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Study on Stability of a Novel Binder System Based on Palm Stearin in Metal Injection Moulding Application

Muhammad Hussain Ismail[1,4], Norhamidi Muhamad[2], Aidah Jumahat[1],
Istikamah Subuki[1] & Mohd Afian Omar[3]

[1]Faculty of Mechanical Engineering
Universiti Teknologi MARA (UiTM), Malaysia
[2]Faculty of Engineering
Universiti Kebangsaan Malaysia
[3]Advanced Materials Research Centre (AMREC)
SIRIM Kedah
[4]Email: hussain305@salam.uitm.edu.my

ABSTRACT

Metal Injection Molding (MIM) is a wellestablished technology for manufacturing a variety of complex and small precision parts. In this paper, fundamental rheological characteristics of MIM feedstock using palm stearin are theoretically analyzed and presented. The feedstock consisted of gas atomized 316L stainless steel powder at three different particle size distributions and the binder system of palm stearin (PS) and polyethylene (PE). The powder loading used was 60vol % for all samples (monosize 16 µm, monosize 45 µm, and bimodal 16 µm + 45 µm) and the binder system of 40vol % (PS/PE = 40/60). The viscosity of MIM feedstock at different temperatures and shear rates was measured and evaluated. Results showed that, the feedstock containing palm stearin exhibited suitable rheological properties by increasing the fluidity of feedstock in MIM process. The rheological results also showed a pseudoplastic flow characteristics, which poses higher value of shear sensitivity (n) and lower value of flow activation energy (E), that are both favourable for injection molding process. The green parts were successfully injected and

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exhibited adequate strength for handling by optimizing the injection pressure and temperature.

Introduction

Metal injection molding (MIM) is a new net shaping technology of powder metallurgy which is developed on the basis of conventional plastic injection molding [1-10]. This new technology is increasingly being accepted in manufacturing sectors as a suitable and cost effective method for high performance parts. This process starts with the mixing of metal powder with a binder to form a feedstock, which the binder is mainly consisted of polymeric materials. The feedstock after mixing is granulated to form pellets. It is then followed by injection molding to form green parts. After molding, the binder is removed through debinding and followed by sintering to produce parts of full or near full density.

Binder is used as a temporary vehicle for homogeneity packing a powder into desired shape and sustaining the particles in that shape until presintering. Today, there are many binder systems in use, such as wax, thermoplastic, thermo setting based binder, solid polymer solution (SPS) and water based systems [1-6]. Those binder systems have disadvantages such as high viscosity, low decomposition and are not available locally. Generally, the developing of a new binder is to overcome some problems associated with the existing systems and to fulfill the requirement of the MIM process. Iriany [6] found that the possibility of using palm oil as one of the binder components of a binder in MIM application. Detail rheological properties has been reported and discussed in the previous work [1,6]. Palm stearin consists mainly of glycerides made up of a range of fatty acid. Many binders in use consist of fatty acid, mainly stearic acid but only in small quantities [1]. Besides the suitable constituent, palm stearin also has many suitable properties, such as low cost, low viscosity and high decomposition temperature. It easily dissolves into organic solvent, a chemically passive and easily available in Malaysia.

The homogeneity level of the feedstock prepared can be measured qualitatively by viscosity measurement [2-6]. It is required for allowing the particles to flow into the die cavity without any difficulties. Mold filling with the MIM feedstock is dependent on viscous flow of the mixture into the die cavity. This requires specific rheological characteristics [7]. This paper discusses the rheological properties and reports on the viscosity of feedstock by using palm stearin as a binder. The viscosity of feedstock was measured and evaluated at different temperatures. The effect of different particles size distribution on the rheological results was also investigated. Then, the value of shear sensitivity, *n* and the flow activation energy, *E* were obtained in order to determine the relation of MIM feedstock to shear rate and temperature. Injection moulding was carried out using the feedstock with the most appropriate rheological properties. Moulding parameters such as injection temperature and pressure were adjusted and optimized until the desired green parts were produced.

Experimental Procedure

The powder used in this study was a 316L grade stainless steel and chemical composition of the powder is tabulated in Table 1. Three different powder samples based on different particle sizes distribution is shown in Table 2. The mixture of 50 %, 16 µm and 50 %, 45 µm was prepared using a rotary dry mixer at a speed of 80 rpm for 100 minutes. The mean particle size distribution was determined using a Coulter LS 130 Laser Particle Size Analyzer. The binders system comprised of palm stearin (PS) and polyethylene (PE) with the fraction of 40/60 by weight percentage. The powder loading used was 60 vol % and the remaining 40 vol % contained binder system of PS/PE.

Mixing was carried out using a Z-blade mixer for preparing the MIM feedstock at three different powder distributions as stated. During mixing, PE, powder and PS were added gradually into the mixer at 160 °C for

Table 1: The Chemical Composition of the 316L Stainless Steel Powder

Elements	Weight (%)
C	0.026
Si	0.58
Mn	1.43
P	0.030
S	0.012
Cr	16.4
Ni	10.4
Mo	2.08
Fe	balance

Table 2: The Particle Size of Stainless Steel

Sample	Powder	Mean particle size (µm)
A	100% 16 µm	11.10
B	100% 45 µm	29.42
C	50% 16 µm + 50% 4 µm	19.73

the time duration of 2 hours. Then, the heater was switched off and the mixer blade was kept rotating for half an hour until the pelletized feedstock was produced.

A capillary rheometer model (Shimadzu CFT500D) was used to investigate the rheological properties of the feedstock. The viscosity of the fine feedstock was measured at varying temperatures and shear stress. The diameters of the rheometer capillaries were 1 mm and the lengths of the capillaries were 10 mm. Samples were placed in the rheometer barrel and allowed to preheat for 120 s under 3, 4, 5 and 6 MPa testload before initiating testing.

The tensile specimen was prepared using a vertical injection moulding model MCP HEKGMBH. During moulding, the temperature and injection pressure were adjusted until the optimum conditions were obtained. The injection specimens were then judged and the dimensions were measured for dimensional analysis.

Results and Discussion

Rheological Result

In general, MIM feedstocks are considered to be shear thinning or pseudoplastic fluids. The main characteristic of a pseudoplastic fluid is that viscosity decreases with the increase of shear rate. Empirical studies have shown that the shear rate during molding usually ranges between 100 and 10,000 s^{-1} and maximum viscosity for molding is 1000 Pa.s at the molding temperature [2-4]. Figure 2 shows the viscosity of the feedstocks as a function of shear rate at three different particle sizes and temperatures. It clearly shows that, with the increasing of shear rate, the viscosity decreases which exhibit the pseudolastic flow, which is common for MIM feedstocks. This could be due to particle orientation and ordering with flow as breakage of particle agglomerates with released of fluid binder [7]. From the figure, it clearly shows that all feedstocks exhibit good rheological behaviour, except for feedstocks B and C especially at the lower temperature 125 °C and lower shear rates. It shows that, both feedstocks, B and C are not feasible for injection molding, particularly at a lower temperature such as 125 °C. They exhibited higher viscosity, which are almost greater than 1000 Pa.s particularly at lower shear rate. Additionally, the variation of viscosity versus shear rate is almost linear, which is an indicator of feedstock stability. A non-linear flow behavior of the feedstocks, i.e. a sudden drop and increase as the shear rate increases, is also noticeable.

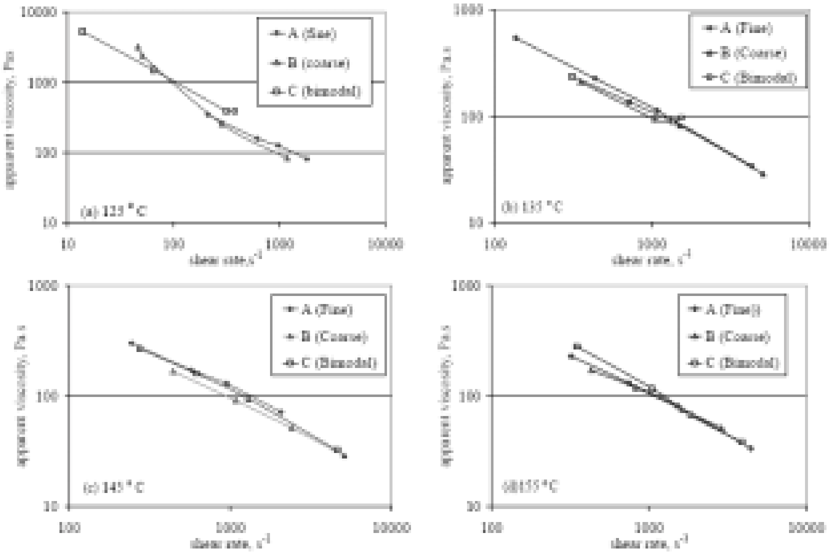


Figure 1: Viscosity of the Feedstock as a Function of Shear Rate at Diffrent Temperature and Particle Size Distribution

The viscosity data shown in Figure 1 indicates the flow ability of the MIM feedstock. The lower the value of viscosity, the easier it is for a MIM feedstock to flow. Nevertheless, too low the viscosity tends to slumping phenomenon, whereby the feedstock is too soft to handle. The viscosity of the feedstock A can be considered as the most appropriate for injection, since all data obtained indicates small changes of viscosity at all tested temperature and pressure. It should be clear in mind that the theoretical range of viscosity of the feedstock is within the range of 10

Pa.s and 1000 Pa.s at all temperature tested [2-7]. It is clear that the temperature of 125 °C was not suitable to provide adequate flow ability especially for feedstocks B and C.

A MIM feedstock is generally considered to be pseudoplastic fluid, which indicates a decrease of viscosity with increase on shear rate and temperature. For a pseudoplastic fluid, it can be expressed by the following equation [2-4];

$$\tau = K\gamma^{n} \tag{1}$$

where τ is the shear stress, γ. The shear rate, n the flow behavior index and K is a constant. The most important rheological property for metal powder feedstock is viscosity (η) , which is defined by;

$$\eta = \frac{\tau}{\gamma} \tag{2}$$

The general shear rate-dependence of viscosity could be described by equation 3.3:

$$\eta = K\gamma^{n-1} \tag{3}$$

The value of n indicates the degree of shear sensitivity and pseudoplasticity, which is smaller than 1. The lower value of n, the more quickly the viscosity of the feedstock changes with shear rate. In turn, the higher value of n indicates the better the rheological stability of feedstock [1]. The viscosities of palm stearin (PS) based feedstock decreasing with shear rate as shown clearly in Figure 1. By applying the Equation 3, the values n results are summarized in Table 3. The higher n value of feedstock A was considered to be most appropriate feedstock for injection molding, since it has better rheological stability and greater pseudoplasticity.

Another important characteristic of MIM feedstock is the temperature-dependence of viscosity. If the viscosity is very sensitive to

Table 3: Shear Sensitivity Value (n)

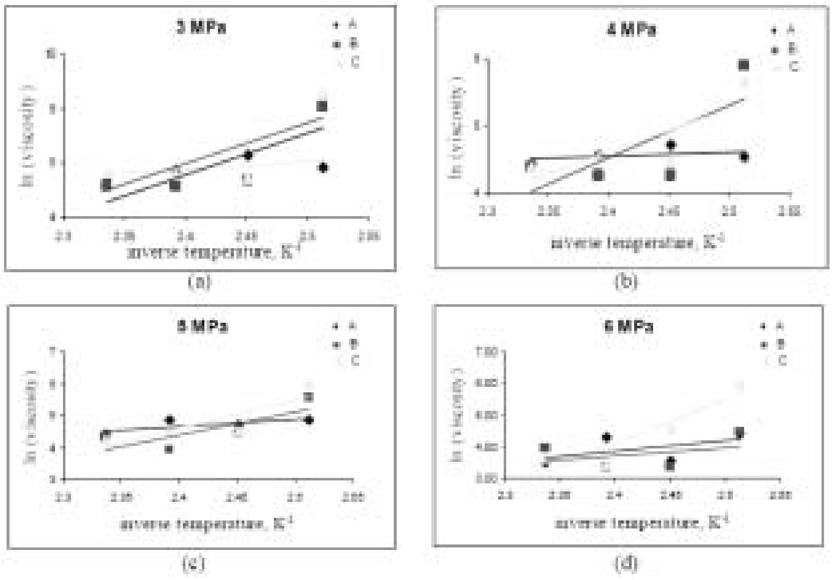
Temperature/°C	A	B	C
125	0.3273	0.1314	0.1891
135	0.7982	0.2637	0.4009
145	0.3318	0.2833	0.2433
155	0.2651	0.3606	0.1590

the temperature variation, any small fluctuation of temperature during moulding results in a sudden viscosity change. This could cause undue stress concentration in the moulded part, resulting in cracking and distortion. In addition, a strong temperature-dependence of viscosity dictates smaller pressure transmission to the cavity, thereby promoting the possibility of the formation of shrinkage-related defects. Therefore, it is important to evaluate the dependence of viscosity to temperature [11]. Normally, as a good approximation, the Arrhenius equation (Eq. 4) can be used as followed;

$$\eta = \eta_0 \exp\left(\frac{E}{RT}\right) \tag{4}$$

where E is the flow activation energy, R the gas constant and T the temperature in Kelvin. The small values of E show a low sensitivity of viscosity to temperature, thereby minimizing stress concentration, cracks and distortion in the moulded parts. According to the Eq. 4, a graph plot as shown in Figure 2; ln (viscosity) versus 1/T at a certain shear rate are obtained.

The graph is fit into straight line. The slope of the graphs indicates the temperature dependence of viscosity, which should be as small as



(a) (b)

(c) (d)

Figure 2: The Relation between Viscosity of Feedstock and Temperature

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 [3].

Based on the slope of graph obtained from Figure 2, the value of *E* is tabulated as shown in Table 4. The flow activation energy for feedstock A is the lowest as compared to feedstocks B and C. This indicates that viscosity of feedstock A is not too sensitive with the changes of temperature. This feedstock can thus be injection moulded in a relatively wide temperature range. Feedstock B and C with higher values of *E* are more sensitive to temperature fluctuation.

Table 4: Flow Activation Energy (E) Data (kJ/mol)

Feedstock	Applied Pressure (MPa)			
	3	4	5	6
A	3.07	1.39	2.00	3.30
B	15.53	15.68	7.12	2.59
C	15.02	13.00	9.01	13.87

The flow activation energy (*E*) data obtained was fairly much lower as compared to many works [2-4, 11]. It is visible that the value of *E* decreases as the mean particle size decrease and that the applied pressure seems to be not influence much on the flow activation energy. For the feedstock C, the value of *E* was not much different at every tested pressure as compared to feedstock B. This was due to the fact that the feedstock C was a bimodal while the feedstock B comprised of 100 % coarse powder. Coarse powder is actually unfavourable in MIM process, since it leads to non-uniform flow during injection [7].

Injection Moulding Evaluation

In injection moulding process, only feedstock A (16 µm) was chosen. This was based on the rheological data obtained, which was believed to be more stable as compared to other two feedstocks (B and C). After several trials and error, the feedstock was successfully injection moulded at the nozzle temperature of 220 °C and injection pressure of 30 MPa. Total cycle time for each injection was between 10 to 20 seconds. Figure 3 shows the tensile shape of the injection moulded parts with a single gate, located at one end of the part. The moulding temperature used

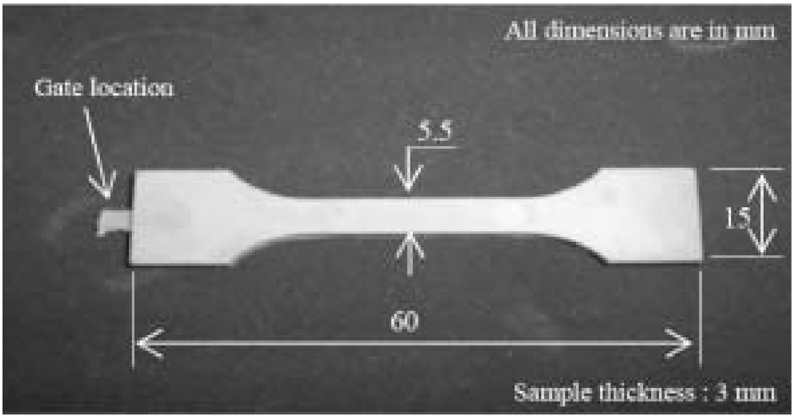
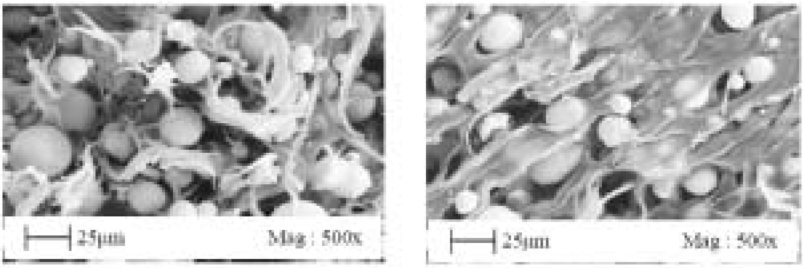


Figure 3: Injection Moulded Part (green part) Tensile Specimen

during the study can be considered as much higher as compared to other reports, which commonly used at the range of 100 °C to 200 °C [4-10]. Nevertheless, during the injection, all injection parts were fairly good and free from normal defects such as short shot, flashes at the parting surface and binder separation. The higher temperature applied was believed to be due to less amount of palm stearin in binder system, which led to high viscosity of the feedstock. In order to make sure the feedstock can be easily flow and moulded, higher temperature was needed to compensate the high viscosity of the feedstock. Figure 4 clearly shows the SEM of the green parts at two different regions; (a) fracture and (b) outer surface. It can be seen that, the binder fills practically all the interstitial spaces



(a) (b)

Figure 4: Scanning Electron Micrograph Shows the Distribution of the PS/PE Binder System on Injected Parts; (a) Fracture and (b) Outer Surface

between the powder particles. It also clearly shows that, in Figure 4(b) outer surface is filled with more binder than those of fracture surface shown in Figure 4(a). Many pores can be seen from Figure 4(a) and the binder flow seems to be non-directional, but in Figure 4(b) it clearly shows that the binder flow is more directional. The presence of pores or so called voids between the particles was due to entrapped air or binder shrinkage during cooling process.

Conclusion

It can be concluded that the viscosity plays a big role in predicting the injected parts by considering the shear sensitivity, *n* and flow activation energy, *E*. From the results obtained, the feedstock based on the palm stearin binder was found to exhibit suitable rheological properties as MIM feedstock. All the feedstock prepared still in a range of pseudoplastic but the fine particle; feedstock A was considered to be the optimum one from the standpoint of flow ability. Bimodal system; feedstock C can improve packing density but it causes higher viscosity that required less binder to attain a viscosity useful for molding. The feedstock comprised of PS/PE was successfully injection moulded at the nozzle temperature 220 °C and injection pressure of 30 MPa with no significant defects observed.

Future Works

In this work it clearly shows that palm stearin has a great potential to be used in MIM process. Its advantages against commercial wax binder system such as it is readily available, inexpensive and environmental friendly has significant impact to the MIM industry. It is expected that the palm stearin can be as used a based binder by increasing its content as much as possible. It is also expected that, this binder can be easily removed during solvent extraction in debinding process. Future works on debinding and sintering has been performed on the injected parts in order to get a clear picture on the feasibility of this binder system in overall process.

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