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PREPARATION AND CHARACTERIZATION OF HYDROXYAPATITE FROM FISHBONE

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ABSTRACT

Hydroxyapatite is the major mineral component of bone and teeth, with a chemical formula of $Ca_{10}(PO_4)_6(OH)_2$. In bones, the minerals are mainly deposited in the form of calcium phosphate compounds with the great majority existing as apatite and only a small amount of them are carbonate containing apatites. Fishbone were converted to hydroxyapatite (HAP) by a heat treatment method at different temperatures and for different conversion durations. X-ray diffraction and Fourier transform infrared spectroscopy analysis confirmed that the samples were mainly highly crystalline hydroxyapatite ceramics. The final product is characterized by X-ray diffraction, scanning electron microscopy_SEM and FT-IR. High temperatures and smaller particle will be produced Hydroxyapatite which is the particle size using 25um with temperature of 1100°C.

Keyword: Hydroxyapatite, Fishbone, heat treatment, particle size and temperature.

INTRODUCTION

Hydroxyapatite (HA, $Ca_{10}(PO_4)_6(OH)_2$) is the major mineral component of bone and teeth. It can promote faster bone regeneration and direct bonding to regenerated bone without intermediate connective tissue and its synthetic form is one of the most widely used biomaterials for reconstruction of the skeleton due to the lack of local or systemic toxicity together with its osteoconductive properties. It is used as an implant material both in its bulk mainly porous form, for filling in or reconstructing bone defects, and as a thin coating on metals, titanium and CoCrMo alloys, for hip, knee, and dental prostheses. Although success rates of these kinds of implants are dependent on bone-implant osteointegration, the success and long-term survival of the implants are also dependent on the prevention of bacterial infection after implant placement. Therefore, there is high clinical demand for synthetic bone substitution materials.[1](Schwartz et.al., 1999).

The main objective of this research is to prepare and characterize the hydroxyapatite from fish bone (*cat fish*) for bone substitution. The main objectives are to prepare hydroxyapitate(HA) from fish bone, to characterize hydroxyapatite from from fish bone(cat fish) and to compared hydroxyapatite from fish bone with synthesis hydroxyapatite.

EXPERIMENTAL METHOD

2.1 Sample Preparation

In the sample preparation steps, fishbone are soaked in the acetone for an hour. Then wash it with distill water and let it dried. The acetone is used to remove the collagens, fats and other impurities.

2.2 Fourier transform infrared spectroscopy (FTIR)

The infrared spectra in this work presented were recorded using the ALVATAR 380 FTIR THERMO NICOLET. The experiment was done in the room temperature to determine the component and phase of the converted fish bone

2.3 X Ray Diffraction (XRD)

X-ray diffraction is now a common technique for the study of crystal structures and atomic spacing. X-ray diffraction is based on constructive interference of monochromatic X-rays and a crystalline sample. These X-rays are generated by a cathode ray tube, filtered to produce monochromatic radiation, collimated to concentrate, and directed toward the sample. The interaction of the incident rays with the sample produces constructive interference (and a diffracted ray) when conditions satisfy Bragg's Law ($n\lambda=2d \sin\theta$).
peak in intensity occurs.

2.4 Tabletop Microscope (SEM)

SEM is used to observe the microstructure of the original bone and converted fishbone. The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample.

RESULTS AND DISCUSSION

3.1 Sample Prepared

Fish bones were obtained from fish (Commercial name: Cat fish). It was then crushed and turn into the powder of hydroxyapatite. The powder of HA are calcined at various temperatures, 800°C, 900°C, 1000°C and 1100°C for 2 h.

3.2 FTIR spectroscopy

According to C.P Yoganand et al., 2009 for FT-IR analysis, in the transmittance mode, shows the presence of carbonate group at around 1410-1450 cm^{-1} and 875 cm^{-1}, hydroxide group at around 3500-3200 cm^{-1}. For phosphate group at 1049-1090 cm^{-1} and 1950-2200 cm^{-1}, 962 cm^{-1} and 560 cm^{-1}.[2]Based on the graph, the value for (a) 713cm^{-1}, (b) 1034 cm^{-1}, (c) 1513 cm^{-1} and (d) 2985cm^{-1}. From the result, it is known that (a) producing hydroxyl ion, (b) phosphate ion while (c) and (d) produced carbonate ion. Compare with the three different sizes, pure HA with the size 25μm has the lowest intensity. So that 25μm size is chosen since it produces lowest intensity.

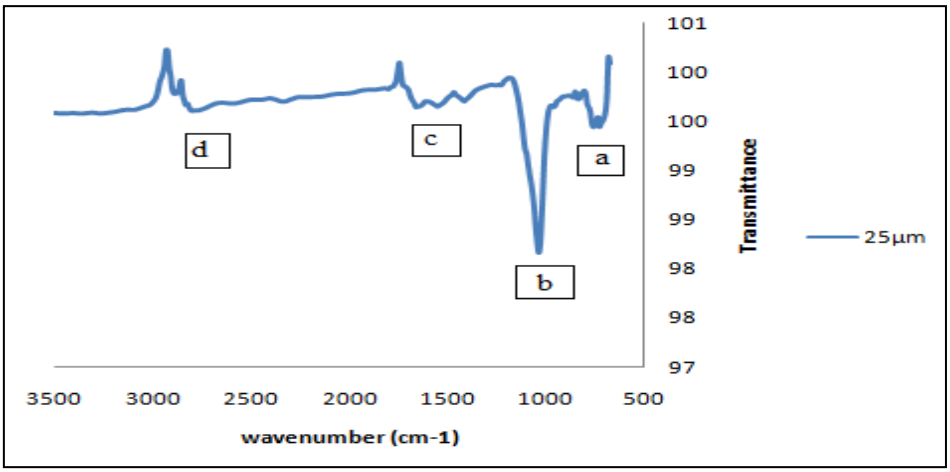

Figure 1: FTIR graph for 25µm of hydroxyapatite before heat treatment

From the graph below, the highest temperature which is 1100°C has the lowest intensity also has the phosphate ion and lowest impurity which is good to produced the best HA. After the heat treatment, the temperature of 1100°C only the phosphate is exist which is at 1026 cm^{-1} which has almost no impurities.

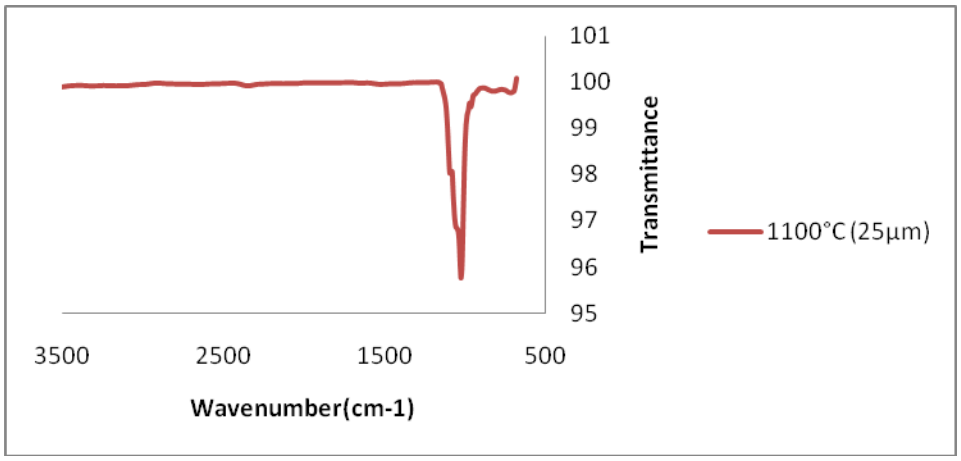

Figure 2: FTIR graph of 25µm with temperature of 1100°C

3.3 X Ray Diffraction (XRD)

The powder was well-crystallized, and all peaks from the XRD pattern were identified as HA. From the graph, the sample with 25µm size has the highest intensity compared to the other sizes which is 90µm and 150µm. The sample with highest intensity has been chosen to sintered at 800°C until 1100°C.

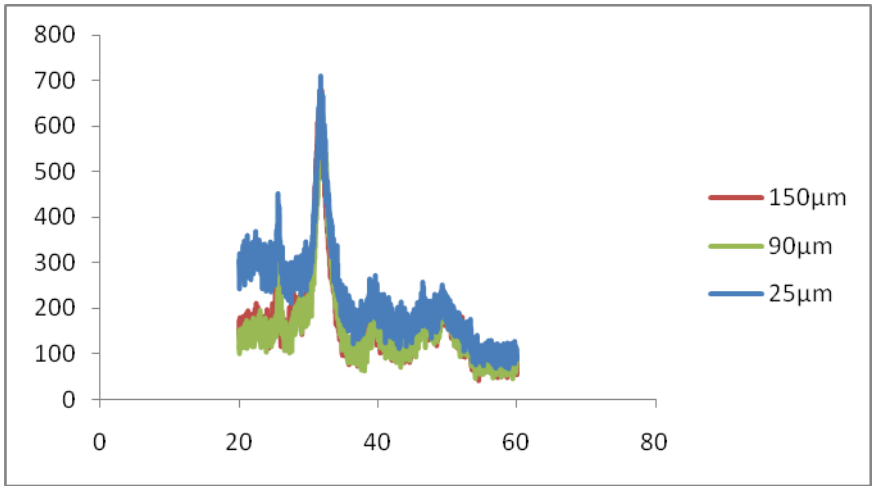

Figure 3: XRD graph of 25um for pure hydroxyapatite before heat treatment

Shipman et al reported that they found that there was a gradual increase in hydroxyapatite crystal size associated with an increase in the heat treatment temperature [25]. The black powder, produced in this study, began recrystallization at about 600 oC without decomposing to any other compound of the calcium phosphate family.

Figures below elicit the XRD patterns of fishbone heated at various temperatures which is 800°C, 900°C, 1000°C and 1100°C. These diffraction patterns show a gradual increase in the degree of sharpness of peaks with increasing heat treatment temperature. These results confirm the previous discussion regarding the effect of the heat treatment temperature on the crystal size of hydroxyapatite.[3] (S. Madhavi et al., 2005).

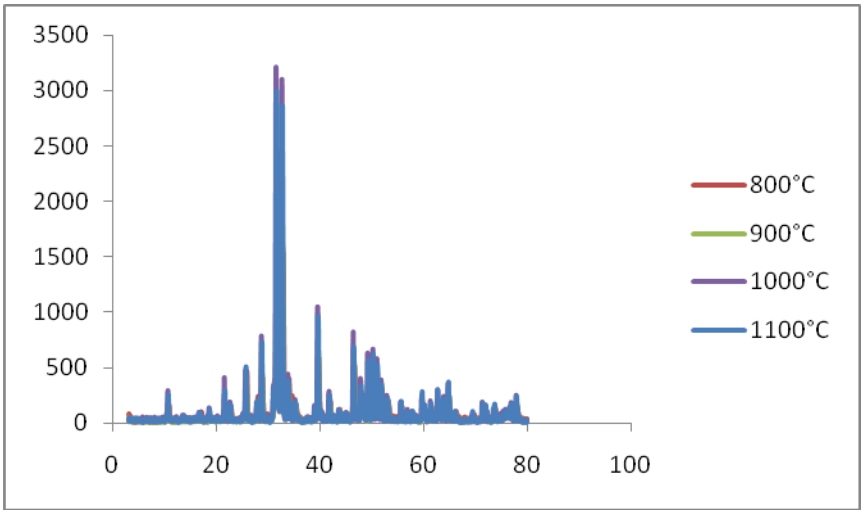

Figure 4: XRD graph of 25µm different temperature of hydroxyapatite after heat treatment

From the graph, it can be seen that the sample with the temperature of 1100°C has the highest intensity compared to the other temperature. The temperature of 1100°C was detected has the minor impurity elements and the most suitable to produce the best HA.

3.4 Tabletop Microscope (SEM)

The morphologies of the pure fishbone are shown in figures below before calcined in three different sizes which are 25µm, 90µm, and 150µm. These SEM images gave insight into the hydroxyapatite structure with respect to particle size and shape. Compared to the three different sizes, it shows that HA microstructure for 25µm has very dense structure which is close to each other. Dense structure has fewer pores than the other two sizes which is 90µm and 150µm.

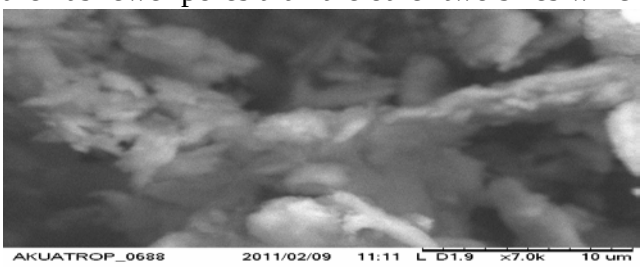

Figure 5: Image For HA before treatment with the size of 25µm

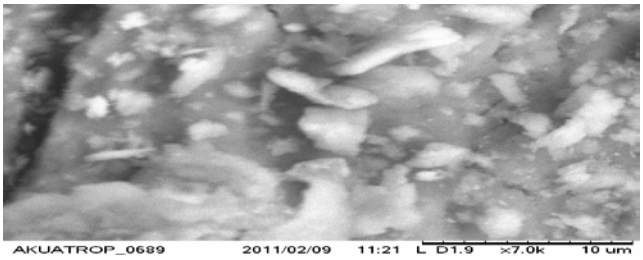

Figure 6: Image For HA before treatment with the size of 90µm

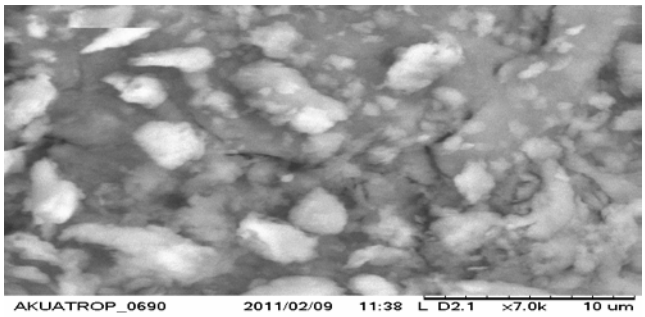

Figure 7: Image For HA before treatment with the size of 150µm

When bone is heated gradually from 200°C to 1600°C, macroscopical (i.e. colour) changes and also microstructural changes occur which include recrystallisation of the bone mineral. The image shows that the high temperature will remove all the impurities.[4] So that, HA with the temperature of 1100°C has fewer pores.

J.LHolden et al.,1995) attempted SEM observations of heat treated human bone and reported that the organic components of the bone tissue are eliminated at 400°C.[5]

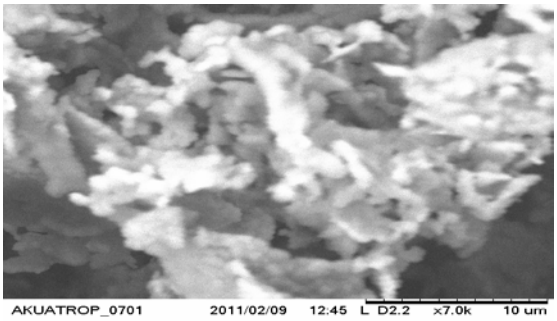

Figure 8: HA with temperature 1100°C

CONCLUSION

This study showed that fishbone can be used as a natural source for production of hydroxyapatite. Hydroxyapatite from fishbone is prepared and characterized using four different instruments which are Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR) and X-Ray Diffraction (XRD). Three different sized will be compared in this study which are 25μm, 90μm and 150μm and the best sized that will produced a good quality of hydroxyapatite is chosen and compared with four different temperatures which are 800°C, 900°C, 1000°C and 1100°C. The best sample will choose which is has same characterization with the natural hydoxyapatite. Based on the FT-IR analysis, 25μm has the lowest intensity compared to the other two sizes which are 90μm and 150μm and the highest temperature of 1100°C has the lowest intensity and lowest impurity is the good quality of HA. For XRD analysis, the smallest particle which is 25μm has the highest intensity compared to the other sizes. The best size to be sintered then has been calcined with the temperature of 1100°C which has the highest intensity. From the SEM analysis, the size with minor of impurities is 25μm which is the most suitable one to produce of HA. Same thing with the temperature, the highest temperature has removed all the impurities. The best temperature that has been used is 1100°C. The process will produce hydroxyapatite acceptable for use in orthopaedic and dental applications. The results of this study showed that the optimum heat treatment temperature to prevent phase transformation of hydroxyapatite. In addition, heating the fishbone at temperatures up to 1100°C indicated the fact that this natural hydroxyapatite is stable at temperatures lower than 1100°C. The highest temperature and the smaller size will produce the best hydroxyapatite.

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REFERENCES

[1] Y. Tanaka, Y. Hirata, R. Yoshinaka & J. Ceram (2003). Process. Res. *Inorganic Materials* 38, 973-984

[2] Yoganand, C.P., Selvarajan V., Jushu Wu & Xue Dong Feng. (2008). Processingof bovine hydroxyapatite (HA) powders and synthesis of calcium phosphate silicate glass ceramics using Dc plasma torch. *Vacuum*. 83:319-325.

[3] S. Madhavi, C., Ferraris & T.J. White. (2005). Solid state chemical. *Material science*. 2838-2845

[4] M. E. Bahrololooma, M. Javidia, S. Javadpoura & J. Ma (2009). Characterisation of natural hydroxyapatite extracted from bovine cortical bone ash. *Journal of Ceramic Processing Research* **10**, 129-138

[5] Yuan Gao, Wen-Ling Cao & Xiao-Yan Wang (2006).Characterization and osteoblast-like cell compatibility of porous scaffolds: bovine hydroxyapatite and novel hydroxyapatite artificial bone. *J Mater Sci: Mater Med* **17**, 815–823

[6] S.J. Lee & S.H. Oh (2003). Fabrication of calcium phosphate bioceramics by using eggshell and phosphoric acid. *Materials Letters* **57**, 4570–4574

[7] K. Prabakaran & S. Rajeswari (2006). Development of Hydroxyapatite from Natural Fish Bone Through Heat Treatment. *Trends Biomater. Artif* **20**, 20-23