Journal of Basic and Applied Engineering Research
p-ISSN: 2350-0077; e-ISSN: 2350-0255; Volume 6, Issue 5; April-June, 2019, pp. 323-326

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

Study and Analysis of Hydroxyapatite Based Composite Materials

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: [1]ranveer2995@gmail.com, [2]vinayforus@gmail.com

Abstract—*Hydroxyapatite is the main inorganic component of bones and teeth. It has been extensively used as an implant material for bone substitute owing to its excellent biocompatible properties. Hydroxyapatite is used as a coating in medical implants as it is biocompatible. Hydroxyapatite based composite materials shows significant properties in the field of biomedical applications. In our present work, Hydroxyapatite was synthesized via wet chemical precipitation reaction. The reactants of the reaction were orthophosphoric acid and calcium oxide powder. Then we prepared four types of Hydroxyapatite based composites with 7.5 wt % each of Zinc oxide (ZnO), Titanium dioxide (TiO_2), Ferric oxide (Fe_2O_3) and Ceria (Ce_2O) via conventional sintering. These composite materials were taken in the form of powder. These powders were further pressed as cylindrical pellets of 10 mm diameter and 3 mm thickness in a hydraulic press. The density of these composite materials was measured by Archimedes principle using DI water and compared with the theoretical density. Subsequently, the porosity of each composite material is calculated. X-ray diffraction (XRD) analysis was carried out to identify different phases. X-ray diffraction (XRD) confirms the phase stability. The hardness of the composite materials was evaluated using Vickers' hardness tester. It was observed that hardness of composite material increased with the addition of Fe_2O_3, ZnO, and CeO_2. While the value of hardness of composite material decreased with the addition of TiO_2. However, the maximum value of hardness increased with the addition of Fe_2O_3.*

Keywords*: Hydroxyapatite, biocompatible, osteoinductive, osteoconductive, bio ceramic.*

1. INTRODUCTION

In orthopedics, hydroxyapatite is used as a coating on bone implants due to its excellent biocompatibility. Hydroxyapatite has calcium phosphate elements which are the same as present in the inorganic parts of the bone. This is the main reason that hydroxyapatite is most used in orthopedic applications. Among the main areas of application, Hap is used as filling material for bone. Another factor to consider is the phenomenon called osteoconductivity, which occurs in materials which promotes the formation of new tissue. It is known that these materials should have high porosity (the order of hundreds of microns) to allow the development of bone within and across them.

The development of new ceramics today must take into account to improve the mechanical properties for a better performance of the implants and also control the level of interaction between the material and surrounding tissue.

Hydroxyapatite becomes one of the most important bioceramic materials for artificial bone owing to its excellent biocompatibility and surface active properties with living tissues. An ideal bone implant should be osteoinductive, reasonable and easy to shape and possessing adequate mechanical properties.

Hydroxyapatite based composite materials for biomedical applications has gained lot of attention because of the high flexibility of apatite structure.

Hydroxyapatite was synthesized 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 hydroxyapatite based composite materials were taken into account for study and analysis. The parameter which we considered is hardness. We desire the hardness in our composite material so that it can easily resist to deformation when subjected to under load. We observed the various results of the addition ZnO, TiO_2 $Fe_2O_3CeO_2$ in HA i.e. Hydroxyapatite. The related study and analysis are carried out and their results are reported.

2. MATERIALS AND METHODS

To prepare the composites, powders were added to HAp in the proportions of 7.5 wt % powder. Hap + 7.5 wt% ZnO, Hap + 7.5 wt% TiO_2, Hap + 7.5 wt% Fe_2O_3, Hap + 7.5 wt% Ce_2O.

For making 1 pellet, we take

1.5gm Hap + 0.1125gm ZnO

1.5gm Hap + 0.1125gm TiO_2

1.5gm Hap + 0.1125gm Fe_2O_3

1.5gm Hap + 0.1125gm CeO_2

1.5gm Hap

For making 5 pellets, we take

7.5gm Hap + 0.5625gm ZnO

7.5gm Hap + 0.5625gm TiO_2

7.5gm Hap + 0.5625gm Fe_2O_3

7.5gm Hap + 0.5625gm CeO_2

7.5gm Hap

The powder mixes were initially hand mixed with mortar and pestle for 2 h. Following this, mixed powders were ball milled using planetary ball mill for 5 h using acetone as mixing medium.

The powders were subsequently pressed as cylindrical pellets of 10 mm diameter and 3 mm thickness in a hydraulic press under the load of 1 ton.

Mixed Powder → Die → Press → Pellets

- Firstly, the mixed powder is poured into the die.
- Secondly, placed the die in Hydraulic Press and set the required parameters.
- Thirdly, apply the load and hold it to a minute for the formation of pellets.
- Lastly, remove the formed pellet from the die.

With the help of Hydraulic Press, the formation of pellets takes place.

Parameters-

- 1 ton
- 1 min holding

"Figure 1 : Pellets Formation"

Sintering of these pellets was carried out at 1250°C for 3 h with the heating and cooling rate of 5°C min^{-1}.The density of these samples was measured by Archimedes principle using DI water. XRD analysis was carried out to identify different phases. The phase dissociation behavior of HA or formation of any reaction product was studied by analysis of XRD data.

The mechanical property, in particular hardness was measured on smoothly polished surfaces using Vickers indentation technique.

Vickers indentation tests were carried out with indent load of 2 kg.

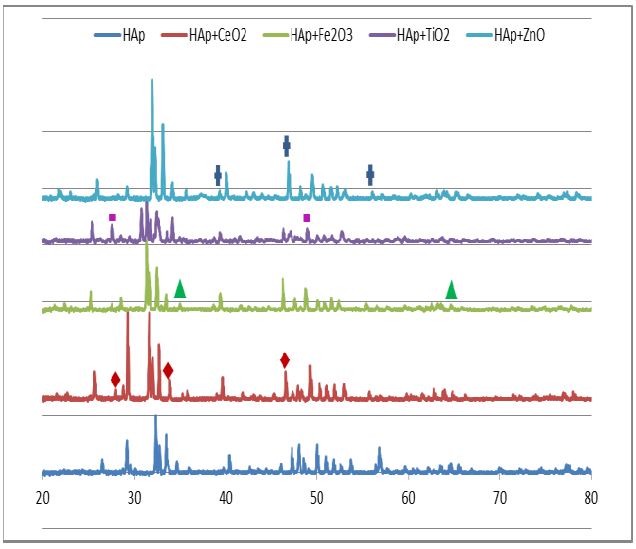
3. RESULTS

3.1 XRD Analysis

XRD analysis was done to confirm the phase stability of the samples. The various peaks formed here reveal the phase confirmation of each sample.

The phase dissociation behavior of Hap or formation of any reaction product was studied by critical analysis of XRD data. X-ray diffraction (XRD) analysis was carried out to identify different phases.

X-ray diffraction (XRD) confirms the phase stability. The comparative analysis of XRD of the given samples is shown below in the figure where the peaks formed for each sample confirms its stability.



"Figure 2 : XRD of HA based composite material"

3.2 Density

The density of these samples was measured as percentage (%) of theoretical density by Archimedes principle using water as a fluid. Archimedes density was measured by the Archimedes density measurement setup.

The density of these composite materials was measured by Archimedes principle using DI water and compared with the theoretical density. Subsequently, the porosity of each composite material is calculated.

Theoretical density was measured and after that the porosity of each sample is calculated.

The values which we got from our experiment are shown here in the tabular form.

In this particular experiment the value of the porosity is maximum in HA/CeO_2. However, the minimum value of porosity is in HA/ZnO.

"Table 1: Density and Porosity of Samples"

Sample	Archimedes'	Theoretical	Porosity
Hap	3.02 g/cc	3.14 g/cc	3.82%
Hap/TiO_2	3.02 g/cc	3.20 g/cc	5.63%
Hap/Fe_2O_3	3.15 g/cc	3.23 g/cc	2.48%
Hap/ZnO	3.19 g/cc	3.24 g/cc	1.54%
Hap/CeO_2	3.04 g/cc	3.27 g/cc	7.03%

Let, Archimedes Density = ρ_a

Theoretical Density = ρ_t

Then, Porosity = $(1- (\rho_a / \rho_t)) * 100$

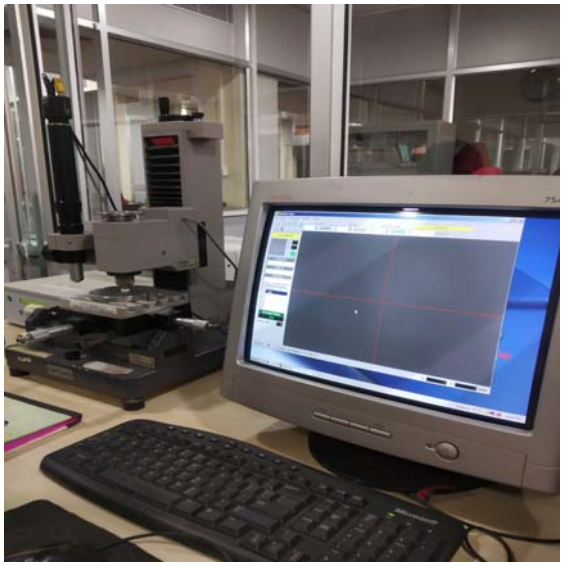
3.3 Hardness

Hardness testing was done by Vickers hardness tester.

Vickers Hardness Test

Parameter –

Load Selection - 200gm Desired Load



"Figure 3 : Vickers Hardness Tester"

Following table shows the result of the each sample undergone through testing.

"Table 2: Hardness Values of Samples"

Sample	Hardness(GPa)
Hap	2.9
Hap/TiO_2	1.9
Hap/Fe_2O_3	7.4
Hap/ZnO	3.8
Hap/CeO_2	3.8

4. CONCLUSION

The density measurement of the given samples revealed the value of porosity in each sample. X-ray diffraction (XRD) confirms the phase stability. It was observed that hardness of composite material increased with the addition of Fe_2O_3, ZnO, and CeO_2. While the value of hardness of composite material decreased with the addition of TiO_2. However, the maximum value of hardness increased with the addition of Fe_2O_3.

REFERENCES

[1] Saha, Naresh, et al. "Sintering, microstructure, mechanical, and antimicrobial properties of HAp-ZnO biocomposites." *Journal of Biomedical Materials Research Part B: Applied Biomaterials* 95.2 (2010): 430-440.

[2] Li, H., K. A. Khor, and P. Cheang. "Titanium dioxide reinforced hydroxyapatite coatings deposited by high velocity oxy-fuel (HVOF) spray." *Biomaterials* 23.1 (2002): 85-91.

[3] Bajpai, Indu, Kantesh Balani, and Bikramjit Basu. "Spark Plasma Sintered HA-Fe 3 O 4-Based Multifunctional Magnetic Biocomposites." *Journal of the American Ceramic Society*96.7 (2013): 2100-2108.

[4] Rao, R. Ramachandra, and T. S. Kannan. "Synthesis and sintering of hydroxyapatite–zirconia composites." *Materials Science and Engineering: C* 20.1-2 (2002): 187-193.

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

[6] Heimke, G., B. d'Hoedt, and W. Schulte. "Ceramics in dental implantology." *Biomaterials and Clinical Applications* (1986): 93-104.

[7] Suchanek, Wojciech, et al. "Hydroxyapatite ceramics with selected sintering additives." *Biomaterials* 18.13 (1997): 923-933.

[8] Kido, H., and S. Saha. "Effect of HA coating on the long-term survival of dental implant: a review of the literature." *Journal of long-term effects of medical implants* 6.2 (1996): 119-133.

[9] Aoki, Hideki. "Medical applications of hydroxyapatite." *Ishiyaku Euro America* (1994): 13-74.

[10] Shepherd, Jennifer H., David V. Shepherd, and Serena M. Best. "Substituted hydroxyapatites for bone repair." *Journal of Materials Science: Materials in Medicine* 23.10 (2012): 2335-2347

[11] Ogiso, Makoto, Yasuo Yamashita, and Toshio Matsumoto. "Differences in microstructural characteristics of dense HA and HA coating." *Journal of Biomedical Materials Research: An Official Journal of The Society for Biomaterials, The Japanese Society for Biomaterials, and the Australian Society for Biomaterials* 41.2 (1998): 296-303.