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**RHEOLOGICAL INVESTIGATION OF MIM FEEDSTOCKS PREPARED WITH DIFFERENT PARTICLE SIZES**

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**ABSTRACT**

*An experimental rheological study has been performed to the gas atomized 316L stainless steel with particle size of 16 and 31 $\mu$ m, mixed with PMMA, PEG and stearic acid as a binder at 63% of volume. The viscosity and shear rate of the feedstocks were measured via capillary rheometer across  $L/D=10$  capillary die at constant temperature of 130°C. Through the analysis, all feedstocks showed significant pseudo plastic behavior as the flow behavior index, ( $n$ ) was less than one. Moreover, the flow behavior index of the coarse powder was larger than the fine powder particles. The activation energy,  $E$  of each feedstock demonstrates the feedstock sensitivity to the temperature and pressure. The  $E$  value was inversely proportional to the shear rate and still higher at coarser powder. The moldability parameter,  $\alpha$  for each feedstock was also significant, and the value was decreased when smaller particle size was used. The results indicated that the feedstock containing larger particle size (31 $\mu$ m) displayed the best rheological behavior with the highest moldability parameter,  $\alpha$ . However, this feedstock showed high sensitivity to temperature fluctuations; thus, it will possibly freeze in the mold cavity at a wide range of temperature, besides maximizing stress concentration, cracks and shape distortion.*

**Keywords:** Metal injection molding, rheology, particle size, feedstocks.

**INTRODUCTION**

Metal injection molding (MIM) is a new net shaping technology of powder metallurgy combined with the basic of conventional plastic injection molding. The technology is capable of producing high-performance and complex shaped metal at low cost [1,2]. Generally, MIM is composed of four main processes, namely a mixing process to prepare a homogenous metal-binder mixture, an injection molding process, a debinding process and finally a sintering process.

In MIM process, mold filling depends on the viscous flow which requires specific rheological characteristic. The viscosity is related with the shear rate and it plays an important role so that it follow pseudoplastic behaviors where the viscosity should decrease with increasing shear rate during injection [1,3]. A high viscosity makes molding difficult. Rheological analysis can be made to quantify the homogeneity of the feedstock using capillary rheometer.

It is known that a feedstock viscosity with low temperature sensitivity and low sensitivity to shear thinning behavior is desired [4]. Thus, parameters such as flow behavior indexes,  $n$ ; flow activation energy,  $E$ : and moldability parameter,  $\alpha$  are also the important parameters in rheological investigations.

Many experimental data about rheological characteristics of MIM feedstocks are available, mainly of seeking suitable feedstock formulations with desirable rheological behaviors. For example, Huang et. al [5] has conducted an investigation on the effects of shear rate, solid volume fraction and temperature on the rheological behavior of Fe/Ni MIM feedstocks, prepared by readily available polymer binder systems. The results indicate

that the binder system which had stable relationships in both the shear-rate dependence and temperature-dependence of the viscosity, could be selected.

Karatas et.al [6] has studied the rheological properties of the ceramic feedstocks, using polyethylene (PE) and three waxes (carnauba, bees wax and paraffin) with steatite powder. The experiment concluded that the formulation proposed met the specific requirement and suitable to be injected molded. The flow behavior index (n) parameters were determined to be less than 1, showing a pseudoplastic behavior.

While in order to evaluate the influence of TiC on the rheological behavior and stability of 316L stainless steel MIM feedstock, Khakbiz et. al [4] has performed an experimental rheological study via capillary rheometer method. They concluded that the rheological behavior of the feedstocks highly depends on the blend composition. The addition of TiC particles to the stainless steel powder increases the viscosity of the feedstock at relatively low shear rates, i.e.  $<500\text{s}^{-1}$ . Furthermore, the feedstock instability increases, particularly at higher solid loading. Nevertheless, with increasing shear rate and temperature, the viscosity decreases and the instability of the feedstock improve.

Krauss et. al [7] has conducted an experiment using capillary rheometer to analyze the rheological behavior of alumina molding feedstocks containing polyethylene glycol (PEG), polyvinylbutyral (PVB) and stearic acid (SA) having different powder loadings. Some of the feedstocks showed a pseudoplastic behavior of flow behavior index,  $n < 1$  and their viscosity also displayed a strong dependence on the shear rate. The results indicate that the feedstock containing a lower powder loading displayed the best rheological behavior.

This paper evaluates the effect of different particle sizes on the rheological behavior and moldability of 316L stainless steel feedstock with binder system comprises of PEG, PMMA and stearic acid. This involves the measurement of the influence of temperature and shear rate on the viscosity of MIM feedstocks, and also the moldability parameter by using a capillary rheometer technique. The capillary test, which gives the fundamental rheological data of a viscous fluid, is currently realized as the best approach to predict the flow behavior during injection molding [4]. Instead of getting information about viscosity, this instrument can also be used to reveal stability and homogeneity of feedstocks and the extent of powder-binder separation.

## EXPERIMENTAL PROCEDURES

The metal powder used in the present study was gas atomized 316L stainless steel powder (ANVAL, Sweden). The characteristic of this powder are reported in Table 1, while Table 2 shows the chemical composition of the metal powder.

Table 1: Characteristics of metal powder used in this study

Characteristics	
Identification	SUS 316L
Powder source	Anval 316 Stainless Steel, Sweden
Tap density, $\text{g/cm}^3$	4.09
Apparent density, $\text{g/cm}^3$	2.82
Pynometer density, $\text{g/cm}^3$	7.96

Table 2: Chemical composition of metal powder used

C	Si	Mn	P	S	Cr	Ni	Mo	Fe
0.027	0.44	1.39	0.026	0.01	16.7	10.4	2.09	balanced

The binder system composed of polyethylene glycol, (PEG) as the major component (73%), polymethyl methacrylate, (PMMA) as backbone polymer (25%), and stearic acid (SA) as surfactant (2%). The surfactant acts as a lubricant that enhances the dispersion of powder in the binder during mixing, as well as enhanced powder loading and green strength [9] without sacrificing the flow properties of the mixtures.

In this study, 63% in volume of powder loading with particle size of  $16\mu - 31\mu$  were used. The remaining volume ratio for binder system was PEG, PMMA and SA at 73, 25 and 2.

Table 3: Formulation of feedstocks

Feedstocks Solids loading, vol (%)	Particle size ( $\mu$ )	Feedstock abbreviation
63	16	F63_16
63	31	F63_31

Table 3 illustrates the composition of the feedstocks used in this study. Two feedstocks were prepared using a sigma type blade mixer with a rotation frequency of 25 rpm. Figure 1 shows the mixing sequence. After mixing, the dough will be removed from the mixer and left at room temperature. When the dough's temperature drops down to about 60°C, subsequently it will be fed into a crusher to produce homogenized granules.

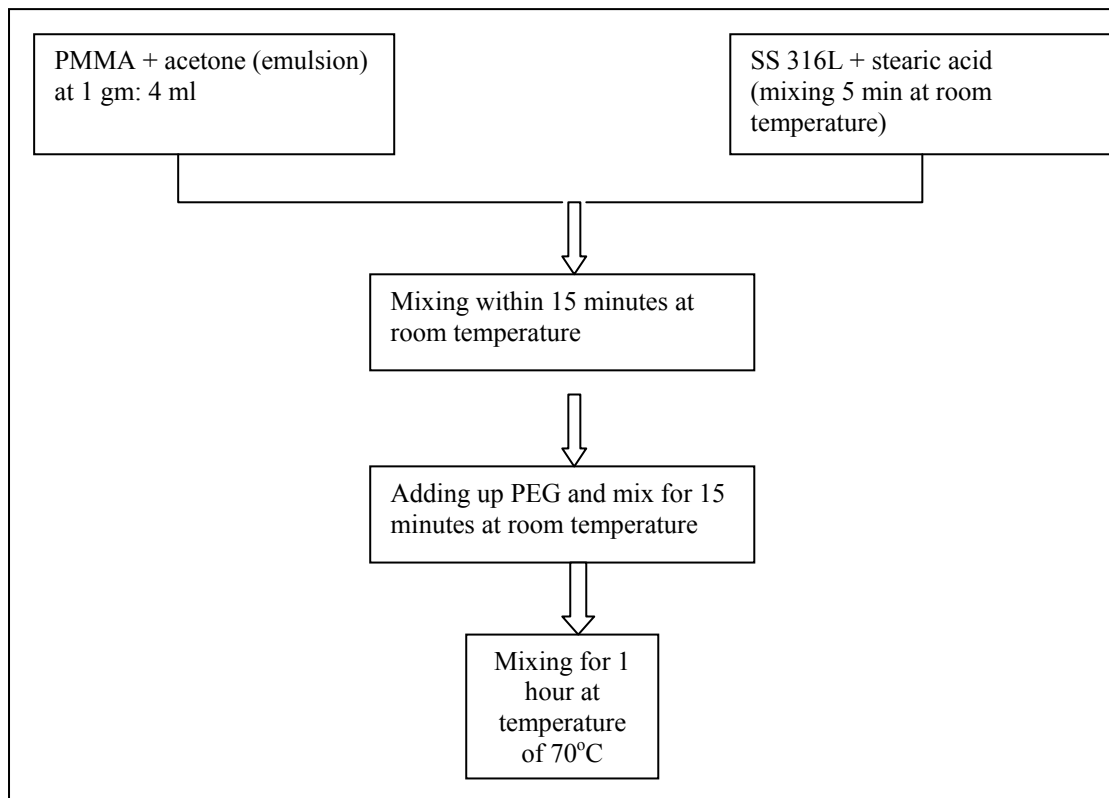


Figure 1: Flow chart of mixing process

The rheology behavior of the formulations was tested using a CFT-500D Shimadzu capillary rheometer, which measures the viscosity resistance when melted materials pass through the die orifice. In order to monitor the flow, a die ( $L/D=10$ ) was attached to the bottom of the extruder barrel. The test was conducted at constant capillary temperature of 130°C and the load applied to the tester was from 30-60 kgf.

The barrel was filled with the feedstock and then pressed lightly with the piston. Then, it was left in the barrel for about 10-15 minutes to attain thermal equilibrium. The pressure drop across the die was recorded in order to calculate the shear stress at the die wall. Meanwhile, the flow rate through the capillary was calculated using the relation provided by Japanese Industrial Standard, JIS K7210 [10]:

$$Q = \frac{0.4}{t} (cm^3 / s) \quad (1)$$

where  $t$  is the time for the piston to travel from 3 mm point to 7 mm point in the barrel. The shear rate,  $\gamma$  was calculated using the equation:

$$\dot{\gamma} = \frac{32Q}{\pi D^3 * 10^3} (s^{-1}) \quad (2)$$

where  $D$  is the die diameter, 1 mm. The tester will display the value of viscosity (Pas), shear rate ( $s^{-1}$ ) and the flow rate ( $cm^3/s$ ). Then, the viscosity versus shear rate graph was plotted. Based on Power Law equation (Eq. 3), the slope ( $n-1$ ) was calculated from the graph to determine the flow behavior index,  $n$  for each feedstock formulations:

$$\eta = K \dot{\gamma}^{n-1} \quad (3)$$

where  $\eta$  is the viscosity,  $K$  is the constant. While the Arrhenius's equation is applied to determine the activation energy,  $E$  for the samples:

$$\eta = \eta_o \exp(E / RT) \quad (4)$$

where  $\eta_o$  is the viscosity at reference temperature,  $R$  is the gas constant and  $T$  is the temperature in Kelvin unit. According to Eq. (4), a group of plots of  $\ln \eta$  vs.  $1/T$  at a certain shear rate can be obtained. The graphs tend to fit into straight lines. The slopes of the graph indicate the temperature dependence of viscosity, or the activation energy,  $E$ . Large values of  $E$  show a high sensitivity of viscosity to temperature.

Moldability parameter ( $\alpha$ ) of binder binder formulations and PIM feedstocks are calculated from Eq. (5) as proposed by Weir et al [6,7]:

$$\alpha = \frac{10^9 (n)}{\eta_o (E / R)} \quad (5)$$

where  $\eta_o$  is the apparent viscosity at the reference shear rate (100, 10 000 and 100 000  $s^{-1}$ ),  $n$  is the flow behavior index,  $E$  is the flow activation energy and  $R$  is the universal gas constant.

## RESULTS AND DISCUSSIONS

### Analysis of viscosity dependence to shear rate

Figure 2 shows the correlation of viscosity of feedstocks and shear rate at injection temperature of 130°C. The feedstock F63\_31 as shown in this figure exhibits highest viscosity while feedstock F63\_16 is the lowest at this injection temperature. Powder-binder separations occur at high shear rate on the feedstock F63\_31. However, all the feedstocks exhibit a shear thinning or pseudoplastic behavior, which is common for MIM feedstocks. The viscosity of a pseudoplastic substance decreases as the shear rate increases (shear thinning). This could be due to particle orientation and ordering with flow as well as breakage of particle agglomerates with release of fluid binder [2].

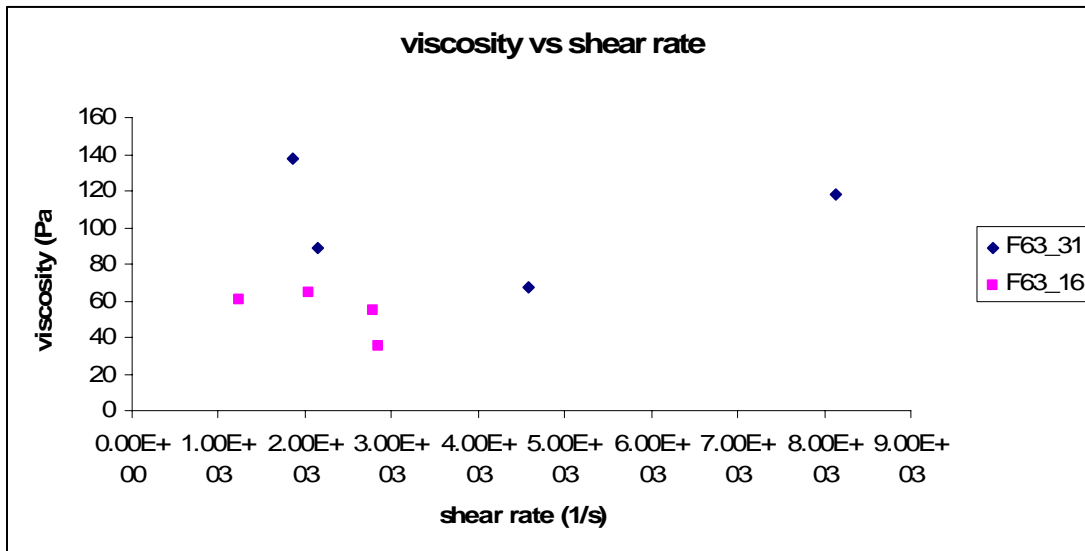


Figure 2: Viscosity of MIM feedstocks as a function of shear rate at temperature of 130°C.

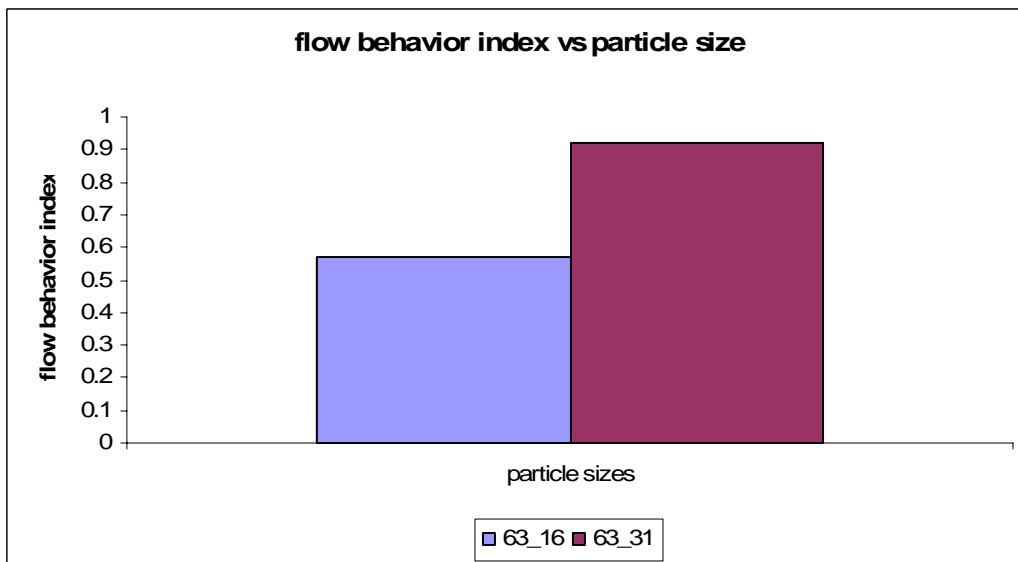


Figure 3: Diagrams show the flow behavior index of feedstocks at 130°C as a function of particle size.

The Power Law equation (eq. 3) can be used to explain the relation between viscosity to shear rate at a given temperature.

Figure 3 shows the flow behavior index of feedstocks at 130°C. All the feedstocks exhibit a shear thinning or pseudo-plastic behavior, as the value of  $n$  is smaller than 1. The lower the value of “ $n$ ” means the more viscosity dependence to the shear rate or showing a greater pseudo-plasticity behavior of the feedstocks [4]. However, some molding defects such as jetting are associated with low “ $n$ ”. Jetting are undesirable since it is the source of severe defects, including weld lines and other imperfections in the final molded part [7]. From the figure, it shows that the  $n$  value of feedstock F63\_16 is smaller than F63\_31. This means that the viscosity is more sensitive to shear rate in the case of a smaller particle size of feedstock, and show the best pseudo-plastic behavior. But this is undesirable because a lower value  $n$  could leads to the slip flow phenomena, which can cause molding defects. Thus F63\_31 was the best feedstock since it has a highest value of  $n$ , or lowest sensitivity to shear thinning behavior.

### Analysis of temperature dependence of viscosity

One of the important characteristic of MIM feedstock is temperature-dependence of viscosity. Pure binder has a viscosity that usually varies exponentially with absolute temperature  $T$  [2]. Therefore, the effect of temperature on the viscosity of feedstock was evaluated based on equation (4)

By plotting  $\ln \eta$  versus  $1/T$  graph at a certain shear rate, the slope that obtained from the graph indicate the value of  $E/R$ , where  $R$  is the universal gas constant.

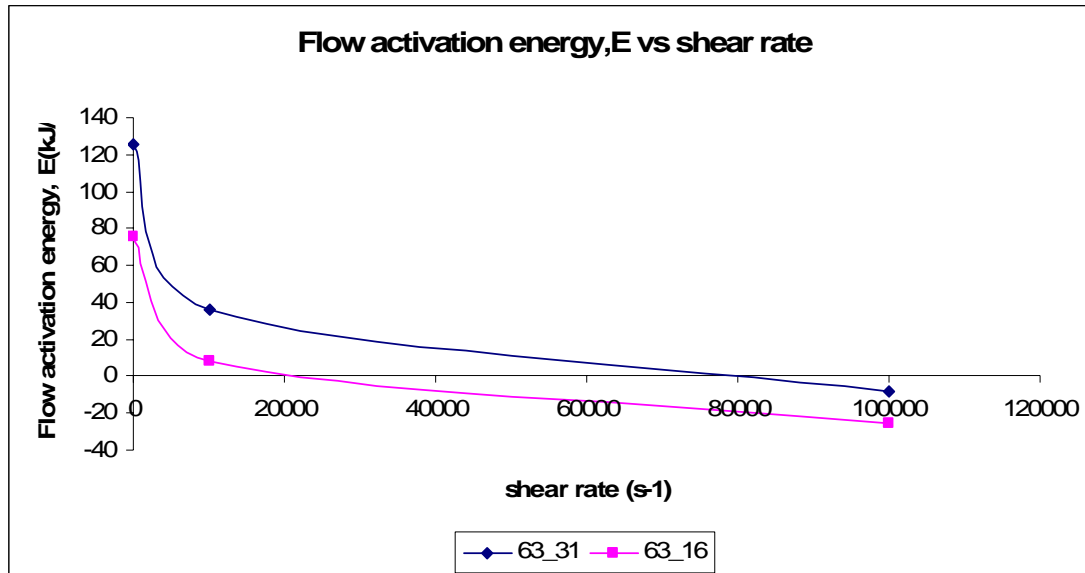


Figure 4: Flow activation energy of PIM feedstocks as a function of shear rate at different particle sizes

The flow activation energy of tested feedstocks as a function of shear rate is shown in Figure 4. For both particle sizes, it is visible that the values of  $E$  decreased as the shear rate increases, and the activation energy was found to be higher, particularly at bigger particle size. A high value of  $E$  indicates a strong temperature-dependence of the feedstocks to the viscosity. Therefore, any small fluctuation of temperature during molding a bigger particle size results in a sudden viscosity change. This could cause defects in the molded parts, such as cracking and distortion due to the undue stress concentration appeared [4]. High activation energy of feedstock F63\_31 indicates a drastic viscosity increase upon cooling, and thus feedstock F63\_31 required a more accurate temperature control during injection molding. Otherwise, mold temperature distribution will cause non-uniform flow, which induced internal stresses.

In addition, feedstock with high sensitivity to temperature is also sensitive to pressure [2]. As pressure increases, a polymer becomes more viscous. The value of  $E$  is also found to be low at high shear rate, and visible for both particle size. The small value of  $E$  shows a low sensitivity of viscosity to temperature, thereby minimizing stress concentration, cracks and distortion in the molded parts. Thus, the 16 $\mu$  particle size of feedstocks at high shear rate (F63\_16) is the best feedstock to be injected molded, as this feedstock is less sensitive to temperatures.

### Analysis of moldability parameter, $\alpha$

In order to establish a general molding index, the following formula can be used to calculate moldability parameter ( $\alpha$ ) as proposed by Weir [4,7] as shown by equation (5).

Results obtained from Weir equation are tabulated in Table 4. (The reference shear rate used in this study is 100 and 10,000s<sup>-1</sup>).

Table 4: Moldability parameter,  $\alpha$  from this study

Shear rate (s-1)	Feedstock	$\alpha$ value
100	F63_31	-1.89E+03
100	F63_16	-3.63E+03
10,000	F63_31	-3.32E+04
10,000	F63_16	5.17E+05

From Table 4, it is shown that the value of  $\alpha$  is proportional to the shear rates, particularly for feedstock F63\_16, contrasts with F63\_31, where the  $\alpha$  value is decreased with the increasing shear rate. In the absence of problems such as jetting or high residual stresses, the higher value of  $\alpha$  is desirable since feedstocks with low  $n$  values are prone to powder-binder separation. Thus, the higher the value of  $\alpha$ , the better the rheological properties. As seen, F63\_16 gives the highest value of  $\alpha$ , particularly at high shear rate, showing the best feedstock behavior to be injected molded.

## CONCLUSIONS

The rheological behavior of gas atomized 316L stainless steel powder with particle size of 16 and 31 $\mu$ m, mixed with PMMA, PEG and stearic acid as a binder has been investigated with powder loading of 63 % volume over a wide range of shear rate.

All feedstocks are possible to be injection molded as the flow behavior index indicates pseudo plastic or a shear-thinning behavior. Moreover, the flow behavior index of the coarse powder was larger than the fine powder particles. Feedstock F63\_31 is the best feedstock since it has a highest value of  $n$ , or lowest sensitivity to shear thinning behavior. The high sensitivity to shear thinning behavior is undesirable because it leads to the slip flow phenomenon, the cause of molding defects. The viscosity of bigger particle size feedstocks are more dependence to temperature effect (higher value of  $E$ ) and this is not suitable for injection molding since any small fluctuation of temperature during molding results in a sudden viscosity change. Thus, feedstock F63\_16 is a more suitable feedstock, because it has a lower viscosity dependence to temperature. The moldability parameter,  $\alpha$  for each feedstock was also significant, and F63\_16 showing the best feedstock behavior to be injected molded, since it has the highest value of  $\alpha$ . A higher value of  $\alpha$  is desirable since feedstock with low value of  $n$  are prone to powder-binder separation.

A feedstock with a good homogeneity, high stability, a high flow behavior index ( $n$ ), low activation energy ( $E$ ) and high moldability parameter ( $\alpha$ ) were found to have optimum injection molding characteristics. The finer particle size shows the lowest sensitivity of the viscosity to the temperature. Further, the moldability parameter is also high, thus exhibits the feedstock F63\_16 as the best feedstock to be injected molding over a wide range of shear rates.

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