

## Microspheres of BCP produced by Snowballing technique for multipurpose application

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**INTRODUCTION:** Biphasic calcium phosphates (BCP), a combination of hydroxyapatite (HAp) and  $\beta$ -tricalcium phosphate ( $\beta$ -TCP), are widely used in dental, orthopedics and drug delivery systems (DDS) nowadays. In an effort to broaden the range of application, architecture and microstructures are major aspects of concern. Considering manufacturing granules of spherical shape, micro-scale size of less than 1mm and loose arrangement still present a challenge, despite the existence of many process to obtain such structures involving several steps [1]. Microspheres are of great interest because they possess a high packing capability combined with the possibility of being injected through less invasive procedures, as bone fillers [2]. The microarchitecture represented by microporosity gives an adsorption potential to microspheres. Snowballing technique is a new process shown to be a suitable method for obtaining ceramic spheres without additives or binders [3]. This work presents the obtaining of microspheres through Snowballing technique.

**METHODS:** Ceramic powders of HAp and  $\beta$ -TCP, HAP-200 and  $\beta$ -TCP-100, respectively, from Taihei Chemical Industrial Co. Ltd., Osaka, Japan, were mixed in a 1:1 by weight composition to form the BCP. The amount of 25g of BCP powder mixture was added to a plastic bottle of ~1000mL (9cm x 17cm) and put in a small ball mill device for transverse mixing at 85 rpm speed for 6 hours. The BCP material were sintered at 1150°C/1 hour and separated by sieve of 1mm and 500 $\mu$ m to form two groups. Microspheres were characterized by scanning electron microscopy (SEM) S-3400N, Hitachi and X-ray diffraction (XRD) D8 Advance, Bruker.

**RESULTS:** The XRD analysis showed that no  $\alpha$ -TCP phase transformation occurred during the sintering process with the BCP. The resulting microspheres were separated into two groups: microspheres ranging between 1mm and 500 $\mu$ m and microspheres with less than 500 $\mu$ m, both groups differing only in microspheres' diameter size. The SEM analysis shows the morphology of the microspheres with round shape and smooth

surface, while under higher magnification the topography of microspheres shows an intricate rough microstructure pattern with microporosity, Fig.1 and Fig. 2. Both groups of samples presented good sintering parameters with neck formation between particles and good mechanical strength allowing the handling of microspheres.

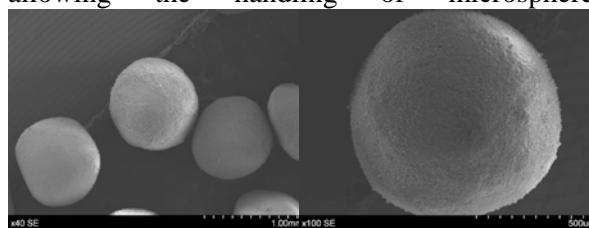


Fig. 1: BCP microspheres ranging between 1mm and 500 $\mu$ m – SEM (1mm bar / 500 $\mu$ m bar)

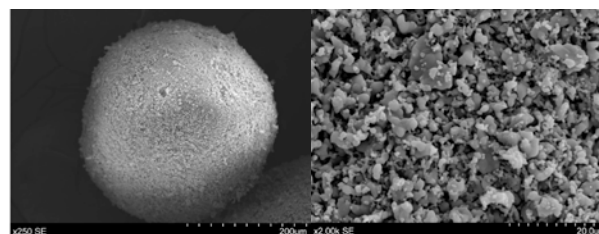


Fig. 2: BCP microspheres with less than 500 $\mu$ m – SEM (200 $\mu$ m bar / 20 $\mu$ m bar)

**DISCUSSION & CONCLUSIONS:** The Snowballing technique allowed to obtain microspheres with less than 1 mm and less than 500 $\mu$ m in diameter without additives or binders. The samples surface presented overt microporosity, which is a desirable feature for protein adsorption, cell attachment and DDS. The round shape gives to the material fluidity, a potential feature for use as an injectable material in less invasive procedures, as vertebral disk filling with material for vertebral fusion (arthrodesis).

**REFERENCES:** <sup>1</sup> M. Bohner, S. Tadier, N. Garderen, et al (2013) *Biomatter* 3:2 e25103-1-15. <sup>2</sup> D.H. Kim, H.H. Chun, J.D. Lee, et al (2014) *Ceramics International* 40:4 5145-55. <sup>3</sup> K.B Violin, T.S. Goia, K. Ishikawa, et al (2014) *Advances in Science and Technology (CIMTEC 2014 - in press)*.

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