



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

Synthesis and sintering-wet carbonation of nano-sized carbonated hydroxyapatite

W. Y. Wong and Ahmad-Fauzi Mohd Noor***

School of Materials and Mineral Resources Engineering, Universiti Sains Malaysia, 11400 Nibong Tebal, Penang, Malaysia

Abstract

Synthetic carbonated apatite ceramics are considered as promising alternative to auto- and allograft materials for bone substitute. In this study, Carbonated hydroxyapatite (CHA) was synthesized by nanoemulsion method. The powder produced was B-type CHA in nano-sized and had 8.25% carbonate content. The CHA samples were made into pellets and were sintered to 800°C. Upon cooling down to 150, 200, 250 and 300°C, carbonation with wet CO₂ was performed on the CHA in a desiccator to re-compensate the carbonate loss due to sintering and improve densification. The aim of this study was to investigate and compare the effect of cooled down temperatures on dense CHA with two kind of wet CO₂ atmospheres: direct wet CO₂ and dry CO₂ through water. Sintered CHA carbonated by using dry CO₂ through water had overall higher amount of carbonate content as compared to carbonation from wet CO₂ directly from tank. D200, sample undergone carbonation by carbonated by dry CO₂ through water at 200°C had the highest carbonate content (3.35%).

© 2016 The Authors. Published by Elsevier B.V. This is an open access article under the CC BY-NC-ND license

(<http://creativecommons.org/licenses/by-nc-nd/4.0/>).

Peer-review under responsibility of School of Materials and Mineral Resources Engineering, Universiti Sains Malaysia

Keywords: Bioceramic ; carbonation ; carbonated hydroxyapatite

1. Introduction

Bone is a rigid hard organ in human body and susceptible to fracture due to injury or diseases¹. One of the treatment to assist in healing is to use synthetic biomaterials as the replacement of the bone lost part²⁻⁵. Human bone consists of inorganic substance which was formed mostly of calcium phosphate⁵⁻⁸. Hydroxyapatite (Ca₅(PO₄)₃OH)

* Corresponding author. Prof., Ph.D.; Tel.: +60 4 5996174; fax: +60 45941011.

E-mail address: srafauzi@usm.my

(HA) is a naturally occurring mineral which is chemically similar to the mineral component of bones and hard tissues in mammals, it is often being used as a substituent of loss part of bone^{6,9,10}.

Currently, the trend of bone substitute material is inclined to regenerative approach where the fast resorption of the implant material and simultaneous filling with newly formed bone and the substituted part, instead of permanently stay at the replaced side^{1,11,12}. However, a stoichiometric of pure HA is chemically stable which results in its bioresorption rate being slow for the replacement of new growing bone tissue^{4,8,11,13}. Thus, carbonated substituted hydroxyapatite ceramics are considered as promising alternative to HA-based bone substitute^{11,14,15}. Inorganic component of human bone contains about β to 8 wt. % of carbonate ions depending on the individual's age^{15,16}. Substitution of carbonate ion promotes the solubility or resorption of HA^{17,18}. Hence, carbonated hydroxyapatite (CHA) is potentially better as its chemical composition approximates bone tissue more accurately.

Carbonate ions can substitute at two sites in HA structure, namely hydroxyl and phosphate ions positions, give rise to A-type and B-type of CHA^{10,15,19}. The amount of A/B ratio in calcium phosphate of human bone mineral is depending on the age, A-type CHA is found in old bone tissue while B-type is in young bone tissue^{10,17}. B-type of carbonate substitution is preferred due to it is commonly found in young tissue, and responsible for the decrease of crystallinity and subsequently increases its solubility^{18,20}.

CHA powder can be produced by nanoemulsion method, which is able to produce nano size CHA particles²¹. Carbonate is incorporated into the structure by using some carbonate ions source during this process²². Sintering CHA at high temperature will cause carbonate to decompose into pure HA and calcium oxide with the released of carbon dioxide^{11,14,23}. The elimination of carbonate ions affect both the biological and mechanical properties, so the processing condition chosen should hinder the decomposition or reduce the amount of carbonate loss²³. In recent research work, the effect of carbonate content and sintering atmosphere has been undertaken to investigate thermal stability of CHA. Heat treatment were performed in various gas atmosphere, such as nitrogen, carbon dioxide, air, water vapour and wet oxygen. The gas atmosphere was revealed to affect the decomposition significantly^{20,24}. Barinov et al.¹⁵ had reported that B-type CHA did not show evidence of decomposition by sintering in wet CO₂.

Consequently in research work by others, the effect of carbonate content and sintering atmospheres has been undertaken to investigate thermal stability of CHA. Heat-treatments were performed in various gas atmospheres, including nitrogen, carbon dioxide, air, water vapor and wet oxygen. The gas atmosphere used was revealed to affect the decomposition significantly²⁴. According to Landi et al.²⁴, thermal treatment in wet CO₂ gave the best result in terms of high carbonate residue with low A/B ratio in the range of the biological CHA. In previous work done by Yanny-Marliana²⁵, CHA with addition of Mg(OH)₂ as the sintering aid and sintered to 800° achieved highest relative density²⁵. Besides, carbonation at 200°C during cooling stage with wet CO₂ was to recompensate the carbonate loss. In this study, two different wet CO₂ atmospheres were used: (1) Wet CO₂ direct from cold tank and (2) Dry CO₂ flowing through water, during cooling at 150, 200, 250 and 300°C.

2. Experimental

2.1. CHA Powder Synthesis

Nanoemulsion method was used to synthesis CHA powder, adopted from Zhou et al.²¹, where it can produce powder in very fine and uniform droplet sizes, typically in the range of 20-200nm. To synthesis the powder, Ca(NO₃)₂·4H₂O (Merck, Germany) provides calcium source and (NH₄)₂HPO₄ (Merck, Germany) provides phosphate source while carbonate source was provided by NH₄HCO₃ (Sigma-Aldrich, USA). The molar ratio of Ca/P was fixed to 1.67 according to the molar ratio in the biological bone, and while PO₄³⁻/CO₃²⁻ is 1. The synthesis was done in room temperature.

The powder obtained was added with Mg(OH)₂ sintering aid. Dry mixing was performed for 5 hours to make sure the powder has a homogeneous composition of mixture. Next, the CHA powder was compacted into pellets using a 13 mm diameter of stainless steel die. The powder was pressed at a pressure of 100MPa and held constant for 120 seconds so that the pressure was uniformly applied on the pellets.

Download English Version:

<https://daneshyari.com/en/article/239799>

Download Persian Version:

<https://daneshyari.com/article/239799>

[Daneshyari.com](https://daneshyari.com)