



Influence of titania content on mechanochemical synthesis of chlorapatite–titania composite nanopowders

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Abstract

The effect of titania content on mechanochemical synthesis of chlorapatite–titania composite nanopowders was studied for the first time. According to the obtained data, the phase compositions, structural features as well as morphological characteristics of the composites were influenced by the titania content. In the presence of 3–5 wt% titania, milling for 5 h resulted in the formation of chlorapatite–titania composite nanopowders. The crystallite size of the samples was around 25 ± 1 and 23 ± 1 nm in the presence of 3 and 5 wt% titania, respectively. With increasing the titania content to 7 wt%, no chemical reaction happened during the milling. The composite nanopowders showed high volume fraction of grain boundaries. Based on the FT-IR results, the products had high chemical purity which is very important in biomedical applications. The TEM images indicated that the composite nanopowder was composed of spheroidal particles with a mean size of around 35 nm. The proposed synthesis strategy provides a facile pathway to obtain novel chlorapatite-based composite nanopowders with high purity and optimal properties.

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1. Introduction

During the last few decades, advances in bioceramics have made significant contribution to the development of modern health care industry and have improved the quality of human life [1]. The superior biocompatibility of calcium phosphates (CaP) has allowed them to be used for various biomedical applications [2]. Besides this field, CaP-based materials have been investigated for other applications such as drug delivery [3], gene therapy [4], chemical sensors [5], catalysts [6], migration barrier [7], electronic [8], chromatography [9], and waste water remediation [10]. Among different forms of CaP, particular attention has been placed to apatites ($M_{10}(XO_4)_6Y_2$) due to their outstanding biological responses to the physiological environment [11]. In apatite formula, M can be substituted by a large number of various ions like Ca^{2+} , Mg^{2+} , Sr^{2+} , Ba^{2+} , Mn^{2+} , Fe^{2+} , Zn^{2+} , Cd^{2+} , Pb^{2+} , H^+ , Na^+ , K^+ , Al^{3+} and so on. In addition, XO_4 can be replaced by PO_4^{3-} , AsO_4^{3-} , VO_4^{3-} , SO_4^{2-} , CO_3^{2-} , and SiO_4^{3-} , while Y can be substituted by

several anions like OH^- , F^- , Cl^- , Br^- , O^{2-} , and CO_3^{2-} . Among them, hydroxyapatite (HAp, $Ca_{10}(PO_4)_6(OH)_2$) is one of the most common materials for artificial bone and tooth applications due to its structural and biological properties [12–14]. Nevertheless, synthetic HAp (s-HAp) has intrinsically high dissolution rate in a biological system, poor corrosion resistance in an acidic environment, and poor chemical stability at high temperature [15,16]. Therefore, many efforts have been made recently to modify the functional specifications of HAp [17–20].

According to literature, the chemical and physical properties as well as bone bonding capability of s-HAp can be sensibly improved by the incorporation of selected ions within the apatite lattice [21]. In fact, these substitutions play a pivotal role in the biological activity, solubility, surface chemistry, morphological features and so on [22,23]. For example, the substitution of OH^{-1} groups by F^{-1} ions in the HAp lattice causes an increase in crystallinity degree and chemical stability as well as a decrease in crystal strain [24,25]. Also, Si-substituted HAp have shown improved biological performances in terms of enhanced bone apposition, bone in-growth and cell-mediated degradation [22]. It has been reported that

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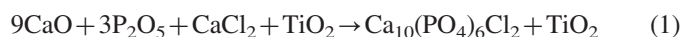
the presence of Mg^{2+} within HAp lattice affects effectively crystallization behavior, thermal stability, and the formation of biphasic calcium phosphates (BCP). Besides, it is well known that trace quantities of cations (i.e. Zn^{2+} and Sr^{2+}) and/or anions (i.e. SiO_4^{3-} , F^- , and CO_3^{2-}) in HAp play an important role in its overall biological performances [26]. In another case, the substitution of Cl^{-1} ions in HAp leads to the formation of an acidic environment on the surface of bone which causes the activation of the osteoclasts in the bone resorption process [27]. On the other hand, the incorporation of bioinert ceramics into the CaP improves their mechanical properties [28,29]. An ideal reinforcing material for the apatite-based composites which satisfies all of the requirements, has not yet been found. Accordingly, synthetic calcium phosphate-based composites are under continuous change. Although a variety of modified-HAp structures were known, only a limited number of studies have been conducted on the substitution of the chloride ion in the apatite [30,31]. So far, the incorporation of Cl^{-1} ions in HAp has been carried out by hydrothermal treatments [32], aqueous precipitation method [30], and mechanochemical process [33]. According to the results of previous studies, the extent of chlorine substitution depends on the concentration of the precursors and manufacturing process parameters [30–33]. Among these methods, the mechanochemical process is a simple dry method to obtain any quantity of powder with controlled microstructure [34]. The powder synthesized using a mechanochemical reaction usually possesses a well-defined structure due to the perturbation of surface-bonded species caused by pressure [35]. This method has benefit of simplicity, reproducibility, and low processing cost [36]. In addition, melting and precisely controlled conditions are not essential and the products have nanostructural characteristics [37]. In the field of bioceramics, high efficiency of this method opens a new way to produce commercial amount of nanostructured calcium phosphate-based materials [38]. Hence, when the mass production of calcium phosphate-based nanopowders is required, mechanochemical method as employed in this research can be served.

According to our recent studies [33,39], chlorapatite nanopowders (n-CAP) can be produced by a facile solid-state process. Results showed that the formation of n-CAP was effectively influenced by the milling time. In addition, during annealing in the range 900–1300 °C for 1 h, the decomposition

of chlorapatite to tricalcium phosphate (β -TCP), tetracalcium phosphate ($Ca_4P_2O_9$), and hydrochloric acid occurred. However, the rate of decomposition increased gradually with increasing the annealing temperature to 1300 °C. In addition, we found that the formation of CAP-based composite nanopowders was effectively influenced by the zinc oxide content [40]. In the present study, the effect of titania content on mechanochemical behavior of the CaO – $CaCl_2$ – P_2O_5 system was investigated. To the best of our knowledge, it is the first study of mechanochemical synthesis of chlorapatite–titania (CAP– TiO_2) composite nanopowders. The phase compositions, structural features as well as morphological characteristics were evaluated to determine the optimum composition.

2. Materials and methods

The raw materials were commercial grade calcium oxide (CaO), calcium chloride ($CaCl_2$), phosphorous pentoxide (P_2O_5), and titanium dioxide (TiO_2). All chemicals were purchased from Merck and used without purification. Fig. 1 shows a schematic view of the mechanochemical synthesis of CAP– TiO_2 composite nanopowders. In all the experiments, the mole ratio of calcium to phosphorous was equal to 1.67 which was in accordance with the stoichiometric Ca/P content in the composition of chlorapatite. Mechanochemical process was carried out in a high energy planetary ball mill at rotational speed of 600 rpm and BPR of 20:1 under ambient air atmosphere using tempered chrome steel vials (volume 125 ml) and balls (20 mm in diameter). To synthesize CAP– TiO_2 composite nanopowders, the different amounts of titania (0, 3, 5, and 7 wt%) were mixed with appropriate amounts of CaO, P_2O_5 , and $CaCl_2$ and then the mixed powders were mechanically activated for 5 h at room temperature without using any process control agent (PCA). The stoichiometry for the overall reaction of mechanochemical synthesis is the following reaction:



X-ray diffraction (Philips X-ray diffractometer (XRD), Cu – K_α radiation, 40 kV, 30 mA and $0.02^\circ S^{-1}$ step scan) was employed to evaluate the phase purity and the crystallographic structural properties of CAP– TiO_2 nanocomposites. The XRD patterns were collected over the 2θ angular range between 10° and 90° at scan speed of $1^\circ/min$. “PANalytical X’Pert HighScore” software

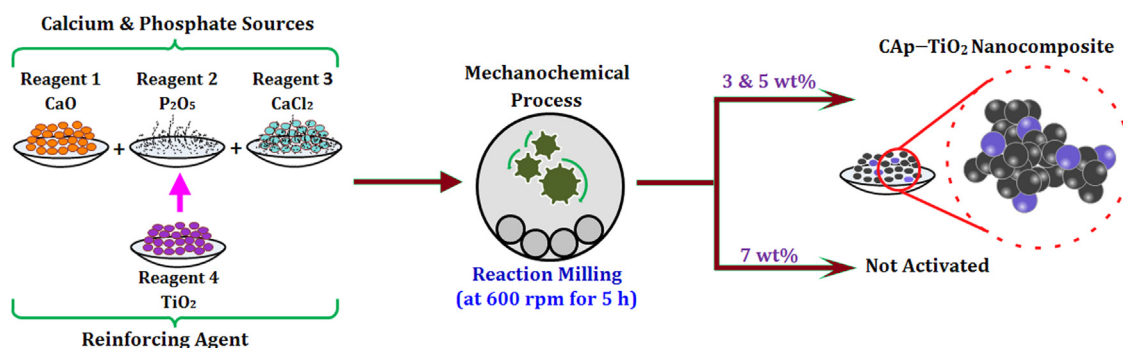


Fig. 1. A schematic view of the mechanochemical synthesis of CAP– TiO_2 composite nanopowders.

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