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Strength development, bioactivity and biodegradability of forsterite nanostructure scaffold

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Abstract

Highly porous forsterite nanostructure scaffolds with high compressive strength were prepared by a two steps sintering method. It was found that the formation of enstatite glassy phase during heat treatment at high temperature is responsible for the strengthening mechanism of the prepared scaffolds. The in vitro bioactivity and degradability of the scaffolds were also determined by immersing them in simulated body fluid (SBF) and Ringer's solution, respectively. The results demonstrated that nanostructure scaffolds with the mean crystallite size of about 33 nm showed suitable bioactivity. Also it was found that the prepared nanostructure scaffolds have a good biodegradability and can release magnesium ions into Ringer's solution. The formation of a small amount of glassy phase (enstatite), which improved the compressive strength of the prepared scaffolds, did not have a detrimental influence on the bioactivity and biodegradability of the forsterite structure in vitro.

Keywords: Forsterite; Biodegradability; Nanomaterials; Scaffolds

1. Introduction

Fabrication of scaffolds with high porosity, similar to the spongy bone of human body, and with high compressive strength is one of the aims of many scientists in tissue engineering [1,2]. Hydroxyapatite is one of the most important bioceramics due to its high bioactivity and osteoconductivity properties. However, the orthopedic applications of hydroxyapatite ceramics have been limited as a result of the low fracture toughness and inappropriate mechanical properties [3,4].

Recently, forsterite has been proposed as a new bioceramic for tissue engineering application [5–9]. It has been proved that nanostructure forsterite has bioactive properties and can form apatite layer on its surface [5,7]. On the other hand, nanostructure forsterite can release magnesium and silicon ions into the biological medium and show good biodegradability [7]. It has been shown that the bioactivity and biodegradability of forsterite ceramics depend on the crystallinity of forsterite structure; with increasing crystallinity the bioactivity and biodegradability of forsterite structure decreased [7].

In our previous paper [10], we synthesized nanostructure forsterite scaffolds with high porosity and high compressive strength almost similar to spongy bone in human body. In the present paper, the mechanism behind the increased strength is studied. Also the bioactivity and biodegradability of the obtained scaffolds were investigated to evaluate the influence of enstatite on the bio-behavior. The obtained results can open a promising approach to utilize forsterite nanostructure scaffolds for load bearing application in tissue engineering.

2. Experimental procedure

Forsterite nanostructure scaffolds were synthesized according to our previous study [10]. First, forsterite powder was synthesized from talc and $MgCO_3$ powders [11]. A slurry of forsterite and ethyl alcohol was prepared and pre-cut sponges were soaked in the slurry. The saturated sponges were then sintered at different temperatures for various holding times [10]. Table 1 shows the heat treatment regimes. To clarify the

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Table 1
designation, heat treatment regimes, porosity and compressive strength of prepared forsterite scaffolds [10].

Designation	Sample A	Sample B	Sample C	Sample D
Heat treatment				
Step 1	HR = 2 °C/min	HR = 2 °C/min	HR = 2 °C/min	HR = 2 °C/min
	T = 300 °C	T = 300 °C	T = 300 °C	T = 300 °C
Step 2	HR = 5 °C/min	HR = 5 °C/min	HR = 5 °C/min	HR = 5 °C/min
	T = 1300 °C	T = 1300 °C	T = 1500 °C	T = 1500 °C
Step 3	T = 1300 °C	T = 1300 °C	T = 1500 °C	T = 1500 °C
	HT = 5 min	HT = 60 min	HT = 60 min	HT = 60 min
Step 4	CR = 50 °C/min	CR = 50 °C/min	CR = 50 °C/min	CR = 50 °C/min
	T = 800 °C	T = 800 °C	T = 800 °C	T = 800 °C
Step 5	T = 800 °C	T = 800 °C	T = 800 °C	T = 800 °C
	HT = 300 min	HT = 900 min	HT = 300 min	HT = 900 min
Step 6	CR = 5 °C/min	CR = 5 °C/min	CR = 5 °C/min	CR = 5 °C/min
	T = 25 °C	T = 25 °C	T = 25 °C	T = 25 °C
Porosity (%)	86	83	80	86
Compressive strength (Mpa)	0.34 ($\pm$0.02)	0.22 ($\pm$0.05)	4.33 ($\pm$0.02)	3.49 ($\pm$0.02)

HR = heating rate, CR = cooling rate and HT = holding time.

designations, for example, the heat treatment regime of sample A is as follows: the sample was heated to 300 °C at the heating rate of 2 °C/min. Then the temperature was increased to 1300 °C at the heating rate of 5 °C/min. At 1300 °C, the specimen is kept for 5 min and then it is cooled down to 800 °C at the cooling rate of 50 °C/min. Subsequently it is kept at 800 °C for 300 min holding time and finally it is cooled down to 25 °C at the cooling rate of 5 °C/min. For this study, we choose 4 different heating regimes based on the lowest and highest compressive strength obtained in our previous study [10].

In vitro bioactivity of the obtained scaffolds was investigated by soaking the prepared samples in the SBF for 1, 2, 4, 7, 14, 21, and 28 days. For this purpose, the prepared scaffolds were soaked in 100 ml SBF without refreshing the soaking medium. This procedure has been widely used to prove the similarity between in vitro and in vivo behavior of certain bioceramic composites. The SBF was prepared according to the standard procedure described by Kokubo and Takadama [12]. The soaking experiment was performed in a shaking bath maintained at 37 °C. After soaking, the samples were gently rinsed with deionized water to remove SBF solutions followed by drying at 100 °C for 24 h. To evaluate the degradation rate, the prepared scaffolds were soaked in 100 ml Ringer's solution (pH 7.40) at 37 °C in a shaking water bath for 1, 2, 4, 7, 14, 21, and 28 days without refreshing the soaking medium. After soaking the samples were dried at 100 °C for 1 day, and the final weight of each sample was accurately measured. The weight loss was expressed as a percentage of the initial weight.

A Philips X'PERT MPD diffractometer with Cu Kα radiation (λ = 0.154056 nm) was used for X-ray diffractometry (XRD) analysis to determine the structure of the obtained scaffolds. XRD patterns were recorded in the 2θ range of 20–80° (a step size of 0.04° and a time per step of 1 s). The transmission electron microscopy (TEM; Leo 912AB) technique was utilized to characterize the morphology and nanostructure of the synthesized scaffolds. The apatite formation on the surface of the samples as a consequence of the precipitation process of calcium phosphate was investigated by Fourier transitioned-infrared spectroscopy (FTIR; Bomem, MB 100), scanning electron microscopy (SEM; Seron, AIS2100), and energy dispersive X-ray (EDX). The concentrations of Ca and Mg ions of the SBF and Ringer's solutions after soaking were determined by an atomic absorption spectrometer (AAS; Perkin Elmer, 2380), and the changes in pH of soaking solutions were also measured at predetermined time intervals (0–28 days) using an electrolyte-type pH meter.

3. Results and discussions

Fig. 1 shows the XRD traces of the prepared scaffolds after different heat treatment regimes. As can be seen, forsterite (XRD JCPDS data file no. 34-0189) was the dominant phase in all the XRD patterns. On the other hand, traces of enstatite (XRD JCPDS data file no. 11-0273) were also observed in the XRD traces. As we showed in our previous study [10], the scaffolds were prepared from single-phase forsterite powder. However after various heat treatment regimes, a very small amount of metastable enstatite can be observed in the XRD patterns. The reappearance of enstatite is reported in previous studies due to its faster kinetics [13–15]. This phase appeared to be stable up to 1600 °C [16]. With the formation of enstatite in the form of glassy phase, new bonds and better attachments were provided between forsterite grains and hence the compressive strength of the scaffolds increased.

By increasing the sintering temperature or holding time, the intensity of XRD peaks increased while their width decreased due to the increase in the grain size with higher degree of crystallinity. It should be noticed that with increasing sintering temperature the compressive strength of the scaffolds increases due to the formation of higher amount of enstatite and better sintering of the structure but this may affect adversely on the bioactivity and biodegradability of the formed scaffolds [7].