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Original Research Paper

Continuous production of hydroxyapatite powder by drip pyrolysis in a fluidized bed

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ABSTRACT

Hydroxyapatite (HAp) powder was produced by drip pyrolysis in a fluidized bed (DPFB) at 913–1113 K with dry air. In this study, HAp powders derived using different concentrations of a solution-type precursor material were compared with those derived using slurry-type precursor materials. From a solution-type precursor containing calcium nitrate and ammonium phosphate dibasic, fine HAp powders with mean particle sizes of approximately 8–40 μm were produced, depending on the solution concentration and reaction temperature. In these cases, bimodal particle size distribution was observed. Spherical alumina of 250 μm was found to be superior to silica sand of 270 μm as the coarse medium particles in reducing contamination of the product powder. The thermal stability of HAp derived from a slurry-type precursor after 1-day aging was improved by DPFB. HAp powders derived from the solution-type precursor material were Ca-deficient, and their Ca/P molar ratio increased with bed temperature. In contrast, the Ca/P molar ratio of HAp powders from the slurry-type precursor material was nearly stoichiometric. Morphology strongly depended on the starting precursor material. The solid collection ratio was closely related to the population of product particle sizes larger than 25 μm.

1. Introduction

Hydroxyapatite (HAp) is a functional material having affinity for living bodies and adsorptivity of water vapor, protein, and ions. The synthesis routes of HAp are categorized into several groups [1] such as precipitation [2], hydrolysis [3], solid state synthesis [4], hydrothermal synthesis [5,6], pyrolysis [7–9], mechanochemical [10], sol–gel [11], and sonochemical syntheses [12]. In the precipitation method, the evolution of monophasic HAp requires long time, during which the initial precipitate of the HAp precursor (usually amorphous calcium phosphate with molar ratio Ca/P = 1.50) gradually crystallizes to HAp (Ca/P = 1.67) [13]. The precipitated HAp powder is then dried, pulverized, and classified to the final product dry powder. This process requires several operation steps, which make it difficult to control the operating conditions for suitable production of the powder.

Pyrolysis, a powder preparation technique via liquid phases, is especially effective for the preparation of HAp-like materials that contain the hydroxyl group in a crystalline structure because the operation is performed in a steam atmosphere. The spray-pyrolysis method can prepare HAp powders directly via liquid phases, thus simplifying the production process [7–9]. However, additional heat treatment of the powder product is often required because the residence time of the product powder in the reactor is ~1 s. Moreover, the low liquid-to-gas ratio of liquid reactant to dispersion gas results in rather low per unit volume productivity of the reactor. Therefore, this process requires a large reactor volume for the mass production of HAp powder.

In the present study, to develop a more compact reactor with fewer operating steps, drip pyrolysis in a fluidized bed (DPFB) was applied to the continuous production of HAp powder. In the DPFB process, a fluidized bed of coarse medium particles provides an isothermal field for simultaneous evaporation of droplets of raw material solution or slurry, pyrolysis of generated precursor powder, and crushing and heat treatment of product powder. The product powder is entrained by gas flow and collected by a gas–solid separator. This process takes advantage of an isothermal operation owing to the fast heat transfer between the fluidized particles and hot gas [14] and a long residence time of the product powder due to its adhesion to the surface of fluidized particles [15]. Because the surface area of the fluidized particles is considerably large, mass production of the product powder carried by exhaust gas is possible owing to the difference in terminal velocity between the coarse and fine particles. The coarse, inert fluidized particles act as a supporter of the precursor or product powder and are instrumental in

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