SHORT CALCIUM PHOSPHATE WHISKERS FOR MEDICAL APPLICATIONS

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[ENGINEERING OF BIOMATERIALS 143 (2017) 17]

Introduction

Synthetic hydroxyapatite (HA) has been successfully used for biomedical (especially orthopedic) applications, because of its close compositionally resemblance to the natural bone [1-3]. HA is one of the components of composites used in bone regeneration very often. The composites usually require an appropriate durability, so most of the researches have focused on the use of HA whiskers as promising reinforcement for such materials [4]. It was also found that the HA whiskers are non-toxic and are compatible with human body. The application of well-crystalized, stoichiometric HA whiskers in resorbable composites for bone regeneration may be limited due to its slow resorption [5], so more appropriable seems to be using resorbable calcium phosphates or whiskers being bi- or triphasic mixtures.
There are a number of different methods for producing calcium phosphate whiskers [6-8]. The most frequently hydrothermal homogenous precipitation is used. But hydrothermal synthesis procedure requires special autoclaves enabling heating of aqueous solutions to high temperatures up to 200°C [9.10].
The present work shows the results of synthesis of calcium phosphate whiskers through the method described in literature [11], by the hydrolysis reaction of β-tri-calcium phosphate (β-TCP) in presence of H_2O_2. The process was carried out in considerable lower temperature than for hydrothermal methods. The formation, morphology and phase composition of whiskers obtained were studied as a function of temperature and time of reaction.

Materials and Methods

Calcium-phosphate whiskers were synthesized in one-pot technique. 4 grams of β-TCP powder (Fluka) were placed in 250 ml capacity Pyrex glass bottle. Then, 100 ml of 30% solution of H_2O_2 (Avantor) was added. The bottle was capped and shaken for 2 min, followed by heating for 8-96 hours in an electric dryer in 90-95°C. The final whiskers from the bottle were filtered, washed with distilled water and dried overnight at 90°C.
The morphology and phase composition of whiskers were analyzed by scanning electron microscopy (SEM) and X-ray diffractometry (XRD), respectively. Functional groups of the samples were identified by Fourier transform infrared spectroscopy (FTIR).

Results and Discussion

Test results indicate that using the above mentioned procedure calcium phosphate whiskers were obtained. As can be seen at FIG. 1, the synthesis led to form whiskers and aggregates with different sizes (from several to tens μm). The morphology and size of these whiskers depend on time and temperature of the reaction. The length and width of whiskers increase with increasing of time and temperature.

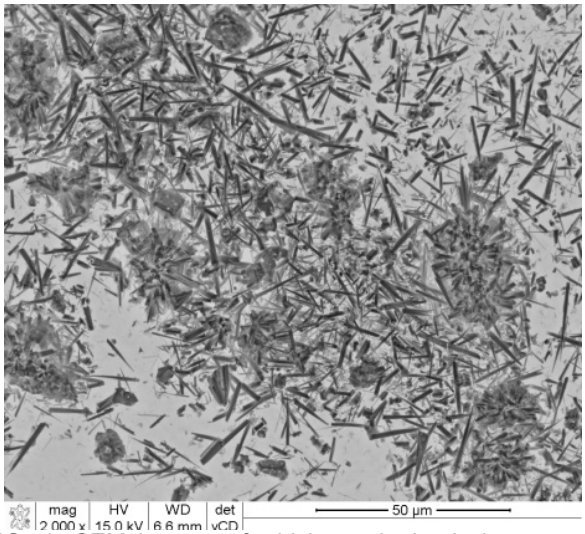

FIG. 1. SEM images of whiskers obtained via one-pot technique.

Phase analysis of the obtained product remains with opposite to the literature's data [11] where produced whiskers were found to be biphasic mixtures of apatitic CaP and octacalcium phosphate ($Ca_8H_2(PO_4)_6 \cdot 5H_2O$). In our tests XRD patterns revealed the triphasic mixture of: hydroxyapatite, calcium pyrophosphate ($Ca_2P_2O_7$) and residue of β-tri-calcium phosphate. These results are confirmed by FTIR spectra. The amount of β-tri-calcium phosphate decreased with the time of reaction up to 15 wt%, the amount of hydroxyapatite increased up to 72 wt%, whereas the content of calcium pyrophosphate is almost stable and decreased only insignificantly from 16 to 12 wt%.

Conclusions

Applying one-pot technique of synthesis using β-TCP powder and 30% solution of H_2O_2, calcium phosphate whiskers were obtained. They were characterized as a triphasic mixture of HA, calcium pyrophosphate and β-TCP.
Test results indicate that there are at least two factors (temperature and time of reaction) that simultaneously affect the morphology and size of the obtained whiskers.
Such whiskers may be useful as promising reinforcing filler for composite of increased mechanical strength.

Acknowledgments

The work was financed from resources assigned to the statutory activity of the Institute of Ceramics and Building Materials in Warsaw.

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