Effect of Palm Stearin binder to the Mechanical Properties of Alumina using Slip Casting Method

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Abstract. Alumina is one of bio-ceramics that have been used as bone replacement material in modern health care industries due to its good mechanical properties. Unfortunately, their potential as bone implant depends upon its strength and compatibility with the biological environment. Since palm stearin possesses low molecular weight, which can prevent residual stress and distortion with abundantly available in Malaysia (low cost). Furthermore, bone implant using palm stearin as a binder has never been studied before. Thus, a study has been carried out to investigate the effect of different contents of Palm Stearin (35 vol%, 36 vol%, 37vol % and 38 vol%) with different sintering temperatures (1300°C, 1350°C, 1400°C, and 1450°C) using a slip casting process to produce porous alumina like the human bone. The samples then were characterized in terms of physical and mechanical properties. The water impregnation test was used to determine the percentage of porosity of the sample whilst Scanning Electron Micrograph (SEM) to determine its pore structure. Three-point bend test and hardness test were used to determine its flexural strength and hardness respectively. It was found that by using 36 % volume of palm stearin and 64% volume alumina which sintered at 1400ºC has porosity of 41.63% with a pore size measured 112μm in diameter. The flexural strength and hardness valued at 5.366 MPa.

1. Introduction

Since 1975, alumina ceramic has proven its bio inertness (it does not interact with the body’s environment apart from causing an initial ‘fibrous tissues’), high wear resistance and excellent biocompatibility (ability of surgical or dental implant to exist in the body with no adverse effect). Even though it has good properties physically and mechanically, but their potential as implant depends upon its strength and compatibility with the biological environment.

This research requires the sample produced from alumina and palm stearin binder using the slip casting method due to cheaper and wide variety of complex shapes can be produced. The samples will be sintered at a certain temperature. Before sintering, the sample will undergo thermal de-binding process to completely remove all binders. This temperature must be higher than the decomposition temperature of the binder. Hence, thermal de-binding and sintering temperature plays a major role to produce a strong and porous sample. According to [1], bone composed of two types i.e. cortical (compact) and cancellous (spongy) bones. Cortical bone has a density of ~2.0 g/cm3 whilst cancellous bone has much lower density; 0.2 g/cm3 [1]. Since alumina has proven its bio inertness, the usage of alumina in bone implant has widen and some examples are hip prostheses ball and sockets, knee prostheses, dental implants and bone screw [2]. The presence of the binder can affect the sintering and...
strength of alumina. Therefore, properties of palm stearin binder are studied and according to [3], palm stearin has a melting point of 61°C and its decomposition temperature is between 288°C - 463°C, environmentally acceptable and low cost [3]. One of the main properties that needs attention is the porosity of alumina that is like the human bone. As mentioned by [4], for bone regeneration, the ideal pore sizes would be in the range of 100 – 400 μm. Its mechanical properties are enough to withstand the in-vivo loads which is a basic requirement of a successful scaffold structure [4]. Porous materials have the advantage of biological anchorage for ingrowth of mineralized tissue into the pore spaces. The architecture of porous implant gives a great impact of bone in growth in the pore [4].

Moreover, the addition of binder will influence the shrinkage phenomenon. When this happened, more areas between the pores are in contact making the pore become smaller. Thus, making the bonding between particle stronger [5]. To test the mechanical properties of the Alumina, 3-point bend and hardness test is used. Brittle material strengths are statistical in nature and different specimen yield different strengths due to variations in the flaws and microstructure [6].

2. Research Work

Suitable methods or procedure is carried out according to the current standard. Flowchart below is the overview of the methodology.

\[\text{Mould preparation} \rightarrow \text{Sample preparation} \rightarrow \text{Alumina + Palm Stearin} \rightarrow \text{Thermal de-binding + sintering} \rightarrow \text{Testing} \rightarrow \text{Physical} \rightarrow \text{Mechanical}\]

2.1 Mold preparation

Typical mold used by the slip casting method is made from plaster of Paris. However, plaster of Paris is not suitable and then it has been changed to silicon mold because the sample sticks to the mold.

2.2 Sample preparation

Important information for materials preparation is to know the pycnometer density of alumina and palm stearin, which are 3.625 g/cm³ and 0.9915 g/cm³ respectively.

2.3 Mixing and Slip casting

Alumina is mixed with Palm Stearin using the mechanical stirrer. Variables that are held constant during mixing pro-cess are:
Temperature: 60 - 64°C Speed: 10 rpm Time of mixing: 6 – 9 minutes.

| Material       | Sample 1 | Sample 2 | Sample 3 | Sample 4 |
|----------------|----------|----------|----------|----------|
| Alumina        | 62 vol. %| 63 vol. %| 64 vol. %| 65 vol. %|
| Palm stearin   | 38 vol. %| 37 vol. %| 36 vol. %| 35 vol. %|

2.4 Thermal de-binding and sintering

Heating rate used for the sintering process is 1°C/min. Before the sample is sintered, it will undergo thermal de-binding process to completely remove all palm stearin in the sample.
2.5 Physical testing

After all samples had undergone a sintering process, their porosity is calculated using equation (1):

\[ P(\%) = \frac{\text{Palumina powder} - \text{Psample}}{\text{Palumina powder}} \times 100 \]  

The microstructure and pore size of alumina is observed using the HITACHI TM30000 Scanning Electron Micrograph (SEM) since different content of palm stearin and will result in different size and arrangement of pore structure.

2.6 Mechanical testing

The machine used for 3-point bend testing is INSTRON 54778 with a static rating ± 10kN and weight of 1.55 kg. The parameters that kept constant are the temperature (23°C), speed rate (20m/s) and humidity (50%).

3. Research Finding

3.1. Sintering

Figure 2 shows that the higher palm stearin content and sintering temperature gives greater shrinkage percentage. Greater shrinkage makes the sample become denser and the bonding between particle is stronger as the pore size becomes smaller. As mentioned by Nampi et. al. (2009), ceramic material shrinks linearly 20%. Hence, at 1450 °C with 38 vol. % of Palm stearin shows the highest percentage of shrinkage which is 25.62%, which is a little bit higher maybe due to the type of binder used.

Figure 2. Effect of sintering temperatures to the percentage of shrinkage of alumina for different binder contents.
3.2. **Porosity**

One of the parameters that affect the porosity and pore size of Alumina is the sintering temperature. Porosity and pore size distribution are decreased when sintering temperatures increase [7]. This can be seen from Figure 3 where alumina that was sintered at 1300 °C with 38 vol. % of palm stearin is the most porous and as the sintering temperature increases, alumina became denser. Additionally, in this study, additionally, the result shows that the porosity for all samples ranges from 35 – 51%. On the contrary, according to L. A. Stanciu et. al. (2001), fast heating rates favor densification instead of grain growth. Furthermore, sintering at higher temperatures can cause possible partial entrapment of the pores within grains [8].

![Figure 3. Effect of palm stearin binder to the porosity of alumina for different sintering temperatures.](image)

3.3. **Flexural properties**

As shown in Figure 4, flexural strength increases with the sintering temperature and the highest flexural strength achieved was 14.51 MPa with 35 vol. % Palm stearin at 1450 °C. Furthermore, as more Palm stearin binder were added, the flexural strength decreases, and the lowest value recorded was 1.534 MPa.

The effect of sintering temperature really gives an impact towards the flexural strength of Alumina. Another factor that contributes to these changes is Palm stearin content. Basically, during sintering, all the binders will decompose leaving behind bars. More binders mean more pores created and hence, decreasing in flexural strength. Porosity has a significant role to influence the flexural strength of sintered Alumina and it is well known that the strength of porous Alumina increases with the decreasing of porosity [9].

![Figure 4. Effect of sintering temperatures to the flexural strength of alumina for different binder contents.](image)
Flexural modulus is the ratio of stress to strain in flexural deformation. From the Figure 5, the modulus increases as sintering temperature increases, which means that, as the sintering temperature increases, Alumina become stronger and makes it harder to bend. The highest value obtained was 86.6269 MPa.

![Figure 5. Effect of sintering temperatures to the flexural modulus of alumina for different binder contents.](image)

3.4. **Hardness**

Hardness increases with the increase in temperature. As shown in the Figure 6 below, at 1450 ºC with the least amount of Palm stearin binder (35 vol. %) is the highest recorded value for hardness which was 79.1 Hv and the lowest value was 17.6 Hv with the highest amount of Palm stearin. A similar trend has been observed by Rana (2009) and Basu et. al. (2005). Hardness value increases along with sintering temperature is because higher sintering temperature offers better densification [10].

![Figure 6. Effect of sintering temperature to the hardness of alumina for different binder content.](image)

3.5. **Microstructural Analysis.**

The microstructure of Alumina mixed with vol. 38% of palm stearin sintered at 1400°C produce pore size of 348μm which is favorable for the bone to grow. Additionally, there’s no presence of palm stearin in the sintered alumina which means that the 3 hours of soaking time is enough to decompose the binder.
Figure 7. Microstructure of Alumina with 38 vol.% palm stearin before and after sintering with pore size 348 μm.

4. Conclusions
The first objective is to find out how the different binder contents can affect the porosity of Alumina; the preliminary result shows that the higher the binder, the porosity amount is increased. The ruptured samples after sintered at temperature 1400°C embedded by the Ytoria was a good learning point to improve the heating method (thermal debinding and sintering).

Greater shrinkage makes the sample become denser and the bonding between particle is stronger as the pore size becomes smaller. Parameters that contribute to shrinkage are the Palm stearin content and sintering temperatures. Therefore, at 1450 ºC with 38 vol. % of Palm stearin shows the highest percentage of shrinkage which is 25.62 %.

The porosity percentage increase as the amount of palm stearin increases. All samples produced porosity ranges from 35 – 51%, which lies in the range for bone porosity (40 – 60%). In terms of pore size, most samples have successfully produced pore sizes ranging from 100 – 350 μm which is ideal for bone regeneration.

Sintering temperature enhanced the strength and hardness of Alumina. It has been proved that the higher the sintering temperature, the stronger the Alumina. The flexural strength and hardness obtained by sample containing 35 vol. % of palm stearin are 14.51 MPa and 79.1 Hv respectively, is good for bone implantation.

Finally, it can be said that palm stearin has proved to be useful and more research can be done soon using palm stearin as a binder to know its full potential.

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