Microhardness Measurements of Hydroxyapatite Synthesized Using Chicken Eggs Shell Precursors

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TOPICS OF PRESENTATION

MEMBERS

INTRODUCTION

MATERIALS AND METHODS

RESULTS

CONCLUSIONS

REFERENCES

Members of Hydroxyapatite (HAP) Synthesized Using Chicken Eggs Shell Precursors's Research Group

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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, HAP is a brittle material that it doesn't show rejections.

- Hydroxyapatite is a biochemical compound that is based in calcium phospate and its chemical formula is given below:

$$Ca_{10}(PO_4)_6(OH)_2 \qquad [1]$$

- The HAP that we have tested is obtained by sol-gel method from the chicken eggs shell, and the resulting particles has been compressed to develop the samples.

- 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.

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.

- 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.

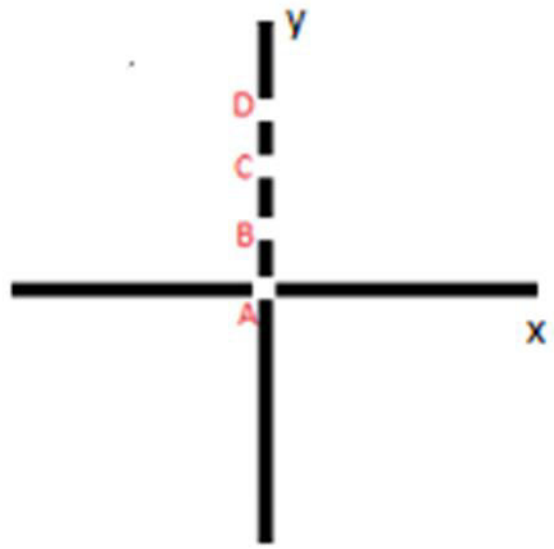
To tested HAP samples using 200 gf for comparison between chicken eggs shell HAP and commercial HAP Vickers microhardness.

- 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.

- The sintering process have been made in a QUIMIS (Mufla Stove) during 2 hours at 1000 °C.

- HAP samples has been tested in a microhardness equipment (Pantec HDX-1000TM) using the indenter force experimental parameter of 200 gf during 15 s and determine Vickers microhardness measurements of different HAP sample.

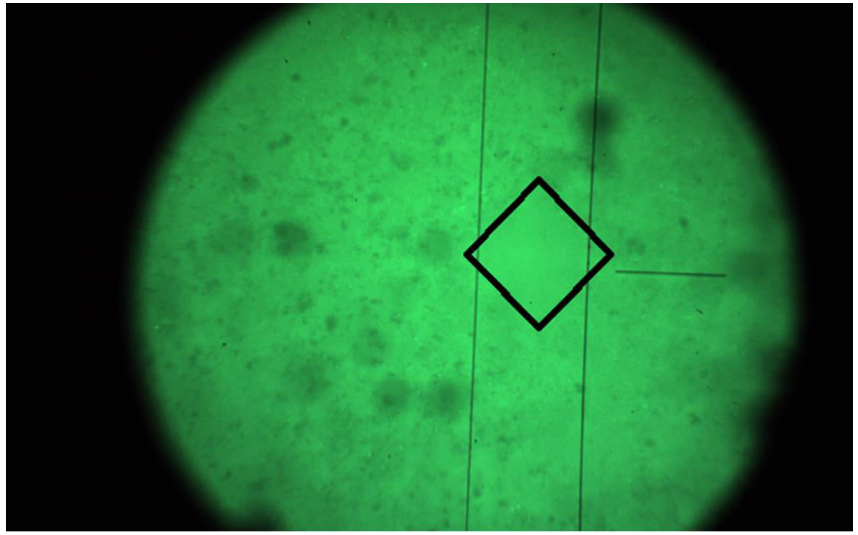
RESULTS



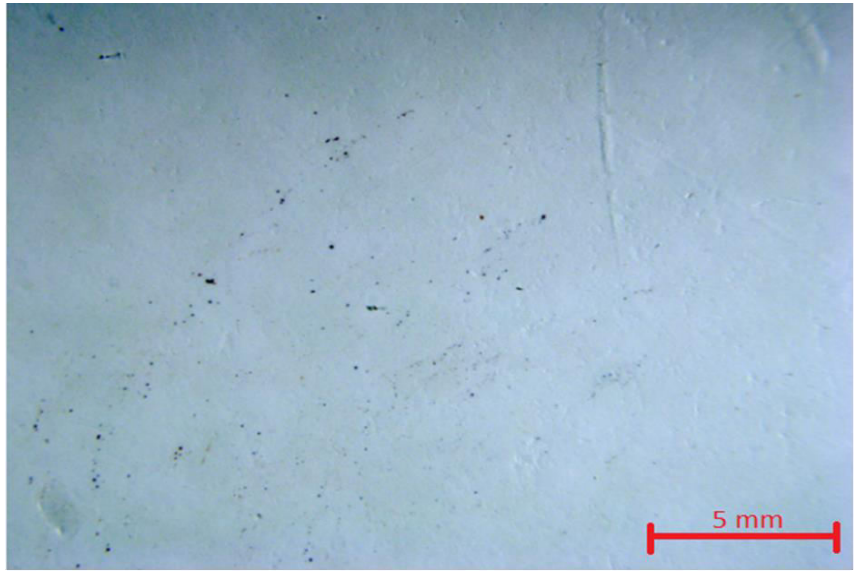
Offset (x;y) representation

Point	Offset (x;y)	Force (gf)	Microhardness (HV)
A	(7,5;7,5)	100	52,25
B	(7,5;8,5)	100	49,07
C	(7,5;9,5)	200	38,46
D	(7,5;10,5)	50	95,10

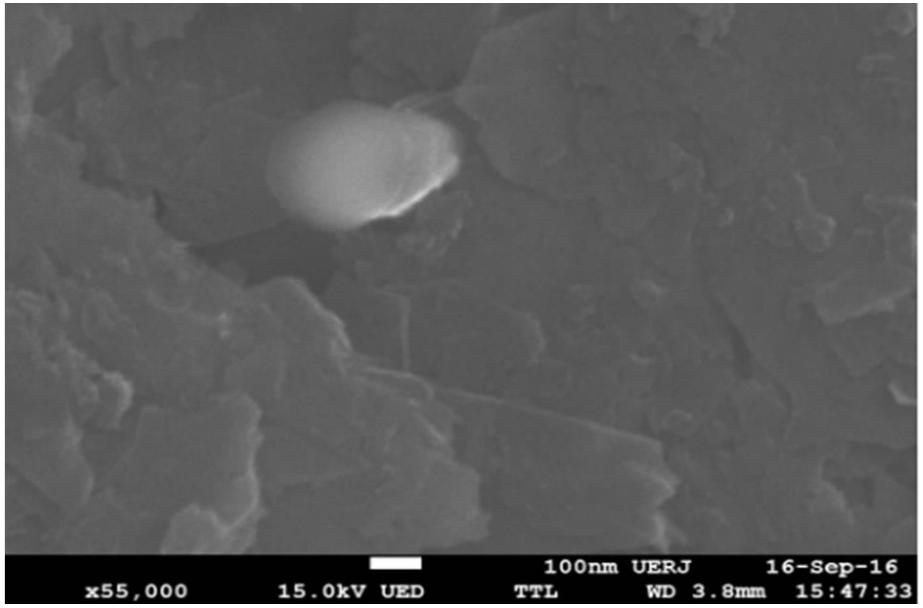
Preliminary microhardness measurements.

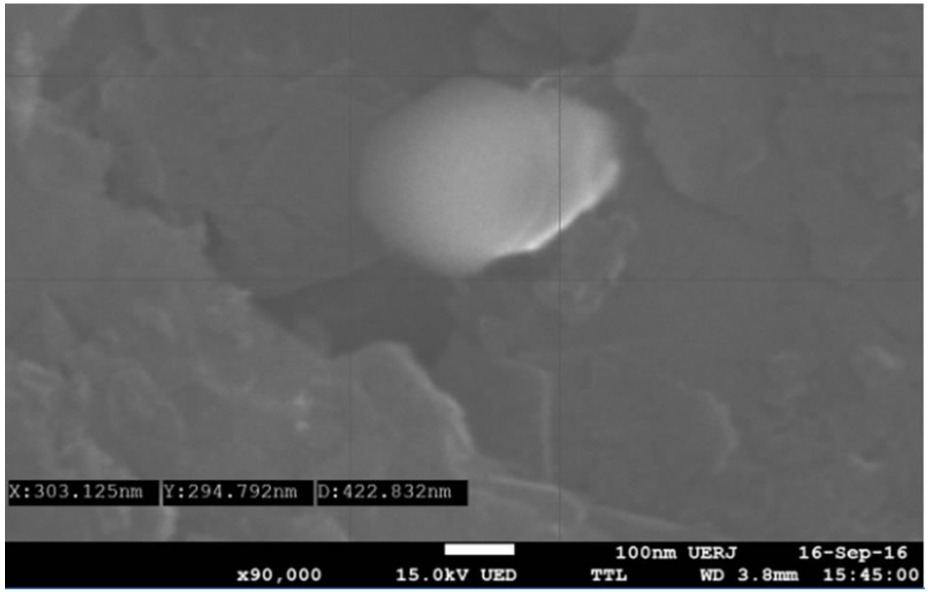


Indentation using 100 gf.



Stereomicroscope image.





Mean size of HAP particles.

Chicken Eggs Shell HAP

Sample 1

Measures	points	Microhardness (HV)
1st	(-3,3)	34.342
2nd	(-3,2)	31.251
3rd	(-3,1)	37.915
4th	(-3,-1)	37.903
5th	(-3,-2)	33.841
6th	(-3,-3)	33.788

Sample 2

Measures	points	Microhardness (HV)
1st	(-3,3)	37.384
2nd	(-3,2)	48.330
3rd	(-3,1)	35.453
4th	(-3,-1)	47.756
5th	(-3,-2)	36.972
6th	(-3,-3)	38.689

Sample 3

Measures	points	Microhardness (HV)
1st	(-3,3)	36.825
2nd	(-3,2)	38.584
3rd	(-3,1)	33.970
4th	(-3,-1)	34.389
5th	(-3,-2)	33.794
6th	(-3,-3)	31.842

Sample 4

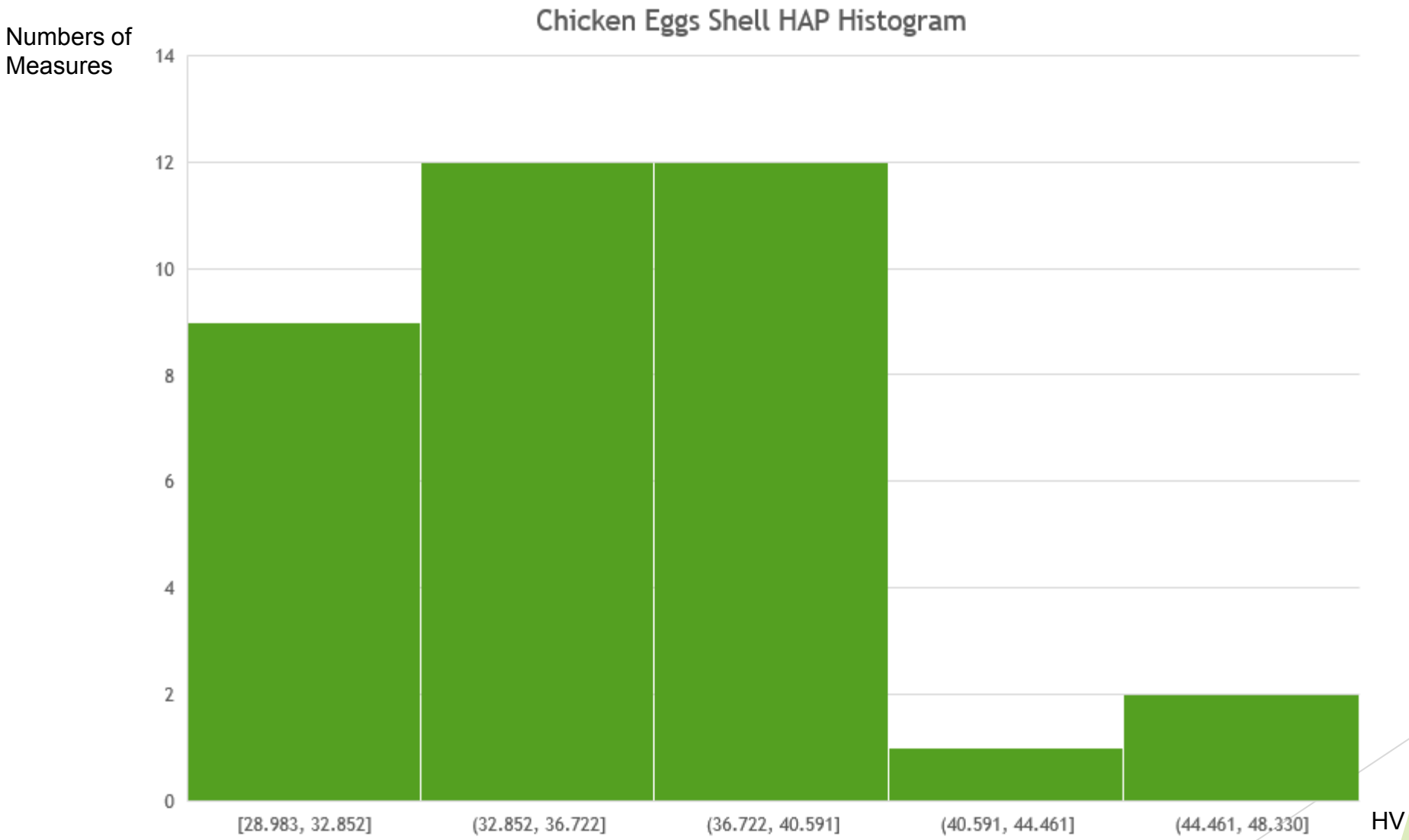
Measures	points	Microhardness (HV)
1st	(3,3)	36.614
2nd	(3,2)	31.347
3rd	(3,1)	35.363
4th	(3,-1)	30.609
5th	(3,-2)	28.983
6th	(3,-3)	30.153

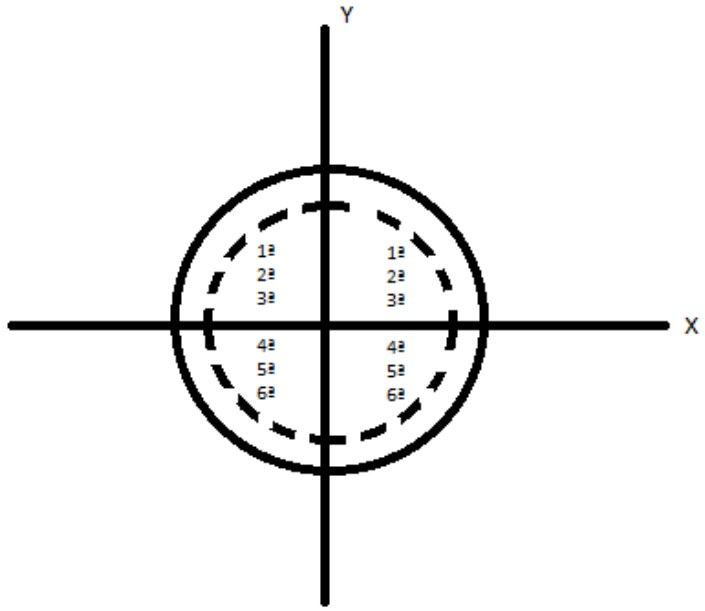
Sample 5

Measures	points	Microhardness (HV)
1st	(3,3)	36.754
2nd	(3,2)	37.832
3rd	(3,1)	40.227
4th	(3,-1)	38.169
5th	(3,-2)	32.729
6th	(3,-3)	33.298

Sample 6

Measures	points	Microhardness (HV)
1st	(3,3)	40.790
2nd	(3,2)	38.196
3rd	(3,1)	36.654
4th	(3,-1)	32.638
5th	(3,-2)	31.585
6th	(3,-3)	35.491


Chicken Eggs Shell HAP Histogram
Numbers of Measures
14
12
10
8
6
4
2
0
[28.983, 32.852]
(32.852, 36.722]
(36.722, 40.591]
(40.591, 44.461]
(44.461, 48.330]
HV



For samples 1,2 and 3, the measures have been made on the left side and for other samples on the right side.

STATISTICS:

Average: 35.846 HV

Standard Deviation: 4.198

Amplitude: 19.347 HV

Sample 1 Commercial HAP

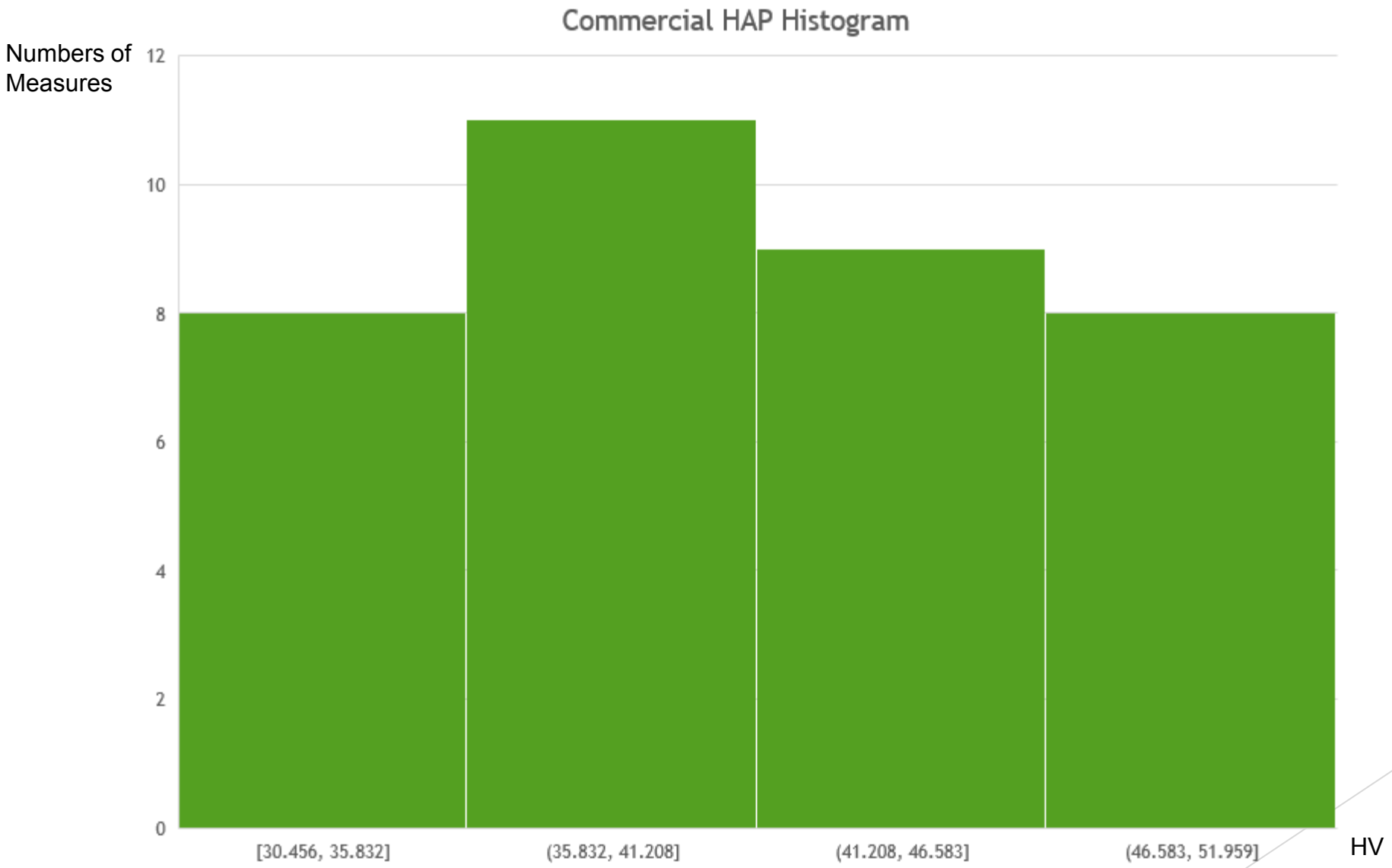
Measures	Points	Microhardness (HV)
1st	(-3,3)	30.456
2nd	(-3,2)	32.250
3rd	(-3,1)	32.034
4th	(-3,-1)	33.180
5th	(-3,-2)	35.182
6th	(-3,-3)	36.611
7th	(3,3)	36.727
8th	(3,2)	40.002
9th	(3,1)	37.689
10th	(3,-1)	41.848
11th	(3,-2)	33.144
12th	(3,-3)	32.362

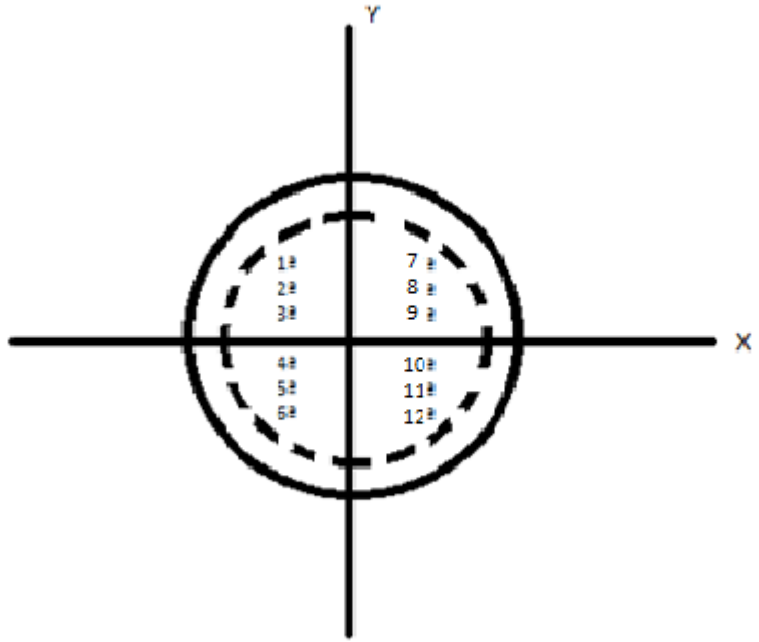
Sample 2 Commercial HAP

Measures	Points	Microhardness (HV)
1st	(-3,3)	44.627
2nd	(-3,2)	41.522
3rd	(-3,1)	36.276
4th	(-3,-1)	47.053
5th	(-3,-2)	51.959
6th	(-3,-3)	47.019
7th	(3,3)	48.719
8th	(3,2)	39.692
9th	(3,1)	47.866
10th	(3,-1)	40.708
11th	(3,-2)	46.018
12th	(3,-3)	50.781

Sample 3 Commercial HAP

Measures	Points	Microhardness (HV)
1st	(-3,3)	39.633
2nd	(-3,2)	43.218
3rd	(-3,1)	43.403
4th	(-3,-1)	40.532
5th	(-3,-2)	34.985
6th	(-3,-3)	36.267
7th	(3,3)	40.594
8th	(3,2)	43.772
9th	(3,1)	48.930
10th	(3,-1)	50.143
11th	(3,-2)	43.286
12th	(3,-3)	42.468


Commercial HAP Histogram
Numbers of Measures
12
10
8
6
4
2
0
[30.456, 35.832]
(35.832, 41.208]
(41.208, 46.583]
(46.583, 51.959]
HV



Measures made on Commercial samples surface

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STATISTICS:

Average: 40.860 HV

Standard Deviation: 5.952

Amplitude: 21.503 HV

CONCLUSIONS

Hence, this 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.

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

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