalline order and a small grain size carbonated apatite similar to the mineral phase of bone in comparison to sintered HA (Yetkinler et al., 1999).

CPCs are exposed to body fluids in vivo. However, the mechanical properties of the cements are often determined experimentally using dry specimens (Koh et al., 2015; Ajaxon and Persson, 2016). The water-saturation state is however a significant factor which could affect the mechanical properties of CPCs (Zhang et al., 2014; Pittet and Lemaître, 2000). In this study, the compressive, diametral tensile and biaxial flexural strength of experimental and commercial CPCs were evaluated in wet (or moist) and dry conditions. The porosity – as assessed by the water evaporation method and helium pycnometry – as well as the morphology and phase composition – as assessed by scanning electron microscopy (SEM) and X-ray diffraction (XRD), respectively; were also evaluated and correlated to the mechanical properties.

#### 2. Materials and methods

Two experimental cements, a brushite cement (Unosson and Engqvist, 2014) and an apatite cement (Ginebra et al., 2004) and two commercial cements (chronOS<sup> $^{\text{IM}}$ </sup> Inject and Norian<sup> $^{\text{IM}}$ </sup> SRS<sup> $^{\text{IM}}$ </sup>) were used in the study. chronOS<sup> $^{\text{IM}}$ </sup> Inject (Synthes GmbH, Switzerland) is a biphasic cement ( $\beta$ -TCP granules are embedded in a brushite matrix) (Bohner et al., 2003), and Norian<sup> $^{\text{IM}}$ </sup> SRS<sup> $^{\text{IM}}$ </sup> (Norian Corp., USA) is a carbonated apatite cement (Constantz et al., 1995). At the time of testing, the available Norian<sup> $^{\text{IM}}$ </sup> SRS<sup> $^{\text{IM}}$ </sup> had expired (expired January 2011, tested March 2015, no unexpired cements available from the supplier). Although XRD was performed to verify the composition, this can be considered a limitation of the study.

#### 2.1. Cement preparation

Fig. 1 presents an overview of the experimental cement preparation methods.

The experimental brushite cement was prepared according to previous work (Unosson and Engqvist, 2014). A liquidto-powder ratio of 0.22 mL/g was used in this process. The starting powder contained monocalcium phosphate monohydrate (MCPM, Scharlau, Sentmenat, Spain) and β-tricalcium phosphate (β-TCP, Sigma-Aldrich, St. Louis, MO, USA) in a 45:55 molar ratio, together with 1 wt% disodium dihydrogen pyrophosphate (SPP, Sigma-Aldrich, St. Louis, MO, USA) for control of the setting time. The particle size of MCPM was sieved to less than 75  $\mu m.$  The liquid was an aqueous solution of 0.5 M citric acid (Acros Organics, New Jersey, USA). To prepare the cement paste, the starting powder and the liquid were mixed for 1 min in a Cap-Vibrator (Ivoclar Vivadent AG, Schaan, Liechtenstein) to allow for more efficient mixing of the two phases. Then the cement paste was filled into rubber moulds using a spatula in order to obtain a cylinder or a disc with the desired size (6 mm in diameter and 13 mm in height for compressive strength (CS) test, 8 mm in diameter and

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