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Effects of fibre reinforcement on the mechanical properties of brushite cement

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Abstract

In this study the effect of structure and amount of polyglactin fibre incorporation into a brushite forming calcium phosphate cement system and the effect of mechanical compaction on the fibre modified system were investigated. In comparison the effect of resorbable polycaprolactone surface coating of cement specimens was investigated. The results showed that, apart from the mechanical properties of the reinforcing material, the structure of the incorporated fibres, regular or random, is crucial for the resulting flexural strength and modulus of elasticity. Fibre reinforcement could also be combined with mechanical compaction of the cement/fibre composite paste leading to a possible 7-fold increase in flexural strength or an almost 5-fold increase in modulus of elasticity. Reinforcement of the tensile surface of cement grafts may ultimately improve strength where required, especially in conjunction with bone fixation devices.

Keywords: Calcium phosphate cement; Fibre reinforcement; Mechanical properties; Brushite

1. Introduction

The development of calcium phosphate cements has led to the production of a new class of ceramic grafting materials, which are mouldable and set at physiological conditions and therefore find application in non load-bearing indications in craniofacial surgery [1], as well as in dental applications such as endodontics [2] and the repair of periodontal bone defects [3].

Despite considerable research efforts resulting in the development of new formulations that either form apatitic or brushite products, many drawbacks associated with their use still exist. For example, Brown et al. [3] repaired periodontal bone defects using an apatite cement and found that 6 months following implantation 11 of 16 patients had exfoliated either part of or the whole implant. The high incidence of clinical failure was thought to be due to the lack of adhesion between the periodontal defect and the implant. The lack of adhesion was thought to result from the lack of flexural resistance of the cement and the absence of macroporosity preventing bone ingrowth.

Although the optimisation of calcium phosphate cements (CPC) has been the subject of many studies [4,5], and improvements in strength have been reported through the use of various additives and by altering mixing parameters etc., the gains in strength have not been such that CPC can be used in load bearing applications. However, in comparison to construction cements, e.g. Portland cement, the compressive strengths of apatitic calcium phosphate cements are high (101 MPa and 52 MPa, respectively

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[5,6]). The incorporation of macroporosity has been the focus of a number of studies; however, macroporosity is detrimental to the mechanical properties of the cement [5,7]. This disadvantage is offset by the cement being physically anchored to the defect site by bone ingrowth.

Fibre reinforcement of CPC was reported by dos Santos et al. [8] who found that incorporating up to 1.6% polyamide fibres had little effect on compressive strength. Another report showed that polyglactin knitted mesh on the tensile surface of CPC increased the work to fracture value by nearly 100 times [9]. Xu and Simon reported the use of resorbable polyglactin fibres to initially improve the flexural strength of the cement and following degradation result in the formation of macroporosity [10].

While fibre reinforcement of CPCs has been reported to increase the ultimate tensile strength (UTS) and work to fracture, the presence of fibre reinforcement did nothing for the actual tensile strength of the cement matrix. Furthermore, the presence of fibres within the matrix could be responsible for a decrease in the compressive strength of the cement by acting as a flaw source. In civil engineering applications of cement, random orientation polymeric fibres are only used to improve the cohesion of the mix so that it can be applied more easily in nonhorizontal applications and to relieve stresses due to expanding gas in the event of fire. Tensile strength improvement is only obtained when the tensile surface (soffit) is reinforced with a high modulus material parallel to the direction of stress. Any other reinforcement serves to prevent shrinkage cracks. Although von Gonten et al. set out to achieve that, their use of a knitted mesh meant that deformation of the mesh would have occurred before the fibres shared any load [11]. As far as we are aware polymeric reinforcement works of CPC to date has served mainly to prevent gross fragmentation following matrix failure and have reported invalid UTS values due to the deformation in the bending tests being more than the thickness of the sample.

In this study, we report a comparative investigation into the mechanical properties of CPC cement with random short fibres added to the mix and continuous parallel fibres set into the soffit of a brushite cement matrix which has very low reported values for diametral tensile strength compared to apatite cements [12], in order to determine whether a significant improvement in tensile properties of the matrix could be attained. Hand mixed and compacted cements were used. The effect of fibre volume fraction was investigated for both modes of specimen preparation. One approach to fracture fixation in maxillofacial surgery is based on the principle whereby biomechanical analyses have identified regions of physiological compression and tension [13] and only regions physiologically placed under tension are fixated. In order to determine if application of this principle to CPC use and hence indication of specific reinforcement was practically possible, soffit reinforcement was effected by application of a thin film of molten polycaprolactone and mechanical evaluation was performed. The melting temperature of this biodegradable polymer is only a few degrees above body temperature and thus could feasibly be coated onto CPC post-setting.

2. Materials and methods

2.1. Calcium phosphate cement and reinforcement

A CPC with brushite (dicalcium phosphate dihydrate), $CaHPO_4 \cdot 2H_2O$, as the end product was produced following a method similar to that of Bohner et al. [12] using β-tricalcium phosphate (β-TCP), $Ca_3(PO_4)_2$ (Plasma-Biotal, UK) and 3.5 M phosphoric acid (H_3PO_4, Sigma–Aldrich, UK), as reactants. The reaction proceeded in accordance with Eq. (1):

$$Ca_3(PO_4)_2 + H_3PO_4 + 6H_2O \rightarrow 3CaHPO_4 \cdot 2H_2O \quad (1)$$

In order to control the rate of the reaction and prevent a sudden setting of the cement paste the phosphoric acid contained 100 mM sodium citrate (Sigma–Aldrich, UK) as additional retardant. The β-TCP comprised spherical particles with a median particle size of 41 μm as determined by laser diffraction particle size analysis (Mastersizer S, Malvern, UK).

One type of fibre reinforcement was used in this study, namely USP size 2–0 Coated Vicryl™ (polyglactin 910) suture (Ethicon, USA). The properties of the polyglactin (PGA) fibre are shown in Table 1.

Additionally the use of a thin layer of polycaprolactone with a molecular weight of 80,000 (Sigma–Aldrich, UK) to reinforce a CPC beam was investigated.

2.2. Fabrication of brushite cement specimens

Three types of test specimen were manufactured: pullout specimens, compression specimens and beam specimens. The details of the specimen geometries and the amounts and location of any reinforcement used are given in Table 2. It should be noted that the percentage volume of random 5 mm fibres is equivalent to the weight of 25 fibres of length 60 mm.

In order to produce hand compacted specimens 3 g of β-TCP was mixed with 2 mL of phosphoric acid/sodium citrate solution and the resulting slurry was compressed by hand into a silicone mould of the required specimen dimensions. After 5 min the specimens were demoulded.

In the case of mechanically compacted specimens 15 g of β-TCP was mixed with 10 mL of phosphoric acid/sodium citrate solution and the resulting slurry was slowly

Table 1
Properties of polyglactin fibre used to reinforce CPC, the diameter range and maximum oversize were obtained from the manufacturer

Diameter range for USP suture size 2–0 (mm)	Manufacturer's maximum oversize (mm)	Modulus of elasticity (MPa)
0.30–0.349	0.004	11,000

The modulus of elasticity was obtained from [9].