

Calcination Effects on the Sinterability of Hydroxyapatite Bioceramics

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Abstract— The sinterability of calcined synthesized HA (700°C to 1000°C) was investigated over the temperature range of 1050°C to 1350°C in terms of phase stability, bulk density, Young's modulus and Vickers hardness. Calcination has resulted in higher crystallinity of the starting synthesized HA powder. Decomposition of HA phase to form secondary phases was not observed in the present work for the calcined powders. The results also indicated that calcination of the HA powder prior to sintering has negligible effect on the sinterability of the HA compacts (up to 900°C). Further treatment at 1000°C was found to be detrimental to the properties of sintered HA.

Keywords— Hydroxyapatite, Calcination, Mechanical properties, Sinterability.

I. INTRODUCTION

The use of hydroxyapatite (HA) as a potential bioactive calcium phosphate ceramics has gained popularity in recent years due to its close chemical resemblance with the mineral components of natural bone and teeth [1], [2]. Several studies have been carried out and the results demonstrated that HA can accelerate initial biological response with host tissues at the implanted site in the body and improves the bone-implant adhesion [3],[4]. Evidence of rapid bone formation and subsequently healing around damaged sites in the body were also observed [5], [6]. Due to these excellent biocompatibility properties, HA ceramics have been widely used in many medical, orthopaedic and dental applications including the augmentation of the jaw, dental implants, spinal surgery, maxillofacial surgery and artificial middle ear implants [1], [2].

However, one of the major setbacks of HA is the poor mechanical properties inherited by the sintered body [6]. Owing to the brittle nature and the low fracture toughness ($< 1 \text{ MPam}^{1/2}$) of the sintered HA, such implants could only be utilised successfully in non-load bearing applications [7]. Therefore, the development of bioactive HA that has improved and ultimately bone-like mechanical properties are desirable. As a result, a great number of studies have been devoted to improve the mechanical properties of HA materials [4-8].

The success of HA ceramic in biomedical application is largely dependent on the availability of a high quality, sintered HA that is characterized having refined microstructure and improved mechanical properties [9]. Intensive works on HA involving a wide range of powder processing techniques; composition and experimental conditions have been investigated with the aim of determining the most effective synthesis method and conditions to produce well-defined particle morphology [10-12]. Various researchers have reported that calcination process would increase the degree of crystallinity of the initial particles [13]. Nevertheless, works on the sinterability of calcined powders are scarce. Thus, the objective of this work is to study the sinterability of synthesized HA powders calcined at various temperatures ranging from 700°C to 1000°C.

II. METHODS AND MATERIALS

In the current work, the HA powder used was prepared according to a wet chemical method comprising precipitation from aqueous medium by slow addition of orthophosphoric acid (H_3PO_4) solution to a calcium hydroxide ($\text{Ca}(\text{OH})_2$) [14]. The synthesized HA powder were calcined in air atmosphere at temperature ranging from 700°C to 1000°C at 10°C/min. and, after a dwell time of 2 h, cooled to room temperature at 10°C/min. The HA powder was compacted into disc and rectangular bar samples and subsequently cold isostatic pressed at 200 MPa (Riken Seiki, Japan) prior to sintering. This was followed by consolidation of the particles by pressureless sintering performed in air using a rapid heating furnace over the temperature range of 1050°C to 1350°C, with ramp rate of 2°C/min. (heating and cooling) and soaking time of 2 hours for each firing. All sintered samples were then polished to a 1 μm finish prior to testing.

The calcium and phosphorus content in the synthesized HA powder were determined by using the Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) technique. The specific surface area of the powder was measured by the Brunauer-Emmett-Teller (BET) method. Nitrogen gas adsorption analysis was performed on a Coulter SA 3100 Analyzer. Samples were outgassed at 150°C for