RESEARCH REPORT

Project ID: RIGS15-139-0139

Project Title: STUDY ON THE NOVEL BINDER BASED BIODEGRADABLE POLYHYROXYALKANOATES (PHA) FOR METAL INJECTION MOULDING APPLICATION

Project Sponsor: RIGS15, IIUM.

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Abstract:

Development of biopolymer binder for metal injection molding (MIM) feedstock play an important roles in improvising the debinding step in MIM process as it is the most time consuming step in the process. The backbone binder used in the industry is petroleum based polymer binder with high debinding time where this condition promotes defect formation to the part along the process. Replacing petroleum based polymer with polyydroxyalkanoates (PHA) biopolymer as the binder is one of idea to reduce the debinding time which reduce the processing cost as well as the formation of defects or failures to the injected part. Therefore, current research was done to provide reliable data on backbone binder mechanical strength, rheological properties of the feedstock and debinding behavior to determine the compatibility of PHA as backbone binder for MIM. The experimental work was divided into 4 main parts, development of backbone binder, rheological study of binder, debinding behavior of the binder and sintering of injected part. First, PHA was plasticized with different composition 1wt% to 5wt% of EPO and tensile tested. The best plasticized PHA was selected for backboned binder application, the binder system used was paraffin wax (PW), plasticized PHA and stearic acid (SA) with ratio 60:30:10 respectively. The second part was done by preparing four feedstock with different powder loading 70vol%, 71vol%, 72vol% and 73vol% and theologically tested. The optimum composition was injected and solvent debound in heptane at 40°C, 50°C and 60°C for 6 hours. The weight loss was measured and analyzed. Next, thermal debinding was set at 500°C with 4 different heating rate 2, 4, 6 and 8°C/min in vacuum atmosphere for 1 hour. The brown part was analyzed optically. The defect free brown part was sintered at 1380°C with heating rate 5°C/min for 3 hours. The density, hardness, shrinkage percentage and microstructure of sintered part was measured and observed. Based on the tensile test, plasticized PHA with 3wt% EPO exhibit high Young's modulus and the most suitable for backbone binder application. It was found that feedstock with 71vol% powder loading was the optimum composition. The optimum condition for solvent debinding in heptane were at 60°C for 60 minutes with 100% of the soluble binder was removed. This finding proved that new binder system reduced the debinding time. Through the experiment, it is found that defect was formed on the green part that thermally rebound. However, for grey parts no defect was formed for all heating rate. The sintered part shows insignificant different of all the properties between the samples, hence it can be conclude that debinding parameter has no significant to the sintered part. The value for density, shrinkage percentage and macro hardness of sintered part was 7.22g/cm^{3}, 6.2% and 67.2 HRB. Thus, it is found that PHA can replace the petroleum based polymer as a backbone binder. However some improvement need to be done on the sintering condition to obtain optimum properties of SS316L MIM sintered part.

Keywords:

plasticizer, EPO, mechanical properties, PHA, binder, MIM

Introduction:

In the present study of backbone binder for metal injection molding (MIM) application small numbers of them are from the renewable natural source. Most plasticizer used were petroleum based polymer such as PMMA (Chua et al. 2011) and polyethylene (Li et al. 2003). Development of biopolymer for backbone binder application will help in reducing the number of petroleum based polymer in large scale as MIM is a giant industry. The previous researcher has discovered a polymer known as polyhydroxyalkanoates (PHAs) that can be produced from renewable and biowaste resource by bacteria fermentation (Mekonnen et al. 2013; Bugnicourt et al. 2014). PHA is a biopolymer that exhibits biodegradable properties at varies environment not only in a composting plant. PHA can be processed into many forms for varies application such as packaging, molded good, films and performance additives (Bugnicourt et al. 2014) . However, pure PHA is a brittle material due to re-crystallization with aging at room temperature. Thus, the mechanical properties of PHA change with time. The mechanical properties of PHA can be modified by adding a plasticizer. The addition of plasticizer enhance the molecular motion and reduce the glass transition temperature of the materials (El-Hadi et al. 2002). Development of PHA blend with natural plasticizer like EPO is very interesting as the blend will be completely biodegradable in the environment and the EPO available in this country can be fully utilized.

Background:

Metal injection moulding (MIM) has been established since 1970's was derived from polymer injection moulding and ceramic injection moulding. In 1990's MIM has grown rapidly due to mass production capability of MIM with low cost compared to the other metal forming process.

The other advantages of MIM are the ability to produce intricate shape product from small to large size with good dimension accuracy tolerance. This advantage make them dominant in production of automotive components, medical equipment and other precision instrument component. Many medical device especially instrument and implant produced by MIM method to reduce the fabrication cost as the metal used are difficult to machine such as stainless steel, cobalt chromium alloy, and titanium alloy. By adopting MIM as the metal forming method no subsequent operation needed in achieving required dimension and shape. Figure 1.1 shows some example of MIM product.

a) b)

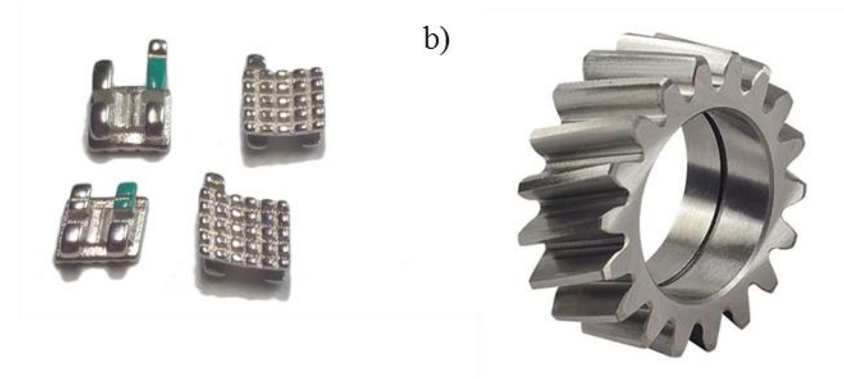


Figure 1. 1 Part produced by MIM a)mono block orthodontic part b) helix gear automotive part.

Objectives:

The general objective of this research is to develop new backbone binder by using PHA. The specific objectives for this research are:

i- To identify appropriate modified PHA as backbone binder for SS316L metal injection molding (MIM) process.

ii- To study the effect of PHA on the rheological properties of the feedstock.

iii- To investigate the effect of debinding parameters to the injected part.

iv- To evaluate the density, shrinkage, hardness and microstructure of MIM final part

Methodology:

The research methodology was planned as follows:

a) Raw materials characterization
b) Backbone preparation
c) Preliminary investigation
d) Feedstock preparation
e) Rheological test
f) Injection molding
g) Debinding
    i) Solvent debinding
    ii) Thermal debinding
h) Sintering
i) Physical and mechanical test

Figure 1.2 shows the overview flowchart of the studies. The flow chart was divided into 3 part according to the objectives to be achieved.

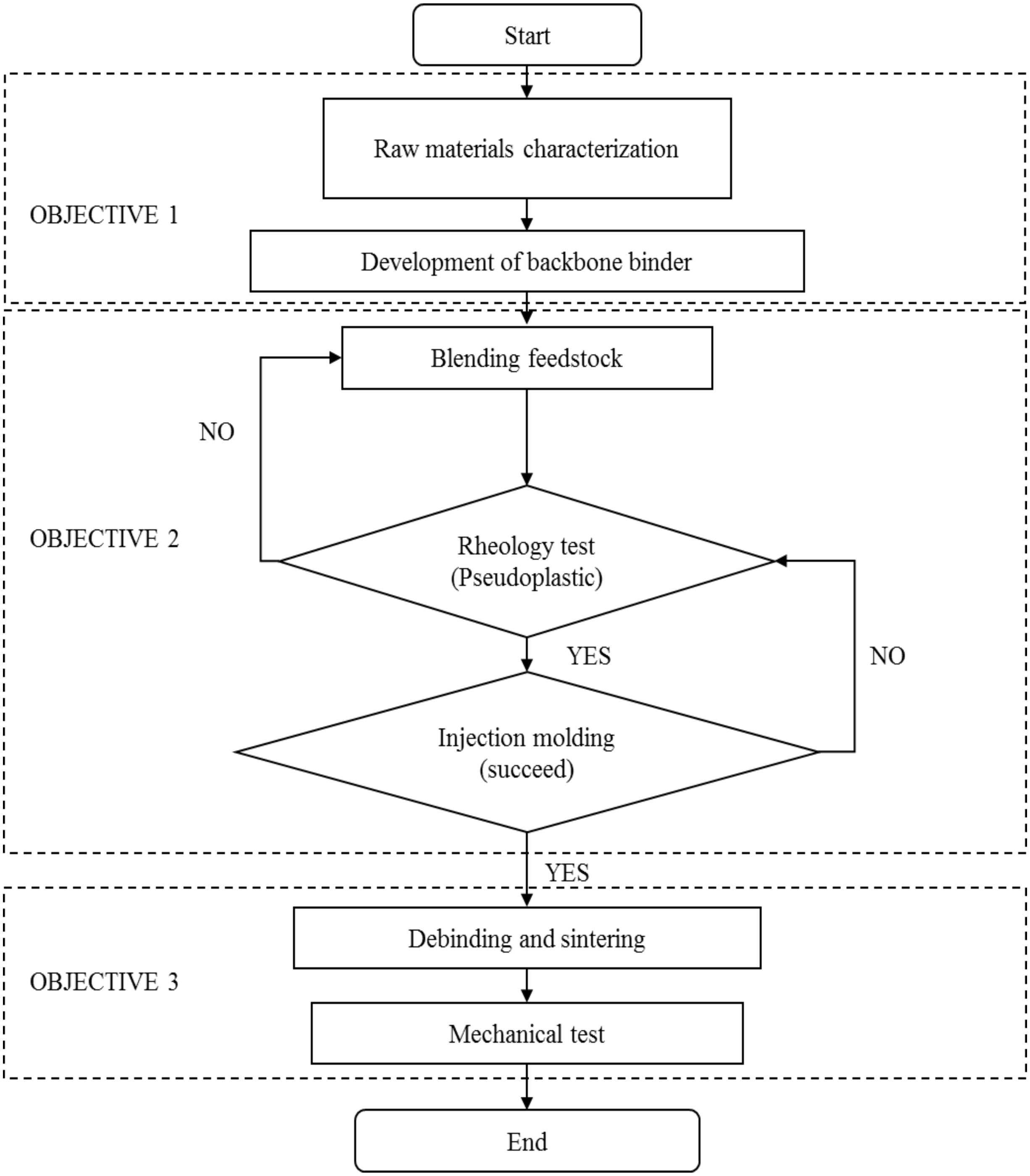


Figure 1.2 Flow chart for the studies

Findings:

Stainless Steel 316L. It is proved that the metal powder used was water atomized in spherical shape which will improve the powder loading and flowability of the feedstock.

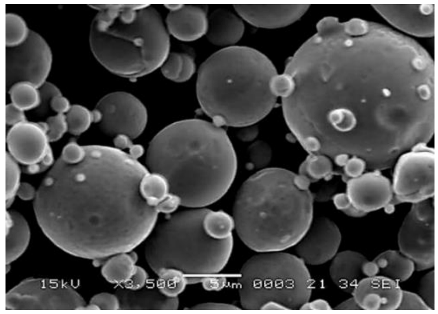


Figure 1.3 Morphology of water atomized SS316L

The SS316L size distribution was recorded in Table 1.1 and Figure 1.4. The average size was determined by D_{50} = 19μm which classified the powder into fine particle that contribute to high packing density of sintered part(Arakida & Miura, 1991).

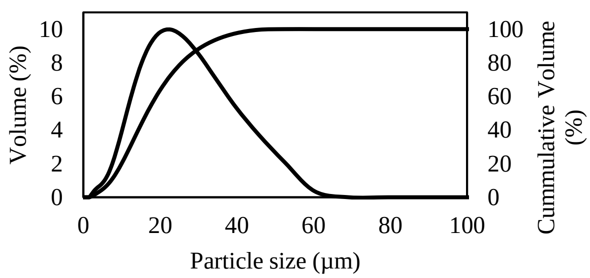


Figure 1.4 SS316L size distribution

Table 1.1 SS316L size

D_{10}	D_{50}	D_{90}
8μm	19μm	36μm

The experimental result of CPVP was illustrated in Figure 1.5. The stable torque indicates the homogeneity between the metal particle and the oleic acid.

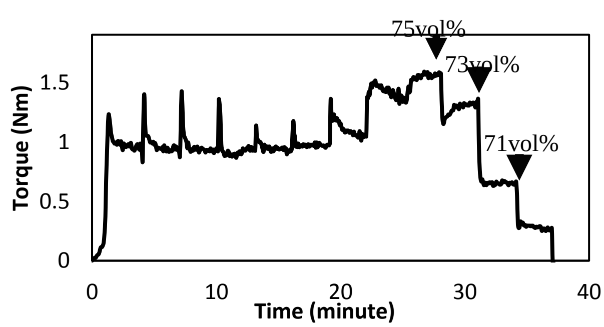


Figure 1.5 Mixing torque graph of SS316L and oleic acid.

Thermal properties of the binder was determined and presented in Figure 1.6 and Figure 1.7.

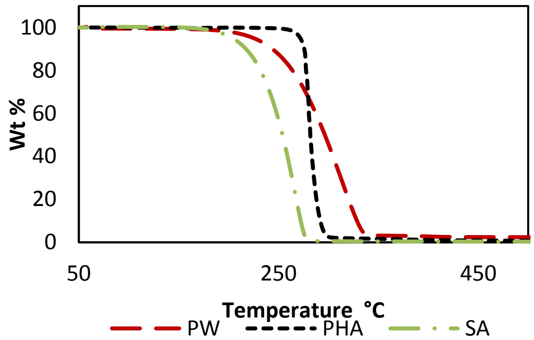


Figure 1.6 TGA curves of binder component

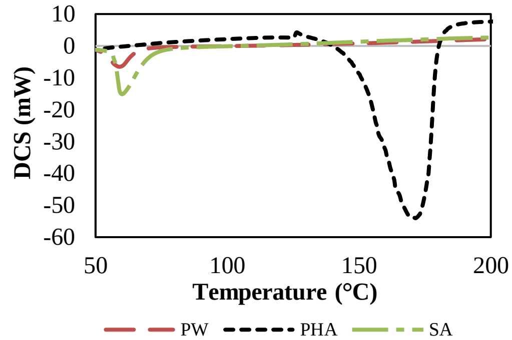


Figure 1.7 DSC curves of binder system component.

Figure 1.8 shows the effect of EPO to the Young's modulus. The value of the properties was summarized in Table 1.2.

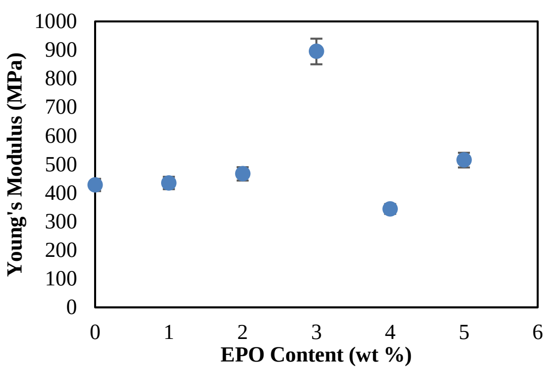
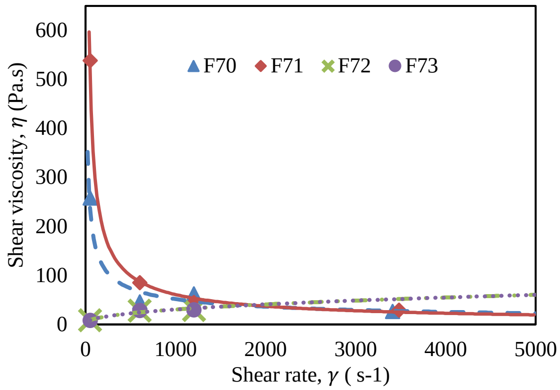


Figure 1.8 Effect of EPO on Young's modulus of PHA

EPO content (wt%)	Young's Modulus (MPa)
0	420
1	428
2	467
3	980
4	344
5	515

Table 1.2 Young's modulus of PHA at different EPO loading

Figure 1.9 shows the relation between shear viscosity and shear rate of the feedstock at the melting temperature, 170°C. Figure 1.10 illustrate the relationship between log η and log γ while the value of n tabulated in Table 1.3.



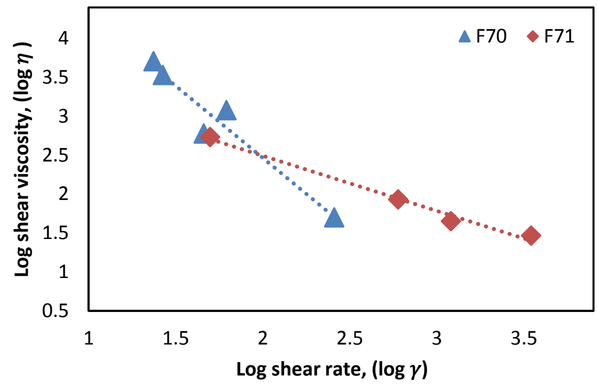
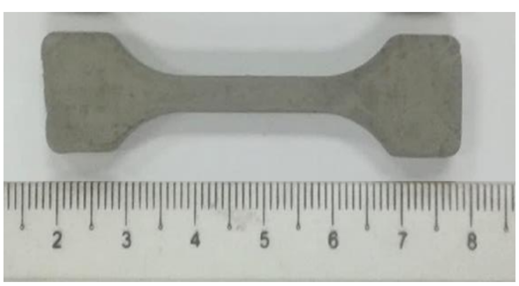


Figure 1.1 Shear viscosity to Shear rate of feedstock Figure 1.2 Log viscosity vs log shear rate for F70 and F71.

Table 4. 1 Flow behavior index, n

Feedstock	Flowability index, n	Correlation coefficient R^2
F70	- 2.421	0.9355
F71	-0.7431	0.9947

Shows in Figure 4.15 the green part have a good surface finish and no defect at 160°C injection temperature, at room temperature mould and pressure 2kN to 5kN. Figure 1.12 shows the SEM micrograph of the green part.



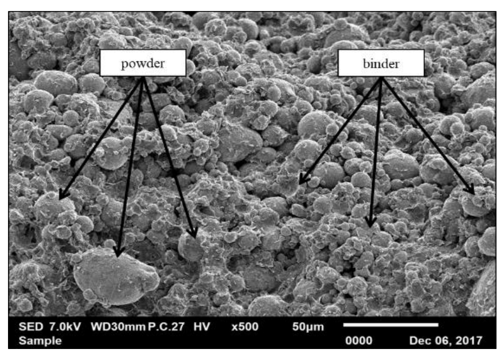


Figure 1.11 SS316L MIM green part. Figure 1.12 SEM micrograph of green part

Figure 1.13 illustrate the kinetic of solvent debinding at three different temperatures 40°C, 50°C and 60°C. Figure 1.14 shows after 6 hours of immersion, the pores channel form at the centre part with no binders left in the space between the particles, the metal particle surface was clean which indicate that the soluble binder was removed.

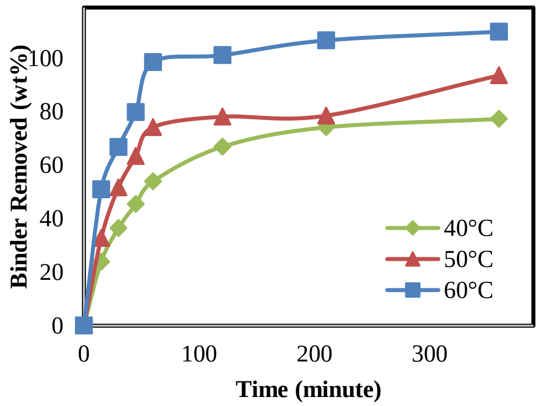


Figure 1.13 Kinetic solvent debinding

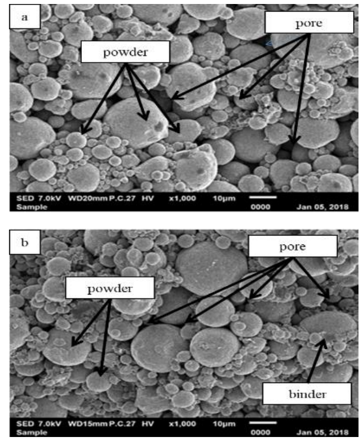


Figure 1.14 SEM micrograph of solvent debound sample after 60 minutes immersion at 60°C a)surface b)center part.

Figure 1.15 shows sample which undergo thermal debound with 8°C/min heating rate. The TGA tested feedstock was observed under SEM and it is found that at 500°C diffusion bond was formed as in Figure 1.16 the bond form provide enough strength to handle the brown part.

Figure 1.15 brown part of 8°C/min heating rate.

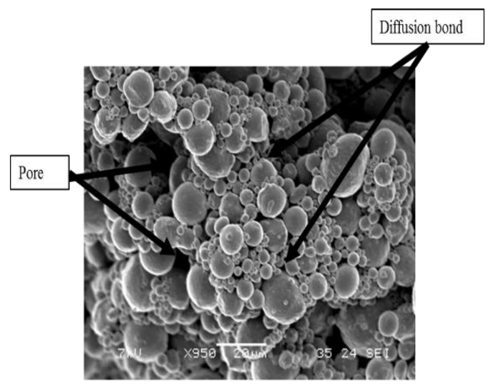


Figure 1.16 Morphology of feedstock heated at 500° C

Figure 1.17 compared the size between green part and sintered part. The part was shrunk due to the diffusion between the metal particles. Figure 1.18 shows the optical micrograph at 100kX magnification of sintered part after been etched using Carpenter etchant.

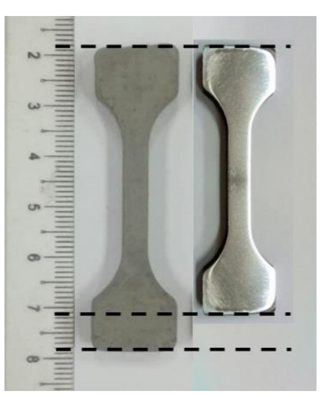


Figure 1.17 (left) green part, (right) sintered part.

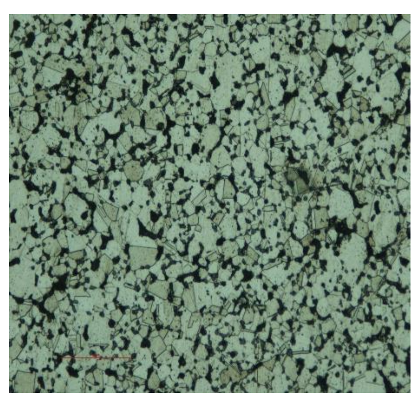


Figure 1.18 SEM micrograph of MIM sintered part

Conclusion:

i- Plasticized PHA with 3wt% EPO content with high Young's Modulus as backbone binder for SS316L metal injection molding (MIM) process.

ii- The 70vol% SS316L powder loading feedstock with 9vol% of PHA exhibit pseudoplastic behavior with shear sensitivity index -2.421.

iii- Solvent debinding at 60°C for 1 hours removed 100% of soluble binder. Thermal debinding at heating rate 8°C/min produce defect free brown part.

iv- The properties of sintered part at sintering temperature 1380°C with heating rate 5°C/min for 3 hours for density, hardness and shrinkage percentage were 91.11%, 67.2 HRB and 6.2% respectively.

Output:

Article

1. Published article: 4
2. Article under review: 1
3. Article under writing:1-2

Conferences (Oral Presentor)

1. ICAMME 2017
2. AMCT 2017
3. PIBEC 2016

Future Plan of the research:

The application of PHA as backbone binder for MIM feedstock enhanced the time consume in debinding step. However, some properties of MIM part need to be considered to meet the MPIF standard some recommendation for future study as follows:.

i- Further study on rheological properties at different temperature was recommended for details behaviour of feedstock with PHA as backbone binder.

ii- Study on optimization for injection parameter to achieves the optimum green part properties and produced better sintered part

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