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The effect of microwave sintering on the microstructure and properties of calcium phosphate ceramic

Chuthathip Mangkonsu^a, Ishikawa Kunio^b, Lila Bunhan^c
Radzali Otman^{d*}, Ahmad-Fauzi Mohd Noor^{a**}

^aSchool of Materials and Mineral Resources Engineering, Universiti Sains Malaysia, Penang 14300, Malaysia

^bDepartment of Biomaterials, Faculty of Dental Science, Kyushu University, 3-1-1 Maidashi, Higashiku, Fukuoka 812-8582, Japan

^cDepartment of Industrial Engineering, Faculty of Engineering, Burapha University, Chonburi 20131, Thailand

^dFaculty of Manufacturing Engineering, Universiti Teknikal Malaysia Melaka, Malacca 76100, Malaysia

Abstract

Despite good ability to improved biological affinity and activity of calcium phosphate (CP) ceramic to the surrounding host tissue when implanted, CP ceramic is capable to encourage direct bond with bone as their chemical compositions are similar to the mineral phase of bone. However, the low mechanical property of CP ceramic restricts use in load-bearing applications. Therefore, the primary aim of this study was to fabricate dense CP ceramic via used of microwave sintering analyze the microstructure and mechanical properties comparison to conventional ceramic sintering. The sintered specimens were characterized by their surface microstructure, density measurement and hardness test. The result demonstrated that the density and hardness values of sintered CP ceramic specimens by microwave sintering were higher than conventional sintering. The microstructures of CP ceramic microwave sintered specimens show also better microstructures (analyzed by FESEM technique), with fines grain size and the present of apatite layer growth on the surface when examined with SBF solution. It can be concluded that the microwave sintering enhanced better microstructure and mechanical properties of CP ceramic.

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* Corresponding author. Tel.: +0-459-410-11; fax: 0-459-410-11.

** Correspondence to: M. N. Ahmad Fauzi, School of Materials and Mineral Resources Engineering, Universiti Sains Malaysia, Penang 14300, Malaysia *E-mail address:* radzali@utem.edu.my (R. Otman.), srafauzi@usm.my (M. N. Ahmad-Fauzi)

1. Introduction

The applications of calcium phosphate (CP) have been used in medical and dentistry for more than 20 years because of their excellent biocompatibility, bioactivity, and osteoconduction characteristics¹. Despite their biological properties being excellent, the disadvantage such as the low degradation rates (in the case of hydroxyapatite, HA), poor mechanical properties, and low osseointegration ability limit their clinical performance². Many attempts have been studied to overcome these disadvantages by used of various techniques to fabricate better property of CP ceramic. Sintering is necessary for all ceramic bodies to produce a microstructure with the required properties. Sintering greatly affect the final product properties since it enhance the optimal chemical composition and final microstructure design, which influence the mechanical performance and the biological behavior of calcium phosphate ceramics³. Generally, the purpose of sintering is to produce sintered part with reproducible and, if possible, designed microstructure through control of sintering variables. In general terms, microstructural control is defined as a control of grain size, sintered density and distribution of other phases including pores. Commonly, the final purpose of microstructural control is to prepare an entire sample with fine grain structure⁴.

During the last 15 years, several researchers have investigated the use of microwave energy to sinter ceramic materials⁵. Microwave sintering has attracted great attention due to its various advantages. Since the performance of microwave heating encourages the transfer of energy directly into the materials, thus more volumetric heating and thus permitting higher heating rate obtained⁶. Consequently, the advantages of microwave sintering in terms of energy consumption and processing cost are reduced, enhanced diffusion processes, very rapid heating rates and significantly reduced processing times as well as improved physical and mechanical properties⁷. The fundamental difference between microwave heating and conventional radiant heating is the volumetric heating. Conventional furnace heats sample using surface heating mechanism, and depending on the heating rate, creates a large thermal gradient from the surface to the center of the sample, particularly having poor thermal conductivity⁸. In contrast, microwave heating is a fast sintering process where energy can be deposited volumetrically throughout the material rather than relying on thermal conduction from the surface⁹. Herein, this study was carried out by using microwave and conventional sintering to fabricate dense CP ceramic. In order to demonstrate the effect of microwave sintering to the microstructure and mechanical properties of sintered CP ceramics, the same condition and parameters were used for both types of sintering.

1. Experimental procedure

1.1. Specimens preparation

The raw material used in this study was hydroxyapatite precursor powder as synthesized by a wet precipitation method in the Rekagraf Laboratory (USM). Calcium phosphate powder was pressed by uniaxially cold pressing into a cylindrical pellet form in a 12 mm diameter stainless steel die (with an applied pressure of 100 MPa) using a Specac manual hydraulic press machine.

1.2. Densification study

The specimens were sintered at different temperatures of 1200, 1250, 1300, 1350 and 1400 °C with 10 °C/min heating rate and 2 hours soaking time. For microwave sintering, the specimens were sintered in Syno Therm microwave furnace, while conventional sintering was sintered in Lenton muffle furnace. The apparent density was measured using Archimedeian principal on six specimens. In the microwave sintering, the sintered specimens at 1400 °C were cracked and surfaces were damaged specially on the edge area of the specimens. This defect resulted in the inability to measure the dimensions of specimens and could not be use for subsequent test. Therefore, for microwave sintering, the firing process was terminated at 1350 °C.

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