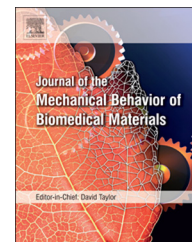


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## Research Paper

# The compressive modulus and strength of saturated calcium sulphate dihydrate cements: Implications for testing standards



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## ABSTRACT

Calcium sulphate-based bone cement is a bone filler with proven biological advantages including biodegradability, biocompatibility and osteoconductivity. Mechanical properties of such brittle ceramic cements are frequently determined using the testing standard designed for ductile acrylic cements. The aims of the study were (1) to validate the suitability of this common testing protocol using saturated calcium sulphate dihydrate (CSD), and (2) to compare the strength and effective modulus of non-saturated and saturated CSD, in order to determine the changes in the mechanical behavior of CSD upon saturation. Unconfined compression tests to failure were performed on 190 cylindrical CSD samples. The samples were divided into four groups having different saturation levels (saturated, non-saturated) and end conditions (capped and non-capped). Two effective moduli were calculated per sample, based on the deformations measured using the machine platens and a sample-mounted extensometer. The effective moduli of non-saturated groups were found to be independent of the end conditions. The saturated and capped group showed no difference in the effective moduli derived from different measurement methods, while the saturated and non-capped group showed a significant difference between the machine platen- and extensometer-derived moduli. Strength and modulus values were significantly lower for saturated samples. It was assumed that the existence of water in saturated CSD alters the mechanical response of the material due to the changes in chemical and physical behaviors. These factors are considered to play important roles to decrease the shear strength of CSD. It was proposed that the reduction in CSD shear strength evokes local deformation at the platen-sample boundary, affecting the strength and effective moduli derived from the experiments. The results of this study highlighted the importance of appropriate and consistent testing methods when determining the mechanical properties of saturated ceramic cements.

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## 1. Introduction

Compressive vertebral fractures arise from bone fragility due to osteoporosis or metastasis. The vertebral fractures result in back pain which may lead to neurological deficits in severe cases. Clinically, vertebral fractures are treated using conservative therapies, external braces, vertebroplasty or spinal fusion surgeries, depending on the severity of the fracture and the age of the patient. Vertebroplasty is a percutaneous procedure first introduced in 1987 (Galibert et al., 1987) to treat hemangioma. The procedure consists of augmenting the damaged vertebral body, typically with a poly(methyl methacrylate)-based bone cement. The usage of acrylic cements in vertebroplasty is associated with potential risks and complications related to toxicity (Dahl et al., 1994), exothermic curing reaction (Deramond et al., 1999) and bioinertness of the final biomaterial. Alternative ceramic bone cements, commonly based on calcium phosphate (CPC) or calcium sulphate (CSC) (Bohner, 2010; Heini and Berlemann, 2001), have also been developed, with additional advantages over acrylic bone cements. Ceramic bone cements have superior biological properties of biocompatibility, osteoconductivity, osteoinductivity, biodegradability (Huan and Chang, 2007; Liu et al., 2013; Nilsson et al., 2004; Yang et al., 2012) and non-exothermic setting reactions. A major disadvantage of ceramic bone cements is its sub-optimal mechanical properties of low shear strength (Blatter et al., 2009) and low fracture toughness (Morgan et al., 1997), which may lead to fragmentation under cyclic loads (Wilke et al., 2006).

There are multiple studies reporting positive results on the aforementioned biological properties of ceramic bone cements, together with reports on its cytotoxicity (Huan and Chang, 2007; Liu et al., 2013), injectability (Huan and Chang, 2007; Liu et al., 2013; O'Hara et al., 2010) and strength (Charriere et al., 2001; Drosos et al., 2012; Huan and Chang, 2007; Lewis et al., 2006; Liu et al., 2013; Van Lieshout et al., 2011; Yamadi and Kobayashi, 2009; Yang et al., 2012). The moduli of ceramic cements (Charriere et al., 2001; Crawford et al., 1998; Drosos et al., 2012; Hing et al., 1997, 1999; Van Lieshout et al., 2011; Welch et al., 2002) were also reported in various studies. However, many studies report the moduli of non-saturated ceramic bone cements (Drosos et al., 2012; Lewis et al., 2006; Wang et al., 2003; Yamadi and Kobayashi, 2009) or bone-cement composites from *ex vivo* studies (Crawford et al., 1998; Hing et al., 1997, 1999; Welch et al., 2002). In other words, studies comprehensively reporting the modulus of pure saturated cements are rare (Charriere et al., 2001; Van Lieshout et al., 2011).

CSCs have a long history of clinical use since its first report in 1892 (Dressmann, 1892). They are known to undergo a complete and quick resorption without significant inflammatory responses (Huan and Chang, 2007; Liu et al., 2013). A typical CSC consists of calcium sulphate hemihydrate ( $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$ ) powders, which react with water to form calcium sulphate dihydrate ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) according to Eq. (1) (Thomas and Puleo, 2009).



The phase of calcium sulphate hemihydrate (CSH,  $\alpha$ - or  $\beta$ -) determines the final density and solubility of the resulting

calcium sulphate dihydrate (CSD).  $\beta$ -phase CSH is known to yield a less dense CSD by precipitation of irregular crystals with interstitial and capillary pores (Thomas and Puleo, 2009) as compared to the denser  $\alpha$ -phase CSH.

The strength of wet CSD is known to be lower than dry CSD (Andrews, 1946). According to Andrews (1946), the strength of CSD is the force required to overcome the frictional resistance between the interlocked CSD crystallites. Andrews pointed out that the existence of water reduces the strength of CSD, as it reduces the frictional resistance between the interlocked crystals. In addition to his findings, dissolution of CSD with addition of water also plays a role in the reduction of the strength of wet CSD. Thus, given that the physical behavior, hence the mechanical properties of wet/saturated CSD differ from dry/non-saturated CSD, the most appropriate protocol to test saturated CSD may also differ from those designed for non-saturated CSD.

The most common testing protocol used to determine the compressive properties of ceramic cements is derived from the international testing standard developed for (ductile) acrylic cements (ISO5833:2002). This two page long testing standard outlines a general method to prepare the testing samples (e.g. filling cylindrical molds and grinding plane-parallel ends) and basic compression test protocol (e.g. the range of displacement rate and suitable boundary condition). The benefit of an abstract mechanical testing protocol is that it provides the flexibility to adapt the test according to specific needs or applications. Therefore, many previous studies reporting the compressive strength of ceramic cement follow the sample preparation method outlined in ISO 5833:2002 (Drosos et al., 2012; Huan and Chang, 2007; Liu et al., 2013; O'Hara et al., 2010; Van Lieshout et al., 2011).

Ceramic cements are known to be brittle. Therefore, the experimentally obtained material parameters are highly sensitive to porosity, pore sizes, pore shape, boundary conditions and angularity between the sample end surfaces. Brittle materials exhibit small deformations until rupture and are very sensitive to flaws within the samples. A small misalignment of the end surfaces (end conditions) or the existence of macro-pores, for example, may lead to premature crack initiation and propagation, and lower derived compressive strength and modulus values than for flawless samples. Further, other factors including chemical composition (Liu et al., 2013; Nilsson et al., 2004; Vyas et al., 2008), liquid-to-powder ratio (Nilsson et al., 2003; O'Hara et al., 2010; Zhang et al., 2011), mechanical and chemical conditioning prior to testing (Huan and Chang, 2007; Liu et al., 2013), the phase of the starting components and crystal shapes (Thomas and Puleo, 2009) also influence the strength and modulus. Thus, the testing standard for ceramic cements may require additional refinement, compared to the standards set for acrylic cements, to produce reliable results.

In the field of civil engineering, especially in the studies of concretes and cements, factors influencing the mechanical properties of ceramics have been extensively studied. As a focus of study, the influences of intrinsic and extrinsic factors on the compressive strength and modulus (Banfill, 2006; Gesoglu et al., 2002) of concretes and cements were carefully examined. Based on this, testing methods quantifying the strength and modulus of brittle materials (ASTM Standard

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