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CHARACTERIZATION OF SUSTAINABLE BINDER WITH HYDROXYAPATITE VIA POWDER METALLURGY ROUTE

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ABSTRACT

Injection molding is a method to produce part and intricate the serial production of net-shape, functionalised parts and structured surfaces in a large batches. Nowadays, there are millions of tons of plastic bags in around the world that's damage to environment and the researchers believe that waste can be used to develop new beneficial product. Thus, this research extensively investigate the potential used Hydroxyapatite (HA), waste plastic bag Low-Density Polyethylene (LDPE) as primary binder and suitable secondary binder, Palm Stearin in injection molding. A simplified hydrothermal method of synthesizing Hydroxyapatite powder is described. Basically, there are four stages that involved which is mixing, molding, debinding and lastly is sintering. Nowadays, due to an exploration of application "Green Technology", this research will be become most of strongly finding for future research in biomedical application. The volume ratio of HA, waste plastic bag LDPE, Palm Stearin combination in feedstock was investigated. Feedstock with two composition 30% and 40% powder loading was injected by injection molding machine with several injection parameter. These finding supports the development of "Green Technology" by using waste plastic bag (LDPE) and Palm Stearin as binder system in injection molding.

Keywords: injection molding, hydroxyapatite, waste plastic bag, palm stearin, implants.

INTRODUCTION

Injection molding is a method to produce part and intricate the serial production of net-shape, functionalised parts and structured surfaces in a large batches. There are four processing steps in injection molding; mixing, injection, debinding and sintering. The injection molding process calls for the production of a homogeneous feedstock consisting of micrometre scale metal particles and organic binders. Then, the feedstock is injected into a patterned mould cavity to replicate a defined surface patterned green part. The molded green part is sintered at an elevated temperature and the final metal part is obtained in metal particle fusion.

Nowadays, both medical and material sciences are needed to provide solutions to the problems for human body. Some of the problem are related to bone degredation, dental etc. Hydroxyapatite (HA) is a material that has significant research application, especially in the biomedical field. HA, with the chemical formula $Ca(PO_4)_6(OH)_2$ is the main component of both bone and teeth. Various method have been employed for the preparation of HA either from natural source (coral and bone) or through chemical synthesis.

It is highly bioactive nature allows new bone to grow from the existing bony walls into the implants made from synthesis HA, thus making the prosthesis stable within a very short period of time [1]. Its unique bioactivity promotes rapid bone growth interfacial fixation that has potential for biomedical applications [2].

The beneficial biocompatible properties of Hydroxyapatite are well documented. It is rapidly integration into the human body, while at the same time the body is none the wiser as to the invasion by a foreign body, albeit a friendly invasion. Its most interesting property is that Hydroxyapatite will bond to bone forming indistinguishable unions [3, 4, 5, 6].

In this study, injection molding technique was chosen because there are some of the advantages using such as parts can be designed and manufactured without any design restrictions. In addition, almost all design changes are possible within the shortest development cycle and turnaround time. Other than that, this technology is the best viable process for producing miniature parts economically [7, 8].

It is well known that this process is ideal for producing complex-shaped components as well as parts that require assembly or multiple steps to put together. Last but not least, it is most beneficial in high volume production of small precision parts with complicated design geometry. The process lends itself to automation where high volumes and consistent quality are required.

GUIDELINES FOR MATERIAL PREPARATION

Raw materials

In this study, there is a few things needs to be done to prepare the HA as a ceramic powder and cutting plastic bag (LDPE). There are mixing all the material, stirring under heating and drying. Two main raw material that need to mix together are Calcium Oxide (CaO) and Ammonium Di-Hydrogen Phosphate ($(NH_4)H_2PO_4$) as the pre-cursors for Calcium and Phosphorus respectively. Distilled water is used as solvent. Then the material was heated and dried to produce the pure HA powder. The density of the HA is: 2.2809 g/cm^3. The polymer used was waste plastic bag (LDPE). The melting point and density of waste plastic bag (LDPE) were measured to be 109.9ºC and 0.910 g/cm^3, respectively.

Processing of LDPE-HA composite

A material mixing is a process mixed up the ceramic powder and binder systems with a correct volume ratio by using Plastograph Brabender at temperature 150°C with speed is 30 rpm. The time needed for compounding is around 90 minutes, depending on the amount of HA incorporated. The selected ratio are 70 % for plastic bag (LDPE) and 30% for Palm Stearin with two different powder loading which are 0.3 and 0.4. Injection molding was carried out using the horizontal screw injection molding machine. The main operating temperatures used include: nozzle, 165°C, front, 165°C, medium, 160°C, rear 1, 155°C and rear 2, 150°C. The cooling time is 20s.

Tensile testing

The strength of the green part was focused on determination of green strength of un-sintered compacted powder metallurgy materials. The experiment is conducted by using Universal Testing Machine. In this test, tensile testing was performed according to the ISO 527-5A (MS) procedures at room temperature using Universal Testing Machine. This machine was setting to get the data to be analyzed where the grip lengths of the green part are 25 mm, while the speed is 5mm/minute.

Debinding

The powder loading of 0.4 have seven samples and each one are placed in the different beakers. Each beaker filled with 30 ml heptane until each of the sample is submerged in the heptane. All beakers are placed in an oven with a temperature of 70ºC [9]. The function of heptane liquid is it works to remove Palm Stearin which is contained in the sample. The debinding process is carried out for 5 hours and the weights of each sample are collected for every one hour.

RESULTS AND DISCUSSIONS

Density of green part

The density of the green part is determined by using Mettler Toledo X 64 apparatus to investigate their performance in order to attain the better quality of green part. Density determination is important in terms of the determination of mineral phases present in samples. All green part with powder loading 0.3 and 0.4 were involved in this testing. Table 1 shows the density of green part for every powder loading percentages.

According to Table-1 shows the density of the green part for every powder loadings. The green part with 0.4 powders loading was in highest density compared to green part with 0.3 powder loading. According from the data above, the density of each sample is not consistent. The average density of powder loading 0.3 is 1.4110 less than density of powder loading 0.4 which is 1.4968. In this tested, this circumstance are assumed due to the highest percent of Hydroxyapatite in all samples for powder loading 0.4 in their composition compared to all samples for powder loading 0.3.

Table-1. Density of green part.

Sample	Powder loading 0.3	Powder loading 0.4
1	1.4107	1.4941
2	1.4041	1.4911
3	1.4112	1.4636
4	1.4027	1.4711
5	1.4066	1.4908
6	1.4203	1.5248
7	1.4216	1.5421
Average	1.4110	1.4968

Porosity of green part

Porosity is defines as being full of tiny holes that the air or water can get through. The density data is important in terms of the determination of mineral phases present in ceramic materials and also in the calculations to obtain the actual porosity of the material. According to the Table-2, the porosity of all sample in both powder loading have less than 1%. The amount of interstitial empty spaces in the materials produced by powder compact, such as ceramics, have a major impact on their properties. Thus, it has an effect on mechanical properties, chemical properties and mechanical erosion of the material.

Table-2. The porosity of all samples.

Sample	Powder loading 0.3 (%)	Powder loading 0.4 (%)
1	0.1823	0.2180
2	0.1831	0.2630
3	0.1847	0.1023
4	0.1854	0.0392
5	0.0880	0.1364
6	0.2260	0.2304
7	0.0810	0.1038

Analysis maximum stress and maximum strain of the green part powder loading 0.3

Figure-1 shows the graph of stress vs. strain for powder loading 0.3 for the LDPE-HA composite developed. The maximum stress value is 6.0578 MPa from sample 7 and minimum in sample 1 of 4.6852 MPa. Strain as ratio of the length also experienced as stress reduction powder loading 0.3 where the maximum strain is 3.690% in the sample 4 and the minimum strain of 2.950% in the sample 6.

It can be seen that the composite materials exhibited both ductile and brittle behavior, depending on the amount of HA incorporated into the plastic bag LDPE polymer. The increasing the amount of HA resulted in the composite losing its ductility as seen from the composite failure occurring in the elastic region, typical of brittle mode failure [10].

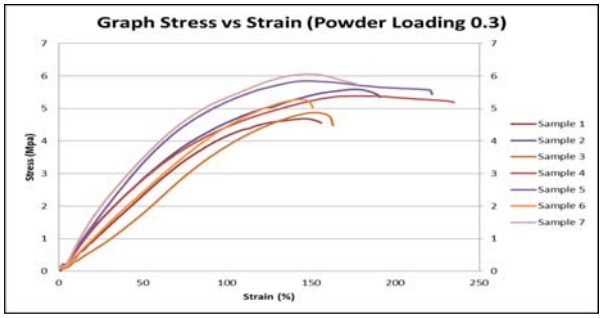


Figure-1. Graph stress vs strain (Powder loading 0.3).

The tensile properties of composites were dependent on the HA content. The reinforcement effect of HA is clearly evident, however, at the expense of tensile strength. This resulted from the inherent characteristics of monolithic HA, a synthetic ceramic material that is typically characterized by its high stiffness and brittleness [10].

Analysis maximum stress and maximum strain of the green part powder loading 0.4

Figure-2 shows the graph of stress vs. strain for loading powder 0.4. This graph is same with graph powder loading 0.3 but as shown in the figure above, the stress for all specimens is not consistent. The maximum stress value is 6.4598 MPa from sample 7 and minimum in sample 3 of 4.9410 MPa. While, the maximum strain for powder loading 0.4 is 3.440% in sample 7 while the minimum strain is 1.765% in the sample 3.

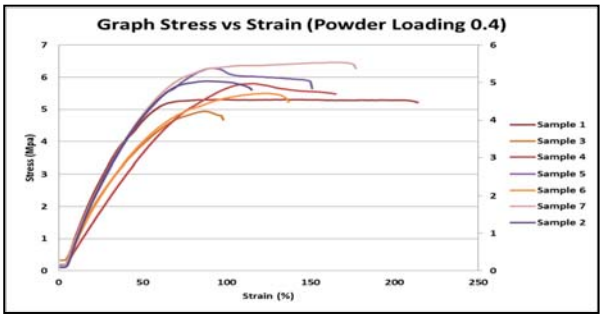


Figure-2. Graph stress vs strain (Powder loading 0.4).

Regarding from the data, the value of stress for powder loading 0.4 is higher than powder loading 0.3 because while injection molding process, the grain boundaries of the material for powder loading more compact than material powder loading 0.3.

However, the amount of HA and plastic bag LDPE polymer still affect strength of the composite materials for 0.4 powder loading which are both ductile and brittle behavior. This situation is same with 0.3 powder loading where the increasing of the amount of HA resulted in the composite losing its ductility and can be categorized as typical of brittle mode failure [10].

Solvent debinding

In this research, powder loading 0.4 are selected to carry out for the debinding process. It can be seen that the green part with higher powder loading has a high density compared to lowest amount of powder loadings. By this circumstances, the green part with higher powder loading are assumed to give a better outcomes for better density value.

The use of different solvent debinding time is also the main factors that will differentiate the weight loss of the tested samples. Weight loss for every samples occurred rapidly at the beginning of the process and slow decrease at the end of the process. This is because most of the Palm Stearin is removed after debinding time between three to four hours of the process is carried out.

Figure-3 presented the graph weight against time that plotted based on the result. The graph pattern of each sample shows are similar pattern which is the weight are reduce by increment of time. The weight of the sample are reduce when the debinding time are increased.

Scanning electron microscopy (SEM) analysis

In this research, this samples of HA are scales for resolution 10 μm at 10kV. The results of image are zooming in to 1500x about 10μm across the whole field to view. The distance of the detector and the SEM holder of a sample is 10 mm at z-axis.

Figure-4 represents the image that catches out from SEM. There are three variables of magnification that uses while conducted the experiment whereas 500x, 1000x and 1500x in order to obtain the clear images and validate the discussion process.

From the figure below, it can be seen in the chain of chemical bonds for Hydroxyapatite more compact and homogenous.

According to Figure-5 shows the difference of green part for each powder loading. The green part images for powder loading 0.3 shows, most of the Hydroxyapatite are covered with LDPE and Palm Stearin whereas acted as primary and secondary binder system. Besides, for this composition powder loading, the binders are monopolizing the structure region due to the high percentages is Palm Stearin.

From the image powder loading 0.4, the Hydroxyapatite powders are more cloaked and homogenous compared as before. However the pattern of images is not much different as before. Hydroxyapatite, LDPE and Palm Stearin was able to bonding with each other and it can be seen through on SEM microstructure images.

According to Figure-6, the microstructure image of powder loading 0.4 green parts was demonstrated whereas the cavities are formed. This image is captured from the green part samples that under test for solvent debinding process in five hours continuously. The cavity as in image is occurring due to losses of weighted of green part. Reduction of secondary binder based on the proportion of time.

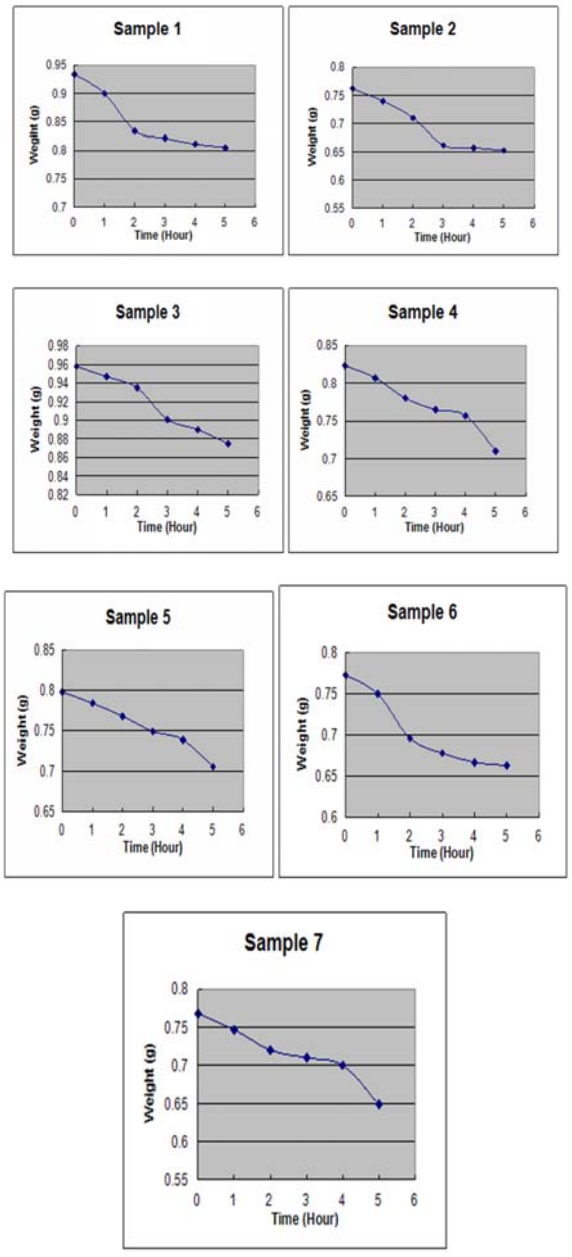


Figure-3. Graph effect of solvent debinding on weight loss of green parts (Sample 1 to 7) with powder loading 0.4 immersed in heptane for various times.

It can be seen after one hour in which the cavity is constructed. After three hours, the cavity is deep as before and in five hours, Palm Stearin are soluble and leaves from the green part, so that's the cavity are constructed. At this crossing, Palm Stearin assumed totally removed from the part and was been proven with the weighting of the green parts. In theoretically, solvent debinding process works by removal the Palm Stearin as a secondary binder system in this current research. So that, with this SEM images, it has proven the Palm Stearin are soluble while debinding process and leaves the green part.

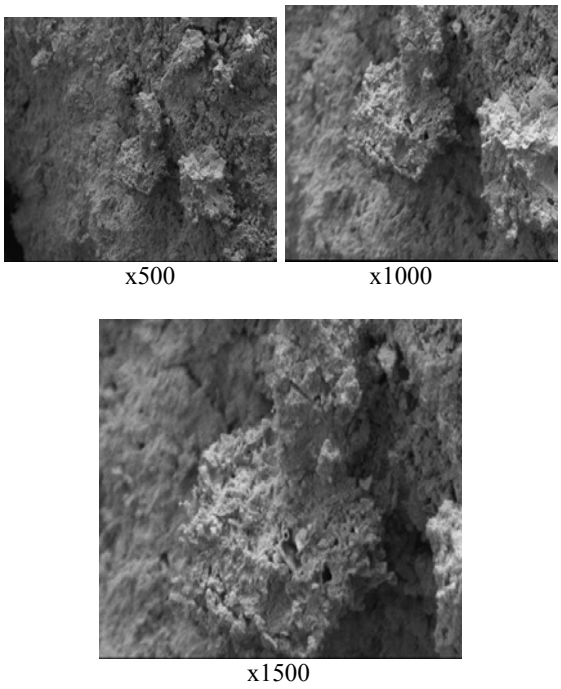


Figure-4. SEM analysis for pure HA.

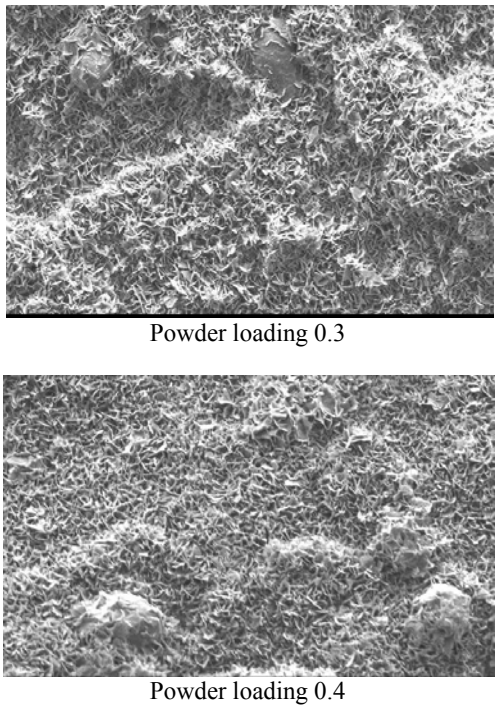
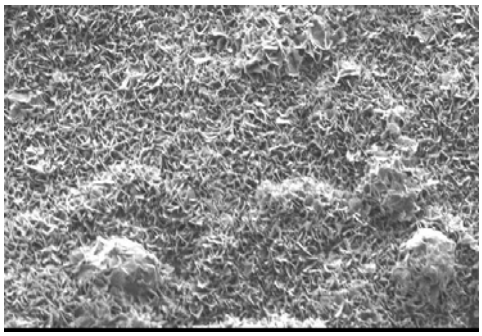
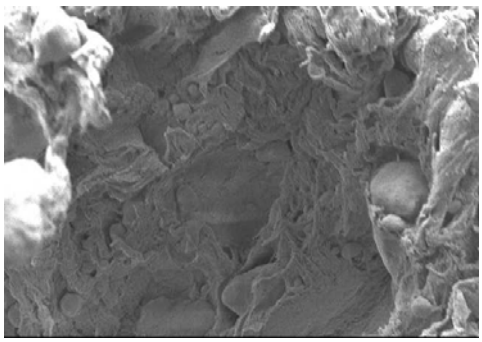


Figure-5. The images of microstructure for green part with different powder loading, 0.3 powder loading, and 0.4 powder loading.

In more understanding, the increment of solubility and diffusivity at higher temperature, the binder dissolve in heptane and leave the compact and then causing the weight loss.



Before debinding



After debinding

Figure-6. SEM images of powder loading 0.4 green part for solvent debinding process before and after five hours.

CONCLUSIONS

A new bioactive LDPE-HA composite was developed using the hydrothermal synthesis using three main mixture of Calcium Oxide, (CaO) Ammonium Di-Hydrogen Phosphate, $(NH_4)H_2PO_4$ and distilled water, H_2O and compounding with binders, where binders are 70% plastic bag (LDPE) and 30% Palm Stearin. The mechanical characteristics observed are tensile strength for powder loading 0.3, 6.0578 MPa and powder loading 0.4, 6.4598 MPa. As known, the research related to "Green Technology" is still new and is growing rapidly. This is because there are many benefits and advantages which can protect the environment and also reduce cost. Hopefully these findings of this research could contribute for future research to explore this application by using plastic bag (LDPE) and Palm Stearin as binder system in injection molding.

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