Rheological Study of MIM Water Soluble Binder System

Muliaty Samsuri
Politeknik Sultan Azlan Shah
Email: muliaty@psas.edu.my

Siti Atiqa Al Zahra Mat Darus
Politeknik Sultan Azlan Shah
Email: siti_atiqa@psas.edu.my

Mohd Bukhari Md Jahi
Politeknik Sultan Azlan Shah
Email: mohd_bukhari@psas.edu.my

Abstract
Although many methods are used for characterization of Metal Injection Moulding (MIM) parts, but the process of metal injection molding (MIM) has provided an alternative for the manufacturer to produce small and complex parts. MIM is an acronym for metal injection molding, a process for manufacturing metal parts. MIM combines the design freedom of plastic injection molding with the performance of metal. This research will provide an introduction to metal injection molding by briefly reviewing the basic process steps by using a composite binder system whose main constituents are polyethyle glycol (PEG), which are water soluble binder system. This also covered Hostamont EK583, a ready made binder and finely dispersed polymethyl methacylate (PMMA). Besides, feedstock at three different PEG% weight binder composition; 65%, 75% and 85% powder loading were studied in order to find the best homogeneity of feedstock. From the results, it shows that the increasing of the PEG% composition would increase the viscosity. The lower the value of viscosity, the easier it is for a MIM feedstock to flow. Result also shows that the flow of the feedstock is found as pseudoplastic flow and at the composition of PEG 75% posses the best homogeneity feedstock.
Keywords: Metal injection molding, binder, water soluble binder

1.0 Introduction

New applications of parts and materials place new demands on manufacturing techniques. Generally, variety of techniques and methods are used for each manufacturing process. Besides the cost of economical consideration, time also is the main aspect in the production process, which a good production needs to be done in a short time with a low cost. MIM is a new powder metallurgy technique combination of conventional plastic injection moulding and powder metallurgy, which has developed into a serious competitor to the classical production methods such as fine casting, stock machining and stamping (Pratabrao et al., 2018). Till now, MIM is an internationally recognized and used production method, which is supported by an extensive set of ISO standards. MIM open new possibilities for designers and product developers to use it for an economical production of complex metal parts. MIM has been used to form complex shape forming ability (Tun, et al., 2018). It combines the attributes of powder metallurgy (e.g. low cost, simplicity, flexibility of composition selection and inexpensive raw materials) and plastic injection moulding (e.g. ability to manufacture complex parts and rapid production) to manufacture small-to-medium sized intricate components and is particularly suited to mass production (Dehghan-manshadi, Bermingham, Dargusch, Stjohn, & Qian, 2017; Dilawer et al., 2017). Complex shapes that are produced using MIM process can be formed inexpensively to nearly full density through the use of a polymer powder combination (Norhamidi et al., 2000). Therefore, there are not necessary to any secondary machining process.

In addition, the added value of MIM part depends on the complexity of the geometry and the number of functions it contains. Usually, the design of MIM can integrate functions, which require several parts in a convention construction method. The reduction in the number of parts and assembly steps together with the choice of the optimum material increases the quality and lower the final cost (Torralba, 2002). Generally, there are five steps involved in MIM process that are mixing, palletizing, injection molding, debinding and sintering (Royer, Barriere, & Bienvenu, 2018). Their basic processes are as follows. First, metal powders with specific particle size and range and shape are mixed with binder to prepare a moldable feedstock. Then the feedstock flows and fills into a mold under heat and pressure to form green parts. Subsequently, the binder is sintered to nearly full densification through sintering process (Norhamidi et al, 2001). Throughout the MIM process, binder system plays an integral role that holds the metal powder together and needs to be removed as completely as possible afterwards before sintering (Zhang et al., 2018).

In MIM process, the rheological properties of the feedstock are key features, which influence the steady flow and the uniform filling into the mold (Ahmad et al., 2017). This research will characterized behaviour of metal injection molding (MIM) binder and feedstock, study thermal properties of binder and feedstock, and find the best condition of binder used in MIM process.

2.0 Experimental Details

2.1 Materials

The binder used in the experiment was PEG and PMMA. For PEG, there are two types of binders used that are PEG 3000 and PEG 6000. While for PMMA type of binder's used is PMMA 15000. Besides, the test also was performed on Hostamont EK 583. Variety of binders is used to get the comparison of all binders. This is important to have a good characteristic of binder to form a high quality product.

2.2 Feedstock

There are three types of feedstock that used in the experiment. There are feedstocks for samples 65%, 75% and 85% PEG binder composition. For every feedstock, 5% Stearic acid was included as a surface actives agent and the balance is the % composition for PMMA. The composition of the binders in this feedstock is listed in Table 1.

Table 1: Weight composition of the binder constituents for different feedstock

Composition	Feedstock 1	Feedstock 2	Feedstock 3
PEG	65%	75%	85%
PMMA	30%	20%	10%
Stearic Acid	5%	5%	5%

2.3 Characterization of MIM Binder and Feedstock

2.3.1 Thermogravimetric Analysis (TGA)

The TGA instrument used can capable of heating or cooling rates up to at least 20 ± 1 °C/min and of automatically recording he difference in heat flow between the sample and reference material. Then, the result will be recorded on a chart by computer analysis software for weight-recording apparatuses which suitable graduations for measurement of weight differential against temperature or time.

2.3.2 Differential Scanning Calorimeter (DSC)

In this method, a sample and a reference substance are put on a metal connected to a furnace which is a heat sink, and the temperature difference at the sample and reference substance is proportional to the temperature of the sample and reference substance. Besides, transitions measured by DSC include melting, curing and cure kinetics, onset of oxidation and heat capacity. DSC can capable of heating or cooling rates up to at least 20 ± 1 °C/min and of automatically recording the difference in heat flow between the specimen (sample) and reference material. The normal operating temperature range is from ambient to 600 °C.

2.3.3 Capillary Rheometer

Capillary rheometers are used to measure shear viscosity from low to high shear rates, directly simulating conditions experienced during processing. It is also used to measure melt fracture and die swell which are often due to elastic properties that manifest themselves at high shear rates during processing. The data is usually presented as a graph of shear stress against shear rate at constant temperature.

3.0 Results and Discussion

a. Analysis of Differential Scanning Calorimeter (DSC)

The DSC analysis was used to investigate the melting behaviour of the binder, PEG and PMMA. All the data for the onset melting point and the peak melting point is shown in Table 2. From the table, it shows that the value of onset melting point for PEG 3000 and PEG 6000 is lower than the value of onset melting point PMMA 15000 and hostamont ek 583. This is because the PEG binder is a water-soluble binder system. But, the values of melting point for all binders are important during mixing process and for the water leaching process.

Table 2: Result for DSC testing for all types of binder used

BINDER	**DSC**
Peg 3000	Onset = 51.80 ^{o}c Peak = 61.67 ^{o}c
Peg 6000	Onset = 57.32 ^{o}c Peak = 72.33 ^{o}c
Pmma 15000	Onset = 98.68 ^{o}c Peak = 108.67 ^{o}c
Hostamont ek 583	Onset = 101.60 ^{o}c Peak = 107.30 ^{o}c

b. Analysis of thermogravimetric (TGA)
From the TGA analysis, the value of the temperature evaporation range as shown in Table 3 can be used as a suitable temperature range during debinding process. At this range, all the binder will be removed. But in this research, it can be concluded, from TGA curves for all samples, the % of the weight of the binder that is melted is around 92 %. This means that not all binder is removed from the feedstock. This thing happened because of there might be unbalance evaporation range of the temperature that is used while the TGA machine is run. So, there will be a little bit binders balanced that is not removed successfully.

Table 3: The summarized value for TGA testing for PEG % composition

BINDER	**TGA**
Peg (65 % weight)	280.00 – 435.00 ^{o}C
Peg (75 % weight)	315.59 – 467.18 ^{o}C
Peg (85 % weight)	225.58 – 459.44 ^{o}C

c. Rheological Analysis
Rheological test is done to determine the flow properties and the level of homogeneity of binder and feedstock at different value of

temperature from 60 °C until 100 °C for PEG 85%, 70 °C to 110°C for PEG 75% and 110 °C to 150 °C for peg 65 %. The temperature range that have used is the range of binder melt. Binder should possess a lower viscosity than feedstock. The viscosity of MIM feedstock is quite higher than binder because of the high volume fraction of powder particles. The MIM feedstock is generally considered to be a pseudo plastic flow, which indicates a decreasing of viscosity with increase shear rate temperature. From the observation, both the Figure 1 and 2 shows the effect of temperature on viscosity at different pressure. It shows the fluctuation phenomenon at pressure 1 Mpa is not followed the pseudo plastic behavior.

The viscosity of feedback must decrease when the temperature is increased. But in this case, this situation is not occurred. The fluctuation may be due to improper handling or monitoring the analysis or because of the composition itself. The fluctuation can be affected the selection to determine the homogeneous feedstock.

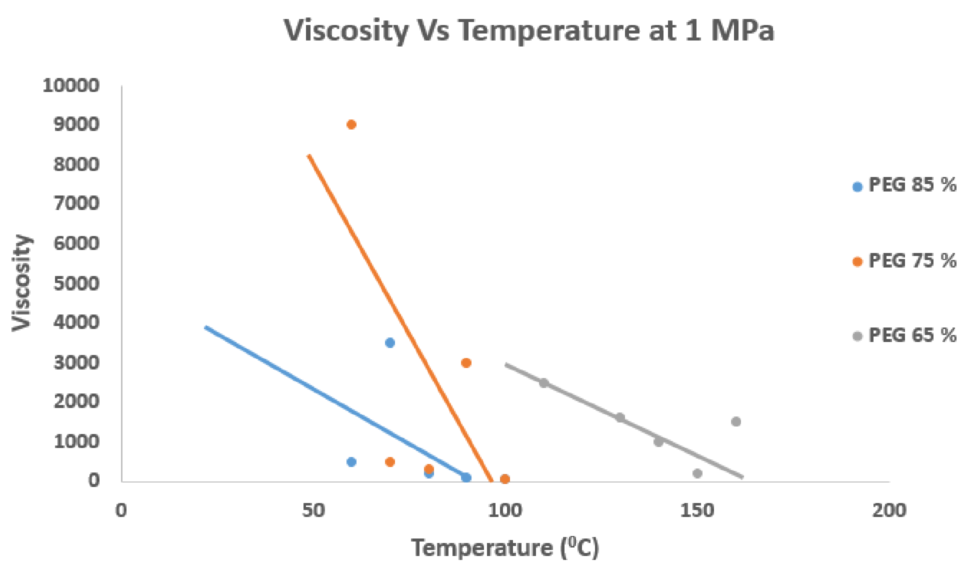

Figure 1: Effect of temperature on viscosity at 1 Mpa

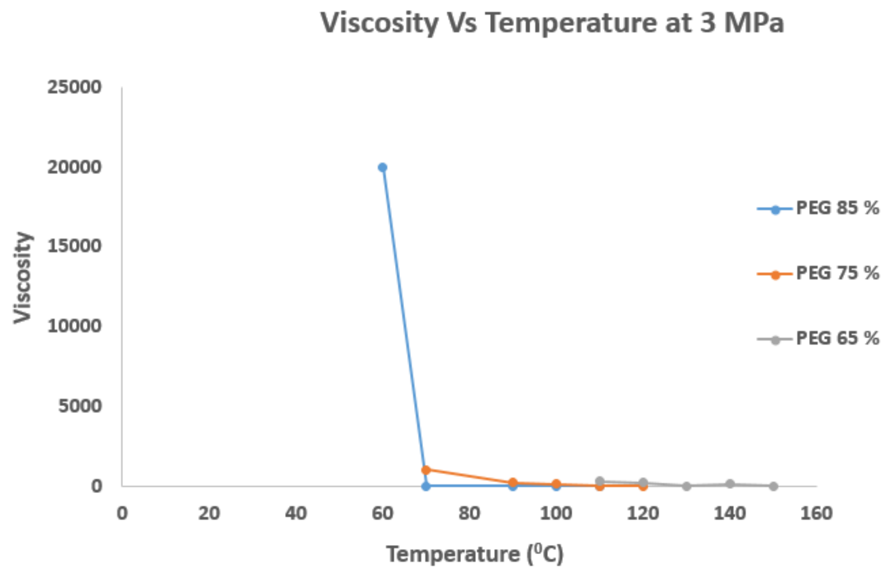

Figure 2: Effect of temperature on viscosity at 3 Mpa

d. Effect of Temperature on Shear Rate

The Figure 3 shows the relation between shear rate and temperature. It shows the shear rate will increase as temperature increase but in this case the graph fluctuates and is not stable. Compared to Figure 4, the graph is quite stable and constant increased between shear rate and temperature. The best condition is at feedstock PEG 75% composition at pressure 3 Mpa through temperature 70 °C to 110 °C. It shows the increasing of temperature will increase the shear rate.

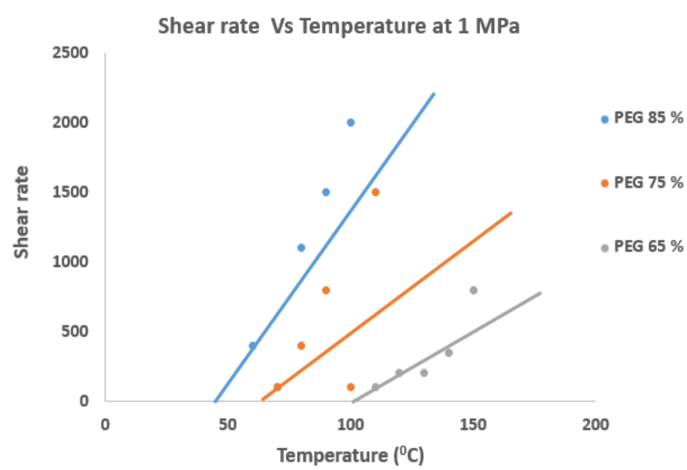

Figure 3: The relation between shear rate and temperature at 1 Mpa

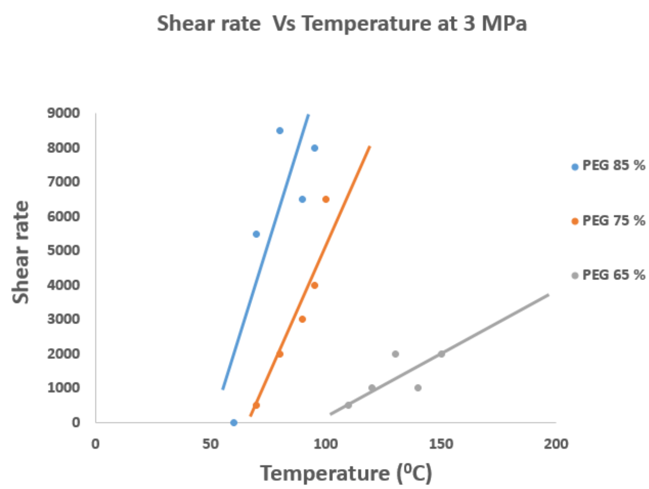

Figure 4: The relation between shear rate and temperature at 3 Mpa temperature

e. Effect of shear rate on viscosity

Figure 5 shows the relation between viscosity and shear rate at 85% PEG composition. It shows that, the increasing of temperature and shear rate will reduce the viscosity, which follows the pseudo plastic flow. When the shear rate is increase, the viscosity is decrease. It also showed that from the three graph relation between viscosity and shear rate, it was found that the curve are the same with each other, but it just differ at the viscosity value because of the differential of percentage of PEG composition which is will increase the value of viscosity at all of the temperature.

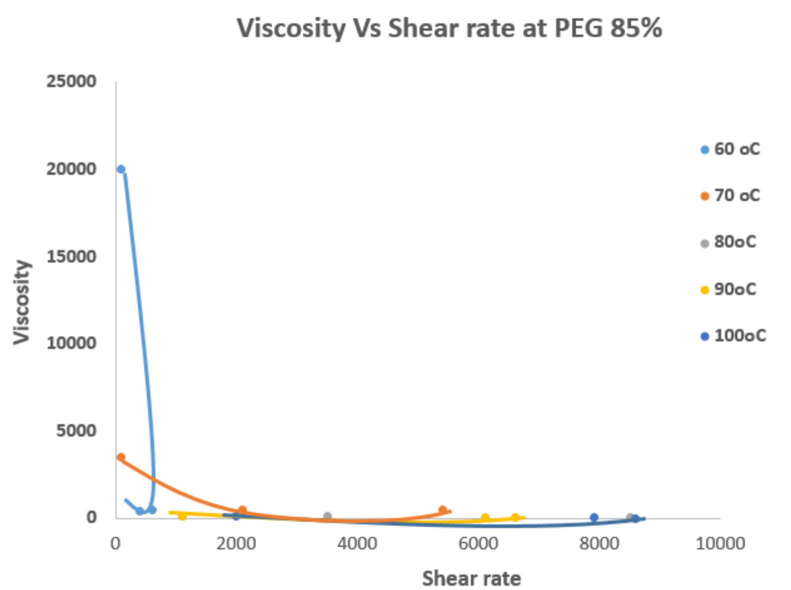

Figure 5: The relation between viscosity and shear rate at 85% PEG composition

While, Figure 6 shows the relation between viscosity and shear rate at 75% PEG composition. From the figure below, all the feedstocks follows the pseudo plastic flow curve, when the shear rate increase, the viscosity is decreased. But at the temperature 70 °C and 100 °C at pressure 1 Mpa, it shows very high viscosity about 9000 Pa.s and 3000 Pa.s that is not suitable for the mim process. For the temperature 80 °C, 90 °C and 110 °C the viscosity were below 1000 Pa.s which is suitable for injection molding process.

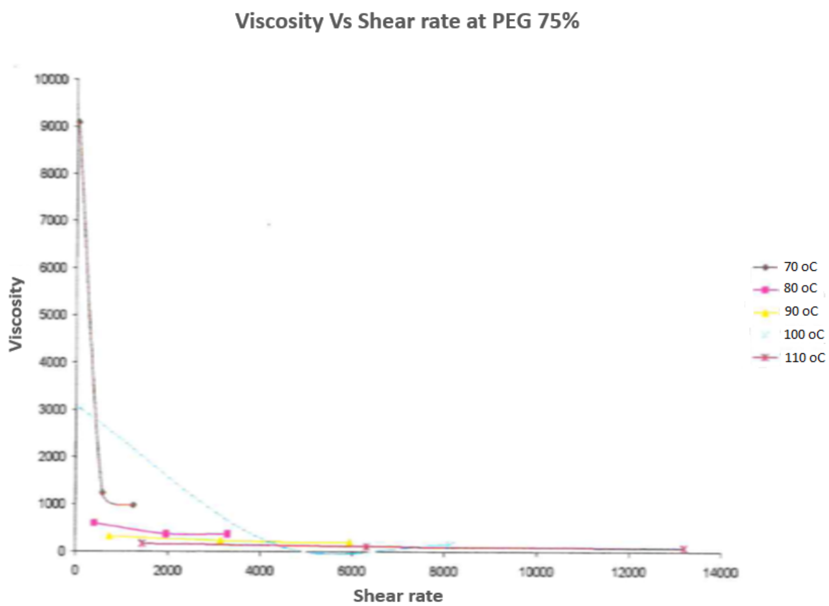

Figure 6: The relation between viscosity and shear rate at 75% PEG Composition

Figure 7 shows the relation between viscosity and shear rate for PEG 65% composition. The increasing of the temperature and shear rate will reduce the viscosity which is followed the pseudo plastic flow. But at pressure 1 MPa, in the temperature 110 °C, 120 °C and 130 °C the viscosity is high which is greater than 1000 Pa.s.

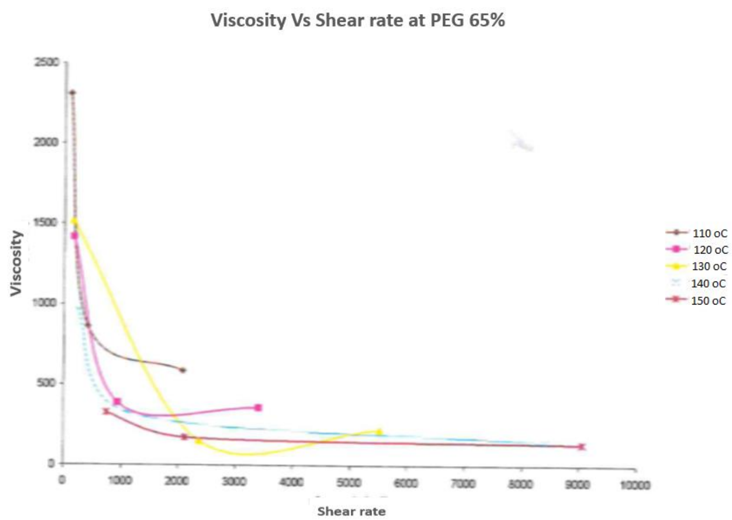

Figure 7: The relation between viscosity and shear rate at 65% PEG composition

4.0 Conclusions

From the TGA and DSC analysis, it shows that the PEG was suitable for water soluble system since the peak melting temperature not too high compared to the other binders such as PMMA and Hostamont EK583. This is because the water soluble binder provides low viscosity during the injection molding operation and the PEG also can be dissolved in water. By using the rheological test, the parameters behaviour like viscosity, shear rate and flow rate were determined. The viscosity at the moulding temperature must be such that the mix flows smoothly into the die without any segregation. While, the viscosity should be as constant as possible over a range of temperature. From the rheological study, it can be concluded that all the data from all three PEG % compositions followed by pseudoplastic behaviour which have viscosity in range of 10 Pa.s to 1000 Pa.s. It is also can be conclude that the 75% PEG is the most suitable PEG composition since the rheological results showed that their data is more reasonable. It can be concluded that the most homogeneous feedstock can improve process ability and part performance.

References

Ahmad, R. N. (2017). Rheological Properties of Titanium Niobium Based Feedstocks for Metal Injection Moulding, *2*(1), 139–149.

Dehghan-manshadi, A., Bermingham, M., Dargusch, M., Stjohn, D., & Qian, M. (2017). Metal Injection Moulding of Titanium and Titanium. *Powder Technology*. http://doi.org/10.1016/j.powtec.2017.06.053

Dilawer, M., Goswami, A., Matthews, S., Li, T., Yuan, X., & Cao, P. (2017). Modi fi cation of PEG / PMMA binder by PVP for titanium metal injection moulding. *Powder Technology*, *315*, 243–249. http://doi.org/10.1016/j.powtec.2017.04.004

Norhamidi Muhamad, Iriany, Ahmad Kamal Ariffin, Jaafar Sahari (2001). “Rheological Study of Feedstock for Metal Injection Moulding Process”. Universiti Kebangsaan Malaysia.

Norhamidi Muhamad, Muhammad Hussain Ismail, Nor Hafiez Mohamad Nor, Ahmad Kamal Ariffin Mohd IhsN, Jaafar Sahari (2000). “Importance of Rheological Behaviour in Metal Injection Moulding For Prediction of Injected Part”. Universiti Kebangsaan Malaysia.

Royer, A., Barriere, T., & Bienvenu, Y. (2018). PT. *Powder Technology*, (2017), #pagerange#. http://doi.org/10.1016/j.powtec.2018.05.047

Torralba, J. M. (2002). Effect of Residual Carbon on the Sintering Process of M2 High Speed Steel Parts Obtained by a Modified Metal Injection Molding Process, *33*(June), 1843–1851.

Tun, U., Onn, H., Raja, P., & Pahat, B. (2018). Parameter Optimization of Metal Injection Moulding : A Review, *5*(6), 100–114.

Zhang, H., Hayat, M. D., Qu, X., Jadhav, P. P., Wang, X., & Cao, P. (2018). Study of a Binder System for Ti-MIM : A Potential Low Temperature Backbone Polymer, *770*, 206–213. http://doi.org/10.4028/www.scientific.net/KEM.770.206