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Mechanochemical synthesis of nanohydroxyapatite bioceramics

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Nanosized hydroxyapatite powder has been synthesized by the mechanochemical method using a dry mixture of calcium hydroxide and diammonium hydrogen phosphate. The effect of mechanochemical process on powder properties has been investigated. Three rotation speeds of 170 rpm, 270 rpm and 370 rpm have been employed with 15 hours milling time. Characterization of the nanopowders has been accomplished by Fourier transform infra red, X-ray diffraction and transmission electron microscopy analyses. The samples have been prepared and sintered in air at varying temperatures ranging from 1050-1350 °C. Results show that increase in rotation speed (370 rpm) increases the crystallite size (9-21 nm). Agglomerate formation with irregular shapes is found in the samples prepared at 270 and 370 rpm. The sintering process influences the stability of powder by yielding TCP phase at all the sintering temperatures. At 370 rpm, the sample sintered at 1250 °C shows the maximum relative density of 95.3% as well as hardness of 5.3 GPa.

Keywords: Mechanochemical synthesis, Nanomaterials, Hydroxyapatite, Bioceramics

Hydroxyapatite (HA) is commonly used for a number of biomedical applications in the forms of granules, blocks[1], coatings and dense bodies[1-5] for bone augmentation. HA has also been found useful for drug delivery and antibiotics[6]. It exists naturally in human bone as crystals within collagen. The high strength is necessary for reliable implant in the body[7]. Many improvements have been made to overcome the limitation of HA in loading application by controlling microstructures via novel sintering techniques or utilization of nanopowders[8]. Development of dense HA ceramics with superior mechanical properties is possible if the starting powder is stoichiometric with improved powder properties such as crystallinity, agglomeration and morphology[1-3]. Nanoscale grain size in dense sintered materials is a desired parameter to enhance the mechanical and biological properties of HA-based bioceramic materials[9]. Several methods have been applied for synthesizing HA nanocrystalline powder like wet precipitation[10], sol-gel[11], hydrothermal[12] and mechanochemical[13].

Mechanochemical ball milling has been used since 1922 wherein the materials components are synthesized by deformation process through ball-particle, particle-wall, and particle-particle collisions[14] at a particular time, leading to the chemical reaction between particles to form new nanosize composites or powders. It is a simple and low cost method compared to other techniques, and has recently received attention as an alternative route in preparing materials characterized by better biocompatibility with natural bone[13]. Synthesis of HA through mechanochemical milling can be in either a wet medium[15] or under dry condition[13]. The dry mechanochemical method is reported to be more beneficial than the wet mechanochemical method due to faster reactions in absence of water. In addition, the dry condition provides a lower level of contamination by the mill material[16], whereby powders obtained can be used directly without filtering and drying[16] stage, as compared to under wet conditions. Some studies have used dry mechanochemical method in producing calcium phosphate bioceramics under various milling times[13], fluoride substituted HA[17] and nanocomposite HA[18]. In this work the properties of HA prepared via the mechanochemical method was investigated.