

The Effect of Cutting Parameters on Surface Roughness When Milling Bio Ceramic

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Abstract

This study investigates the mechanical properties of bio-ceramics, especially hydroxyapatite (HAP) as well as the effect of milling parameters on surface roughness quality, using the method known as Taguchi being utilised for optimisation. Dry mixing, compression, and sintering at 1200°C were employed to prepare HAP samples mixed with 1% Polyvinyl Alcohol (PVA) and 4% Polyethylene Glycol (PEG) binders. SEM, XRD, modulus of rupture, density, porosity assessments, Vickers hardness, and surface roughness test were just a few of the comprehensive tests that were conducted out to evaluate the mechanical properties and identify the materials. The Taguchi approach was utilised all over the machining process on a CNC milling machine in order to determine the best combination of cutting speed, feed rate, and depth of cut for the best possible surface smoothness. The findings showed that surface roughness is highly influenced by feed rate, with lower rates resulting in finer finishes. Cutting speed had very little effect, whereas the depth of cut demonstrated a considerable influence. The best values for the machining parameters were found to be 27 rpm, 0.023 feed rate, and 0.05 depth of cut which is going to combination of parameter 3. The findings show that feed rate during HAp milling has the greatest effect on surface roughness, with lower rates producing better finishes. The optimum combination of cutting speed, feed rate, and depth of cut for a perfect surface finish is successfully determined by the study.

1. Introduction

Biomaterials encompass natural or artificial substances designed to interact with biological systems, facilitating the repair, replacement, or improvement of various tissues or organs within the body. These materials can range from simpler examples like urinary catheters to more intricate devices such as internal glucose sensors. They may also include permanent fixtures like cardiovascular stents or stainless-steel implants utilized for bone replacement. The diverse components of biomaterials collectively contribute to their role in the overall bodily system. There are four categories for biomaterials which is biocomposites, metals, polymers, and ceramics. Strong metals that are utilized in load-bearing implants, such as titanium and stainless steel, can corrode and break down bone. The biodegradability and flexibility of polymers, such as polyethylene, make them useful in medical disposables for medication delivery. Blending materials together, biocomposites offer unique features tailored to certain applications. Since they are compatible and maintain bone tissue, ceramics especially hydroxyapatite (Hap) are preferred for use in dental implants[1].

Hydroxyapatite (HA), or calcium phosphate with the chemical formula $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$, is a bioceramic material widely employed for regenerating hard tissues due to its outstanding biocompatibility, high

osteoconductivity, and chemical similarity to the apatite found in human hard tissues. Despite its excellent biological activity, the mechanical properties of HA bioceramics fall short of human bone, limiting their clinical applications such as in prosthetic teeth and bones. Furthermore, HA's slow degradability hinders complete bone replacement and remodeling, severely restricting its clinical usability. Therefore, there is a pressing need to enhance the mechanical characteristics and degradability of HA-based bioceramics[2].

To enhance the mechanical, biocompatible, and bioactive characteristics of Hydroxyapatite (HAp), polymer composites are essential. Commonly employed binders include Polyethylene Glycol (PEG) and Polyvinyl Alcohol (PVA). Polyvinyl alcohol (PVA) is chosen for its non-toxic and biodegradable attributes. Being water-soluble, biodegradable, chemically and thermally stable, PVA exhibits high strength and optical transparency in water. Its porous structure allows for easy incorporation of fillers like HAp, facilitating a hierarchical arrangement to aid in bone resorption. Polyethylene glycol (PEG) is another significant material in tissue engineering, appreciated for its non-toxic, biodegradable, biocompatible, and water-soluble nature. Additionally, blending PEG with PVA enhances the toughness and mechanical properties of the composites. Leveraging this advantage, PEG-blended PVA emerges as a promising candidate for improving bonding formation and cell adhesion in HAp[3].

Surface quality holds particular significance in nearly all machining processes, including milling. In milling, the performance of the milling components is evaluated based on surface quality. Surface roughness is a widely employed parameter for assessing product quality in the machining process. The factors influencing surface roughness include surface hardness, machining conditions, workpiece material, and tool geometry. The quality of the final product in milling operation such as surface finish depends on machining parameters such as cutting speed, depth of cut and feed rate. Achieving the smooth surface, require careful or the right settings of parameters, and changing these settings based on the material being machined and the machining process can have a great effect on the product's final surface quality[4].

Understanding the mechanical properties of bio-ceramics and the impact of cutting parameters during milling is essential for optimizing their manufacturing processes. Despite the significance of this research area, there is a lack of comprehensive studies addressing the relationship between bio-ceramic properties and milling parameters. Thus, there is a need to systematically investigate the mechanical properties of bio-ceramics and analyze the effects of cutting parameters during the milling process.

2. Methodology

2.1 Sample preparation

Dry mixes of HAp powder and different amounts of binders composed up of 1% PVA and 4% PEG are used. The compaction method is used to prepare the samples for this experiment. For this study, eight samples have been prepared. The sample, called as the "green body," is then sintered at 1200°C for two hours after being compacted with one ton of weight and held for one minute.

2.2 Mechanical properties test

2.2.1 Shrinkage

The shrinkage test is a commonly used measurement technique for studying the effect of temperature on bioceramics, as most ceramics suffer shrinkage following the sintering process. In order to perform the shrinkage test, the HAp sample will also be measured. Before the sample is subjected to the sintering process, the shrinkage test is performed by first measuring the HAp sample's width, length, and depth with a vernier caliper. The first stage, which measures the HAp sample's width, length, and depth, should be repeated after it has been sintered. Using the following equation 1, it is straightforward to compute the percentage of shrinkage.

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

2.2.2 Modulus of Rupture (MOR)

A three-point bending test can be used to determine the bioceramic's modulus of rupture (MOR). As MOR shows the HAp sample's ability to withstand pressure experienced in vivo, it is an essential factor to take under consideration. When applying HAp, the bioceramic material needs to be strong enough to withstand the mechanical pressures produced by the human body. To make sure the sample is suitable for withstanding mechanical forces, the MOR value must be determined. To determine the yield strength or flexural strength of the HAp sample, MOR is calculated using equation 2 below.

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

Two supports at the same level are placed parallel to each other and 15 mm away to conduct the test. The HAp sample are placed at the center of the supports. The loading fixture should then be moved closest to the HAp sample's surface. Before applying load at a rate of 1 mm/min and starting the testing, ensure that all values in the computer are 0. A predetermined maximum load must be attained or the sample must fracture before data can be collected.

2.2.3 Porosity and Density

The mechanical properties and biological effectiveness of HAp are significantly impacted by its porosity and density. The Archimedes buoyancy technique is the method used for the porosity and density test, which is carried out in the Ceramic Laboratory, G2, UTHM. The HAp sample is submerged in distilled water as part of the procedure. During the holding period of the submerged HAp sample, the temperature of the distilled water is regulated and set to room temperature, which is 23 °C. A dry sample of HAp is weighed and heated to 1000 milliliters of distilled water in order to perform this test. The HAp sample then sits on a rack before being placed in the beaker. After two hours of heating, the HAp sample is allowed to remain in the beaker filled with room temperature distilled water for a full twelve hours. Next, the weight of the HAp sample under liquid, wet, and dry conditions is recorded. Regarding wet weight, take filter paper to wipe out any remaining water from the HAp sample's surface before weighing. Equations 3 and 4 can be used to calculate the density of the HAp and determine the porosity of the HAp sample.

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

$$\text{Percentage of porosity} = \frac{(W_{\text{wet}} - W_{\text{dry}})}{(W_{\text{wet}} - W_{\text{soaked}})} \times 100\% \quad (4)$$

2.2.4 Vicker Hardness

Vickers Hardness testing is a testing technique that is frequently used for small components, delicate areas, or surface depth analyses. This method uses an optical measurement device and involves measuring and converting the indentation made by a diamond indenter into a hardness value. The HAp sample is set up on stage, and the lens is positioned closest to the sample's surface to begin testing. 1.961N (HV0.2) is the test force used for indentation. To determine the mean hardness of the HAp sample, the sample is indented five times.

2.3 Powder Characterization Analysis

2.3.1 Semi-Electron Microscope (SEM)

The SEM uses a focused stream of high energy electrons to create magnified images that can be analyzed more closely. The scanning electron microscope (SEM) may conduct analysis at specific points on the material and facilitate observation under wet, low, and high vacuum states. To get the magnified image, the HAp sample must be coated before being placed under high vacuum. Once the sample is in a high vacuum state, begin adjusting the lens power until the HAp sample's microstructures are clearly visible. 1500x magnification is employed to view the microstructure.

2.3.2 X-Ray Diffraction (XRD)

A common technique for determining out the crystal structure is X-ray diffraction (XRD). Thus, the crystalline phases found in the HAp sample may be identified and the chemical composition can be revealed by the use of X-ray Diffraction (XRD) analysis. The XRD analysis method measures the intensity of scattered radiation in relation to its subsequent direction by pointing an x-ray beam at the sample. When the beam splits, the sample's crystalline structure is revealed by the scatter, also known as the diffraction pattern. The Material Science Laboratory has access to the XRD Analysis machine.

2.4 Machine Compatibility

2.4.1 Machining Parameter

Table 1 *Machining Parameter*

Machining Parameter	1	2	3
Cutting Speed, CS (m/min)	27	41	14
Feed Rate, f (mm/rev)	0.046	0.069	0.023
Depth of cut, d (mm)	0.1	0.15	0.05

Table 2 *Combination of parameters based on Taguchi Method*

Cutting Speed, Cs (m/min)	Feed Rate, f (mm/rev)	Depth of cut, d (mm)
27	0.046	0.10
27	0.069	0.15
27	0.023	0.05
41	0.046	0.15
41	0.069	0.05
41	0.023	0.10
14	0.046	0.05
14	0.069	0.10
14	0.023	0.15

2.4.2 Surface Roughness Test

A device that can be used to quickly and accurately evaluate a material's surface roughness or texture is called a surface roughness tester. The Mitutoyo SJ400 machine model is the one in use. This device utilizes a detector with a measuring range of up to 800µm and a hand-held tester called the perthometer PAV-CV to provide high accuracy measurements.

3. Result and Analysis

This chapter presents the findings from the material testing that was done. The mechanical properties test and the powder characterization analysis test are the two different tests that are carried out. In terms of powder characterization analysis, semi-electron microscopy and X-ray diffraction tests are carried out. The testing for mechanical properties includes density, porosity, vickers hardness, surface roughness, modulus of rupture, and shrinkage. Thus, in order to reach a conclusion for this study, all of the data that were collected were examined, evaluated, and discussed.

3.1 Mechanical Properties Test Analysis

3.1.1 Shrinkage

By comparing the HAp sample's length, weight, width, and thickness, four parameters were used to indicate the shrinkage test. Since the green body shrinks after sintering, it is done in between the sintered body and the green body. We can conclude from the result that there is variation among the eight hydroxyapatite samples mixed with 1% PVA and 4% PEG binder. Shrinkage encompasses around from 15% to 25% of the total. There is a difference in the amount of weight, length, width, or thickness shrinkage between samples.

Table 3 Percentage of shrinkage of HAp sintered body

Sample		Percentage of Shrinkage (%)			
PEG (4%)	PVA (1%)	Weight	Length	Width	Thickness
Sample 1		17.92	23.56	23.30	20.73
Sample 2		16.83	23.73	23.15	21.73
Sample 3		18.38	23.38	21.93	23.17
Sample 4		17.31	23.57	23	17.51
Sample 5		18.11	23.21	20.52	23.30
Sample 6		23.74	22.86	23.30	18.92
Sample 7		23.75	22.89	22.68	20.90
Sample 8		23.67	22.87	21.84	18.84

3.1.2 Modulus of Rupture

Equation 2 above is used to determine the yield strength of the HAp sample based on the maximum stress and maximum strain that were measured. Table 4 illustrates the yield strength value for the HAp sample that was calculated. According to the Modulus of Rupture (MOR) test yield strength data. Sample 1 and Sample 3 have slightly lower values of 29.18 N/mm² and 27.68 N/mm², respectively, while Sample 2 shows the highest yield strength at 38.99 N/mm². In overall, the minimum value for yield strength of hydroxyapatite samples mixed with 4% PEG and 1% PVA binders is 27.68 N/mm² and the maximum value is 38.99 N/mm². On average, yield strength value obtained is 32.31 N/mm². The mechanical properties of the material are improved by the combination of hydroxyapatite and binders. In general, yield strength is an important property that determines the highest stress a material can withstand before permanently deforming. Apart from that, higher HAp yield strength provides better support for tissue integrity and failure resistance. It is necessary for the design of components and structures in engineering.

Table 4 Yield Strength of HAp samples

Sample	Yield Strength N/mm ²
1	29.18
2	38.99
3	27.68
4	33.38

3.1.3 Porosity and Density

The equations 3 and 4, which have been addressed in the previous chapter, are used to calculate the experimental porosity and density value. The experimental porosity and density value obtained are displayed in Table 4. As for density, the minimum value for density of hydroxyapatite samples mixed with 4% PEG and 1% PVA binders is 2.774 g/cm³ and the highest value is 3.1321 g/cm³. On average, density value obtained is 2.930 g/cm³. Higher density in hydroxyapatite (Hap) samples indicates better material integrity and compactness. The findings of the porosity test show variation across the three hydroxyapatite samples mixed with binders 1% PVA and 4% PEG. Sample 3 has the lowest porosity which is about 0.88, whereas Sample 2 has the highest porosity just over 0.96. These variations from the result still can be described as the same since all of it is around 0.9 range. Largest porosity may indicate a porous structure that reduces mechanical strength, whereas lower porosity may indicate a better density and possibly increased strength. These insights have importance for applications where material performance things, such as implants or bone grafts. In overall, from the result in Table 4, we can see that density is higher than porosity which means that the hydroxyapatite mixed with 4% PEG and 1% PVA binders has a better or increasing in strength.

Table 5 Porosity and Density of HAp samples

Sample	Percentage of porosity %	Density (g/cm ³)
1	0.937	2.822
2	0.977	3.1321
3	0.901	2.7740

3.1.4 Vickers Hardness

In order to improve the accuracy of the hardness data, the samples are indented five times at different points with the same constant load. The average value of hardness obtained is displayed in Table 6 below. It is observed that the highest value of mean hardness for hydroxyapatite samples mixed with 4% PEG and 1% PVA binders is 178 HV and the lowest is 165 HV. On Average, mean hardness value obtained is 172.347 HV. A greater Vickers hardness value for hydroxyapatite (HAp) samples shows a denser and stronger material, which can make it more suitable for load-bearing biomedical applications such dental implants or bone grafts. Porosity, grain size, and the addition of additives or binders can all affect HAp's hardness, affecting its mechanical properties in order to meet the needs specific biomedical applications.

Table 6 Mean Hardness value of HAp samples

Sample	Mean Hardness (HV)
1	165.009
2	174.03
3	178.003

3.2 Powder Characterization Analysis

3.2.1 Semi-Electron Microscope (SEM)

The HAp microstructure can be seen at the 1500x magnification of the SEM micrograph. The SEM image for the microstructure of the HAp sample is displayed in Figure 1. The granules show size variation and distribute themselves and there is some roughness everywhere in the sample in a hidden pattern at 1500x magnification. With specific areas showing porosity which can affect the mechanical strength, the phase's continuity appears to be improved by the presence of 1% and 4% PEG binders. The distribution and size of these gaps play an important part in defining the mechanical strength and biocompatibility of the material.

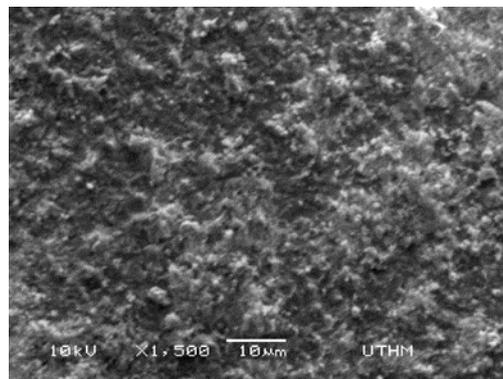


Figure 1 SEM Micrograph of sintered HAp sample

3.2.2 X-Ray Diffraction (XRD)

By comparing the hydroxyapatite diffraction patterns obtained in the lab with JCPDS standards, the phases were identified. Using an X-ray diffraction with $\lambda = 1.5406$ radiation generated at a voltage of 40 kV and a current of 30 mA, the phases were identified. At a step size of $0.02^\circ 2\theta$, data were gathered in the 2θ range of $10.835-81.704^\circ$.

Based on the Figure below which is the analysis of the Taguchi method, the main factor influencing machining surface roughness is feed rate, with lower rates producing smoother surfaces. Cutting speed is relatively unaffected, therefore it may be adjusted with flexibility. The ideal machining parameters are a depth of cut of 0.05, a feed rate of 0.023, and a cutting speed of 27 for the best surface quality and stability which belongs to parameter 3. The "Smaller is Better" approach is applied to these settings in an effort to maximize mean Signal to Noise (S/N) ratios.

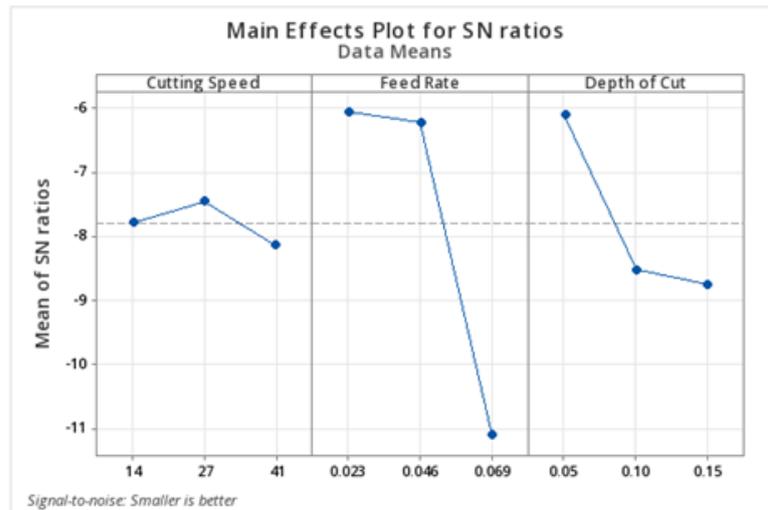


Figure 3 Taguchi Method graph result of Signal to Noise Ratio for surface roughness using 'Smaller is Better' approach

4. Conclusion

In overall, the HAp sample was produced by a process that involved mixing, compressing, sintering, and machining. Subsequently, a series of tests were carried out to determine the mechanical properties of the HAp sample and the impact of surface roughness cutting parameters on the HAp sample when it was milled in combination with 4% PEG and 1% PVA binders. Through a variety of tests, involving x-ray diffraction, semi-electronic microscopy, shrinkage, modulus of rupture, Vickers hardness, surface roughness, porosity, and density, the mechanical parameters of the HAp sample were determined. Each experiment is carried out on the sintered body of HAp. Tests for shrinkage revealed variances of 15% to 25%, indicating significant dimensional changes following the sintering process. The MOR tests verified the toughness of the material by showing yield strengths ranging from 27.68 N/mm² to 38.99 N/mm². The densest and most structurally sound of the HAp samples with 4% PEG and 1% PVA binders, Sample 2 is best suited for high-strength applications, according to the porosity and density experimental investigation. Sample 3's lower density and porosity are more appropriate for lighter material requirements. In general, density is higher than porosity, which means that the binders have improved strength, which is important for implant and automotive applications. With values ranging from 165 HV to 178 HV and an average of 172.347 HV, the Vickers hardness test the results for HAp samples with 4% PEG and 1% PVA binders show an advantageous relationship between hardness and material density. Higher hardness and density are required for durability and effectiveness in load-bearing biomedical applications, which suggests the samples are suitable for these kinds of applications. The variation in hardness also demonstrates how porosity, grain size, and binder content affect the mechanical qualities, which is crucial for modifying HAp for specific uses in biomedicine. For the effect of cutting parameters on the HAp sample, the most important component influencing surface roughness, according to research on hydroxyapatite samples machined on a CNC, is feed rate. The ideal parameters were found to be 0.023 mm/rev, 27 m/min for cutting speed, and 0.05 mm for cut depth. These settings provide the best surface quality and process stability and have been proven by experimental findings that reveal surface roughness values near to the estimated 1.189 μm . The study emphasises that, in order to obtain the required level of surface smoothness, feed rate and depth of cut optimisation are crucial, with cutting speed being the least significant factor. All of the experiments done on sintered hydroxyapatite ceramics have finally showed exactly the mechanical properties of the HAp sample combined with 4% PEG and 1% PVA are. The parameter that has been most influenced, the parameter that is best based on Taguchi method analysis, and the parameter combination with the lowest surface roughness have all been determined. Each of the originally set objectives was achieved.

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References

- [1] S. Ajmal, F. Athar Hashmi, and I. Imran, "Recent progress in development and applications of biomaterials," *Mater. Today Proc.*, vol. 62, pp. 385–391, 2022, doi: 10.1016/j.matpr.2022.04.233.
- [2] Z. Jawad, F. Mohammed, and E. Al-hassani, "Effect of Cordierite Additions on Mechanical Properties of Hydroxyapatite Used in Medical Applications," *Eng. Technol. J.*, vol. 41, no. 6, pp. 0–0, 2023, doi: 10.30684/etj.2023.138142.1375.
- [3] S. Rajan, K. Marimuthu, C. B. Ayyanar, and M. E. Hoque, "Development and in-vitro characterization of HAP blended PVA/PEG bio-membrane," *J. Mater. Res. Technol.*, vol. 18, pp. 4956–4964, 2022, doi: 10.1016/j.jmrt.2022.04.130.
- [4] D. K. Yadav, N. K. Dixit, D. Agarwal, and S. K. Khare, "Optimization of surface roughness by design of experiment techniques during CNC milling machining," *Mater. Today Proc.*, vol. 52, no. 2022, pp. 1919–1923, 2022, doi: 10.1016/j.matpr.2021.11.565.