



Influence of different amount of Na₂O additive on the structure, mechanical properties and degradability of bioactive wollastonite

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Received 15 June 2015; received in revised form 28 August 2015; accepted 16 September 2015

Available online 25 September 2015

Abstract

Wollastonite (CaSiO₃) ceramics with various Na₂O contents (0, 5, 10, 15 and 20 mol%) were successfully prepared by sintering sol–gel derived precursors. The ceramics are composed of β-CaSiO₃. With increasing Na₂O content, Na₂Ca₂Si₃O₉, Na₄Ca₄Si₆O₁₈ and Na₂CaSi₃O₈ are observed. The particle size distribution obeys the normal distribution. Na₂O decreases the total pore volume of wollastonite powder, and increases the apparent porosity of wollastonite ceramic block. Na₂O increases the bending strength of wollastonite, and the elastic moduli of all ceramics match that of cancellous bone of human body. The thermal expansion coefficients (TECs) of the ceramics are all close to that of titanium alloy (Ti6Al4V). The degradation rate is increased with increasing Na₂O content. All samples show the ability of apatite induction in simulated body fluid (SBF) solution.

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Keywords: A. Sol–gel processes; C. Mechanical properties; E. Biomedical applications; Wollastonite; Na₂O

1. Introduction

Bioactive ceramics are reported to be able to stimulate bone regeneration [1,2]. Hydroxyapatite, due to its chemical similarity to the inorganic component of hard tissues, is more widely used in the clinic applications [3,4]. However, the poor mechanical properties of hydroxyapatite limited its applications [5]. Calcium silicate based bioceramics, because of their superior bioactivity, have been studied as potential substitutes for hard tissue. Silicon plays an essential role in the formation of apatite layer [6]. Wollastonite is the most common calcium silicate biomaterials proposed for bone tissue regeneration [7]. Moreover, pure calcium silicate ceramics are greatly induced apatite layer on calcium silicate surface after soaking in SBF

[8]. However, the limitation of the ceramics is their poor mechanical properties that restrict some load bearing implant application [9–11].

In recent years, some related researches [12–16] have reported the effect of additives on the properties of biomaterial, and the additives improved the properties of biomaterial. Taddei [17] and Wu [18] studied the effect of the fluoride content on the properties of calcium silicate-based materials. Padilla [19] studied the influence of the phosphorus content on the bioactivity of sol–gel glass ceramics. Ma [20] studied the effect of the magnesia content on the degradability and bioactivity of sol–gel derived SiO₂–CaO–MgO–P₂O₅ system glasses, which showed that the degradation rate of glass decreases with increasing magnesia content and the formation of apatite layer on glass surface is retarded.

Our previous papers [21,22] studied the influence of Na₂O, MgO, ZnO or ZrO₂ on mechanical properties, degradability and in vitro bioactivity of wollastonite. These additives all improved the bending strength of the ceramic. The addition of Na₂O increased the degradation rate of the ceramic, and MgO,

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ZnO or ZrO₂ decreased the degradation rate. However, there are few literatures about that how the Na₂O content affects the properties of bioactive wollastonite. Therefore, in this work, different amount of Na₂O additive has been incorporated into wollastonite using sintering sol–gel derived precursor, and we mainly discuss the influence of different amount of Na₂O additive on the structure, mechanical properties and degradability of bioactive wollastonite.

2. Experimental

Ceramics of composition (50–*x*)CaO–*x*Na₂O–50SiO₂ (where *x* is 0 mol%, 5 mol%, 10 mol%, 15 mol% and 20 mol%) were prepared by sintering sol–gel derived precursor. The sol–gel derived precursor was prepared by calcium nitrate tetrahydrate (Ca(NO₃)₂·4H₂O), tetraethyl orthosilicate ((C₂H₅O)₄Si, TEOS) and sodium nitrate (NaNO₃). The detailed processes have been described in our previous work [21]. For preparation of ceramic powders, the stabilized powders were sintered at 1000 °C for 1 h with a heating rate of 5 °C/min, which was determined by thermogravimetric and differential thermal analysis (TG/DTA) of the dried gel. For preparation of ceramic blocks, the mixture of stabilized powders and appropriate amount of polyvinyl alcohol water solution binders was uniaxially pressed at 200 MPa and then the compact samples were also sintered at 1000 °C for 1 h with the same heating rate.

The thermogravimetric and differential thermal analysis (TG/DTA) were carried out by a SDT 600 TA instrument from room temperature to 1000 °C with a heating rate of 10 °C/min under an air atmosphere. X-ray diffraction (XRD, Shimadzu XRD-6100) was used for analyzing the crystalline phase. Fourier transform infrared spectroscopy (FTIR) was recorded using a Bruker Optics VERTEX-70 FTIR spectrometer. The pore size distribution of the ceramic powders was analyzed by a Barrett Joyner Halanda (BJH) method using Surface Area Analyzer (SAA, BECKMAN COULTER, SA3100). The granularity of the powders was measured by LS13 320 laser diffraction particle size analyzer. The microstructure and element composition were studied using a Scanning electron microscope (SEM, Hitachi S-3400N) and an energy dispersive spectrometer (EDS, Horiba EMAX x-act).

The apparent porosity of the ceramic blocks was measured by the drainage method according to Archimedes principle, and the number of trials was 3. The results of the apparent porosity were evaluated using the following equation:

$$K = \frac{m_2 - m}{m_2 - m_1} \times 100\%$$

where *K* is the apparent porosity, *m* is the quality of the sample after drying in the air (g), *m*₁ is the quality of the sample after absorbing water in the water (g) and *m*₂ is the quality of the sample after absorbing water in the air (g).

The three-point bending method was used to test the bending strength and elasticity modulus of the ceramics using RGD-5 type electronic tensile machine (span was 30 mm, the

movement speed of indenter was 0.5 mm/min). The experimental results were calculated from three testing samples. The calculating equation of the bending strength and elasticity modulus is listed in our previous work [22]. The thermal expansion coefficient (TEC) was measured by thermal expansion coefficient tester (PCY-III-1000) on squareness specimens of 50 × 6 × 6 mm³ from 20 °C to 500 °C with a heating rate of 5 °C/min. The experiment was repeated three times.

The degradability was performed in tris–(hydroxymethyl)–aminomethane and hydrochloric acid (Tris–HCl) buffer solution with a pH value of 7.4 at 36.5 °C, using triplicate samples according to the ISO 10993-14 standard. The bioactivity was evaluated by in vitro assays in simulated body fluid (SBF) as described by Kokubo et al. [23]. The volume of the solution was 22 ml and the solution was continuously replaced every 2 days.

3. Results and discussion

3.1. Thermal property analysis

Fig. 1 shows the thermogravimetric and differential thermal analysis (TG/DTA) curves of dry gel powders without Na₂O after being dried at 120 °C. From the TG curve, it is observed that two obvious weight losses occurred. An initial process in the range of room temperature to 136.17 °C is attributed to the volatilization of residual water and ethanol, corresponding to a weight loss of 13.05%. The second process occurred at 564.47 °C with a weight loss of 41.12%, which might be caused by the thermal decomposition of nitrates used in the experiment. When the temperature was higher than 800 °C, the TG curve became stable and the sample mass remained unchanged. From the DTA curve, the exothermic peak at 935.97 °C corresponding to the emergence of crystallization was detected. The sintering temperature should not be below 935.97 °C. High sintering temperature and long maintaining time can make the crystallization and densification more sufficiently [24,25]. However, too long sintering time can increase the agglomeration formation resulting in a larger

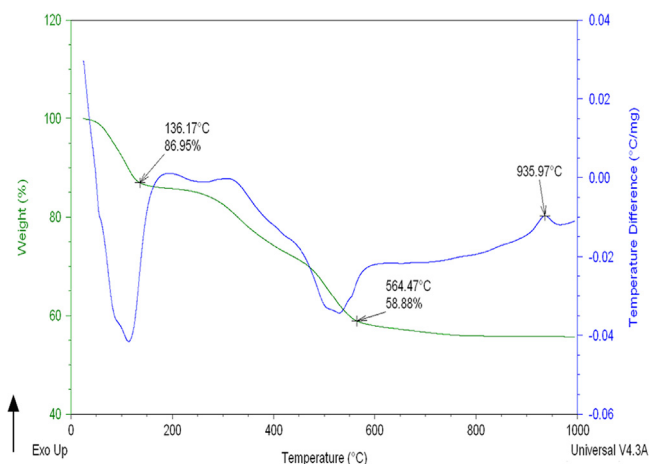


Fig. 1. TG/DTA curves of dry gel powders without Na₂O after being dried at 120 °C.

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