Journal of Basic and Applied Engineering Research
p-ISSN: 2350-0077; e-ISSN: 2350-0255; Volume 6, Issue 5; April-June, 2019, pp. 319-322

http://www.krishisanskriti.org/Publication.html

Preparation and Characterization of Hydroxyapatite Powder

Ranveer Singh Soni[a] and Dr. Vinay Pratap Singh[b]

[a]PG Student, Harcourt Butler Technical University, Kanpur (UP)-208002
[b]Assistant Professor, Harcourt Butler Technical University, Kanpur (UP)-208002
E-mail: [a]ranveer2995@gmail.com, [b]vinayforus@gmail.com

Abstract—*The present work deals with the preparation and the subsequent characterization of HAP. Hydroxyapatite is used as a coating in medical implants as it is biocompatible. Hydroxyapatite is a naturally occurring mineral of calcium apatite. This calcium phosphate salt is similar to the human hard tissues and is found in bone and the enamel of teeth. Hence, hydroxyapatite is used in orthopedic and dental applications. In the present work, HAP has been prepared by chemical precipitation techniques. Chemical precipitation technique is one of the important techniques for preparing HAP powders. Hydroxyapatite was prepared via wet chemical precipitation reaction. The reactants of the reaction were orthophosphoric acid and calcium oxide powder. The media was deionized water and ammoniated water was added to maintain the pH of the solution. The prepared hydroxyapatite was calcined and then characterized. The various types of characterization techniques have been used to study the properties of HAP powders. The prepared powder was characterized using X-ray diffraction to get the phase purity of the sample. Scanning electron microscope further gave information about the size of the agglomerates. Energy dispersive spectroscopy was used to determine the Calcium by Phosphorus ratio.*

Keywords- *Hydroxyapatite, orthopedic, bioactive, osteoconductive, biocompatible*

1. INTRODUCTION

Hydroxyapatite [$Ca_5(PO_4)_3(OH)$] is a naturally occurring mineral of calcium apatite. The Ca/P ratio of hydroxyapatite is 1.67. Hydroxylapatite is the hydroxyl end member of the complex apatite group. The OH- ion can be replaced by fluoride, chloride or carbonate. It crystallizes in the hexagonal crystal system. It has a specific gravity of 3.08 and is 5 on the Mohr hardness scale. Pure hydroxyapatite powder is white. This calcium phosphate salt is similar to the human hard tissues and is found in bone and the enamel of teeth. Hence, hydroxyapatite is used in orthopedic and dental applications.

In orthopedics, hydroxyapatite is used as bone scaffolds and as a coating on bone implants due to its excellent biocompatibility and bone bonding ability. It forms an osteoconductive layer on the implants so that bone growth can take place into the layer. This helps in anchoring the implant and transferring the entire load to it.

Hydroxyapatite is the main mineral component of bone. Carbonated-calcium deficient hydroxyapatite is the main mineral of which dental enamel and dentin are comprised. .Consequently HA was readily considered as a bioactive material for artificial bone substitution because of its biocompatibility, and chemical and biological affinity with bony tissue.

In my present work, Hydroxyapatite has been prepared using wet chemical precipitation method and then characterized to determine phase purity, crystal and agglomerate size, and Ca/P ratio.

2. PROCEDURE

14.67 grams of CaO powder was weighed using an analytical weighing machine and was added to 750 mL of deionised water. The mixture was simultaneously heated (temperature set to 70°C) and stirred using a magnetic stirrer. The temperature of this solution was constantly monitored using a thermometer. 1000 mL of deionised water was taken in a burette and 9.5 mL of orthophosphoric acid was added to it. When the temperature of the CaO and water mixture reached 70°C, then the mixture of orthophosphoric acid and water was added drop by drop from the burette into the beaker containing CaO and water.

The pH of the solution was monitored using a pH paper, and when the solution turns acidic, 40 mL of ammoniated water is added. When all solution from the burette had completely drained into the beaker, heating and stirring was stopped and the beaker was left to cool for about 40 minutes. The prepared solution was then filtered using a filter paper to get the hydroxyapatite powder. The filtered powder was dried in the oven for 24 hrs at 60°C and then calcined in the furnace to remove the volatile impurities.

3. STEPS INVOLVED IN PREPARATION

- 14.67 grams of CaO powder weighed and added to 750 mL of deionised water.

- The mixture heated (temperature set to 70°C) and stirred using a magnetic stirrer.
- Solution of 1000 mL of deionised water in 9.5 mL of orthophosphoric acid added to the mixture of CaO and H_3PO_4 drop by drop using a burette when its temperature reached 70°C.
- When the solution turned acidic, 40 mL of ammoniated water added.
- When all the solution had drained, mixture left to cool for 40 minutes.
- The prepared solution was filtered using a filter paper.
- The filtered powder dried in the oven for 24 hrs at 60°C.
- Powder calcined in the furnace.

4. RESULTS

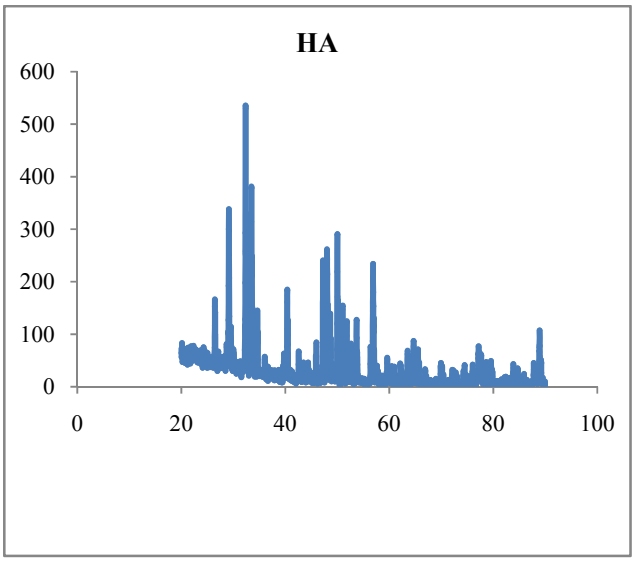
The calcined powder was crushed using a mortar and pestle. Now this powder was characterized further using x-ray diffraction, scanning electron microscope and energy dispersive spectroscopy.

4.1 X-Ray Diffraction

Phase analysis was studied using the room temperature powder X-ray diffraction. Samples are scanned in a continuous mode from 20° – 90° with a scanning rate of 0.02 (degree) / 1 (sec). Particle size in agglomerated or aggregated systems like hydroxyapatite powders is one of the most important parameters controlling ceramic processing.

In this study, particle sizes of the powders were investigated using XRD. Each method of measurement senses a particular aspect of particle size. The profile broadening by powder X-ray diffraction senses mono crystalline domains. Laser diffraction method determines only the agglomerated. The HAP sample has a clear bimodal particle size distribution.

The prepared powder was first characterized for phase purity by x-ray diffraction and the data showed that the prepared powder was Hydroxyapatite.



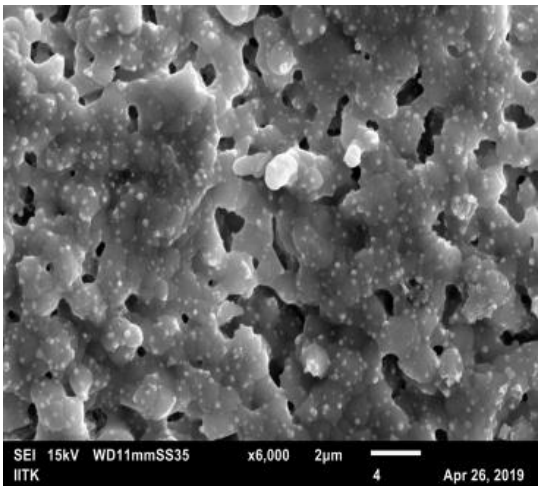
"Figure 1: X-ray Diffraction of HA(Hydroxyapatite)"

4.2 Scanning Electron Microscope

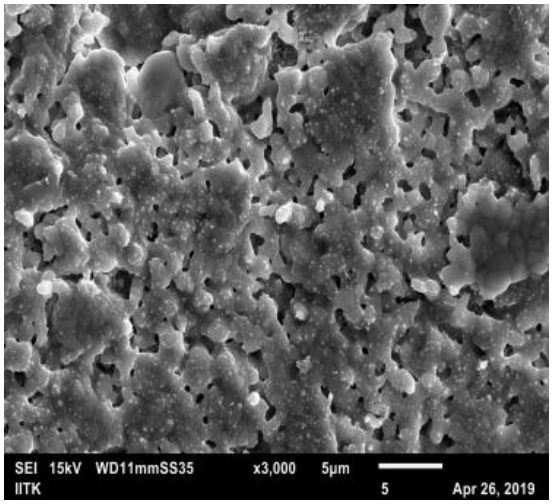
Microstructural features were studied using (Scanning Electron Microscope).It was used to get the size of the agglomerates of HA.

Understanding the sintering behavior of hydroxyapatite powders is important, because this allows designing ceramics with controlled grain growth, microstructure and mechanical properties.

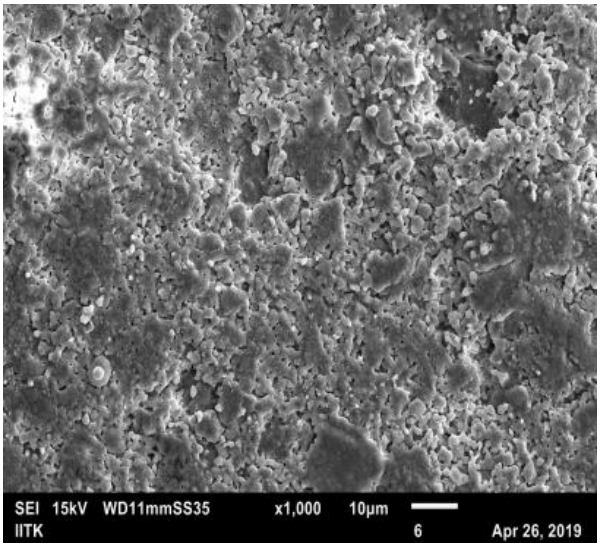
In the present study, the effect of powder characteristics on densification, microstructural development and mechanical properties of HAP were studied.



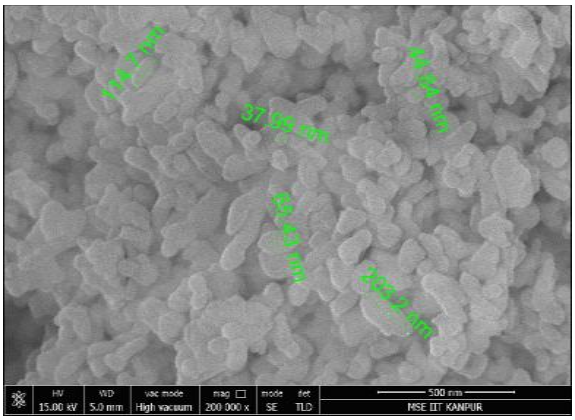
"Figure 2 : SEM at 6000 Magnification"



"Figure 3 : SEM at 3000 Magnification"



"Figure 4 : SEM at 1000 Magnification"

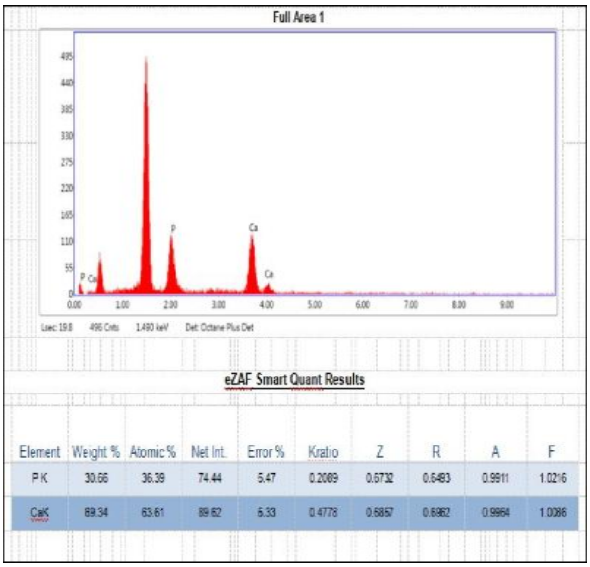


"Figure 5 : SEM at 500 Magnification"

The size of the agglomerates were typically around 40-200 nm.

4.3 Energy Dispersive Spectroscopy

It was performed to check the Ca/P ratio of the prepared powder. The Ca/P ratio was found to be around 2 that is much greater than the actual value of 1.67.



Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z	R	A	F
P K	30.66	36.39	74.44	5.47	0.2089	0.6732	0.6483	0.9911	1.0216
Ca K	69.34	63.61	89.62	5.33	0.4778	0.6857	0.6962	0.9964	1.0086

"Figure 6 : EDS of HA(Hydroxyapatite)"

5. CONCLUSION

The present work deals with the synthesis and characterization of the powder. The size of agglomerates was around 40-200 nm.

The densification of HAP was the result of volume diffusion and grain boundary diffusion.

XRD results indicate there is an improvement in crystallinity of HAP after calcination.

EDS data suggests that Calcium Phosphate might be present in the powder as the Ca/P ratio is approximately 2 instead of 1.6.

REFERENCES

[1] Elliott, J. C_, P. E. Mackie, and R. A. Young. "Monoclinic hydroxyapatite." *Science* 180.4090 (1973): 1055-1057.

[2] Rivera-Muñoz, E. M., et al. "Growth of Hydroxyapatite Nanoparticles on Silica Gels." *Journal of nanoscience and nanotechnology* 11.6 (2011): 5592-5598.

[3] Rivera-Muñoz, Eric M., Rodrigo Velázquez-Castillo, and P. Munoz-Alvarez. "Mechanical characterization of hydroxyapatite-based, organic-inorganic composites." *Materials science forum*. Vol. 539. Trans Tech Publications, 2007.

[4] Oonishi, H. "Orthopaedic applications of hydroxyapatite." *Biomaterials* 12.2 (1991): 171-178.

[5] Monma, H., and T. Kamiya. "Preparation of hydroxyapatite by the hydrolysis of brushite." *Journal of Materials Science* 22.12 (1987): 4247-4250.

[6] Rao, Rayasa R. "Current Trends in Processing and Shaping of Bioceramics." *Current Trends on Glass and Ceramic Materials*(2013):3

[7] Gotman, Irena. "Characteristics of metals used in implants." *Journal of endourology* 11.6 (1997): 383-389.

[8] Sendax, Victor I. "The Dental Clinics of North America.Hydroxyapatite-Coated Implants." *Implant Dentistry* 1.2 (1992): 163.

[9] Ravaglioli, Antonio, and Adriano Krajewski. *Bioceramics: Materials· Properties· Applications*. Springer Science & Business Media, 1991.