Available online at www.sciencedirect.com

ScienceDirect

Procedia Chemistry 19 (2016) 174 – 180

Procedia Chemistry

5th International Conference on Recent Advances in Materials, Minerals and Environment (RAMM) & 2nd International Postgraduate Conference on Materials, Mineral and Polymer (MAMIP), 4-6 August 2015

Properties of Calcium Phosphate Scaffolds Produced by Freeze-Casting

Z.A. Shazni[1*], M. Mariatti[2], A. Nurazreena[3] and K.A. Razak[4]

[1] School of Materials & Mineral Resources Engineering, Engineering Campus, Universiti Sains Malaysia, Nibong Tebal, Penang, Malaysia.

*amyrashazni@yahoo.com

Abstract

The pore structure of three-dimensional scaffolds applied in tissue engineering may influence the mechanical properties and cellular activity. As the optimal pore size is dependent on the specifics of the biomaterial or tissue engineering application, the ability to alter the pore size over a wide range is necessary for several scaffolds in order to meets the requirements of the applications. The aim of this study is to develop methodologies to produce calcium phosphate scaffolds with acceptable pore size and defined pore-channel interconnectivity. The pore size of calcium phosphate scaffolds is established during the freeze-drying fabrication process. In this process, material suspension is simply frozen and then dried by freeze-drier, which able to produce material with unique porous architectures, where the porosity is almost a direct replica of the frozen solvent crystals. There are two different method of freeze-casting carried out in order to study the effect of freezing temperature by which in the first method; sample being soaked with liquid nitrogen (-196 °C) for about 10 minutes before been place inside a freezer (-40 °C). In the second method, the sample was directly placed inside a freezer for casting at temperature of -40 ^{o}C. The results show that the pore size of the scaffolds decreased as the freezing temperature was reduced. Taken together, these results demonstrate that the methodologies applied in this study can be used to produce a range of calcium phosphate scaffolds exhibiting better compressive strength, approximately 665-875 KPa for 54-64.3% of porosity with mean pore size from 102-113 µm. The methods developed in this study provide a basis for the investigation on the effects of different freezing temperature in freeze-casting process on the porosity, morphology, and compressive properties of the calcium phosphate scaffolds.

Keywords: Scaffolds; Calcium Phosphate; Freeze-casting.

---

doi:10.1016/j.proche.2016.03.090

1. Introduction

Porous scaffolds are used extensively in tissue engineering to provide a three-dimensional structure on which tissue cells can growth. The pores' structure of these scaffolds has shown significant effect on both mechanical properties and cellular activity[1]. In biomaterials and tissue engineering applications, the pore size of scaffolds must be large enough to allow infiltration of the cells toward the center of the scaffolds, which being small enough to provide sufficient density for cellular attachment[2]. Besides that, pore diameter influences the ability of diffusion process to occur within the scaffolds such as diffusion of nutrients and waste products[3]. The optimal pore size is depends on the specifics tissue engineering applications. Bone tissue repair for example, required pore size ranged in 100-150 µm which is substantial for bone ingrowth[4]. Besides that, endothelial cells show favorable attachment to pores in the range of 20-80 µm[5]. Furthermore, scaffolds must be made up of sufficient mechanical strength in order to provide support during cell growth. Therefore, for scaffolds to use in multiple applications, the ability to alter the pore size over a wide range is essential.

In this project, freeze-casting method is used to fabricate porous calcium phosphate scaffolds, which have been used in a variety of tissue engineering studies[6]. In this process, slurry of calcium phosphate is simply frozen and then dried by freeze-drier. This produces a continuous network of ice crystals throughout the body of scaffolds which able to produce scaffolds with unique porous architectures, where the porosity is almost a direct replica of the frozen solvent crystals[7-8]. As the pore structure of the scaffolds mirror the ice crystal structure formed during freezing, therefore the pore structure can be controlled by altering the freezing process used during freeze-drying such as varying the freezing temperature[8].

Previous work has investigated the effects of freezing temperature used in the freeze-casting process. It is noted that, the freezing temperature influenced the pore size. The results show that, the pore size decrease as the freezing temperature increase[9].

In this study, two different method of freeze-casting were carried out in order to study the effect of freezing temperature. In the first method, sample being soaked with liquid nitrogen (-196 °C) for about 10 minutes before been place inside a freezer (-40 °C). In the second method, the sample was directly placed inside a freezer for casting at temperature of -40 °C. Extremely low temperature of liquid nitrogen results in rapid formation of ice nuclei and the growth of small ice crystals. However, freezing process at -40 °C, results in slow ice nucleation and nuclei tend to grow into larger ice crystals which lead to the production of materials with large and random pores[10].

2. Materials and Methods

2.1 Preparation of calcium phosphate scaffolds

The porous inorganic scaffolds were produced by the freezing of calcium phosphate slurries. Slurries were prepared by mixing 50 wt% distilled water with 4 wt% of polyvinyl alcohol (PVA) as binder and 4 wt% of polyethylene glycol (PEG) as plasticizer and 50 wt% of β-TCP powder loading. Slurries were homogeneously mixed by using mechanical stirrer for 4 hours at 600 rpm. Then, homogeneous slurries were de-aired inside a vacuum dessicator with pressure supplied at 0.09 MPa to remove bubbles.

Freezing of the slurries was done by pouring them into a cylindrical polypropylene (PP) mold. In order to study the effect of freezing temperature, the slurry in the polypropylene mold was soaked into liquid nitrogen (-196 °C) for about 10 minutes before been place inside a freezer (-40 °C). This is called as a first method. Meanwhile, in the second method, the sample was directly placed inside a freezer at temperature of -40 °C. Next, frozen samples were freeze dried using FreeZone® 4.5 Liter Benchtop Freeze Dry Systems, Labconco at very low vacuum pressure 0.200 mBar and at low temperature of -47 °C. Sintering of the green bodies was done in an air furnace using Carbolite CWF 1100 at temperature 1100 °C.

Apparent porosity was derived from the density measured by Archimede's method. The microstructure of the samples was analyzed by scanning electron microscopy (SEM) (Model: Hitachi TM3000 Table Top).