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Foamed high porosity alumina for use as a bone tissue scaffold

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Abstract

Porous alumina can be used as a synthetic bone scaffold in tissue engineering. Using a direct foaming method a foamed alumina was produced using in situ evolution of gases by calcining blends of ammonium sulphate and aluminium sulphate with varying ammonium mole fraction (AMF). The effect of foaming heating rate was observed by varying the foaming heating rate from 100 °C/h to 600 °C/h. The effect of sintering temperature was also observed by varying the sintering temperature from 1500 °C to 1600 °C. The resulting porous structure exhibited high mechanical strength with high levels of porosity and high average pore size. Foamed alumina with the optimal conditions was fabricated using 0.33 AMF with a foaming heating rate of 100 °C/h followed by sintering at 1600 °C. The porous alumina produced attained a high porosity value of 94.39%, an average pore size of 300 μm and the highest strength amongst all samples of 384.3 kPa. Interconnected porosity was observed using micro-CT.

Keywords: A. Powders:chemical preparation; B. Porosity; D. Al_2O_3; E. Biomedical applications

1. Introduction

Synthetic bone tissue scaffold materials are seen as an off-the-shelf option that may provide a three dimensional construct for bone forming cells that behaves as a vehicle to deliver osteogenic cells and growth factors in tissue engineering.

Porous alumina can be used as a synthetic bone tissue scaffold material or a porous ceramic prosthetic device. The success of a porous scaffold depends on its ability to provide a functional balance between mechanical strength, pore size, interconnectivity of the porous structure and properties of osteoconductivity [1,2].

Porosity is an important design characteristic to satisfy an application as a synthetic bone scaffold material. An optimal pore size 300 μm is required for bone formation and vascularisation [3]. Scaffold porosity of at least 90% is required to provide high surface area for cell interaction, sufficient space for extracellular matrix regeneration [4]. Open porosity and interconnected porosity is very important for cell infiltration and large surface area for cell anchorage leads to better tissue adhesion [5,6].

Various methods are available to produce a porous ceramic material and have been reviewed by Studart et al. [7]. These include the replication technique, use of a sacrificial template and the direct foaming technique. The replication technique uses ceramic slurry applied to polymer foam and upon sintering the polymer template is burnt out. The use of a sacrificial template incorporates sacrificial additives of any shape to produce porous ceramics whereby the sacrificial additives may burn out, decompose or evaporate. For both these methods pyrolysis of the polymer or differences in thermal expansion coefficients at increasing temperature can occur weakening the material by inducing cracks.

The direct foaming technique involves dispersing gas into a ceramic suspension, known as foaming, and subsequently sintering at high temperatures to obtain high strength porous ceramics. The dispersal of gas during foaming creates bubbles in the ceramic suspension. The direct foaming technique offers an easy, cheap, and fast alternative to produce macroporous ceramics with both open and closed porosities in the range of 40–97%. The porous structure usually exhibits improved mechanical strength.

Foaming can be achieved by the introduction of gases externally via mechanical agitation or by the use of in situ evolution of gases through thermal decomposition or chemical reaction. The authors

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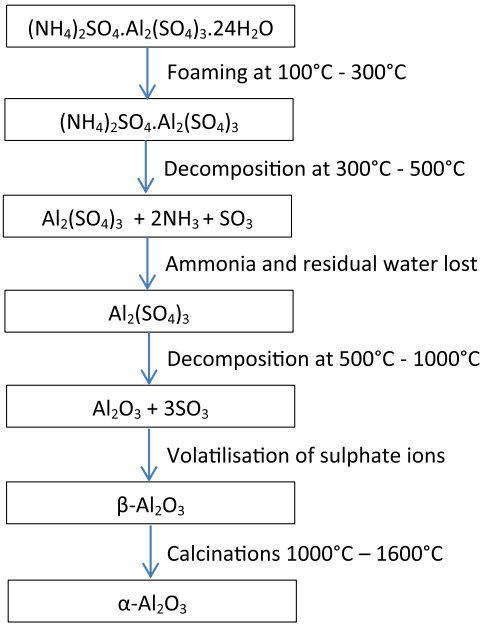

Fig. 1. Chemical synthesis of aluminium sulphate and ammonium sulphate solution.

have published a preliminary study to produce foamed porous alumina by foaming [8]. Using in situ evolution of gases, porous alumina could be synthesized by calcining blends of ammonium sulphate and aluminium sulphate with varying ammonium mole fraction (AMF) (see Fig. 1). High levels of porosity of 94% were achieved with high mechanical strength of 237 kPa which is significantly higher than a biodegradable biomaterials tissue scaffold of similar porosity levels.

The key focus of this study is on the synthesis of the foamed alumina by varying AMF, foaming temperature and sintering temperature and characterising this material using SEM, measurement of porosity, compressive strength and micro-CT. Parameters of interest in this study are presence of open and/or closed porosity, pore size distribution, average pore size, pore cell wall thickness, pore connectivity and mechanical strength.

2. Materials and methods

2.1. Synthesis of porous alumina

Porous alumina was synthesised using the chemical breakdown of ammonium sulphate and aluminium sulphate salt solutions. This method is known as the direct foaming of sulphate salt blends. The sulphate mixture undergoes a complex heating cycle in which it is first volatised, calcined and finally sintered, producing a strong porous structure.

Aluminium sulphate and ammonium sulphate were homogeneously mixed in an air tight bag before distilled water was added. The sulphate salt blends, known as alumina precursor, were dissolved in water and heated. An additional preheating phase was introduced for sets 2, 3, 4 and 5. Preheating involved heating the alumina precursor to a viscous liquid prior to the foaming step. For foaming, the solution reaches boiling temperature and foaming occurs with the evaporation of excess water content. This is known as foaming and foaming temperature was varied. With increasing temperature, the ammonium starts to decompose causing ammonia and residual water to be lost. In the final decomposition state, sulphate ions were volatised and porous alumina was obtained. The green body porous alumina is then removed from the crucible and sintered in a high temperature furnace, where the transformation β- to α-alumina occurs. The sintering temperature was also varied.

Table 1
Composition of salt used.

Ammonium mole fraction (AMF)	Ammonium sulphate (g)	Aluminium sulphate (g)	Distilled water (g)
0.09	13.21	666.45	132
0.33	13.21	133.29	36
0.50	13.21	66.645	24
0.66	26.42	66.645	36
0.91	132.1	66.645	132

The parameters of interest in this study are the ammonium sulphate mole fraction in the aluminium/ammonium sulphate blend, referred to as the ammonium mole fraction (AMF), foaming temperature and the sintering temperature.

To attain a specified AMF range, the compositions are listed in Table 1.

Foaming temperature and sintering temperature were also varied with AMF. Foaming temperatures of 600 °C/h and 100 °C/h were tested as were sintering temperatures of 1500 °C and 1600 °C with varying AMF.

2.2. Characterisation

2.2.1. Density

A direct measurement method (AS1774.5) was used to determine the density of porous alumina so as to preserve integrity of the internal cellular structure. The bulk density (D_b) of porous alumina was calculated using the equation shown below. Each sample was cut into a cubic shape and dried at 110 °C before cooling to ambient temperature. The dried mass (m_D) of the samples was then immediately recorded and measurements, for bulk volume (V), taken with a pair of digital Vernier callipers for calculation.

$$D_b = \frac{m_D}{V} \times 10^3$$

2.2.2. Porosity

The porosity value was determined according to AS1774.5 using the equation below. A theoretical density (ρ_r) of 3.894 kg/m^2 was used to calculate the bulk density (ρ_b) of porous alumina.

$$\text{Porosity } (\%) = \left[\frac{\rho_r - \rho_b}{\rho_r}\right] \times 100\%$$

2.2.3. Uniaxial compression test

The mechanical strength of porous alumina was determined in a universal tensometer (ELF3400, BOSE). Samples of one