The Fabrication Porous hydroxyapatite Scaffold Using Sweet Potato Starch as a Natural Space Holder

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The Fabrication Porous *hydroxyapatite* Scaffold Using Sweet Potato Starch as a Natural Space Holder

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Abstract. A study has been conducted to evaluate the effect of sintering temperature on the mechanical and physical properties of hydroxyapatite ceramics. In this study the fabrication of porous hydroxyapatite derived from bovine bone obtained from food scraps of local restaurant. Bovine bone is cleaned and boiled to remove the fat content. Then cut into small pieces to facilitate the process of making powder. Further calcined with a temperature of 600°C, then crushed with mortar to produce powder. Sweet potato is used as a Space holder which act as pore makers in this HA ceramic. A ball milling process with a speed of 225 rpm rotation for 1 hour was used to mix raw material. The powder is sintered with temperature of 1050, 1100 and 1200 ° C then the powder is characterized by XRD to evaluate the formation of HA and β-TCP phases. The results of this study obtained a hydroxyapatite sample with a maximum shrinkage 44.46% at a temperature of 1200°C and the highest porosity was 49.35% at 1050°C and the best compressive strength of 4.41 MPa achieved at 1200°C. Morphology of sample was observed using SEM and reveal porosity size of about 17.5µm and 12.5µm. Interconnected porous was clearly observed on the sintered samples. Porosity is expected to facilitate the circulation of body fluids and encourage the growth of cells.

1. Introduction

Bone, collectively called the skeleton, are the hard tissues in the human body that performs as a protective and supporting structure. Bone is also organ that dynamically perform the remodeling process and adapt to the working loaded by changing its form. Bone is composed by a complex structure with organic and inorganic constituents. Over service time bone is sometimes unable to perform its function properly because of a disability that may be caused by illness, accident or age factor. In an effort to overcome/cure defects in the bone researchers introduced material implants called biomaterial [1].

Biomaterial is intended to replace and/or remodeling body part through direct interaction with organs. In general, a material must have the ability to conform with the environment where it is placed (biocompatibility), not harm the body (bioinert), and act as a medium to cell or tissue growth (bioactivity) [2, 3]. In addition, biomaterial must also be able to integrate with the surrounding tissue (osterointegrity) and provide an adequate the mechanical properties [4].

Hydroxyapatite (HA, $Ca_{10}(PO_4)_6(OH)_2$) is a type of calcium phosphate mineral containing hydroxide [5]. The chemical composition and crystal structure hydroxyapatite is similar to that of bones and teeth. Hydroxyapatite is nontoxic material to be used as an implant material because of its non-toxic nature, bioactive, has biocompatibility with adjacent tissue, non-corrosive, and can encourage new bone growth in its porous structure [6]. However, HA has weaknesses that are brittle, not osteoiconductive, low mechanical properties and low structural instability when mixed with body fluids [7].

In accordance with the natural conditions of bone, there is a porous structure with bone surface area is wider and smaller than the compact bone mass. Porous bone is composed of trabeculae or plates that form flat webbing and contain collagen fiber. Currently to provide engineering tissue and drug delivery systems applications researchers have developed porous hydroxyapatite [8, 9]. In tissue engineering applications, porous structures allow for bone and surrounding tissue growth and facilitate the flow of vitamins and nutrients to the bones and other parts of the body through the tissues and blood vessels calipers [10].

Several methods have been developed to produce porous hydroxyapatite. Yao et al. developed a method of slip casting to make porous HA with controlled porosity [11]. The Camphene Freeze Checking Method also developed to produce porous HA with interconnected pores [12]. Another method of producing porous ceramics through the polymeric sponge replication method [13]. The polymeric sponge replication method performed by impregnating cellulosic sponges with HA slurries however the results show an interconnected-pores with poor mechanical strength for load bearing applications. Porous Ti6Al4V / HA composites through powder compaction with the application of two types of space holder such as Sodium Chloride (NaCl) and polymethyl methacrylate (PMMA) [14]. In this study, high porosity was achieved up to 43.9%. In addition, interconnections pore with applicable sizes are formed with appropriate pore sizes (2–25 μm). Among these methods powder metallurgy via powder sintering methods is a conventional processing that has been widely used to produce ceramic material because of its simplicity in process. In addition, powder compaction with applying a material as space holder provided simple method and introduced high porosity level

Fabricating porous structures with the space holder method, the size of the porosity formed depend on the temporary particles added to the matrix. This temporary particle will act as a space holder that will leave marks as pores. Some researchers also implemented another type of space holder to produce porous material i.e. saccharose [15], salt [16], carbamide [17], corn starch dextrin [18], tapioca starch [19] and magnesium powder [20]. The main objective of this work is fabricating porous hydroxyapatite Al2O3 composite with the application of space holder materials (i.e. green bean starch) through the powder compaction method.

2. Materials and Method

Hydroxyapatite (HA) is derived from cow bone obtained from local market which is from the leg or femur bone part. The bovine bone is cleaned from the remaining food stuck by washing and dripping with a wire brush then boil it for 5 hours to clean up the fat content of the bone and clean the marrow and tendon easily. Subsequently, dried the bovine bone under the sun rise until white color and reduce the size by grinding into small pieces. The calcination process was performed using electric furnace with 3°C/min heating rate up to 600°C then hold for 30 minutes and cooled in the furnace. The process of calcination is done in order to decompose organic substances and water contain.

Raw material for space holder that is sweet potato (*Ipomoea batatas L.*), sweet potato then peeled, cleaned, cut and dried for powder making process. The space holder is then crushed by using mortar and sieved according to the desired size.

The 80%wt of calcined hydroxyapatite was mixed with 20%wt of space holder by using ball milling at 225 rpm for one hour. A unidirectional compaction process with pressure of 2000 Psi was applied to produce cylindrical sample with size length 12 mm and diameter 10 mm. The samples were then fired in conventional electrical furnace up to 600°C and hold for 60 minutes to completely burn out of space holder then continued until 1050, 1100 and 1200°C respectively. Sintering process was done with 5°C/min heating rate and 3 hours holding time final desired temperature.

The calcined hydroxyapatite and sintered samples are characterized by using XRD machine (Rigaku MiniFlex 600), while space holder was investigated it weight lost process using Thermo Gravimetric Analyzer (TGA). Apparent density was calculated using Archimedes' law and morphology of the samples was scanning using Scanning Electron Microscope (SEM) type Inspect S50 from FEI Company. Mechanical properties were verified to spot compressive strength of the samples via Hydraulic Universal Material Tester.

3. Results and Discussion

Thermo gravimetric analyzer (TGA) testing was performed to determine the rate of weight change on the temperature function of sweet potato powder from room temperature to 600°C. As can be seen from figure 1. at the initial temperature of 49.78°C the weight of 100% powder of sweet potatoes.

Weight loss began at a temperature of 200.2°C with a weight of 94.88% and at a temperature of 255.55°C a very noticeable weight loss of 77.74% weight. At 350.03°C temperature the sweet potato powder weighs 1.24% until the final temperature 586°C the weight of the sweet potato is 0.99%. Weight reduction does not reach 0% because at 600°C temperature sweet potato powder is still remaining in form of ash from green bean powder itself. The burn of this starch powder will create pores on the hydroxyapatite specimen.

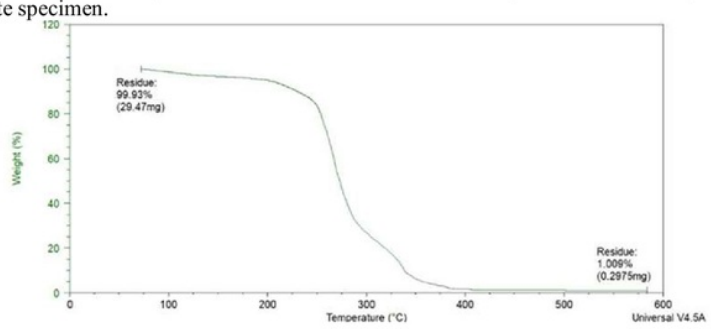

Figure 1. Curve of sweet potato starch thermo gravimetric analyzer result

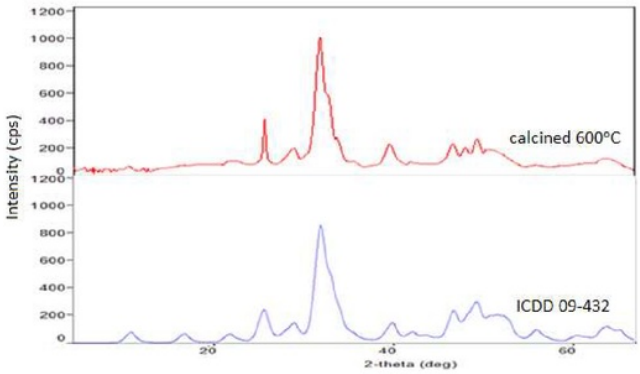

Figure 2. XRD result of calcined bovine bone

As depict in figure 2 we can see that an identical XRD of both calcined and ICDD 09-432 standard peaks respect to ordinate 2θ degree. It is confirm that calcined bovine bone produced hydroxyapatite powder.

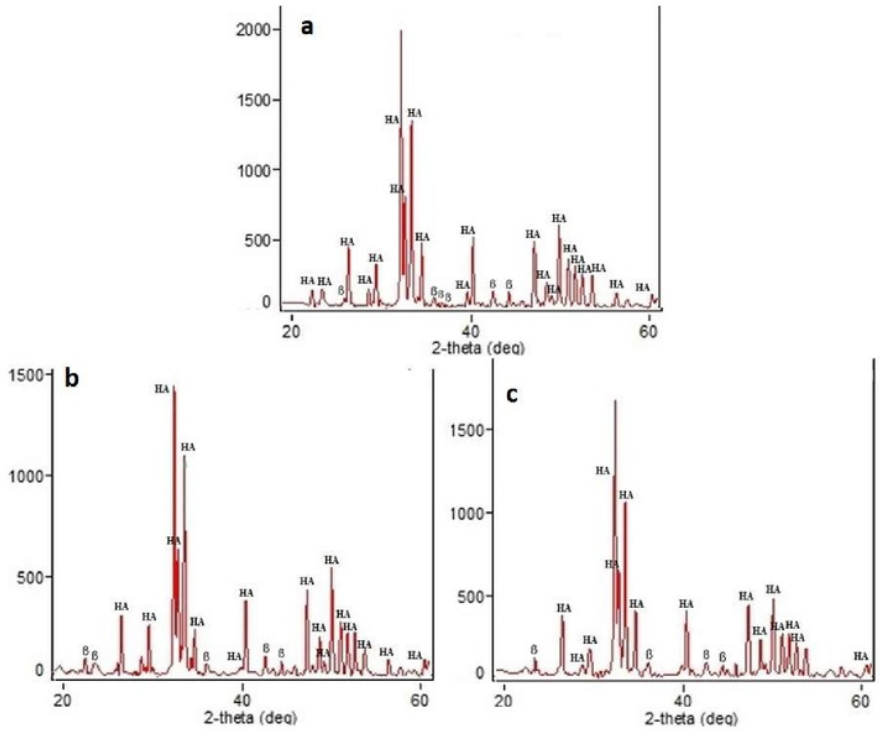

Figure 3. XRD result of samples sintered at a) 1050°C, b) 1110°C and c) 1200°C

Figure 3 shows XRD result of samples sintered t various temperature. The results show an increase in peak intensity and decreases in peak width indicating increased crystallinity and crystallite size. Shifting at the peak position is observed from standard positions. The results show a dehydroxyl process in hydroxyapatite during heating. The bovine powder which calcined at 600°C is initially pure hydroxyapatite (fig. 2) for all sintering regime is experiencing decomposition process in to β-TCP.

Shrinkage occurs due to the solidification of the specimen after sintering. The loosening of specimens occurs due to diffusion of hydroxyapatite during sintering process. From the sintering results (fig. 4) The smallest shrinkage is present at a temperature of 1050°C and increase at a temperature of 1200°C. The result shows that high temperatures resulting higher diffusion of samples. Theoretically the shrinkage during sintering is homotetik due to isostatic suppression of the green body. Both density and shrinkage measurements thus show that almost full density is achieved after sintering at high temperatures.

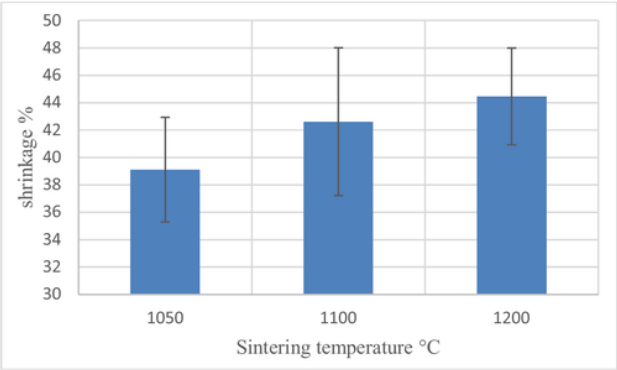

Figure 4. Shrinkage samples at various sintering temperature

Density testing using Archimedes theory. Apparent density and porosity result depicted in figure 5. At sintering temperature variations i.e. 1050, 1100 and 1200°C the average of apparent density is 1.6, 1.62 and 1.29 g/cm^3 respectively. From the fig. 5.b can be seen decreasing the porosity trend. At a temperature of 1050°C there is a maximum porosity of 49.35% and at a temperature of 1200°C the minimum porosity is 38.65%. It can be concluded that the higher the temperature the less of porosity occurs. This is because porosity will occur at low temperatures and when the higher temperatures porosity disappeared and the HA diffuses. The results obtained are in accordance with research that has been done previously that the higher temperature generated porosity will decrease and the longer sintering time will result in a decrease in the porosity also.

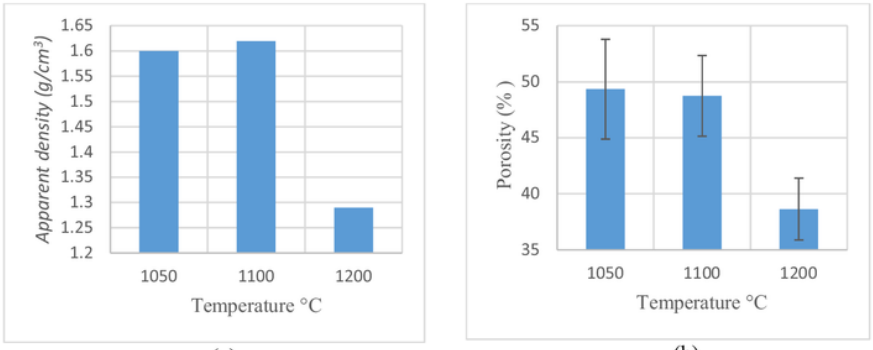

Figure 5. a) Apparent density and b) Porosity at various sintering temperatures

Compressive test with sintering temperature variations of 1050, 1100 and 1200°C have been done to evaluate the compressive strength of the sample. From the test results (fig. 6), compressive specimen increased in compressive strength from 1.73 MPa to 1.85 MPa and last 4.41 MPa. It can be seen also that the maximum compressive strength results are found at a temperature of 1200°C and the lowest strength is at a temperature of 1050 ° C. The higher the temperature produce a good compressive strength because at high temperatures HA particles increasingly bonded or diffused with each other and also at high temperatures decreased porosity as the results of previous density testing.

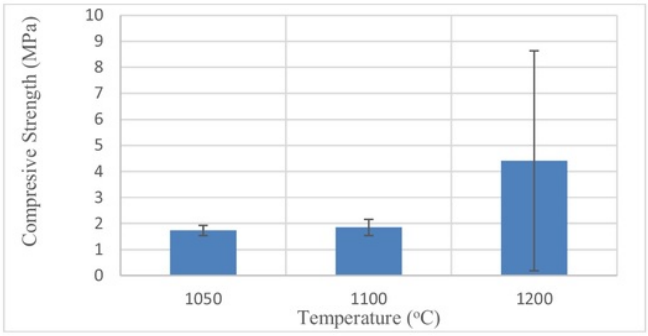

Figure 5. Compressive test result with sintering temperature variations of 1050, 1100 and 1200°C

Examination of microstructure using Scanning Electron Microscopy (SEM) equipment used to see the morphology of micro structure. SEM results are shown in figure 6 for hydroxyapatite samples sintered at 1050°C. SEM examination is done to find out how much porosity that occurs in the sample HA 1050 ° C. It is can be seen also from the figure an interconnected porosity in the sample. The greater the porosity that occurs, the more mechanical forces decrease in the sample.

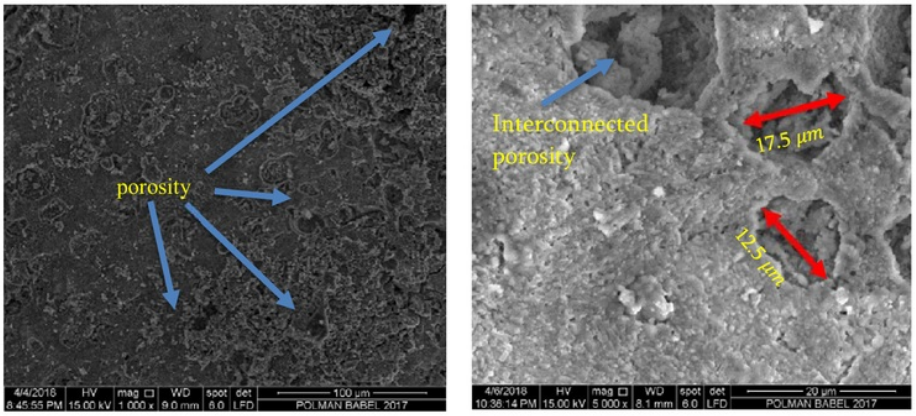

Figure 6. Typical morphology of samples sintered at 1050°C at different magnification

4. Conclusion

Based on the results of research the effect of temperature on the physical and mechanical properties of hydroxyapatite as explained, we conclude that the experimental results showed that porous hydroxyapatite were successfully fabricated using natural space holders. The largest sample shrinkage occurs at a temperature of 1200°C by 44.46%, the higher the temperature will result in high shrinkage. Most porous samples is present in samples that sintered at 1050°C, and the lowest porosity value is at 1200°C sintering temperature. This is because at a temperature of 1200°C the hydroxyapatite has fused together. Mechanical strength with compressive tests obtains the maximum results in samples with a temperature of 1200°C which can withstand loads of 4.41MPa. Interconnected porous was clearly observed on the sintered samples. Porosity is expected to facilitate the circulation of body fluids and encourage the growth of cells.

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