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The Effect of Binder on Bioceramic Surface Roughness when Milling using Ball Nose End Mill

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Abstract

Nowadays, the increasing number of road accidents has led to a rise since the accidents may result in permanent disability that often require surgical intervention. As a purpose to replace bone, a type of bioceramic which is hydroxyapatite (HAP) is chosen since it is a chemical substance that has similar characteristics to the mineral found in human bone and hard tissues. The water-soluble binder is used to make the substances hold together which is PEG and PVA. Four (4) samples of hydroxyapatite with different amounts of binder are made to examine the effects of binders on the bioceramic mechanical properties. on bioceramic surface roughness when milling using ball nose end mill. The sample is prepared by using dry mixing and compaction method whereas the ratio of PEG and PVA used are from 1% to 4% by weight. After compaction, the samples are sintered in the furnace for a total of 22 hours with 2 hours of soaking period in which the heating and cooling rate is 120°C/hour. The temperature set for the soaking period is 1200°C. The HAp sintered body is then machined by CNC milling machine using ball nose end mill cutter with a constant machining parameter of cutting speed, feed rate and depth of cut which is 27m/min, 40mm/rev and 0.1mm. Other than that, the sample undergoes mechanical properties testing such as modulus of rupture, vickers hardness, porosity and density. From the test, it is observed the amount of binder used influences the mechanical properties and surface roughness of the sample. Sample 4 which contains PEG 4 PVA 1 has the highest yield strength, hardness value and surface roughness. Other than that, the highest porosity obtained is sample 3 which contains PEG 3 PVA 2 and sample 4 has the highest density. In conclusion, the hydroxyapatite (HAp) samples with different amounts of binder used influence the sample compatibility and mechanical properties.

1. Introduction

A biomaterial is essentially a substance that has been modified for use in a medical setting. Biomaterials is a synthetic material that is used to repair, recreate, or replace the anatomy of limbs or bone that are permanently or temporarily bonded to living tissue and serve a more interactive function. Biomaterials are grouped into four classifications which are polymers, metals, ceramics, and composites.

Ceramics that are used for repair and replacement of damaged parts of the musculoskeletal system are referred to as bio-ceramics. Bio-ceramic possesses a unique combination of properties such as high intrinsic strength, biocompatibility and versatility. Bio-ceramics are utilized in numerous medical procedures [1].

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Hydroxyapatite (HAp), is a representative bio-ceramic that can be found in bone tissue and has been used in numerous medical applications due to its bioactivity and osteoconductive properties in vivo. Bone regeneration is supported by its ability to conduct and induce the growth of bone, which enhances its significance as a valuable substance in the field of tissue engineering. [2].

Binders are substances used to bind inorganic and organic particles and fibers to form strong, hard and flexible components. The binding action is caused by chemical reactions that occur when the binder is heated, mixed with water or other materials or exposed to air. Polyethylene glycol (PEG) and polyvinyl alcohol (PVA) is the most common polymeric binders used in medical industries nowadays. These polymers binders have excellent biological inertness and good comprehensive performance. [3]

Milling refers to a machining technique where a cutter that rotates is utilized to deliberately eliminate material from a workpiece and it has been chosen to be the part of the machining process. A CNC milling machine is used to run the machining process. By applying the milling process, high precision and complex design of product can be produced. Other than that, the milling process offers benefits of low cost, faster processing time and good surface finish. Surface integrity is necessary because the surface characteristics of machined HAp have a substantial effect on implant quality. The mechanical characteristics of HAp ceramic are significantly improved by the presence of scale grains, and this material may find use as an implant in a number of different applications.

Nowadays, the statistics of road accidents are increasing day by day. Other than road accidents, there are also accidents that occur at the workplace especially in the construction sector. These accidents will result in permanent disability such as bone fracture that will require bone replacement. HAp is commonly used as a bone implant material and as a bioactive. Along with the various biomedical applications, numerous HAP synthesis techniques for producing HAp powder have been developed. Therefore, the development of bone tissue engineering depends on the binder's ability to form HAp when milling with a ball nose end mill to achieve the proper surface roughness. Other than that, investigating the influence of different binder percentage use and the surface roughness of HAp can optimize the machining process and improve its mechanical properties.

2. Methodology

2.1 Sample Preparation

HAp powder is mixed with different amounts of binders consisting of PEG and PVA. The samples in this experiment are prepared via compaction method. There are four (4) samples that have been prepared for this research according to the different amount of binders used. The sample that has been compacted with 10 tons of load with a holding time of 5 minutes called as green body is then sintered at 1200°C with 2 hours of soaking profile time.

2.2 Mechanical Properties Test

2.2.1 Shrinkage

Shrinkage test is a measurement method used to analyze the effect of temperature on the bioceramic and this testing is common since most of the ceramics will undergo shrinkage after the sintering process. Thus, the HAp sample also will be measured for shrinkage test. The first step for shrinkage test is done by measuring the HAp sample on its width, length and depth by using a vernier caliper before the sample undergoes the sintering process. After the HAp sample has been sintered, repeat the first step which measures the HAp sample on its width, length, and depth. The percentage of shrinkage can be easily calculated by using the equation 1 below

$$\text{Percentage Shrinkage} = \frac{\textit{Wet length @ weight before} - \textit{Dry length @ weight after}}{\textit{Wet length @ weight before}} \times 100\% \quad (1)$$

2.2.2 Modulus of Rupture (MOR)

The modulus of rupture (MOR) of a bioceramic can be determined using a three-point bending test. MOR is a crucial aspect to be considered since it reflects the ability of the HAp sample to tolerate pressure occurring in vivo. As for the application of HAp, the bioceramic must be able to withstand the mechanical applied stresses that are generated by the human body. The MOR value needs to be determined to ensure the suitability of the sample to withstand mechanical forces. MOR are calculated by using equation 2 below to obtain the yield strength or flexural strength of the HAp sample.

$$Yield\ Strength = \frac{Max\ Stress}{(1 + Max\ Strain)} \quad (2)$$

The testing is conducted by placing the same level of two supports with a distance of 15mm gap and parallel with each other. The HAp samples are placed at the center between the supports. Then, move the loading fixture nearest to the surface of the Hap sample. Ensure all values in the computer are 0 before starting to apply load at rate 1 mm/min and run the testing. To collect data, wait until the sample fractures, or a predetermined maximum load is reached.

2.2.3 Porosity and Density

The mechanical properties and biological performance of HAp are greatly influenced by the porosity and density of the HAp. The method used for porosity and density test is Archimedes buoyancy technique where it is done at a Ceramic Laboratory, G2, UTHM. The technique uses distilled water to submerge the HAp sample. The temperature of the distilled water is controlled and set to room temperature which is 23°C during holding time of the submerge HAp sample. To conduct this testing, 1000ml of distilled water is heated and weighed dry sample of HAp. Next, a rack is used to place the HAp sample before submerging it in the beaker. The beaker is then being heated for 2 hours before the HAp sample is being hold for 12 hours in the beaker with distilled water at room temperature. Then, the dry weight, wet weight and weight in liquid for HAp sample is recorded. As for wet weight, remove the residual water on the surface of the HAp sample using filter paper before weighed. The density of the HAp can be calculated using equation 3 and equation 4 to find the porosity of HAp sample.

$$\rho = \frac{\text{dry weight}}{\text{wet weight} - \text{weight in liquid}} \quad (3)$$

$$Percentage\ of\ porosity = \frac{(Wwet - Wdry)}{(Wwet - Wsoaked)}\ x\ 100\% \quad (4)$$

2.2.4 Vickers Hardness

Vickers Hardness testing serves as a method of testing commonly employed for small components, delicate sections, or surface depth examinations. This approach relies on an optical measurement system and entails employing a diamond indenter to create an indentation, which is then measured and converted into a hardness value. To run the testing, the HAp sample is placed at the stage and place the lens nearest to the surface of the sample. The test force applied for indentation is 1.961N (HV0.2). The sample is being indented 5 times to obtained the mean hardness of the HAp sample.

2.3 Powder Characterization Analysis

2.3.1 Semi-Electron Microscope (SEM)

A concentrated stream of high energy electrons is utilized by the SEM to produce magnified image for analysis. SEM is capable of performing an analysis at the selected point locations on the sample and enables observation in high vacuum state, on low vacuum state and in wet conditions. The HAp sample needs to be coat before the sample is put under high vacuum state to obtain the magnified image. After the sample is under high vacuum state, start adjusting the lens power until the microstructures of the HAp sample can be seen clearly. The magnification used to see the microstructure is 1500x.

2.3.2 X-Ray Diffraction (XRD)

X-ray Diffraction (XRD) is a common technique that is used to determine the crystal structure. Thus, X-ray Diffraction (XRD) analysis plays a role to identify the crystalline phases present in the HAp sample and reveal the chemical composition information. XRD analysis method is performed by directing an x-ray beam to the sample and assess the intensity of scattered radiation in relation to its subsequent direction. Upon the beam's

separation, the scatter or referred as diffraction pattern, unveils the crystalline structure of the sample. The XRD Analysis machine can be used at Material Science Laboratory.

2.4 Machine Compatibility

2.4.1 Machining Parameter

Table 1 *Machining Parameter*

Cutting Parameter for CNC Milling		
Cutting Speed, *V*	Feed Rate, *f*	Depth of Cut, *d*
27 m/min	40mm/rev	0.1mm

2.4.2 Surface Roughness Test

A surface roughness tester is a machine that can be used to quickly and accurately determine the surface texture or surface roughness of a material. The machine model used is Mitutoyo SJ400.This machine consists of high accuracy measurements with a hand-held tester name perthometer PAV-CV and a detector that has a measuring range up to 800µm.

3. Results and Analysis

This chapter discuss about the results obtained from material testing conducted. There are two different tests conducted which are powder characterization analysis and mechanical properties test. As for powder characterization analysis, the tests conducted are X-ray diffraction and semi-electron microscope. For mechanical properties test, the tests conducted are shrinkage test, modulus of rupture, surface roughness, vickers hardness, porosity, and density. Thus, all the data obtained were observed, analysed and discussed to get a conclusion for this research.

3.1 Mechanical Properties Test Analysis

3.1.1 Shrinkage

The shrinkage percentage of HAp sample is calculated as stated in equation 1 above. The percentage of shrinkage of HAp sintered body is as shown in Table 2. The difference in percentage of shrinkage is due to the vaporization of water and binder in the HAp sample when sintering at 1200^{o}C in which the particles will closely pack and decrease towards the end process.

Table 2 *Percentage of Shrinkage*

Sample	Binder		Percentage of Shrinkage (%)	
	PEG	PVA	Weight	Length
1	1%	4%	18.37	20.76
2	2%	3%	12.18	21.42
3	3%	2%	13.26	21.40
4	4%	1%	23.38	21.10

3.1.2 Modulus of Rupture (MOR)

From the maximum stress and maximum strain of the HAp sample obtained, the yield strength of the HAp sample is calculated using the equation 2 above. The yield strength value for HAp sample obtained from the calculation are shown in Table 3. It is observed that sample 4 has the highest yield strength which is 32.36 N/mm^2 while sample 1 has the second highest yield strength which is 29.77 N/mm2. As for sample 2 and sample 3, the yield strength obtained are range between 19 N/mm^2 to 24 N/mm^2. These values indicate the amount of load that can be applied before it deforms plastically. A higher yield strength demonstrates a higher ability of the HAp sample to resist deformation under applied stress and it can be advantageous for any application that requires stronger and more resilient material. Other than that, higher yield strength of HAp offers better support for tissue integrity and resistance to failure. As for HAp, other important factors also need to be considered especially in terms of its biocompatibility and bioactivity.

Table 3 *Yield Strength of HAp Sample*

Sample	Binder	Yield Strength (N/mm^2)

	PEG	PVA	
1	1%	4%	29.77
2	2%	3%	19.12
3	3%	2%	23.31
4	4%	1%	32.36

3.1.3 Porosity and Density

The experimental porosity and density value is calculated using equation 3 and equation 4 as mentioned in chapter above. Table 4 shows that experimental porosity and density value obtained. As for density, sample 4 has the highest density value that is nearest to the theoretical density value of HAp which is 3.16g/cm^3 in which will impact its mechanical properties. However, porosity also will affect HAp bioactivity, mechanical properties, and biodegradability. The percentage of porosity and density data obtained vary depending on the percentage of binder added to the sample. The difference in percentage of porosity is caused by the effect of the compression load applied and sintering temperature.

Table 4 *Porosity and Density of HAp sample*

Sample	Binder		Porosity	Density
	PEG	PVA	(%)	(g/cm^3)
1	1%	4%	2.18	2.9639
2	2%	3%	0.73	2.9496
3	3%	2%	2.81	3.0287
4	4%	1%	1.92	3.0309

3.1.4 Vickers Hardness

The samples are being indented 5 time with different point location and same constant load to increase the accuracy of hardness results. Table 5 below shows the mean hardness value obtained. It is observed that sample 4 has the highest hardness value which is 447.272HV, and it is followed by sample 3, sample 2 and sample 1 which the hardness value obtained is 421.652HV, 334.764HV and 319.78HV respectively.

Table 5 *Mean Hardness Value of HAp Sample*

Sample	Binder		Mean Hardness
	PEG	PVA	(HV)
1	1%	4%	319.78
2	2%	3%	334.74
3	3%	2%	421.562
4	4%	1%	447.272

The mean hardness value for each sample varies due to the different ratio of binder used in which it can affect the HAp biocompatibility and bioactivity. Factors such as compression load, sintering temperature and sintering duration also may affect the difference of hardness value. However, this factor does not give an effect to the HAp sample since all the HAp sample undergoes the same compression load, sintering temperature and sintering duration. In addition, other factors need to be considered even the HAp has highest mean hardness such as porosity and density to determine what is the best composition that offers better tissue integration and improves wear resistance. Other than that. increasing in density leads to higher hardness value.

3.2 Powder Characterization Analysis

3.2.1 Semi-Electron Microscope (SEM)

The SEM micrograph reveals the HAp microstructure by using 1500x magnification. Figure 1 shows the SEM micrograph obtained for HAp sample microstructure. Overall, it can be observed that all sample has rough and radical groove surface. Other than that, all the sample has pores which are not a full solid of HAp sample. The pores show at the SEM micrograph are approximately same as the percentage porosity data obtained from mechanical properties testing.

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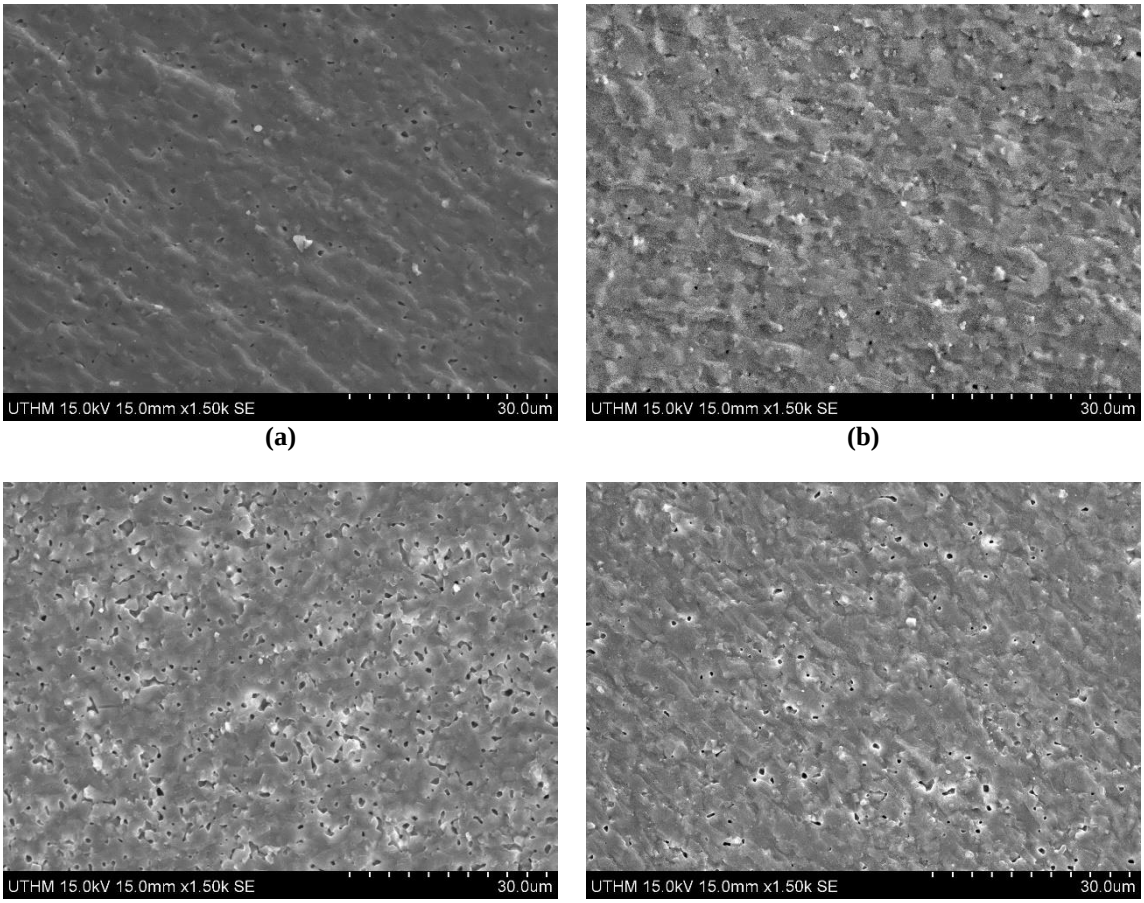

Fig. 1 *SEM Micrograph (a) Sample 1 (b) Sample 2 (c) Sample 3 (d) Sample 4*

3.2.2 X-Ray Diffraction (XRD)

Phases identification was achieved by comparing the diffraction patterns of hydroxyapatite obtained in laboratory with JCPDS standards. The phases identification was carried out by using a X-ray diffraction with Cuka=1.5406Å radiation generated at a voltage of 40 kV and a current of 30 mA. Data were collected in the 20 range of 10.835-81.704 °, with a step size of 0.02°20. The data obtained shows that all sample has the same formation of nanocrystalline of $Ca_{10}(PO_4)_6(OH)_2$ and the peak pattern obtained are same as the JCPDS standard.

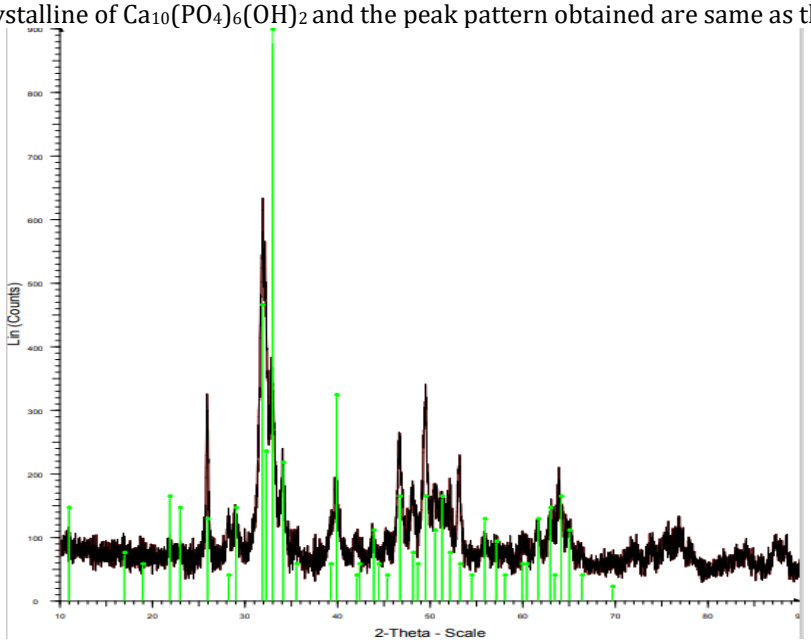

Fig. 2 *Peak Pattern of HAp*

3.3 Machine Compatibility

3.3.1 Surface Roughness Test

Surface roughness tester was conducted after the HAp sample is machined using ball nose end mill cutter. The testing was done to determine which different composition of binder added gives a rougher surface roughness. One cutting parameter was used for the machining process which is 27 m/s for cutting speed, 0.040 mm/rev for feed rate and 0.1 mm for depth of cut. The surface

Table 6 *Surface Roughness of HAp after machining*

Sample	Binder		Ra
	PEG	PVA	(µm)
1	1%	4%	0.434
2	2%	3%	0.895
3	3%	2%	0.876
4	4%	1%	1.981

4. Conclusion

In conclusion, the effect of binder has a significant impact on the bioceramic surface roughness when milling using a ball nose end mill. Mixing, compressing, sintering and machining process was conducted to produce HAp sample before various testing were conducted to determine which binder percentage produces the best surface roughness when milling using ball nose end mill. Two different binders were used in this research with different percentages of binder used which is polyvinyl alcohol (PVA) and polyethylene glycol (PEG). The binders used have affected the mechanical properties of HAp based on the collecting data. The effect of binder on the surface roughness was determined through various testing conducted such as shrinkage, modulus of rupture, x-ray diffraction, semi electron microscope, vickers hardness, surface roughness, porosity and density. All testing is done on the HAp sintered body. As for porosity and density, sample 4 (PEG 4 PVA 1) has high potential to be the best mixture percentage of binder since it has the nearest density value to the theoretical density value of HAp and it has 1.92% for its porosity. Other than that, sample 4 also gives the highest surface roughness which is 1.981µm when tested and it also has the highest yield strength which is 32.36N/mm^2. Therefore, it is more suitable for any application that requires a stronger application. Besides that, the observed vickers hardness indicates that Sample 4 has the highest hardness value. Manufacturers and researchers can make informed decisions to achieve the desired surface finish for bioceramic components by understanding the impact of binders. However, in order to fully assess the applicability of bioceramics, especially hydroxyapatite, other factors including bioactivity, tissue integration, biocompatibility, and specific application must be taken into consideration. Finally, the objective of this research finding was achieved as the binders effectively affected the bioceramic surface roughness when milling using ball nose end mill. Other than that, the binders also affected the bioceramic on various factors when tested.

Acknowledgement

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Appendix A: MOR Data

Sample	Binder		Max Stress (N/mm2)	Max Strain (%)
	PEG	PVA		
1	1	4	57.5036	0.93136
2	2	3	31.9654	0.67152
3	3	2	30.5318	0.30955
4	4	1	43.0068	0.32895

Appendix B: Porosity and Density Data

Sample	Binder		Dry weight (g)	Weight in liquid (g)	Wet weight (g)
	PEG	PVA			
1	1%	4%	1.8551	1.2429	1.8688
2	2%	3%	2.0305	1.3471	2.0355
3	3%	2%	1.5089	1.0247	1.5229
4	4%	1%	1.5767	1.0665	1.5867

Appendix C: Vickers Hardness Data

Sample	Indent Point	H Length	V Length	Average Length	Hardness (HV)
1 (PEG 1 PVA 4)	1	34.1436	32.7695	33.4566	331.289
	2	35.9892	33.1357	34.5624	310.428
	3	35.0664	34.051	34.5587	310.495
	4	33.9590	33.1357	33.5474	329.498
	5	34.8818	33.5018	34.1918	317.194
2 (PEG 2 PVA 3)	1	32.4826	31.6711	32.0768	360.401
	2	32.4826	33.5018	32.9922	340.68
	3	32.6671	34.2341	33.4506	331.407
	4	34.5127	33.5018	34.0073	320.646
	5	33.9590	34.0510	34.0050	320.688
3 (PEG 3 PVA 2)	1	32.8517	27.4605	30.1561	407.774
		28.6068	30.3896	29.4982	426.165
	2	32.6671	28.0097	30.3384	402.887
	3	29.3450	28.3759	28.8604	445.208
	4	31.9289	27.0944	29.5116	425.778
	5	32.8517	27.4605	30.1561	407.774
4 (PEG 4 PVA 1)	1	30.2678	26.179	28.2234	465.532
	2	26.5766	30.9388	28.7577	448.394
	3	28.9759	29.2912	29.1336	436.9
	4	30.2678	26.7282	28.498	456.604
	5	27.684	31.1219	29.403	428.931

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