Microhardness Measurements of Hydroxyapatite Synthesized Using Chicken Eggs Shell Precursors

Author: Marcelo Vitor Ferreira Machado[1] *marcelovfmachado@gmail.com*
Advisor: José Brant de Campos[1]

1State University of Rio de Janeiro

PPG-EM Seminars: season **2017**
www.ppg-em.uerj.br

July 05, 2017

Keywords: Hydroxyapatite, Microhardness, Chicken Eggs Shell.

1 Introduction

Considering the advance in the biocompatible materials researches, hydroxyapatite (HAP) have been revelead a important alternative to bone grafts and orthodontic implants. Because of its similarity with inorganic phase of bone tissues and its osteoconducting property [1], HAP is a brittle material that it doesn't show rejections.

In general, one of the most important features for those applications is the search to the improvement of the sintered HAP mechanical properties, obtained by the use of different chemical methods and precursor materials. Concerning microhardness measurements, other mechanical properties can be determined from them, for example, fracture toughness (KIC) and an analysis of the strain hardening effects[2], in the plastic behavior.

2 Materials and Methods

A Field Emission Scanning Electron Microscope (JEOL JSM 7100F) has been used to obtain the mean size of hydroxyapatite particles.

Samples of hydroxyapatite could be conformed by uniaxial compression method using a uniaxial hydraulic press equipment (Contenco Pavitest), where we have used a average compression force equal to 4000 kgf for 20 minutes, that provide an average stress compression of approximately 77.44 MPa during that time. The sintering process have been made in a QUIMIS (Mufla Stove) during 2 hours at 1000 °C. Theses equipments have been used to determine the experimental parameters that would be for comparison between chicken eggs shell HAP and comercial HAP.

HAP samples has been tested in a microhardness equipment (Pantec HDX-1000TM) to define the indenter force experimental parameter (200 gf) during 15 s and determine some preliminary Vickers microhardness measurements of this HAP sample. Finally, we have acquired a stereomicroscope (Discovery.V8 ZEISS) image of the HAP specimen's surface, on which we could note some irregularities and impurities. Theses equipments have been used to determine the experimental parameters that would be for comparison between chicken eggs shell HAP and comercial HAP.

To determine the microhardness of HAP samples for that comparison, the only change have been in hydraulic press equipment. Samples of hydroxyapatite could be conformed by uniaxial compression method using a uniaxial hydraulic press equipment (EVA 5052), where we have used a average compression force equal to 4000 kgf for 20 minutes, that provide an average stress compression of approximately 254.91 MPa (this samples have been compressed using a more narrow matrix than one) during that time.

3 Results

The experimental parameters to Vickers microhardness test determined have been 200 gf to indenter force (among 50 gf, 100 gf and 200 gf, this last parameter has shown the best microhardness impression) and 15 s to the time indentation.

After those experimental determination, the microhardness measurements in chicken eggs shell HAP and commercial HAP have been measured. To the chicken eggs shell HAP the measures have been determined using 6 samples and the results are:

Average: 35.846 HV

Standard Deviation: 4.198

Amplitude: 19.347 HV

Then, to the commercial HAP microhardness measures, 3 samples have been used and the statistics data are:

Average: 40.860 HV

Standard Deviation: 5.952

Amplitude: 21.503 HV

3.1 Graphics

Histograms relating the number of measures (vertical axis) with the respectives Vickers microhardness range to each kind of HAP (horizontal axis) have been plotted as shown below:

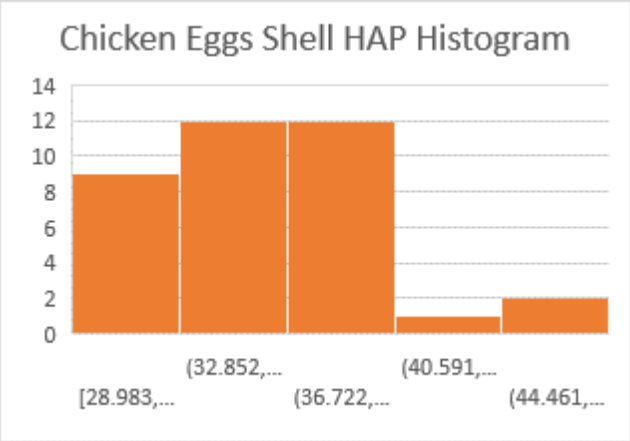

Figure 1: Chicken eggs shell HAP histogram.

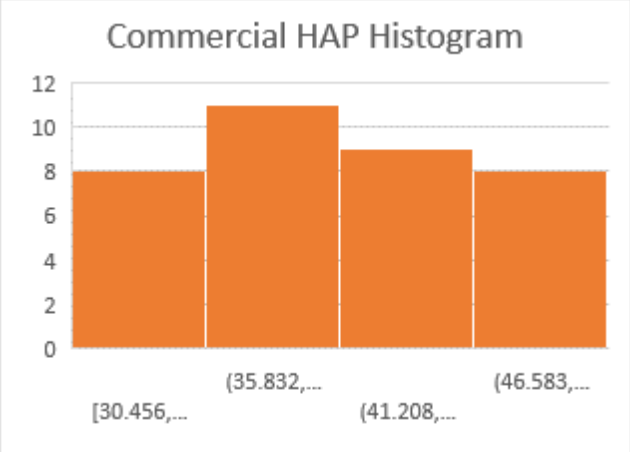

Figure 2: Commercial HAP histogram.

4 Conclusions

The research is going on using the indenter force experimental parameter defined (200 gf) upon the hydroxyapatite samples to determine microhardness measurements as we can observe in the sequence.

Then, doing a simple statistics data analysis we can observe that the commercial HAP microhardness average is greater than chicken eggs shell HAP microhardness average, as we have already expected, but the measurements to comercial HAP microhardness show a greater dispersion around the average than chicken eggs shell HAP. The value of standard deviation obtained to the commercial HAP samples is great enough to contemplate the chicken eggs shell HAP samples average.

This research is ongoing with other experiments to analyse why this microhardness behavior to chicken eggs shell have been shown.

5 Acknowledgments

The author would like to give a special thanks for the advisor of this research José Brant de Campos and all members of our hydroxyapatite research group, they are: Vitor S. Ramos, Marilza S. Aguilar, Bruno Di Lello, Saulo Acioli and Nataly Cristiane de Campos.

References

[1] Andrade, A. L., "Synthesis, *Caracterization, Tests and Use of HAP-91 Obteined from Calcite*" (1998).

[2] G. Muralithran, S. Ramesh, Ceramics International 26 (2000) 221 – 230.

[3] Th. Zisis, A. E. Giannakopoulos, International Journal of Solids and Structures 48 (2011) 3217 – 3231.

[4] D. B. Marshall, B. R. Lawn, Journal of Materials Science 14 (1979) 2001 – 2012.

[5] S. S. Scherrer, J. Robert Kelly. G. D. Quinn, K. Xu, Dental Materials 15 (1999) 342-348.

[6] Gomes L. C., Di Lello, B. C., Campos,J. B., Sampaio, M. A., *" Synthesis and Calcium Phosphate Caracterization Using Chicken Eggs Shell as Precursor"*

[7] Ferreira, J. R. M., da Rocha, D. N., Louro. L. H. L., Prado da Silva, M. H., *"Phosphating of Calcium Carbonate for Obtaining Hydroxyapatite from the Ostrich Egg Shell"*