



Nano-alumina/hydroxyapatite composite powders prepared by in-situ chemical precipitation

Congyi Zhang^a, Xihua Zhang^{a,*}, Changxia Liu^b, Kai Sun^a, Jianjun Yuan^a

^aKey Laboratory for Liquid–Solid Structural Evolution and Processing of Materials (Ministry of Education), Shandong University, Jinan 250061, China

^bKey Laboratory of Advanced Manufacturing and Automation Technology, Ludong University, Yantai 264025, China

Received 22 July 2015; received in revised form 23 August 2015; accepted 24 August 2015

Available online 30 August 2015

Abstract

Nano-alumina/hydroxyapatite composite powders for biomedical applications were synthesized by in-situ chemical precipitation. The effects of synthesis parameters and calcination on the properties of the composite powders were studied. The morphology, chemical composition and thermal behavior of the composite powders were investigated by field emission scanning electron microscope (FESEM), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR) and differential scanning calorimetry (DSC). The results showed that crystallinity of the composite powders progressively increased with increasing the synthesis and calcination temperatures. During calcination, the hydroxyapatite was transformed into calcium phosphate (β -TCP) at 1100 °C, while the aluminum hydroxide was converted to γ -alumina and θ -alumina. In addition, aggregations were observed for samples heated at 1000 °C and above. The as-synthesis alumina powders with an average particle size of approximately 30 nm were well distributed around the HA particles.

© 2015 Elsevier Ltd and Techna Group S.r.l. All rights reserved.

Keywords: Hydroxyapatite; Alumina; Composite powders

1. Introduction

Hydroxyapatite (HA) is the main inorganic component of human bones and teeth, which is considered to be the most promising materials for bone replacement and repair [1]. In recent years, HA has attracted considerable attention due to its excellent biocompatibility and bioactivity, as well as fluorescence [2], magnetism [3] and adsorption properties [4]. However, the poor mechanical properties of synthesized HA, especially low strength and brittle nature restrict its applications [5]. With the purpose of overcoming these disadvantages, reinforcements such as ceramic particles, graphene and carbon nanotubes have been used to improve the mechanical properties of HA [6–9]. Being a bio-inert

material, Al_2O_3 exhibits excellent mechanical properties and wear resistance. Moreover, mechanical properties of HA and HA coatings can be improved by addition of Al_2O_3 [10–13].

A variety of methods have been reported for the preparation of HA composites such as chemical precipitation process [14], sol–gel process [15,16] and high energy ball milling [17]. Compared with other preparation methods, the in-situ chemical precipitation has been widely studied for synthesis of HA composites due to mild conditions, low cost, ease of work-up and promising large-scale production. So far, several HA matrix composites such as TiO_2/HA [18], $\text{Fe}_3\text{O}_4/\text{HA}/\text{chitosan}$ [19] and chitosan/carbon nanotubes/HA [20] have been prepared by in-situ chemical precipitation. This paper aimed to study the feasibility of producing the nano- $\text{Al}_2\text{O}_3/\text{HA}$ composite powders via in-situ chemical precipitation. The effects of synthesis conditions and calcination on the structure and morphology of the prepared powders were also investigated.

*Corresponding author. Tel.: +86 053188365056.

E-mail address: zhangxh@sdu.edu.cn (X. Zhang).

¹Present/permanent address: Shandong University, No. 17923, Jingshi Road, Jinan, Shandong Province, PR China.

2. Experimental

2.1. Materials

Calcium nitrate tetrahydrate ($\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$), diammonium hydrogen phosphate ($(\text{NH}_4)_2\text{HPO}_4$) and aluminum nitrate nonahydrate ($\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$) as raw materials were used to prepare the HA/ Al_2O_3 composite powders. 30% ammonia solution was added to adjust the pH values. Polyethylene glycol (PEG600 and PEG1500) and cetyltrimethyl ammonium bromide (CTAB) acted as dispersants. All the chemicals purchased from Shanghai, China were analytical grade and used without further purification.

2.2. Synthesis of nano- Al_2O_3 /HA composite powders

Nano- Al_2O_3 /HA composite powders were prepared by in-situ chemical precipitation. In order to discuss the preparation conditions, 20 wt% of Al_2O_3 was chosen in this work. 0.2 M $(\text{NH}_4)_2\text{HPO}_4$ aqueous solution (360 mL) and 0.2 M $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ aqueous solution (295 mL) were successively added drop-wise to 0.2 M $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ aqueous solution (600 mL) at different temperatures (30, 50 and 70 °C), while constantly stirring. The pH was maintained at 10–11 with ammonia solution during reaction. The resulting slurry was aged at room temperature for 24 h. Then, the precipitate was dried at 80 °C for 24 h followed by repeatedly washing with deionized water and absolute alcohol.

To investigate the effects of surfactants on the morphology of Al_2O_3 /HA precursors, 500 mg of PEG600, PEG1500 and CTAB were dissolved in the 0.2 M of $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ aqueous solution, respectively. Then, subsequent precipitation process at 70 °C was performed. The nano- Al_2O_3 /HA precursors obtained with PEG1500 were calcined at 500, 900, 1000 and 1100 °C for 1 h with a heating rate of 10 °C/min, respectively.

2.3. Characterization

X-ray diffraction (XRD) measurements were carried out using Rigaku Dmax-2500PC to determine the crystalline phases present in the composite powders. Fourier transform infrared (FT-IR) spectroscopy was recorded by the KBr pellet technique using a Bruker tensor 37 spectrometer for the range 400–4000 cm^{-1} . Field-emission scanning electron microscope (FESEM) study was obtained on a Hitachi SU-70. The differential scanning calorimetry (DSC) was performed using NETZSCH DSC 404 C from 90 to 1200 °C with a heating rate of 10 °C/min.

3. Results and discussion

3.1. Phase analysis

The XRD patterns of nano- Al_2O_3 /HA precursors synthesized at different temperatures are shown in Fig. 1. As seen, the XRD patterns of the samples prepared at 30 and 50 °C

exhibited broad peaks, indicating lower crystallinity of the structure. While the crystallinity of the precursors progressively increased with increasing synthesis temperatures. The XRD results confirmed the existence of HA (JCPDS 09-0432) and bayerite (JCPDS 20-0011), and the crystallinity of the precursors showed a sharp increase at 70 °C. Research has been reported that 60 °C is a transition temperature. Below that temperature the HA crystals are monocrystalline, while above this temperature the HA crystals become multicrystalline [21].

Fig. 2 shows XRD patterns of nano- Al_2O_3 /HA composite powders calcined at different temperatures. The crystallinity of the samples showed great improvement after calcination. However, the beta-calcium phosphate (β -TCP) phase was detected at 1100 °C due to the dehydroxylation and decomposition of HA [22]. Besides, the XRD spectra did not contain any peaks related to Al_2O_3 , while it could be observed that the peaks of HA shifted slightly to the lower reflex angles. In

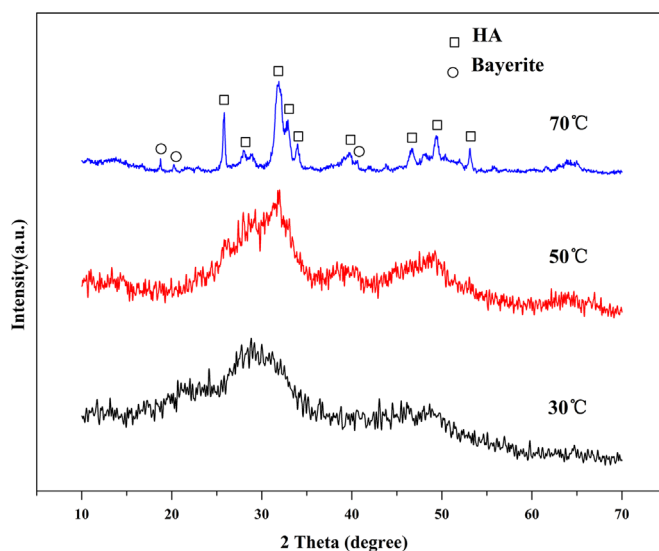


Fig. 1. XRD patterns of nano- Al_2O_3 /HA precursors at different temperatures.

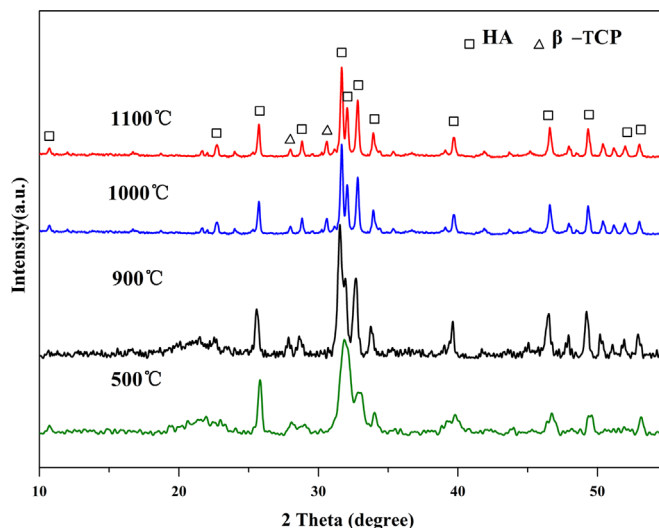


Fig. 2. XRD patterns of nano- Al_2O_3 /HA composite powders after calcination.

Download English Version:

<https://daneshyari.com/en/article/1459430>

Download Persian Version:

<https://daneshyari.com/article/1459430>

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