

Weight loss, ion release and initial mechanical properties of a binary calcium phosphate glass fibre/PCL composite

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Abstract

Composites comprising a biodegradable polymeric matrix and a bioactive filler show considerable promise in the field of regenerative medicine, and could potentially serve as degradable bone fracture fixation devices, depending on the properties obtained. Therefore, glass fibres from a binary calcium phosphate ($50\text{P}_2\text{O}_5 + 50\text{CaO}$) glass were used to reinforce polycaprolactone, at two different volume fractions (V_f). As-drawn, non-treated and heat-treated fibres were assessed. Weight loss, ion release and the initial mechanical properties of the fibres and composites produced have been investigated. Single fibre tensile testing revealed a fibre strength of 474 MPa and a tensile modulus of 44 GPa. Weibull analysis suggested a scale value of 524. The composites yielded flexural strength and modulus of up to 30 MPa and 2.5 GPa, respectively. These values are comparable with human trabecular bone. An 8% mass loss was seen for the lower V_f composite, whereas for the two higher V_f composites an approximate 20% mass loss was observed over the course of the 5 week study. A plateau in the degradation profile at 350 h indicated that fibre dissolution was complete at this interval. This assertion was further supported via ion release studies. The leaching of fibres from the composite created a porous structure, including continuous channels within the polymer matrix. This offers further scope for tailoring scaffold development, as cells from the surrounding tissue may be induced to migrate into the resulting porous matrix.

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1. Introduction

Absorbable polymers have played an important role in areas of clinical medicine [1], in both tissue engineering (TE) and drug delivery applications, and represent an important growth area for temporary applications such as repair and regeneration of healing tissues [2].

Composites comprising a biodegradable polymeric matrix with bioactive fillers also show considerable promise in the field of regenerative medicine, potentially in orthopaedic procedures for hard tissue repair and reconstruction. Their potential replacement of metallic bone plates

offers the possibility of reducing problems such as stress shielding, and would reduce the need for revision surgery.

Poly- ϵ -caprolactone (PCL), a semicrystalline aliphatic polyester, has been widely studied as a potential biomaterial. It has mainly been used in thermoplastic polyurethanes and resins for surface coatings. It also serves to make stiffeners for shoes and orthopaedic splints, and biodegradable packaging, sutures and fibres. Initially, it was recognized that PCL can be degraded via a hydrolytic mechanism and later also under physiological conditions. Under certain circumstances, cross-linked PCL can be degraded enzymatically, and low-molecular-weight fragments of PCL are taken up by macrophages and degraded intracellularly [3].

Studies on materials for bone regeneration have focused increasingly on materials that are resorbable. PCL and cal-

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cium phosphate (CaP) powder particles have previously been used to generate composite scaffolds with precise architectural features via injection moulding of thermoplastic porogens fabricated by DDP (drop on demand printing). Varying PCL:CaP ratios were investigated and an ultimate compressive strength value of over 10 MPa was seen for PCL and 25 MPa seen for the 80:20 composite, along with a compressive modulus of over 250 MPa for PCL with 400 MPa for the 80:20 composite [4].

A range of PCL/hydroxyapatite (HA) composites were fabricated with HA contents of 0, 20, 40 and 60% by mass. The highest tensile yield strength (approximately 17 MPa) was obtained for the 20% HA sample, with the highest tensile modulus value (1.5 GPa) seen for the 60% by mass composite [5].

Chen et al. investigated PCL/chitin (natural) fibre composites as potential bone substitutes. Composites with fibre contents of 0, 15, 35, 45, 55 and 65% by mass were produced and mechanically tested. The highest tensile strength and tensile modulus was obtained for the 45% by mass composite, with values of approximately 20 MPa and 1.9 GPa, respectively [6].

Phosphate-based glasses (PBGs) are of huge interest due to their hydrolytic degradation mode and a chemical composition, which closely matches that of the inorganic phase of bone. Many formulations are biocompatible, and subtle alterations to their composition permits a wide variation of mechanical and thermal properties [7].

The conversion of PBG into fibres [8] was an interesting advancement, and these fibres have been evaluated for their potential as *in vitro* guides for muscle cells [9] and have also been studied to investigate the effects of seeding human oral osteoblasts, oral fibroblasts and hand flexor tendon fibroblasts [10,11]. Similar forms have also been evaluated for their potential to reinforce collagen. Here, the channels formed upon PBG fibre degradation could potentially assist with angiogenesis [12].

Although PBG composites potentially involve a broad range of glasses, polymers and coupling agents, the limited data relating to the physiological response to these systems is universally positive. This includes biocompatibility studies of three degradable phosphate glass fibres and of several different degradable polymers studied by Andriano et al. [13]. Similar studies conducted with a PCL/Bioglass[®] composite (previously investigated by Corden et al. [14,15]) showed fibre surfaces treated with silane did not confer any negative biological responses [16]. *In vivo* trials by Lowry et al. [17] with a bioabsorbable PCL/glass implant in a rabbit humerus fracture model showed these materials to be well tolerated with minimal inflammation.

Features of absorbable polymeric biomaterials that make them of use in TE include: (i) acting as a mechanical scaffold for transplanted cells; (ii) acting as a mechanical or permeability barrier, to guide tissue regeneration; (iii) temporarily bearing mechanical loads during tissue healing or regeneration; and (iv) acting as a remodelable provisional

matrix into which cells from the surrounding tissue could be induced to migrate [18].

Therefore, based on the above (namely iii and iv), a binary (50/50 calcium phosphate) PBG fibre/PCL composite has been investigated, which could potentially serve as a degradable bone fracture fixation device, depending on the properties obtained. Weight loss, ion release and the initial mechanical properties of the fibres and composites produced have been investigated.

2. Materials and methodology

2.1. Phosphate glass and fibre production

The glass composition (50 mol.% P₂O₅ and 50 mol.% CaO) was produced using CaHPO₄ and P₂O₅ (Sigma–Aldrich, UK) as starting materials. The precursors were blended, placed in a 100 ml volume Pt/5% Au crucible type BC 18 (Birmingham Metal Company, UK), heated in air at 300 °C for 1 h and then melted at 1100 °C for 2 h. The resulting glass was poured into a graphite mould (pre-heated to 350 °C) and left to cool to room temperature. Continuous fibres were produced by melt-draw spinning using a dedicated in-house facility (see Table 1 for codes used in this study).

Fibres were used as-drawn, non-treated (NT) and heat-treated (HT). The heat treatment consisted of heating the fibres up to 5 °C below the T_g (529 °C) and holding the temperature for 2 h, and then cooling to room temperature. The fibres were treated prior to non-woven random mat production.

2.2. Assembly of non-woven random mat fibres

Randomly orientated non-woven mats were produced from (10 mm) chopped fibres. The fibres were separated into 3 g batches and dispersed within a water/Cellosize solution. Cellosize (hydroxyethyl cellulose-based material obtained from Univar Ltd., UK, used as a fibre binding agent) polymer granules were blended into 4 litres of deionized water and were continuously agitated at 500–600 rpm for 10 min. The fibres were added and left to disperse for 10 min before extraction using a fine mesh strainer, before rinsing with deionized water to remove any residual binder.

Table 1

Fibre volume fractions of the composites investigated in this study for as-drawn or heat-treated fibres

Composite sample code	Volume fraction of fibres (%)	Mass fraction of fibres (%)	Fibre drawn type
Low V_f	6	18	As drawn
Higher V_f (NT)	17	38	As drawn
Higher V_f (HT)	18	39	Heat-treated

V_f = % volume fraction of fibres; NT = non-treated fibres; HT = heat-treated fibres. The HT fibres were treated for 3 h at 5 °C below T_g .

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