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Effect of Calcination on The Properties of Hydroxyapatite from Patin Fish Bone for Biomedical Use

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Abstract: Most of the inorganic components in human bones are calcium phosphate, which occurs naturally. Hydroxyapatite (HAp) has been used extensivelyto rebuild bone. is the chemical formula for HAp, and the Ca/P ratio is 1.67? This study is to extract HAp powder from patin fish bone and investigate the effect of calcination on the properties of HAp. The extraction process involved cleaning, boiling, drying, grinding, and milling process to obtain micron size powder of the patin fish bone. The characteristics of produced HAp powder was characterized withScanning Electron Microscope (SEM), Energy Dispersive X-ray Spectroscopy (EDS), X-ray Diffraction (XRD), and Fourier Transform Infrared Spectroscopy (FTIR). EDS and XRD testing confirmed that calcium and phosphorus elements existed in the HAp samples. The best hydroxyapatite is from *patin* fish bone sample with a calcination temperature 900 with Ca/p ratio of 1.64.

Keywords: Hydroxyapatite, Ca/P, Calcination

1. Introduction

To regenerate bone, hydroxyapatite (HAp) has been frequently used. Most of the inorganic elements in human bones are a naturally occurring form of calcium phosphate. HAp chemical formula is with a Ca/P ratio of 1.67. The introduction should describe general information on the subject matter area of study. It is usually arranged in such a manner to gradually bring to focus the specific motivations of the current study, the research questions, the problem statements, the hypotheses, the objectives, as well as the expected outcome [1].

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Biomaterials refer to the complete body system and are made up of several parts. Biomaterials are used in dentistry and medical applications for involvement with living tissue. These are frequently connected to hip replacement implants, cardio-vascular reinforcement implants, and dental fillings. Many recipients, including the elderly with longer life expectancies and younger persons with cardiac issues, trauma, or genetic abnormalities, benefit from these applications by improving their quality of life [2]. Bones have a protective role, particularly in important regions like the body and brain where an injury could be fatal. It is arranged in these places to absorb the most energy with the least amount of damage to the bone itself. The structure of bones is arranged hierarchically, from nanometer to millimetre scales. Bone is predominantly made up of carbonated apatite, which makes up around 65% of its weight. However, because the bone is a living tissue, it also contains an organic component that makes up roughly 20 to 25% of its makeup [3].

Calcium phosphate has been utilized in biomaterials, particularly for the regeneration of tough tissues like bone and teeth [4]. Due to its controlled breakdown, which enables bone formation, biphasic calcium phosphate, a compound made up of hydroxyapatite and β–tricalcium phosphate is suggested as a suitable choice for the creation of bone scaffolds. In bodily fluid, hydroxyapatite, is thermodynamically stable in its crystalline state and closely resembles bone mineral. Without creating any local or systemic toxicity, inflammation, or foreign body reaction, hydroxyapatite can integrate into bone [5].

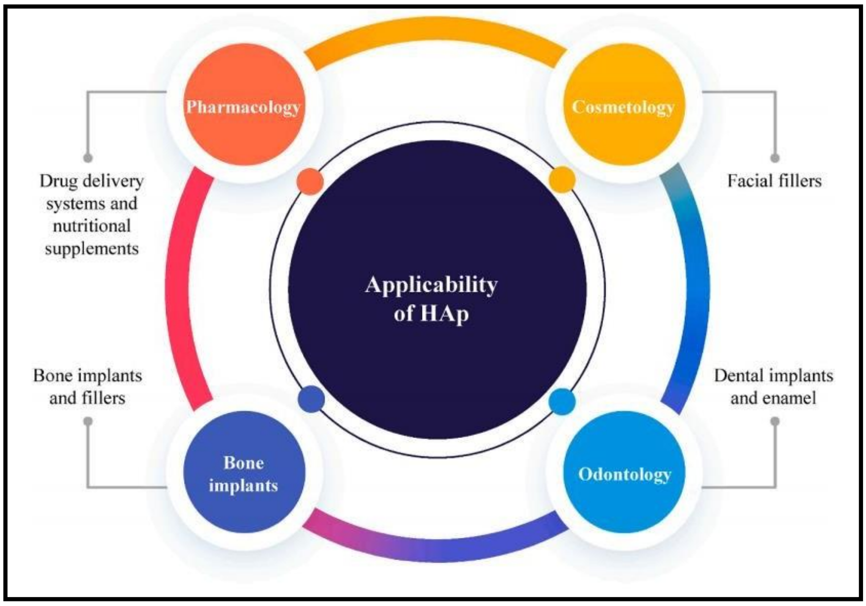

Figure 1: Applications of biomedicals of Hap [6]

2. Materials and Methods

Figure 2 shows the methodology of the extraction of hydroxyapatite from *patin* fish bone. There are preparation of materials needs to be done before carrying out calcination procedure and characterization and testing.

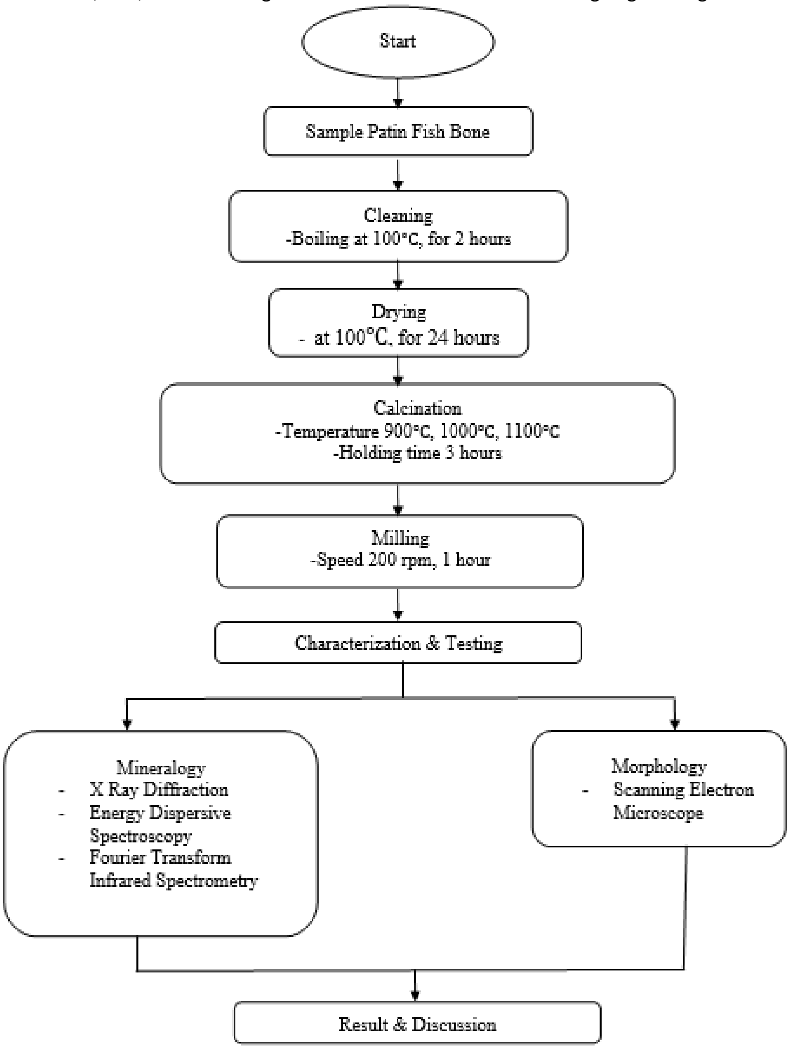

Figure 2: Methodology chart

2.1 Materials and Apparatus

The main materials that are used in this study are *patin* fish bones, using the bones from the whole body,from head to tail. The sample powder of hydroxyapatite from *patin* fish bones was done using heat treatment calcination with 3 hours holding time with a cooling rate of 5. Figure 3 shows the calcination profile for producing Hap with three different temperatures.

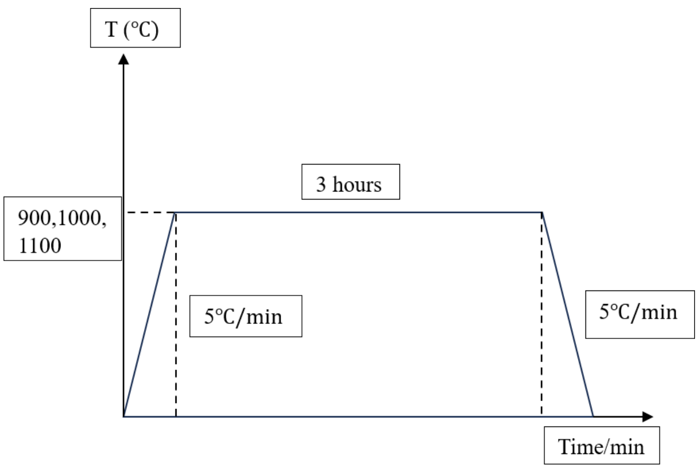

Figure 3: Calcination profile for producing HAp

2.2 Methods

To produce sample powder of patin fish bone for hydroxyapatite, patin fish were used and the bones from the whole body were cleaned and boiled at 100 for 2 hours. After boiling the bones, it is dried by using drying oven at 100 for 24 hours. Then it is being grind and milled for 2 hours at 200 rpm speed. After being milled, it goes through calcination process, Figure 4 shows patin fish bone before calcination and Figure 5 shows patin fish bone after calcination.

Figure 4: Patin fish bone before calcination

Figure 5: Patin fish bone after calcination

2.3 Testing Methods

The extraction and the characterization of the HAp powder was assessed using Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), X-ray Diffraction (XRD) and Fourier Transform Infrared Spectrometry (FT-IR).

3. Results and Discussion

3.1 SEM analysis of Hap

Figure 6, Figure 7, and Figure 8 show SEM analysis of sample powder patin fish bone temperature 900, 1000 and 1100 with the magnification of X3500. The morphology surface of 900 shows large pores in the powder, the shape of the powder is large with rough edges and surfaces. For 1000morphology surface, the shape of the powder is small with round shapes and surfaces. Based on morphology surface of 1100 the sample powder analysis shows an irregular rough surface with large pores.

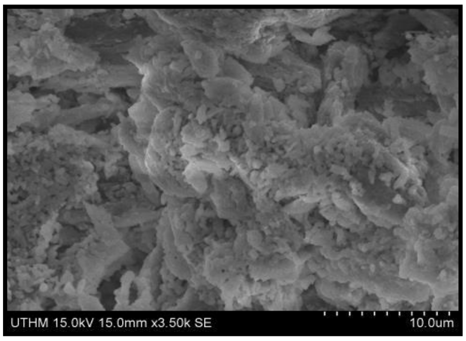

Figure 6: Sample powder 900

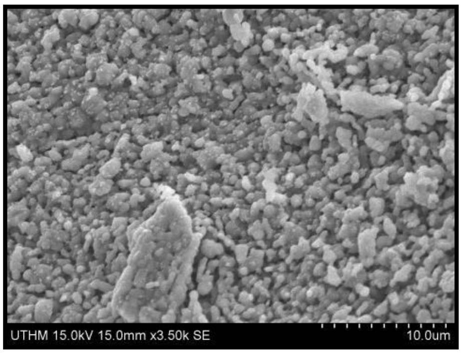

Figure 7: Sample powder 1000

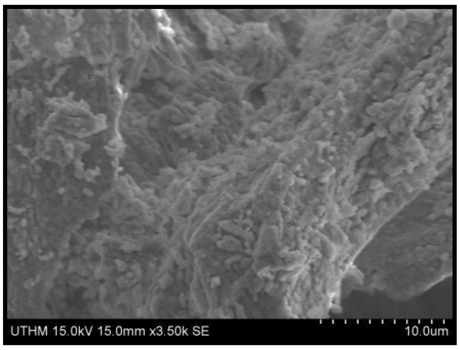

Figure 8: Sample powder 1100

3.2 EDS analysis of HAp

Based on the electron diffraction of the HAp powder from SEM analysis, the weight percentage and the atomic percentage of the element composition were calculated. The molar ratio of Ca/P value were calculated using Eq.1:

$$Molar\ ratio\ of\ \frac{Ca}{P} = \frac{Molar\ ratio\ of\ ca}{Molar\ ratio\ of\ P} = \frac{(\frac{Weight\ percentage\ of\ ca}{molecular\ weight\ of\ ca})}{(\frac{Weight\ element\ of\ P}{molecular\ weight\ of\ P})} \qquad Eq.1$$

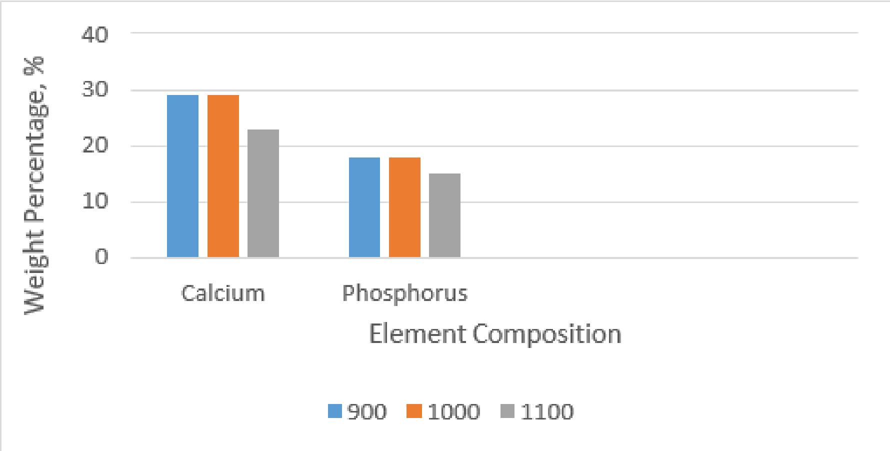

Figure 9: Bar chart of weight percentage, % against element composition

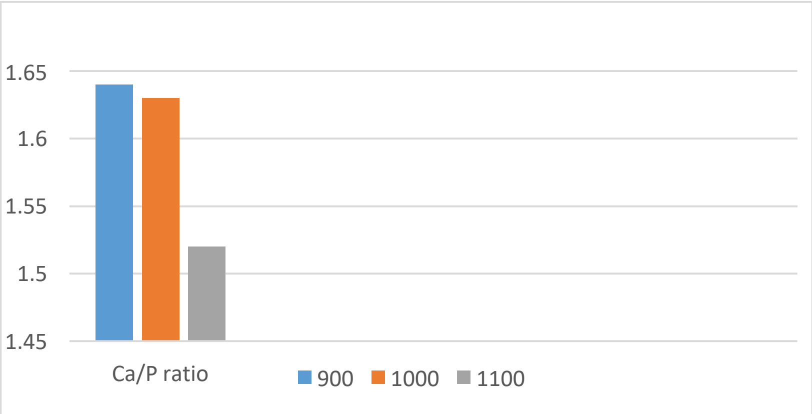

Figure 10: Bar chart of Ca/P ratio of Hydroxyapatite

From Figure 9, it shows that for the Calcium element, the Calcium (Ca) element was significantly low in *patin* fish bone powder of temperature 1100℃ for only 14.97%. From the SEM analysis, it shows that there are many large pores in the powder sample, affecting the sample powders.Figure 10 shows the Ca/P ratio with the most suitable sample for hydroxyapatite is the 900℃ temperatures with 1.64.

3.3 XRD analysis of HAp

The produced HAp powder from patin fish bone after calcination at temperature 900°C, 1000°C, and 1100°C respectively were characterized using XRD analysis. The data was collected in the range of 10°to 70°, with step size of 0.020°. The narrower the crystalline peak, the sharper the crystalline structureof the diffraction. It shows in the result of 900°C. This means that from calcination process, it succeeds in extracting HAp from patin fish bones. As the temperature increases, resulted in sharper and narrower crystalline peak diffraction pattern of Calcium Phosphate, and it shows that the crystallinity of the sample powder increases proportional to the calcination temperature.

3.4 FTIR analysis of HAp

Figure 11 shows the characteristic band for the chemical present appeared along range 1410.67 cm^{-1} to 628.62 cm^{-1}. The analysis recognizes the presence of CH with bending vibration 1087.86 cm^{-1} for 1000℃ and 1087.70 cm^{-1} for 1100℃. Phosphate groups are recognized at the peak of 1022.85 cm^{-1} for 1000℃ and 1020.78 cm^{-1} for 1100℃.

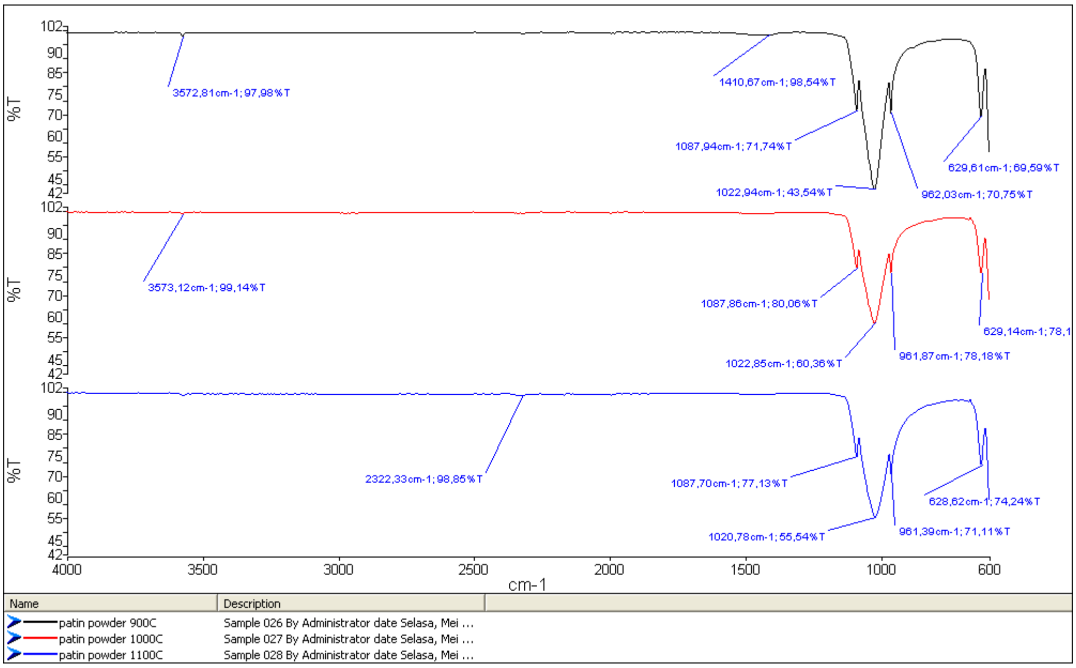

Figure 11: Ftir analysis

4. Conclusion

The patin fish bone wastes may be utilized for biological applications, such as bone and dental implants, according to this study. The HAp powder was extracted using calcination procedures. The Ca/P ratio increased proportionally with the increase in calcination temperature, according to the EDS results of the generated HAp powder. This indicates that the natural HAp was successfully extracted from the patin fish bone by the calcination procedure. It is to keep in mind that additional elements, like the initial material's chemical makeup, surface morphology, and particle size, might influence the effect of calcination on how HAp behaves.

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