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The Influence Of Palm Stearin Content On The Rheological Behaviour Of 316L Stainless Steel Mim Compact

Mohd Afian Omar[a] , Istikamah Subuki[b], Norsyakira Abdullah[a] and Mohd Fahies Ismail[a]

[a]Advanced Materials Research Centre (AMREC), SIRIM Berhad, Lot 34, Jalan Hi-Tech 2/3, 09000 Kulim Hi-Tech Park, Kedah, Malaysia

[b]Faculty of Mechanical Engineering, University Technolgy MARA, 40450 Shah Alam, Selangor

Corresponding email: afian@sirim.my

Abstract

Metal Injection Molding (MIM) is a growth technology for producing metal component. Feedstock preparation for MIM is a very crucial step since deficiencies in quality of the feedstock cannot be corrected by subsequent processing adjustments. Hence, it is important that the feedstock is homogeneous and free from powder-binder separation or particle segregation. One of the great concerns in the quality of the feedstock is the formulation of binder. The present mixing study was conducted with 316L stainless steel with a locally binder system; palm stearin and polyethylene. This study investigates the influence of the PS content in the binder formulation via rheological characteristics and various mixing conditions. The results show that good homogeneity of the feedstock prepared for injection moulding of 316L SS powder with the palm stearin as a based binder system.

Keywords : Metal Injection Moulding; palm stearin; rheological behaviour; torque rheometer

1 INTRODUCTION

Metal Injection Moulding (MIM) has emerged as the up and coming of age technology, which combined plastic injection moulding and conventional powder metallurgy (P/M) technology [1-2]. It is a cost effective, near net shape forming process of small intricate and precise parts.

The MIM process basically begins with mixing selected powders and binder in the correct proportions. Very fine powder of typically less than 20 µm are usually preferred with multi-components binder comprising of polymer and other additives. A suitable formulation of binder is mixed with the metal powder (usually referred as feedstock) is granulated and injection moulded into the desired shape. The binder acts as temporary vehicle for homogeneously packing a powder into desired shape and holding the particles in that shape until the beginning of sintering; that will be removed by solvent extraction and thermal debinding method. Finally the part incline to sintered at an elevated temperature to produce a high final density.

Feedstock preparation for MIM is a crucial step since deficiencies in quality of the feedstock cannot be corrected by subsequent processing adjustments. Hence, it is important that the feedstock is homogeneous and free of powder-binder separation or particle segregation. Furthermore, the selection of binder and the formulation is important characteristics since it promotes the fluidity and rigidity of the feedstock especially during mixing, injection moulding and debinding [1-3].

Many binder systems have been developed in MIM with the aims of reducing cost and shorten the overall debinding time and at the same time maintaining shape integrity during the subsequent processing [3,4]. A new developed binder system consists of palm stearin shows a better rheological properties [5,6]. However the composition of palm stearin was achieving only 40wt.%. The advantages of this bio-polymer binder; palm stearin includes local natural resources and environmental friendly. Due to this consideration, this study investigate the influence of palm stearin content with the aim of using palm stearin as a based binder system.

2 EXPERIMENTAL PROCEDURES

The 316L gas stomised stainless steel powder were used in this study having a mean particle size of 10 µm. The mean particle size distribution was determined using a Coulter LS 130 Laser particle Size Analyser. The particles of the powder were spherical in shape as shown clearly in Figure 1. Five feedstock with the binder system consisted of a locally available binder known as palm stearin and polyethylene was prepared varied between 50 and 80 wt.% as shown in Table 1. The increasing in palm stearin was primarily intended to reduce the viscosity of the binder so that the injection temperature might be lowered. The volume fraction of the powder in these mixtures was kept constant at 65 vol%.

Mixing experiments were conducted in a Brabender Plastogram at 160oC and speed of 50 rpm for 2 hours. When the required mixing temperature was reached, the binder with the composition shown in Table 2 was loaded into the bowl little by little with the powder. The torque value is a measure of the resistance on the rotor blades. By observing the mixing torque values, the homogeneity of the feedstock prepared can be predicted: the lower the value, the better is the mixing [7]. Uniform mixing was assumed to have occurred when the torque reached a steady state value.

After each mixing experiment, the feedstock viscosity was measured using a Shimadzu CFT-500D Capillary Rheometer. During the capillary rheometer test, the feedstock was forcibly extruded through a small cylindrical orifice with a 1.0 mm diameter and 10 mm length (L/D=10). The palletized feedstocks were placed in the rheometer barrel and allowed to preheat for 120s under 1, 2, 3, 4 and 5 MPa test load before initiating testing.

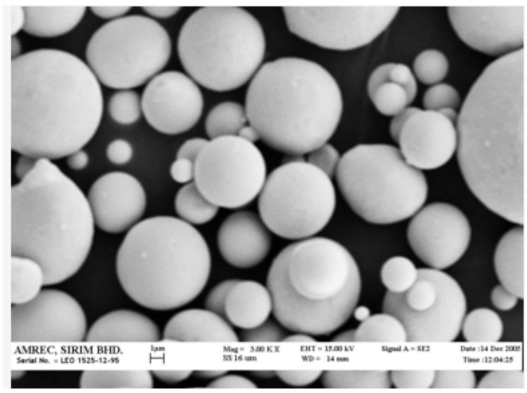

Figure 1 : Scanning electron micrograph of 316L gas atomised stainless steel powder

Table 1 : The binder composition

Binder System	Palm Stearin (wt.%)	Polyethylene (wt.%)
A	50	50
B	60	40
C	70	30
D	80	20

3 RESULTS AND DISCUSSION

1 Mixing at varying palm stearin composition

Variation in the mixing torque and the evaluation of the homogeneity of the feedstock can be determined using a torque rheometer. The mixing torque, which is proportional to the work required to mix the powder and binder, is an indicator of the viscosity of the mixture. The uniformity of the mixture is estimated by the variation in the mixing torque over a period of time (1,2).

The mixing process also depends on the formulation of binder. A curve representing the variation of the applied torque with the feedstock of volumetric 65vol% with different compositions of palm stearin increase from 50% to 80% is defined in this study. Concerning the mixing torque as a function of mixing time (Fig. 2), four different composition of palm stearin of the feedstock can be evaluated. The figure illustrated the maintenance or increment of the mixing which is achieved the maximum torque level followed by the fast decrease of the mixing torque in the first minutes of the test (5-15 min) and reaching the steady state with a constant value after approximately 30 minutes.

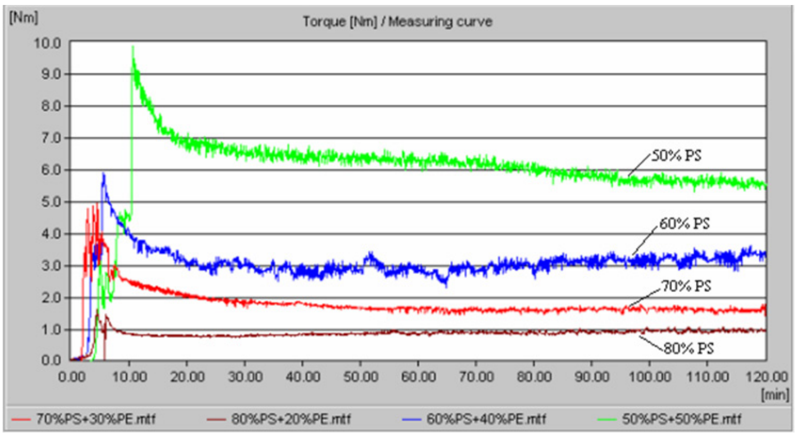

Figure 2 : Mixing torque as a function of mixing time at 50 rpm and 160°C

From the figure 2, varying the composition of palm stearin showed variation in torque level—indicating differences in viscosity of the mixtures. Typically it was observed that the lower composition of palm stearin resulted in higher maximum torque level. Furthermore, it was observed that difference of 4 Nm in constant torque level between the 50% PS and 60% PS, is much higher compared to the difference in constant torque level lower than 1 Nm between 60% PS and 70% PS and so on. This could be due to the fact that friction is created with the lower composition of palm stearin, therefore the resistance on the rotor blades was higher. The torque value was decreased as the increasing composition of palm stearin.

The maximum concentration of palm stearin in the mixture may be related to the transition between the above two types of rheological behaviours. In fact, there is a significant difference in the shape of the curve. Therefore, a simple analysis of the mixing torque as a function of mixing time curves may allow an evaluation of different composition of binder.

By observing the mixing torque values, the homogeneity of a feedstock can be predicted: the lower the value, the better is the mixing (7). Uniform mixing was assumed to have occurred when the torque reached a steady state value. Thus, from the figure 2, the analysis of the results permits to conclude that the most appropriate concentration of palm stearin in binder system is 70%. For the feedstock made with 80% composition of binder system indicates the lowest value of torque which was believed due to powder binder separation during mixing and it also shows an instability phenomena that not sufficient to provide a good flowability of the particles.

However, the method of torque evaluation study suggested is based, essentially, on the higher or smaller difficulty of mixing the inorganic powder with the binder before reaching a steady state. It is after reaching a steady state that the rheological characteristics of the feedstock to be processed should be compared. Thus, a curve representing the variation of the applied torque with the different composition of palm stearin in binder formulation after the establishment of the steady stateis defined in this study. The relationship between the mixing torque applied to the different composition of the palm stearin in the binder formulation is shown in figure 3.

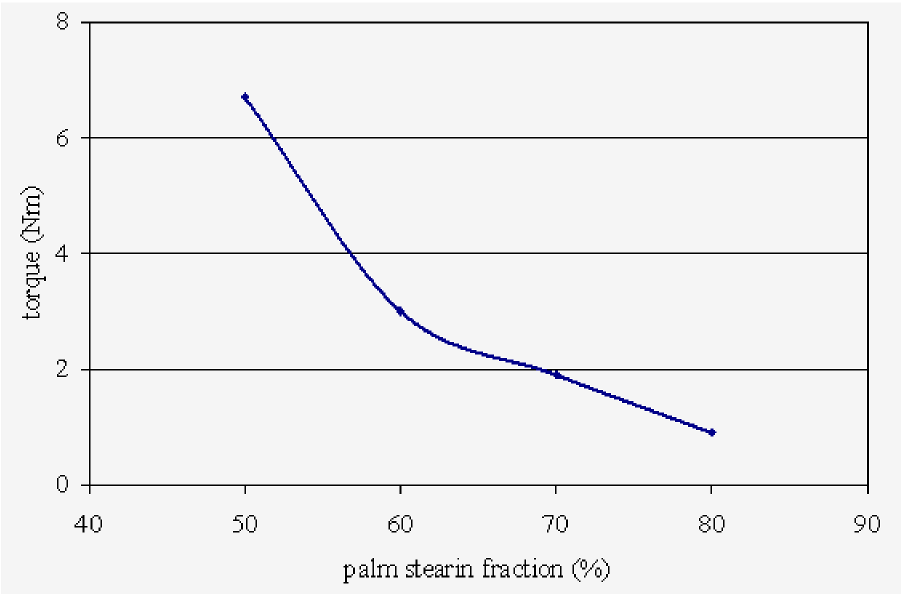

Figure 3 : Mixing torque as a function of palm stearin concentration

According to the figure 3, concerning with the mixing torque variation with the different composition of palm stearin in the binder formulation, it can be concluded that as the concentration of palms stearin increased, the effect of decreasing torque level is more significant. This may be due to the higher concentration of lower molecular weight of binder which expands at a higher temperature, leading to lower viscosity and thus, reduces the torque value. This behaviour explicitly shows, how important the development of optimized feedstock composition is. Concurrently one must be aware to lower the stability in the green state only to an extent, where the part can be demolded and handled without deformation.

2 Rheological characterisation

Initially, for successful moulding, the powder-binder mixtures should have pseudoplastic flow characteristics, since increasing shear rates produce lower viscosities that will assist mold filling. Stability characterisation of the feedstocks were done at 130°C-160°C. These selected temperatures were slightly higher than the melting point of PE (Tm-128.24°C). Higher process temperature was not suitable for PS feedstock since some of the PS component could be evaporated.

2.1 Viscosity

Table 2 shows the viscosity of the feedstock composed of different composition of palm stearin for different pressure applied at 150^{o}C. This temperature was selected according to the stability of the feed stock prepared. Furthermore, previous study conducted by Iriany et al (2002) claimed that this temperature usefulness is for generating a shear rate about 100s^{-1}.

Table 2 : The viscosity data for different composition of PS feedstock (Pa.s)

Feedstock	Applied pressure (MPa) at 150oC				
	1	2	3	4	5
A (50%PS)	57.81	37.96	54.59	29.86	40.66
B (60%PS)	50.8	30.8	31.26	27.78	26.62
C (70%PS)	69.14	56.14	33.99	31.26	37.78
D (80%PS)	32.2	23.53	21.73	27.60	34.64

The results show that by increasing the composition of PS the viscosity was reduced for the all pressure applied. This is more noticeable, since PS has a lower molecular weight than polyethylene. Similar results have been reported by Tseng W.J et al (1999) who found that the powder-binder suspensions decreased their viscosity proportionally to the stearic acid fraction. They claimed that this viscosity is correlated with the enhancement of the microstructural uniformity of particle packing.

As stated before, the viscosity reduced with PS content in the binder composition. In other words, PS had functioned as a modifier to reduce the viscosity and yield stress of the mixture. However, the PS had failed in reducing the viscosity of feedstock C which was increased nearly to the viscosity of feedstock A. Iriany et al (2002) claimed that this phenomena is instable due to excessive concentration of PS in the binder formulation. An excessive concentration of PS in the feedstock system will ease the PS, which do not adsorb onto the powder surface and PE, separating from the bulk material. It should be clear in mind that the theoretical range of viscosity of the feedstock is within the range of viscosity of the feedstock is within the range of 10 Pa.s and 1000 Pa.s at all temperature tested [1-3].

In this study, the measurements were automated. The rheometer would stop if the entire sample had flowed down the capillary completely, but this did not happen to feedstock E which composed 80% wt. PS respectively. It was found that some sample remained in the barrel of the rheometer after the test had been completed. The measurement time of these samples was short; it took about half of the others. This indicated that the binder had flowed out of the mixture leaving powder rich matrix in the barrel, which could not flow anymore since the applied shear stress was not high enough to force the remaining sample to flow. This results was contrast with Iriany et al (2001) who claimed that this phenomena was happened during the PS content increased to 60 wt.%. They conclude that the concentration of PS in the binder formula must not be greater than 45 wt.% for preventing binder separation. Even at temperatures above its melting point, the feedstock will not flow before a critical minimum shear stress (yield stress) is attained.

2.2. Effect of shear rate

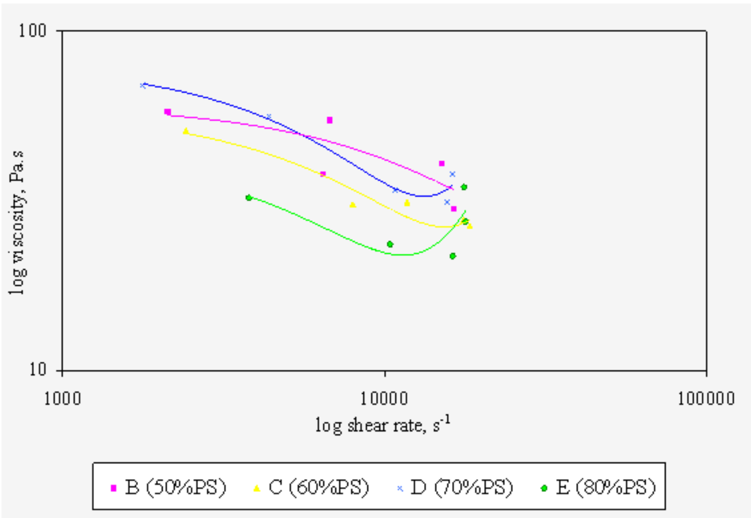

Figure 4 : Viscosities of stainless steel powder mixture with various binder formulation at 150°C

Figure 4 shows the viscosity versus shear rate for different composition of palm stearin in binder formulation. The results show that the viscosity of feedstock decreased with shear rate, indicating pseudoplastic behaviour; same as other MIM feedstock. The pseudoplastic flow can be expressed by the equation 1;

$$\eta = K\gamma^{n-1} \quad (1)$$

the value of n indicates the degree of shear sensitivity and pseudoplasticity. The lower value of *n*, the quicker the viscosity of the feedstock changes with shear rate. In turn, the higher value of *n* indicates the better the rheological stability of feedstock because of the viscosity decreased slowly with increasing shear rate. This higher shear sensitivity is important to produce a complex and delicate product, as it will provide better stability of the viscosity during mold filling. Table 3 lists the value of shear sensitivity of the feedstock prepared. It clearly shows that all data give higher value of *n* but lower than 1 which are suitable for metal injection moulding practice.

Table 3 : Shear sensitivity value (*n*)

Temperature/ ^{o}C	A	B	C	D
150	0.7419	0.6974	0.6572	0.9175

The higher *n* value of feedstock D, 0.9175 was a result of higher PS contents in its formula than the other feedstock prepared and it considered nearly to newtonian and dilatant flow which is not applicable to MIM feedstock. According to Ong et al. (1995), Iriany claimed that palm oil has the properties of Newtonian fluid (Iriany, 2002). However, according to German (1990), dilatant behaviour can occur at the higher shear rate. As we can see for the feedstock E it has a minimum point in the newtonian range and tend to dilatant flow.

German (1997) presented that the shear stress applied to the mixture τ must be corrected for the mixture yield strength τy as equation 2.

$$\tau = \tau y + \eta\gamma \quad (2)$$

where η is the mixture viscosity and γ is the shear rate. According to the equation 2, graph of shear stress versus shear rate have been plotted as shown in figure 5. The figure shows the shear stresses of the stainless steel feedstock with different palm stearin composition at 150^{o}C for different shear rates. The results show a linear relationship for all the mixture, where shear stress increased with increasing shear rate. The yield stress τy can be evaluated from the intercept value of the τ - γ for each feedstock as shown in figure 6 and it become lower with increasing composition of palm stearin in binder formulation.

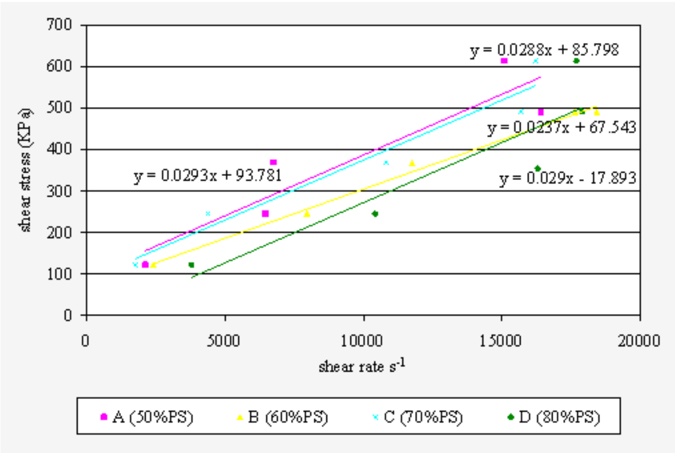

Figure 5 : Shear stress versus shear rate graph for various feedstocks at 150°C

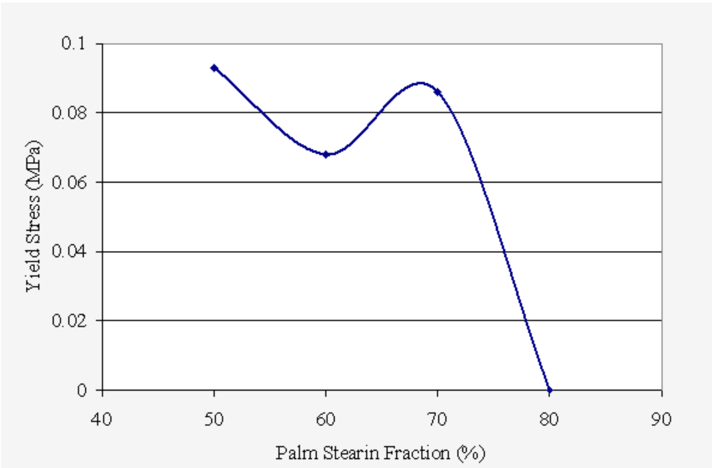

Figure 6 : The yield stress value (τy); MPa

The results indicated that the feedstock containing lower palm stearin in the binder formulation shows a higher yield stress compared to which a higher palm stearin content in the feedstock prepared. Since the yield stress can be considered as the minimum force required to make a relative movement between particles assemblies, the greater value of the yield stress could be considered as a result of the lower fluidity of the lower palm stearin content in the binder formulation. In addition, two interesting condition can be noted. First, the yield stress for feedstock C was higher compared to the feedstock B, which suppose reduce by increasing palm stearin. This make C feedstock have a lower fluidity and it was easy as they flowed into mold cavity during injection moulding. The second interesting to note that the yield stresses for feedstock D which consist higher palm stearin content, 80 wt.% was negative within the limitation of experimental error. The yield stress of feedstock D is very low at temperature 150°C. It was observed during rheological measurement that the D feedstock showed a very low viscosity at 150°C, even they could flow without force induced. The low τy values confirm this and could be drawback as they flowed into the mold cavity during injection moulding. This is more noticeable, since the palm stearin has a lower molecular weight and lower viscosity then the other component.

2.3. Effect of temperature

The behaviour of MIM feedstock is thermally activated, where the viscosity, is expressed by an Arrhenius equation as below:

$$\eta = \eta_o \exp\left(\frac{E}{RT}\right) \quad (3)$$

where E is the flow activation energy, R the gas constant and T the temperature in Kelvin. The value of E should be as small as possible to avoid sharp viscosity changes that reduce the flow ability of the feedstock and cause stress concentrations, cracking and distortion in the molded parts [10]. Figure 7 shows the logarithm scale of viscosity against temperature at 5MPa pressure applied. The data of the flow activation energy in Table 4 indicate that, increasing palm stearin will decrease the flow activation energy. The activation energy of feedstock D consist of 70% PS is the lowest which implies that is the best from the standpoint of temperature sensitivity. The value of D indicates the influence of temperature on the viscosity of MIM feedstock. If the value of E is low, the viscosity is not too sensitive to temperature differences, thus any small fluctuation of temperature during injection moulding practice might not results on sudden viscosity change that can cause molding defect. However, all formulation still considerably low if compared to many rheological studies by other researchers (8-12), so the viscosity of feedstock composed of palm stearin is not a problem besides it can be based binder system.

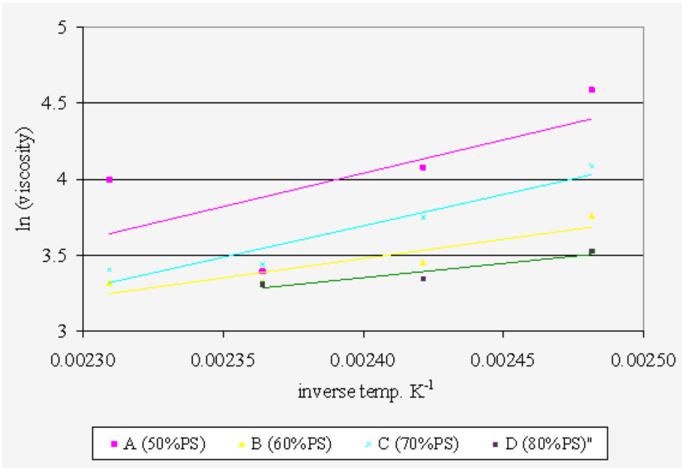

Figure 7 : The relation between viscosity of feedstock and temperature

Table 4 : Flow activation energy at different composition of PS

Feedstock	A	B	C	D	E
Activation energy, E	25.06	13.48	20.92	12.9	24.62

4 CONCLUSIONS

The study has shown clearly that PS component plays several important roles in the composite binder system. The increasing of PS content in the binder system decreased the torque value during mixing and reduced the viscosity for all pressure applied. All the feedstocks pose a pseudoplastic behaviour with the viscosities decreased with shear rate that favourable during injection in MIM application. The rheological data shows that PS can be used as a based binder system with the composition of 50 to 70 wt%.

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