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Effect Of Binder On Bioceramic Surface Roughness When Milling

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Abstract: Too many road incidents result in permanent disability, such as bone fractures requiring bone replacement. Hydroxyapatite (HAP) is a chemical compound with analogous properties to the mineral component of human bones and hard tissues. Because of this, it is commonly used as a bone implant material and, more commonly, as a bioactive coating. Numerous HAP synthesis techniques for producing HAP powder have been developed along with various biomedical applications. This study examines the effects of binders on bioceramic surface roughness when milling. The sample is prepared for machining with varying binder percentages. Using the dry mixing and compaction technique, samples are prepared. The ratio between PEG and PVA samples ranges from 1 to 4% by weight. The samples are sintered in the furnace using a heating rate of 2°C/min and a two-hour soaking period. The temperature is set to a constant 1200 degrees Celsius. All samples were machined using a CNC milling machine with a constant spindle speed of 318.471 rpm, cutting speed of 27 m/s, feed rate of 0.046 mm, and depth of cut of 0.1 mm. All samples are analyzed using SEM, surface roughness, modulus of rupture, XRD, density, and porosity. The quantity of binders that influence the machining of bioceramics was ascertained through sample preparation and testing. The samples with the maximum hardness contain the most PEG, while those with the lowest hardness contain the most PVA. Results were obtained, and the optimal sample composition was (PEG 4 PVA 1). The conclusion is that the produced HAP samples are compatible with machining and that the binders influence machining, hardness, and surface roughness. The composition of the binder influences the density and porosity of the produced HAP samples.

Keywords: Hydroxyapatite (HAP), PEG, PVA, compression, Sintering, Cnc Milling

1. Introduction

Biomaterials are synthetic materials used to repair, recreate, or replace the anatomy of limbs or bones that are permanently or temporarily bonded to living tissue. Although biomaterials can replace or recreate limbs or bones, they cannot completely replace organ function. Polymers, metals, ceramics,

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and composites are the four classifications of biomaterials. Polymers, which can be used for soft and hard tissue applications, are the most abundant and versatile biomaterials.

Bioceramics are biocompatible ceramic materials. Bioceramics are an important and effective subset of biomaterials. Regarding biocompatibility, bioceramics range from ceramic oxides, which are inert in the body, to resorbable materials, which are finally replaced by the body after assisting in repair. Bioceramics are utilised in numerous medical procedures (Bose & Bandyopadhyay, 2013).

Hydroxyapatite (HAP), one of the methods of representative bioceramics that demonstrate exceptional biocompatibility and bioactivity with bone replacement in living tissues, has attracted considerable interest in these materials as good choices for orthopaedic, dental, and maxillofacial applications. Bone regeneration is facilitated by its osteoconductive and osteoinductivity, which make it a valuable material in tissue engineering. The interaction mechanism between the binder and bioceramics can improve HAP's microstructure and mechanical properties (Pokhrel, 2018).

Binders are substances used to attach inorganic and organic particles and fibres to rigid, rigid, and flexible materials. Generally, the binding effect is caused by chemical reactions when the binder is heated, combined with water or other substances, or exposed to air (construction, 2014). Binders commonly used include Polyethylene Glycol (PEG) and Polyvinyl Alcohol (PVA). These polymer binders have excellent biological inertness, are biocompatible, and have good comprehensive performance (Wei et al., 2017).

Milling is one of the manufacturing processes that can produce products. Milling is done where the cutters revolve to remove material from the workpiece toward the angle with the tool axis. By applying the milling process, it may conduct various operations and functions and produce high-precision items ranging in size from small to huge. Existing milling techniques and mechanical properties of porous HAP have disadvantages that result in increased machining expenses and times. The surface qualities of machined HAP significantly impact implant quality, which necessitates surface integrity. With scale grains, the mechanical properties of HAP ceramic are greatly enhanced, and this material has the potential to function as an implant in various applications.

As mentioned in subtopic, in the manufacturing process, milling machines are one of the methods that can be used to produce bioceramic products in the manufacturing process. Bioceramic, which is HAP used to determine biocompatibility combined with percentage binder, enables HAP with porous structures to be bioresorbable and have stronger bonding to the bone. The binder is any material or substance that holds other materials together to form a cohesive whole mechanically, chemically and adhesion or cohesion. Binders are liquid or dough-like substances that harden by a chemical or physical process and bind fibres, filler powder and other particles added into them. At the same time, clarifying that interaction between HAP and percentage binder will give some effect on milling operation and surface roughness. Therefore, the aim of this study is to determine which is the best percentage binder that could produce the require surface roughness when milling the bioceramic machined part.

2. Methodology

2.1 Sample preparation

The samples in this experiment are prepared via compaction of powders technique. The mixture was sintered at 1200°C. In this study, there were four samples that had been prepared for this study according to the percentage of binders. To characterize the hydroxyapatite powder, there are a several instruments have been used in this study such as X-ray Diffractometer (XRD and Scanning Electron Microscope (SEM) and other mechanical testing

2.2 X-ray Diffraction (XRD) Analysis

Powder XRD (X-ray Diffraction) is perhaps the most widely used x-ray diffraction technique for characterizing materials. As the name suggests, the sample is usually in a powdery form, consisting of fine grains of single crystalline material to be studied. The technique is also used widely for studying particles in liquid suspensions or polycrystalline solids (bulk or thin film materials). The term 'powder' really means that the crystalline domains are randomly oriented in the sample. Therefore, when the 2-D diffraction pattern is recorded, it shows concentric rings of scattering peaks corresponding to the

several spacings in the crystal lattice.

2.3 Scanning Electron Microscope (SEM) Analysis

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. In most applications, data are collected over a selected area of the surface of the sample, and a 2-dimensional image is generated that displays spatial variations in these properties. The SEM is also capable of performing analysis of selected point locations on the sample so this approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions

2.4 Modulus of Rupture (MOR)

The modulus of rupture (MOR) is a measure of the strength of a material under bending stress. In the case of bioceramics, which are ceramic materials used in biomedical applications, the MOR is an important property because it reflects the ability of the material to withstand the stresses that are placed on it in vivo. Bioceramics are used in a variety of biomedical applications, such as dental implants, orthopaedic implants, and bone scaffolds. In these applications, the bioceramic must be able to withstand the mechanical stresses that are generated by the patient's body. The MOR is a critical property for determining the suitability of a bioceramic for these applications.

2.5 Density And Porosity Analysis

The importance of density in Hydroxyapatite is to control thermal and mechanical properties. Density of Hydroxyapatite is defined as an object mass per unit volume. The measurement of density is performed following simple calculation by measuring total samples weight and divides it by the total samples volume. The porosity also gives the major effect on the density of the samples and they are formed by the air trapped during compression done. The percentage of porosity is calculated using all the weight that we acquired from the previous test

2.6 Vickers Hardness Test

The Vickers hardness test is a common test often easier to use than other hardness tests since the required calculations are independent of the size of the indent. This test is very important to test the hardness of the sample produced. The diamond indenter can be used for all materials irrespective of hardness including the sintered ceramics.

2.7 Surface Roughness Test Data Analysis

The surface roughness of machined surface was measured using the Surface Roughness Tester (Mitutoyo SJ400). The tester instrument consists of a drive unit name perthometer PAV-CV and a detector.

3. Results/Analysis/Discussion

In this chapter it will discuss the result and data analysis obtained from the experiment to analyze the HAP powder that has been done. The process involved in attaining those results had been discussed thoroughly in the previous chapters. The objective of this chapter is to investigate the crystallinity of the derived hydroxyapatite powder after being subjected to various ranges of binders. All the data are obtained from experimental result will be discussed accordingly. The HAP powder was characterized and recorded using XRD, SEM, MOR and various mechanical testing after sintering. The data analysis have been conducted and tested to find the best percentage binder that could produce the require surface roughness when milling the bioceramic machined part.

X-ray Diffraction (XRD) Analysis

The structural characterization was carried out by using a X-ray diffraction with $\lambda = 1.5406 \text{ \AA}$ radiation generated at a voltage of 40 kV and a current of 30 mA. Data were collected in the 2θ range of $10.835\text{--}81.704^\circ$, with a step size of $0.02^\circ/2\theta/s$. Phases identification was achieved by comparing the diffraction patterns of hydroxyapatite obtained in laboratory with JCPDS standards. The peaks were very sharp and all the measured XRD patterns reveal identical and strong broadening of the lines. Formation of nanocrystalline of $\text{Ca}_5(\text{PO}_4)_3(\text{OH})$, was confirmed by X-ray diffraction (XRD) study. From the result, all samples recognized as hydroxyapatite $\text{Ca}_5(\text{PO}_4)_3(\text{OH})$ in hexagonal phase as it compared to JCPDS (Joint Committee on Powder Diffraction Standards). From this figure, the peak of pattern has start rise at 25° and the highest peak is at 33° .

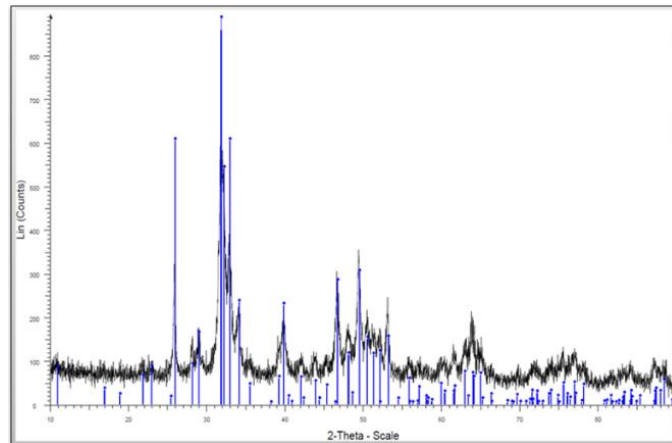


Figure 1 The XRD analyses with the peaks are marked with blue to show the HAP composition

3.1 Shrinkage Test Analysis

The shrinkage percentage of the samples is done by the dimensional measurements done on the thickness, and the diameter of the samples using a Vernier calipers ($\pm 0.02\text{mm}$). All the data was recorded before and after sintering process to acquire the shrinkage percentage. The shrinkage percentage range is from 46.7% to 61.33% and it's around 60% of shrinkage which can be considered as high. This is due to the vaporization of water and the binders.

3.2 Electron Microscope (SEM) Analysis

In this study, the scale that used to see the HAP powder is zooms 1500x. So, from Figure 2 there is particles size below $63 \mu\text{m}$ that could see. The images were acquired and it was clear.

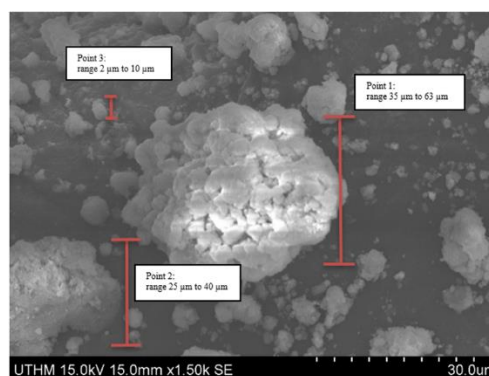


Figure 2 SEM micrograph of HAP powder with magnification of 1500X particle size, scale of below $63 \mu\text{m}$

3.3 Modulus of Rupture (MOR) Analysis

Modulus of rupture (MOR) is a mechanical property that measures the material's resistance to failure under bending or flexural stress. It is also known as the flexural strength or bending strength.

Overall, the observed data suggests that (PEG 1 PVA 4) bioceramics have higher yield strengths and, thus, are likely more suitable for applications that require stronger and more resilient materials. However, it is important to consider other factors, such as biocompatibility, bioactivity, and specific application requirements, to assess the Bioceramics suitability comprehensively.

3.4 Density And Porosity Analysis

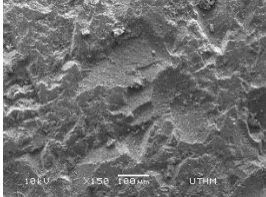
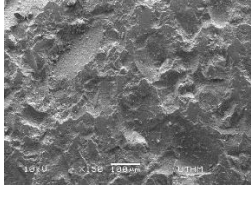
The density acquired which shows that the highest density is the sample with ratio of (PEG 2 PVA 3) in it with the density of 3.0729 g/cm³ and the lowest density is the ratio (PEG 1 PVA 4) 2.9481 g/cm³. The other are in the range of 3.0729 g/cm to 2.9481 g/cm³. The porosity also gives the major effect on the density of the samples and they are formed by the air trapped during compression done. The highest porosity percentage value is 0.93% which is a mixture of binder (PEG 2 PVA 3). The lowest percentage porosity is 0.59% which has a mixture of (PEG 3 PVA 2) the other samples with different ratios are in the range between the 0.59% to 0.93%. The compression load and sintering temperature are a cause that affects the percentage of porosity in samples.

3.5 Vickers Hardness Test

The hardness differs due to the composition of the binders. The hardness is due to the effect of concentration of binders ratio in sample because all other factors such as compression, sintering are having a constant value. The PVA and PEG binders have a huge effect on the samples. The ratio of PEG gives a higher hardness to the materials compared to PVA. The tables above shows the recorded results of the Vickers Hardness test. The hardness of sample 1 which is a mixture of (PEG 1 PVA 4). The maximum reading of hardness recorded is 349.397 HV and the minimum reading of hardness is recorded 332.226 HV. The mean hardness reading recorded was 338.611HV. The hardness of sample 2 which is a mixture of (PEG 2 PVA 3). The maximum reading of hardness recorded is 341.887 HV and the minimum reading of hardness is recorded 323.702 HV. The mean hardness reading recorded was 332.732 HV. The hardness of sample 3 which is a mixture of (PEG 3 PVA 2).The maximum reading of hardness recorded is 351.668 HV and the minimum reading of hardness is recorded as 323.702 HV. The mean hardness reading recorded was 336.574 HV. The hardness of sample 4 which is a mixture of (PEG 4 PVA 1).The maximum reading of hardness recorded is 458.21 HV and the minimum reading of hardness is recorded as 417.14 HV. The mean hardness reading recorded was 435.185HV.

3.6 Surface Roughness Test Data Analysis

It is found that the depth of cut is 0.1 mm and all the samples has the same value of depth of cut because the spindle speed 318.471 rpm and cutting speed 27 m/s and the federate are the same for all the sample with the different binders. The highest surface roughness value recorded is 2.006 um in the sample (PEG 3 PVA 2). The lowest surface roughness recorded is 0.988 um in the sample (PEG 4 PVA 1). It has a range of surface roughness from 0.988 um to 2.006 pm. The ceramic cutting tool with the angle 900 gives a fine surface roughness on the Hydroxyapatite samples.

Sample	After Machining	After Surface Roughness
PEG 1 PVA 4		

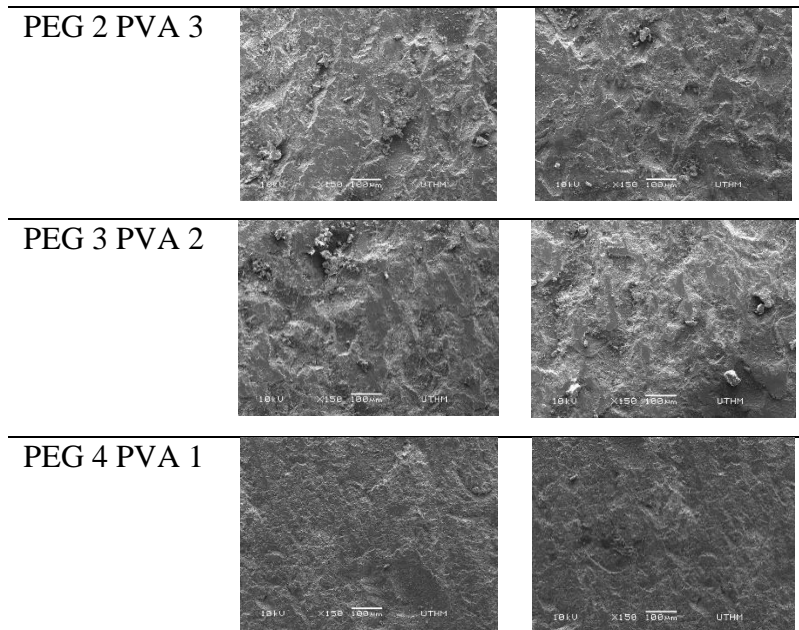


Figure 3: The picture shows SEM micrograph of HAP powder after machining and surface roughness with magnification of 1500X

4. Conclusion

This study concluded the effects of binders on bioceramic surface roughness when milling. The objectives of this study were achieved by mixing, compressing, sintering, machining, and testing various mechanical characteristics to determine which is the best percentage binder that could produce the require surface roughness when milling the bioceramic machined part. Two different binders, Polyvinyl Alcohol (PVA) and Polyethylene Glycol (PEG) were utilized in this study. Both of these binders have affected the mechanical and machining properties of HAP samples. This conclusion can be drawn from the data collected from the various experiments performed on the sample. The machinability of the Hydroxyapatite bioceramic samples is high. Still, the effects of the binders can only be determined through tests such as shrinkage, modulus of rupture, density and porosity, Vickers hardness tests, and surface roughness tests.

In the surface roughness test, we determined that the binders affect the machining and surface roughness. The surface roughness and hardness tests yielded different results based on the mixture proportion. The porosity and density measurement also recorded a range of data indicating numerous. The sample (PEG 4 PVA 1) has a high potential for machining because it has the suitable porosity and density and give the lowest surface roughness 0.988 (μm) of any mixture tested. This mixture's surface roughness is also averagely smooth. The Vickers hardness test on this sample indicates that this mixture of materials is too firm and soft for the machine. The observed modulus of rupture indicates that (PEG 1 PVA 4) bioceramics have more significant yield strengths and are, therefore, more likely to be suitable for applications requiring sturdier and more resilient materials. To exhaustively evaluate the bioceramics suitability, it is necessary to consider additional factors, such as biocompatibility, bioactivity, and application-specific requirements. Finally, it was determined that the binders PEG and PVA effectively affected the bioceramic machining on various factors, as demonstrated by all the tests conducted on sintered hydroxyapatite ceramics. All of the objectives that had been outlined were accomplished.

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