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Synthesis of Hydroxyapatite by Chemical Precipitation Technique and Study of Its Biodegradability

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Abstract- Biomaterials are materials which are either present naturally or synthesized in- vitro which are in contact with biological system and have various medical applications. Biomaterials are widely used now a day due to its non-viable properties and it can also be used as implants like Hydroxyapatite. Hydroxyapatite (Hap) is a biomaterial which is porous and granulated in nature and has wide application in biomedical, dentistry and orthopedics. Hydroxyapatite is biocompatible, non-toxic, non-inflammatory and even non- immunogenic agent. The present report illustrates the synthesis and characterization of Hap powder using precipitation method and evaluation of its biodegradability. Precipitation method is employed because Hap obtained after synthesis from this method will be in the pure form. It was analyzed that the sample is completely biodegradable. It is a compound of great interest mostly because of its chemical similarity to the mineral component of bones and its wide application in various fields.

Index Terms- Biomaterial; Hydroxyapatite; Biocompatible; Biodegradability.

1. INTRODUCTION

Biomaterials have been used from past centuries for body repairs. There are various defects which come across with increase in age resulting in bone injuries as bone regeneration capacity decreases [Rivera-Munoz (2011)]. Defects in oral tissues are also encountered which results from tumors or abnormal development of skeletal had resulted into the challenges for tissue engineering for their restoration [Habibovic and Groot (2007)]. Reports have been published for bone regeneration which used synthetic bone grafts. It was found that bone defects are reconstructed where the synthetic bone-like biomaterials were introduced [Hench (1991)]. Biodegradation of the material was also observed which gives the glimpse of same functionality exhibited as of bone [Moore *et al.* (2001)] [Sousa *et al.* (2010)].

Bio-ceramics have taken great importance due to its properties like biocompatibility, corrosion resistance and primarily because the bone tissue is composed largely of mineral phases, which makes them an important option for replacement of bone or to promote regeneration of it [Eric (2011)]. Different types of biomaterials are classified according to their nature and application. Chronic problems like osteomyelitis require long term usage of antibiotics that may cause several side effects. Biomaterials such as hydroxyapatite are useful for carrying these antibiotics and are also biodegradable in nature [Ginebra *et al.* (2006)]. Hap has been widely used for bone reconstruction due to their osteoconductive property and eventually Hap is degraded due to the activity of osteoclasts [Rumpel *et al.* (2006)].

Hydroxyapatite [Ca_{10} $(PO_4)_6$ $(OH)_2$] (Hap) has gained its interest in research due to its chemical and structural similarity to bone minerals and hence it is also considered as a good material for bone substitutes. Hap can be used as bone integration and have potential to improve cell proliferation and thus improve implant integration and wound healing without generating any immunogenic response [Kumar *et al.*(2010)]. It can be used to understand the properties and physicochemical behavior of biological mineral phases found in humans [Schnettler *et al.* (2003)]. Also, Hap is thermodynamically the most stable calcium phosphate at the pH, temperature, and composition of biological fluids [Sopyan (2003)].

Various methods like sol-gel approach [Agarwal *et al.* (2011)], multiple emulsion technique[Kimura *et al.*(2007)], electrodepositing techniques [Dumielie *et al.*(2008)], precipitation method (Eslami *et al.* 2008) etc. have been employed for the purpose of synthesis of Hydroxyapatite. The present report illustrates the synthesis of Hap using precipitation method due to simplicity of the process and cost effectiveness as well as its biodegradability when used as an implant.

2. MATERIALS AND METHOD

2.1. *Materials*

Calcium nitrate, potassium phosphate and ammonium hydroxide was purchased from E-Merck Pvt. Ltd., Mumbai, India. All the chemicals used were of

analytical grade and deionized water was used for the study.

2.2. *Precipitation Technique*

Titration method was used for the synthesis of hydroxyapatite by precipitation technique. The solution of 0.32M, 40 ml Ca $(NO_3)_2$ and 0.19M, 60 ml KH_2PO_4 was used for the synthesis of hydroxyapatite. Temperature was maintained at 70-85^oC and pH was checked at regular interval and maintained at 9-10 for proper reaction to take place for the formation of Hap. After the complete formation the solution obtained was dried and crushed in order to obtain the powder form.

2.3. *Fabrication of pellets*

Powder was fabricated in the form of pellets by applying pressure of 2 tons for 1 minute using palletizer. 0.8 grams of powder were weighted and pellets were formed using powdered form of Hap. Every time before loading the sample into dye, it was cleaned properly so that sticking doesn't occur and pellets can be formed in an effective manner.

After pellets are formed they were utilized for in-vitro assessment of hydroxyapatite as well as test for its biodegradability.

2.4. *Test for Biodegradation*

Degradation test was determined by soaking the pellets in Tris-HCl buffer solution and pH of the solution was maintained to 7.4 at 37^0 C. Dry weight of the pellets was noted before soaking. Just after soaking the pellets in Tris-HCl buffer its weight were noted down. The pellets soaked in Tris-HCl buffer for 12 days and at regular interval of time the pellets were taken out from the buffer, washed with deionized water and after drying at room temperature were weighed.

3. RESULTS AND DISCUSSION

3.1. *Synthesis of Hydroxyapatite*

Hydroxyapatite was synthesized successfully by the means of chemical precipitation method. White fine powder of Hydroxyapatite was synthesized using chemical root method as explained above. The solution obtained was dried and using mortar and pestle powdered form Hap was obtained. This was then used for the fabrication of pellets and test of biodegradability.

3.2. *Test for Biodegradation*

Successive degradation of the pellets was observed with increase in number of days. It is clearly seen from Table 1 that weight loss is taking place from initial 1.03 g and 1.10 g for pellet 1 and 2 respectively to 0.95 g and 0.98 g. But after 10 days no weight change was observed (Table 1).

Table 1. Consecutive readings for 12 days of pellets immersed in Tris- Buffer solution

S.NO	No. of Days	Weight	
		Pellet 1(g)	**Pellet 2 (g)**
1	0	1.03	1.10
2	2	1.00	1.06
3	4	0.98	1.02
4	6	0.98	1.00
5	8	0.97	1.00
6	10	0.95	0.98
7	12	0.95	0.98

The result obtained by in-vitro assessment depicts the biodegradation of Hap with increase in number of days. It is well known that bone has regular degradation in-vivo; similarly hydroxyapatite also possesses properties of biodegradation and will act like bone when implanted inside the body and also when used as an implant.

4. CONCLUSION

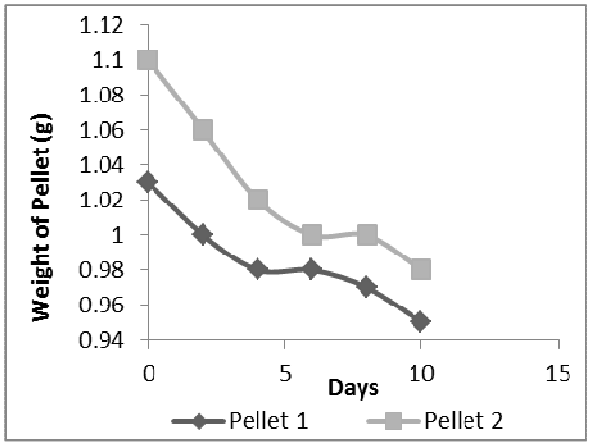

Fig.1. Degradation of Hydroxyapatite Pellets

Hydroxyapatite was synthesized successfully by the aid of chemical precipitation method which resulted into pure, fine and porous form of Hydroxyapatite. The Hap formed from precipitation method was employed for biodegradation test. Weight loss was observed when readings were taking with successive period of time. Decrease in weight gives the confirmation that Hap is showing biodegradability nature but up to certain extent (10days), after that no change in weight was observed. From this it can be concluded that when synthetic hydroxyapatite is used as an implant then it will show biodegradability and also biocompatibility. Further work will be done by employing various other methods for synthesizing Hap in order to get the best suited method. Emphasis will be given on bulk production of Hap and in-vitro assessment of its biocompatibility.

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