Processability Study of Natural Hydroxyapatite and SS316L Via Metal Injection Molding

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***Abstract*-Metal Injection Molding (MIM) process is capable to produces varieties of intricate shape and complex geometry parts in large quantity components at low cost of processing. There are four Major steps in MIM process which is includes of mixing fine metal powders with plastic binder to form a feedstock, injection molding of the green part, extracting the binder, and finally sintering. Thus this paper are concentrate on investigate the processability of Natural Hydroxyapatite and Stainless Steel (SS316L) mixture with an established binder formulation as a feedstock in MIM. The Natural Hydroxyapatite (NHAP) was produced from waste Tilapia Fish Bones. While, Polyethylene (PE) and Palm stearin (PS) acted as a binder system. NHAP/ SS316L mixture was successfully injected and established as an appropriate feedstock in MIM. The green strength of ejected part was determined. The maximum force that could attach is 48.1719 N and the maximum stress equal to 6.02Mpa.**

Keywords- *Natural Hydroxyapatite; Tilapia Fish Bones; SS316L; Feedstock; Metal Injection Molding*

I. INTRODUCTION

Metal Injection Molding is comparatively known as a development of processing technology applied in powder metallurgy processing industries [1]. Moreover , it also define as a modification from plastic injection molding which is replaced it major constituent of plastic to fine metal powder [2]. There are four Major steps in MIM process which is includes of mixing, molding, debinding and sintering [3]. The terms of feedstock is corresponds to the mixture of powder and binder [3]. Hydroxyapatite (HAP) $Ca_{10}(PO_4)_6(OH_2)$ is one of constituent that found in human bone or teeth and the major elements including calcium and phosphorous. The properties of HAP composition is nearly closed to the human bone. In addition, HAP also categorized as high biocompatibility materials and it lead toward an extensive use in medical implant materials. It's called as high biocompatibility due to stoichiometric ratio equal to 1.667 which is considered on the weight of calcium to phosphorous [4, 5]. Earlier investigation on hydroxyapatite (HAP) as a powder in MIM employed by using the HA /SS316L with powder loading of 58%. [6]. The other researcher use the combination of HA/ Ti6Al4V with 78.21 vol % [7]. Both of researchers used the synthetic HA with PE and PS as binder system. The problems of hydroxyapatite powder produced by synthetic process are needed additional chemical to improve the mechanical strength and many chemical involved in process to stable the structures and fixed in human body. This method is rather complicated and required high initial cost to produce HAP. Moreover the HAP from natural is cheaper and uncomplicated compared to synthetic method. Besides, this process also takes a long time and difficult to control the sample of hydroxyapatite produced completely [8]. Based on this problem, the hydroxyapatite powder was produced from the natural sources (Tilapia Fish Bones) by calcination process. [9,10]. NHAP that derived from Tilapia fish bone used to apply as powder in metal injection molding with the combination of metal powder SS316 L and binder system. The combination between SS316L and HAP could enhanced the mechanical properties of the biocomposites by equilibrate their disadvantages of each other. These biocomposites are expected to be applied in numerous applications especially for load bearing implants [6, 11]. Thus this paper are concentrate on investigate the processablity of NHAP / SS316L mixture with the established binder formulation as a feedstock in MIM.

II. MATERIALS AND METHODS

A. *Materials*

Table 1 and fig 1 summarized the combination characteristic of SS316L water atomized powder (Epson Atmix Corp) with irregular shapes as it is compatible with water leaching and high corrosion resistance that was acted as a metal powder. While NHAP from Tilapia Fish bones were used as ceramic powder. The NHAP powder was produced from Tilapia Fish bones by calcination process. Moreover, an established binder system in MIM which is Polyethylene (PE) and Palm stearin (PS) acted as temporary vehicles in for homogenously feedstock and packing powder into desired shaped before sintering stages [12].

TABLE I: COMBINATION OF (SS316L) AND NHAP CHARACTERISTIC

Characteristic	Details
Identification	SS31L PF-10F / $Ca_{10}(PO_4)_6(OH_2)$
Powder source	Epson Atomix Corp / Tilapia Fish bones
Tap density g/m^3	4.06
True pynometer density g/m^3	8.0471/ 3.156
Powder size	D10 = 2.06 µm D50 = 6.553 µm D90 = 17.712 µm

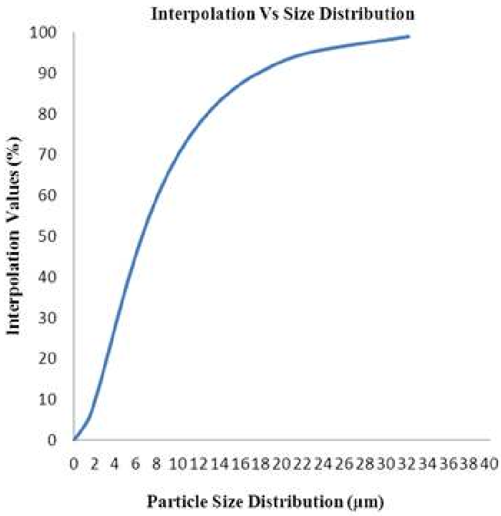

Figure 1: Graph Interpolation vs. Particle Size Distribution for SS316L/NHAP

B. Critical Powder Volume Concentration (CPVC)

The terms of CPVC is represents the maximum volume of powder in fluids where as the mixture of powder condition is uniformly [13, 14] However, the uniformity of the mixture is predicted by the variation mixing torque versus time [14]. The mixing torque is plays an important role in determination the Critical Powder Volume Percentage (CPVP). The maximum torque in evolution curves presented a critical powder loading that can be used for metal powders. It can be obtained by using ASTM 281-31 standard by adding 0.5 ml oleic acid within every 5 minutes of interval time until the maximum torque value were drop. Equation 1 shows the correlation the correlation between volume of oleic acid and torque value:-

$$CPVC = 100 \, x \, (v_f / v_f - v_0) \, \% \qquad (1)$$

Critical loading point can be estimated when all the particles are tightly packed and all voids between the particles are filled with the binder [14, 3]. The optimum powder loading is kept approximately 2-5% lowers than the critical loading [3].

C. Mixing

There feedstock compositions with 63 Vol. % powder loading and binder formulations were mixed. Feedstock was utilized with 60 % PS and 40% PE respectively. The melting point of PE is 120 ˚C and the mixing temperature must be higher than the highest melting point and lower than the lowest of degradation temperature of binder system [12]. Regarding to this, the mixing temperature was set at 150 ˚C. Fig 2 show the Mixing Process While fig 3 Show the Feedstock

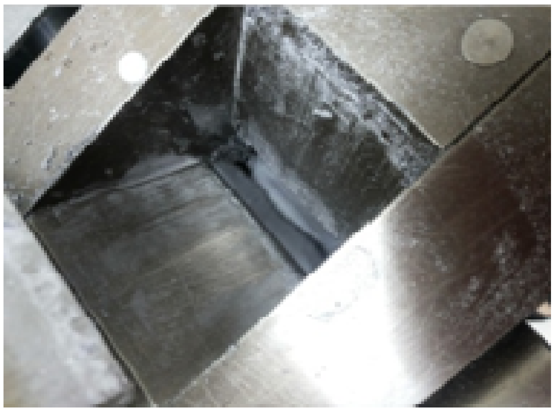

Figure 2: Mixing Process and Feedstock

Figure 3: Feedstock

D. Injection Molding

The next process is injection molding. Generally, the feedstock were feed into the injection molding machine and melt in the heated barrel. Pressure will be applied on the feedstock and it will force the melting material into the mold cavities. The feedstock solidified after the cooling stage and the pressure was released so those, the part will release. In simplest explanation, molding process refer to heating the feedstock in highest temperature that it melts, then following by forcing the melts into the cavity and solidified when it cool and the shape is formed as the mold cavity [7]. A series of trial and error tests were conducted to determine the optimal values of the parameters in the process. The values for injection temperature, barrel temperature, and injection pressure were set at 165 C, 160 C, and 64 Mpa, respectively, which are the optimal values to produce green part without any defect in this experiment. When the mold is filled, the feedstock will extract the heat through the die. The last stage in molding step where as cavity is opened in order to eject the part. Theoretically, the part is rigid at this point so that the temperature is below compared to the flow temperature while mixture process. Fig 4 shows the Nissei 21 Horizontal screw injection molding machine while fig 5 represent the Sample of Green part

Figure 4: Nissei 21 Horizontal screw injection molding machine

Figure 5: Sample of Green Part

III. RESULTS AND DISSCUSSION

A. *Critical Powder Volume Concentration (CPVC)*

The variation of torque while mixing process and the maximum solid loading can be predicted by monitoring the torque rheometer [15]. The homogeneity of the mixture could be reached by determining the critical powder loading. In simplest explanation the density peak at the critical powder loading with the point where the viscosity becomes effectively infinite. In other words, mixture composition near the critical loading are amplified into large viscosity shifts [14]. Figure 6 exhibit the torque value versus mixing time at every point of solids loading. The values of torque were increase when the combinations of SS316L/NHAP powder were added in the mixture and then return to lower steady stated associated with the homogeneity of the mixture. However the mixture is in unstable condition when excessive the critical powder loading point. The critical powder loading for SS316L/NHAP is 68.189 % Vol as the highest peak in fig 6. Basically, higher powder loading also contributes in producing the better shape retention part especially after sintering process. Other than that it also can minimize shrinkage but it will give a barrier while mixing process and it will resulting the inhomogeneous feedstock and the optimum powder loading is around 2% -5% lower than CPVC [3]. 63%Vol is the selected powder loading that uses which is within the optimum powder loading value.

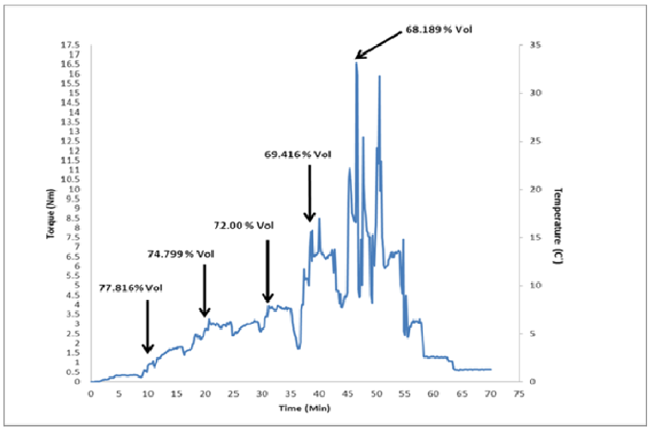

Figure 6: Critical Powder Volume Concentration for SS316L/NHAP mixture

B. *Green Strength Of Injected Part*

The green strength means the mechanical strength of parts where as a compacted powder must have in order to withstand mechanical operations and it is subjected after pressing and before sintering, without damaging its fine details and sharp edges [14]. In this current study, the strength of the green part are analyzed by followed Metal powder Industries Federation (MPIF 15) as test standard recommended, which is focused on determination of green strength of unsintered compacted powder metallurgy materials. The testing methods are called as Transverse Rupture Test Fixture. The experiment are conducted by using Universal Testing Machine that provided by polymer and ceramic laboratory, UTHM. The green parts are place centrally located and perpendicular to the supporting rods where as the top surface faced upwards. In simplest explanations, the green part were supported near the ends and then broken by applying the force at the center of the support The span lengths of the green part are 25.4 mm or equal to 1 inch, while the speed of the Universal testing machine are 0.1 inch /minute. Fig 7 shows the Transverse Rupture Test Fixture methods

Figure 7: Transverse Rupture Test Fixture methods

Based on fig 8, the series of graph show the maximum force that could attach is 48.1719 N and the maximum stress equal to 6.02Mpa. To date there are several biocompatible metallic materials that are frequently used as implanting materials in orthopedic surgery, Medical grade SS 316L is categorized as austenitic chromium - nickel stainless steel which is having properties such as sinterability to high densities and corrosion resistance [16]. Compared to other metallic implants, stainless steels exhibit lower strength and much higher corrosion resistance, but possess greater ductility and lower production [17]. Hydroxyapatite (HAP) structure are contains of calcium phosphate which is had such a great properties in terms of bioactivity and biocompatibility. This is due to their similarity composition to the inorganic tissues of the bone structure. [18]. Metal implant has a higher Young's modulus compared to human bones and this difference is called as stress shielding which is caused by inhomogeneous distribution of bone from metal implant. However the present of the porous structure in the implant could reduce the Young's Modulus [19]. So, in this research with the composition between SS316L and NHAP supposed could produce a porous structure part and it can be tailored to meet specific clinical requirements. Besides the aim of this research is invention the potential of using Tilapia Fish bones to produce powder as an introduction to contribute future development/research such as part in biomedical applications through Metal Injection Molding with cost effectively.

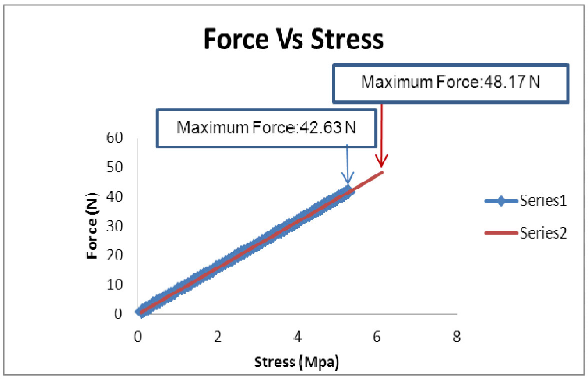

Figure 8: Graph Force Vs. Stress

IV. CONCLUSSION

In summary , the processability of feedstock that combine between Medical grade SS316L and NHAP that produce from waste Tilapia Fish bones were successfully by injected through Metal injection Molding. The feedstock compositions with 63 wt. % powder loading of SS316L/ NHAP and binder formulations were mixed. Feedstock was utilized with 60 % PS and 40% PE respectively. The values for injection temperature, barrel temperature, and injection pressure were set at 165 C, 160 C, and 64 Mpa, respectively, which are the optimal values to produce green part without any defect in this experiment.

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REFERENCES

[1] K. R. Jamaludin, N. Muhamad, M. N, S. Y. M. Amin, Metal injection Molding (MIM) Feedstock preparation with Dry and Wet Mixing: A rheological Behaviour Investigation , Advances In Mechanical, Material And Manufacturing Engineering. 2009

[2] Baiyun Huang, Shuquan Liang, Xuanhui Qu, The rheology of metal injection molding, Journal of Materials Processing Technology 137 ,2003, 132–137

[3] German .R and Bose, Injection Molding of Metals and Ceramics . Powder Industries Federation U.S.A, 1997

[4] Suchanek, W.L. and Yoshimura, M.,Processing and properties of hydroxyapatite- based biomaterials for use as hard tissue replacement implants, J.Mater.Res, 13(1998), 94-117

[5] Sharon kehoe, Optimisation of Hydroxyapatite (HAp) for Orthopaedic Application via the Chemical Precipitation Technique, School of Mechanical and Manufacturing Engineering, Dublin City University, 2008

[6] Ramli, Mohd Ikram, Sulong, Abu Bakar. Arifin, Amir, Muchtar, Andanastuti, Muhammad, Norhamidi, Powder Injection Molding of SS316L / HA Composite : Rheological Properties and Mechanical Properties of the Green Part, Journal of Applied Sciences Research, 8(11): 5317-5321, 2012

[7] Arifin, Amir, Sulong, Abu Bakar, Muhamad, Norhamidi, Syarif, Junaidi, Ramli, Mohd Ikram, Powder injection molding of HA/Ti6Al4V composite using palm stearin as based binder for implant material, Materials and Design 65,2015 , 1028–1034 Contents.

[8] Venkatesan, J. and Kim, S.K, Effect of temperature on Isolation and characterization of Hydroxyapatite from Tuna (Thynnus obesus) Bone., Materials 2010, 3, 4761-4772;

[9] Mustafa, N. Ibrahim, Mohd Halim Irwan , Asmawi, Rosli, Mohd Amin, Azriszul, Hydroxyapatite extracted from waste fish bones and scales via calcination method, International Integrated Engineering Summit (IIES,2014).

[10] Ahmad Norman Khalis Ahmad Fara, Mohd Azha Yahya, Hassan Zuhudi Abdullah, Preparation and Characterization of Biological Hydroxyapatite (HAp) Obtained from Tilapia Fish Bone, International Conference on X- rays and Related Teqchniques In Research And Industry 2014 (ICXRI,2014).

[11] Fan Xin, Chen Jian, Zou Jian Peng, Wan Qian, Zhou Cheng, Ruan Jian Ming, Bone Like Apatite Formation on HA/316L Stainless Steel, Composite Surface in simulated body fluid. Trans.Nonferrous Met.Soc.China 19, 2009, 347-352

[12] Z.Y. Liu , N.H. Loh , S.B. Tor , K.A. Khor , Y. Murakoshi , R. Maeda, Binder system for micro powder injection molding, Materials Letters 48 ,2001, 31–38

[13] Feng-Jiin Liu, Kan-Sen Chou,Determining critical ceramic powder volume concentration from viscosity measurements, Ceramics International 26,2000 , 159±164

[14] M.H.I Ibrahim, N. Muhamad, A.B. Sulong, Rheological Investigation of Water Atomised Stainless Steel Powder for Micro Metal Injection Molding, International Journal of Mechanical and Materials Engineering (IJMME), Vol. 4 ,2009, No. 1, 1-8.

[15] Mutsuddy, B.C., Ford, R.G, Ceramic Injection Molding.London: Chapman & Hall, 1995

[16] Heaney, D.F., T.J. Mueller, "Mechanical properties of metal injection moulded 316l stainless steel using both prealloy and master alloy techniques." Powder Metallurgy, 2004, 47(4): 1-7

[17] Fabrizio Matassi, Alessandra Botti, Luigi Sirleo, Christian Carulli, and Massimo Innocenti , Porous metal for orthopedics implants Clin Cases Miner Bone Metab ,2013, 111–115.

[18] Oleg Prokopiev, Igor Sevostianov, Dependence of the mechanical properties of sintered hydroxyapatite on the sintering temperature Materials Science and Engineering A 431, 2006 , 218–227

[19] Ryan G, Pandit A, Apatsidis DP. Fabrication methods of porous metals for use in orthopaedic applications. Biomaterials 2006;27:2651–70.