

Self-reinforced composites of bioabsorbable polymer and bioactive glass with different bioactive glass contents. Part II: In vitro degradation

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Received 15 January 2007; received in revised form 29 April 2007; accepted 11 June 2007

Available online 5 July 2007

Abstract

The in vitro degradation behavior of self-reinforced bioactive glass-containing composites was investigated comparatively with plain self-reinforced matrix polymer. The materials used were spherical bioactive glass 13–93 particles, with a particle size distribution of 50–125 μm , as a filler material and bioabsorbable poly-L,DL-lactide 70/30 as a matrix material. The composites containing 0, 20, 30, 40 and 50 wt.% of bioactive glass were manufactured using twin-screw extruder followed by self-reinforcing. The samples studied were characterized determining the changes in mechanical properties, thermal properties, molecular weight, mass loss and water absorption in phosphate-buffered saline at 37 °C for up to 104 weeks. The results showed that the bioactive glass addition modified the degradation kinetics and material morphology of the matrix material. It was concluded that the optimal bioactive glass content depends on the applications of the composites. The results of this study could be used as a guideline when estimating the best filler content of other self-reinforced osteoconductive filler containing composites which are manufactured in a similar way.

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Keywords: Bioactive glass; Composite; Hydrolytic degradation; Polylactide; Self-reinforced

1. Introduction

The use of self-reinforced bioabsorbable polymeric implants in fracture fixations has two main advantages. The hydrolytic degradation of the implant material eliminates the removal operation, which is often used with corresponding metallic implants. Thus bioabsorbable implants are the preferred choice when only temporary support for healing tissue is needed. Secondly, because the bioabsorbable implants decompose gradually, the stresses are also transferred gradually to the healing tissue. Therefore stress shielding and weakening of the fixed tissue, which is often noticed with the corresponding metallic implants, are prevented [1–3].

However, most of these bioabsorbable implant materials do not have properties to accelerate or facilitate tissue healing. Such properties as bioactivity or osteoconductivity,

seen as bone bonding ability, would be advantageous for implant materials used in bone fracture implants. Thus in the past few years increasing attention has been paid to developing multifunctional biomaterials. These materials have several different functions in the same device, such as osteoconductivity and biodegradation.

Several different in vitro and in vivo studies have been reported on composites in which various ceramic filler materials have been added to the bioabsorbable polymers [4–11]. The bioceramic filler materials are reported to have an effect on, among other things, mechanical properties, degradation behavior, water absorption, mass loss, morphology, osteoconductivity and structure of the studied composites [5,6,9–11].

It is essential to understand the influence of the added compounds on the hydrolytic degradation of the matrix material. Several studies have been carried out on the degradation of different poly- α -hydroxyacids [2,12] as such, but the added compounds modified the degradation behavior. Some properties of the different material combinations

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forming the composite could be estimated based on earlier studies, but the degradation is dependent on so many factors that the prediction is often complicated.

The aim of this study was to examine the in vitro degradation behavior of self-reinforced poly-L,D-lactide 70/30 and its bioactive glass-containing composites. An earlier study [5] was done using a bioactive glass content of 15–20 wt.%. This filler content proved to be useful. The current study was performed to find out the effect of different filler contents (0–50 wt.%) on the in vitro degradation behavior of the composites. When the degradation behavior of the different composites is known, it is easier to select suitable composites for specific clinical applications.

2. Materials and methods

2.1. Materials and manufacturing

The materials and manufacturing process of the composites studied are similar to those reported in the earlier study [4]. Bioactive glass 13–93 (BaG) spheres (Vivoxid Ltd., Turku, Finland) with a composition of 6 wt.% Na₂O, 12 wt.% K₂O, 5 wt.% MgO, 20 wt.% CaO, 4 wt.% P₂O₅ and 53 wt.% SiO₂ and a particle size distribution 50–125 µm (the manufacturing of the glass and glass spheres have been described by Brink et al. [13–15]) were compounded with high molecular weight poly-L,D,L-lactide with an LL/DL dimer ratio of 70/30 (Boehringer Ingelheim, Ingelheim am Rhein, Germany), abbreviated PLA70 in the present paper, using a twin-screw extruder. The BaG sphere content in the composites varied from 0 to 50 wt.%. The non-reinforced composite rods (diameter approximately 5–6 mm) were further self-reinforced (SR) using solid state die-drawing [16–18] by drawing them through a heated die (65 °C). The draw ratios (DR) achieved were 3.5 and 2.0. The abbreviations, BaG contents, draw ratios and diameters of studied self-reinforced composites are listed in Table 1.

The initial mechanical and bioactivity properties of the composites studied are reported in an earlier study [4].

2.2. In vitro hydrolysis

All gamma sterilized, self-reinforced composites were immersed in phosphate-buffered saline (PBS; ionic concen-

trations Na⁺ 156.2 mM, HPO₄²⁻ 24.9 mM, H₂PO₄⁻ 5.5 mM and Cl⁻ 100.9 mM), pH 7.4, at 37 °C to ascertain in vitro degradation. $V_{\text{solution}}/V_{\text{sample}} > 20$ was used. The buffer solution was changed biweekly and pH was measured (three parallel samples) using a Mettler Toledo MP225 pH-meter (Mettler-Toledo GmbH, Schwerzebbach, Switzerland). If the pH of the buffer solution changed significantly (over 0.2), the buffer solution was changed more often. A follow-up time of 104 weeks was used.

2.3. Mechanical testing

During the in vitro hydrolysis the composites were mechanically tested by three-point bending and shearing using an Instron 4411 mechanical testing apparatus (Instron Ltd., High Wycombe, UK). The composites were tested under wet conditions at room temperature right after removal from buffer solution and deionized water rinsing (to remove the residual salts). At least four parallel samples were tested.

Bending strength and modulus were determined according to standard SFS-EN ISO 178 [19], the calculations of which were modified to the cylindrical samples. The cross-head speed was 5 mm min⁻¹ and the bending span 46–68 mm depending on the diameter of the sample. Shear testing was performed according to the standard BS 2782 method 340B [20] (calculations were modified to the cylindrical samples) using a crosshead speed 10 mm min⁻¹.

2.4. Mass loss and water absorption

To determine the mass loss of the composites, the samples were weighed before hydrolysis. During in vitro hydrolysis, samples were removed from the buffer solution and weighed wet to determine the water absorption (WA). After weighing, the samples were rinsed with deionized water and ethanol to remove residual salts and then dried for 5 days in a vacuum at room temperature. After drying, the samples were weighed and the remaining mass and WA were calculated as follows:

$$\text{remaining mass} = \frac{m_d}{m_0} \cdot 100\%$$

$$\text{water absorption (WA)} = \frac{(m_w - m_d)}{m_d} \cdot 100\%$$

where m_0 = initial dry mass of the sample, m_w = wet mass of the sample during in vitro and m_d = dry mass of the sample after removing from the buffer solution. The weight measurements were done using a Mettler Toledo AG245 (Mettler-Toledo AG, Greifensee, Switzerland) and the results are the average of three parallel samples.

2.5. Molecular weight measurements

The weight average molecular weight (M_w) of the composites was studied using conventional gel permeation chromatography (GPC) with narrow polystyrene standards

Table 1
Abbreviations used, BaG contents, draw ratios and diameters of the self-reinforced composites studied [4]

Composite abbreviation	BaG content: desired/measured ^a [wt.%]	Draw ratio, DR	Diameter [mm]
SRPLA70	0/0	3.5	2.7
SRPLA70BaG20	20/16.1	3.5	3.1
SRPLA70BaG30	30/27.7	3.5	3.1
SRPLA70BaG40	40/37.2	3.5	3.2
SRPLA70BaG50	50/47.7	2.0	4.2

^a By combustion analysis.

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