Synthesis and Characterization of Nanocrystalline Hydroxyapatite by Combustion Method

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***Abstract*— Among various biocompatible materials hydroxyapatite (HAP) is widely used in medical applications. As nanocrystalline Hydroxyapatite is similar in composition and crystal structure of natural bone it can be used as temporary substitute materials for human bone. A simple combustion technique for synthesizing nanocrystalline hydroxyapatite powder from eggshell has been carried out. The resulting powder was characterized using XRD, SEM and FESEM measurements. The particle size was calculated by Debye-Scherrer equation using XRD data. The range of size of resultant HAP powder was between 23nm-75nm. The average particle size was 34 nm.**

***Keywords*— Nanocrystalline HAP, combustion method, Debye-Scherrer's equation, XRD, SEM**

I. INTRODUCTION

HAP is chemically similar to the composite of bones and hard tissues in mammals. As it is useful as versatile material for implantation purposes, biologists and biomaterial scientists has placed considerable attention to this material. It has been proved that HAP is highly biocompatible and bioactive when use in bone implantation. However, the use was limited only to non-loading bearing applications due to its mechanical property such as poor sinterability. Nanocrystalline HAP could be synthesized from eggshell using combustion method. The combustion method is a very simple and inexpensive one among various methods of synthesis of HAP. With this method, it is possible to improve the mechanical properties of the material concerned by controlling the parameters such as particle size, distribution and morphology, thus rectifying the drawback of poor sinterability.

II. EXPERIMENTAL PROCEDURES

The experimental procedure is as follows. Eggshells were boiled in water for 30 min. Water and moisture content of the shell were removed by placing them in a hot air oven for a few minutes. Dried shells were crushed into fine powder using a blender. The obtained powder is then dissolved in HNO_3 forming froth. The solution was kept settle down and then was filtered into a measuring cylinder to get a known volume. The egg solution was added to 1M citric acid with thorough mixing using magnetic stirrer. Its pH value was adjusted to 9.5 by adding 1: 1 NH_4OH. 1M $(NH_4)_2$ HPO_4 solution is added to the above mixture at a rate of 1mL per minute with vigorous stirring. The precipitate of HAP was dissolved by adding HNO_3 until the pH reached to 1. The solution was then stirred until the transparent gel was formed at 70°C. The Gel obtained was kept in a preheated muffle furnace at 250°C. It was found to undergo combustion with a bright flame leaving the black coloured precursor. When this precursor was heated at 900°C

for 1hr, a pure white nanocrystalline hydroxyapatite was obtained.

III. RESULTS AND DISCUSSION

Addition of citric acid to the egg solution contributes as a fuel for combustion and it also makes the solution viscous without any precipitation. When the Gel, which composed of citric acid and the organic constituents of eggshell, undergoes decomposition, it forms into a fluffy mass showing the evolution of CO_2.

The synthesized hydroxyapatite sample was characterized by using powder X-ray diffraction (XRD: RIGAKU-RINT 2000 X-ray Diffractometer). The grain size of HAP powder was determined by Debye –Scherrer formula

$$D = \frac{0.9\lambda}{B\cos\theta}$$

where D = the size of particle

B = full width at half maximum of the peak

λ = the diffraction wavelength (0.154059 nm)

θ = the diffraction angle

The calculated range of particle size of HAP was between 23nm - 75nm. It showed narrow range of particle size. The average particle size of obtained HAP is 34nm. Fig. 1 shows the XRD pattern for Hydroxyapatite. The resulting XRD pattern was found to be exactly coincide with the reference HAP pattern. This indicates that the obtained HAP powder was in good purity. (nearly 100%) A sharp and well defined XRD pattern shows HAP is in good crystallinity as well.

The morphology and size of the nanoparticles were investigated using a scanning electron microscope (SEM) (SEM: JEOL JSM-5610). Fig. 2 and 3 show the SEM images of the sample. SEM photograph of the as-prepared HAP powder shows that individual hydroxyapatite particles formed in globular shape. The nanometric primary particles agglomerated tightly into micrometric aggregates of various shapes and sizes.

Scanning electron microscope measurement for morphology evaluation of the powder was performed on a JEOL FESEM instrument (model JSM 6700F). Fig.4, 5 and 6 show the FESEM images of the sample. The photographs show the particle size distribution is from 125-142 nm.

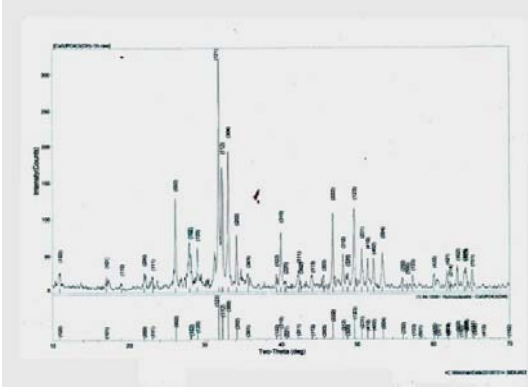

Fig.1 XRD pattern of HAP powder

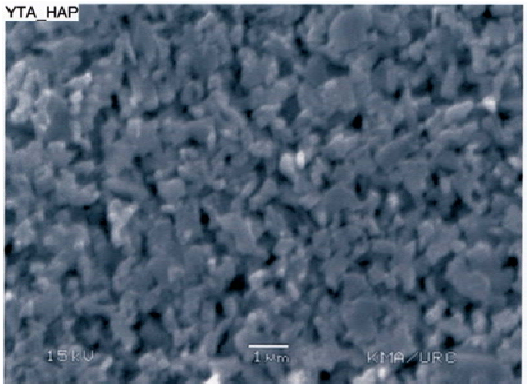

Fig.2 SEM image of the HAP powders

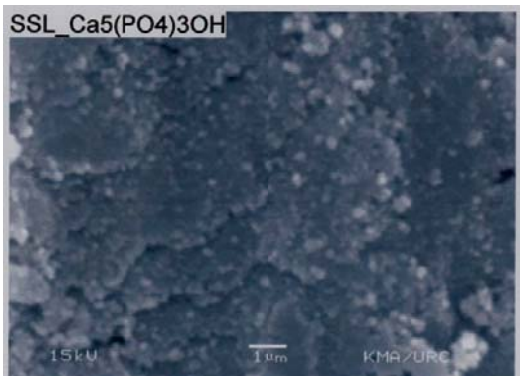

Fig.3 SEM image of the HAP powders

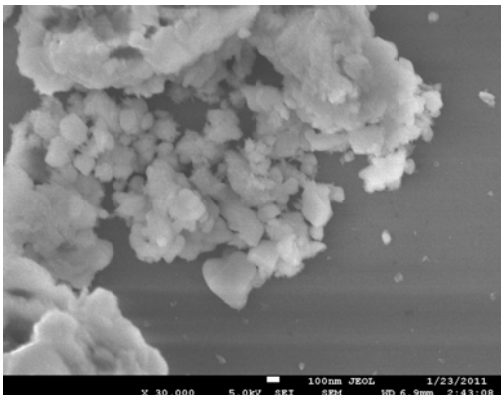

Fig.4 FESEM image of the HAP powders

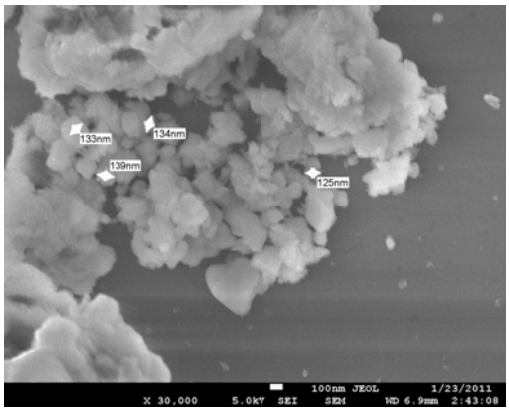

Fig.5 FESEM image of the HAP powders

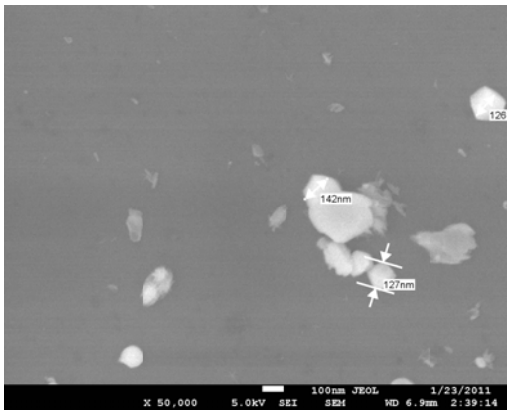

Fig.6 FESEM image of the HAP powders

IV. CONCLUSIONS

In this research, a simple combustion technique was used for synthesizing nanocrystalline hydroxyapatite powder from egg shell. X ray diffraction method was employed to characterize the sample. The particle size was calculated by Debye-Scherrer equation using XRD data. The range of particle size was between 23nm - 75nm. The average particle size of HAP was 34nm. Synthesis of HAP starting from eggshells at a low temperature is a method for producing a useful bioceramic material utilizing easily available and low cost raw materials, the eggshells. This method is the one which enable to control the parameters such as particle size, distribution and morphology for improving the mechanical properties of HAP.

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