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Development of Thermoplastic Natural Rubber (TPNR) as a New Binder in Metal Injection Moulding

Mohd Afian Omar[1*] and Norita Hassan[2]

[1]Advance Material Research Centre (AMREC),
SIRIM Berhad, Lot 34, Jalan Hi-Tech 2/3,
Kulim Hi-Tech Park, 09000 Kulim, Kedah, Malaysia
[2]Department of Manufacturing and Materials Engineering,
Kulliyyah of Engineering,
International Islamic University Malaysia, Jalan Gombak,
53100 Kuala Lumpur, Malaysia

[*]Corresponding author: afian@sirim.my

Abstract: *Since metal injection moulding (MIM) is still new in Malaysia, therefore it is great opportunity for those who carry out the development of new binder system. Perhaps this technology can be exploited in Malaysia in future for the benefits of all industrial sectors. Developing of a new locally binder system, which is beneficial to the local industry is a great concern. Many attempts have been made in developing a new binder system which concentration is given on the cost reduction and shorten the production process. Thermoplastic natural rubber (TPNR) has a possibility as binder component because of many suitable properties it is such as low cost as locally produced, low viscosity, high decomposition temperature and easily dissolves into solvent also chemically passive. Hence, TPNR is worth to consider as a binder component.*

Keywords: TPNR, metal injection moulding, debinding, sintering, binder

1. INTRODUCTION

Metal injection moulding (MIM) feedstock is a mixture of metal powder and binder. The function of the binder system is to provide flow ability of the metal particles and shape retention of the moulded parts.[1] The behaviour of the feedstock depends greatly on the binder formulations and its composition. Suitable behaviours feedstock that refers to its rheological behaviours is one of the key factors to ensure the successful of MIM process.[1–3] Therefore, the rheological, moulding behaviour and sintering characterisation of feedstock is an important factor in the success of good binder system. In these studies, the possibility of using thermoplastic natural rubber (TPNR) as a binder system has been investigated. TPNR is classified as thermoplastic elastomer, it absorb solvent and swell, but do not dissolve; furthermore, they cannot be reprocessed simply by heating. The molecules of thermoplastic rubbers, on the other hand, are not connected by primary chemical bonds. Instead, they are joined by the physical aggregation of parts of the molecules into hard domains.[4] TPNR estimated has a great potential application as a binder system as it is expected ease during injection moulding process with less defect and also considerable motivation to design and develop binders that are based on natural sources which are locally produced. In this study, TPNR backbone polymer was applied with paraffin wax (PW). PW binder used is attributes as a binder such as low viscosity, high decomposition temperature, lower molecular weight to avoid residual stress and distortion, environmentally acceptable, inexpensive and easily dissolved in organic solvent.

2. EXPERIMENTAL

The TPNR binder system with ratio of 55 wt % Paraffin wax, 35 wt % TPNR and 10 wt % stearic acid (SA) was compounded out in a laboratory mixer Brabender Plasticoder at 140°C and at speed of 50 rpm for 12 min according to the adequate time for the component to melt and homogenise. Initially the steel powder was mixed with different formulation of binders while the volume fraction of the powder in the mixture kept constant at 65%. The rheological results in term of shear rate, shear stress, and pseudoplastic behaviours have been presented using a Capillary Rheometer (CFT–500D, Shimadzu) at various temperature and shear rates. The tensile specimens were fabricated in a vertical injection moulding MCP HEK-GMBH. The injection pressure was 300 bar and the mould temperature was 190°C–200°C. Solvent debinding process was performed at different time and temperature in a bath of heptanes in order to remove paraffin wax and SA. Specimens were taken out and dried for two hours after bathing. The sintering process was performed in a controlled vacuum atmosphere with heating rate of 5°C/min in a temperature range of 1320°C–1380°C. The specimens were soaked for two hours and subsequently furnace cooled.

3. RESULTS AND DISCUSSION

3.1 Feedstock Compounding at Different Powder Loading

In the feedstock mixture, it is expected that each powder particle, which should be enveloped by a very thin film of binder, has a tight contact with each other. At the same time, all pores among powder particles are filled with binder or describe as a homogeneous feedstock. But it is very difficult to get that ideal powder-binder mixture. At this stage the evolutions of mixing torque have been studied during the mixing time to understand the effect of powder concentration and powder-binder formulation. Powder loadings ranging from 61 to 67 vol % of gas atomised 316L SS (22 μm) were evaluated by using same formulations of binder system, 55 vol % PW, 35 vol % TPNR, 10 vol % SA. The mixing was carried out at 160°C with rotation speed of 50 rpm for 120 min.

It clearly shows in Figure 1 that the torque value increases as the powder loading increases from 61 to 65 vol %. This explains that greater friction occurred at the higher amount of powder loading that result in a higher viscosity of the mixture. For powder loading of the 65 and 63 vol %, the torque stabilises at a steady level in a short time, indicating a uniform mixing. Meanwhile, the 61 vol % of powder loading took a longer time for the torque value to be stable. Powder loading of 65 vol % was selected based on the previous analysis on further study of feedstock rheological analysis and moulding of feedstock.

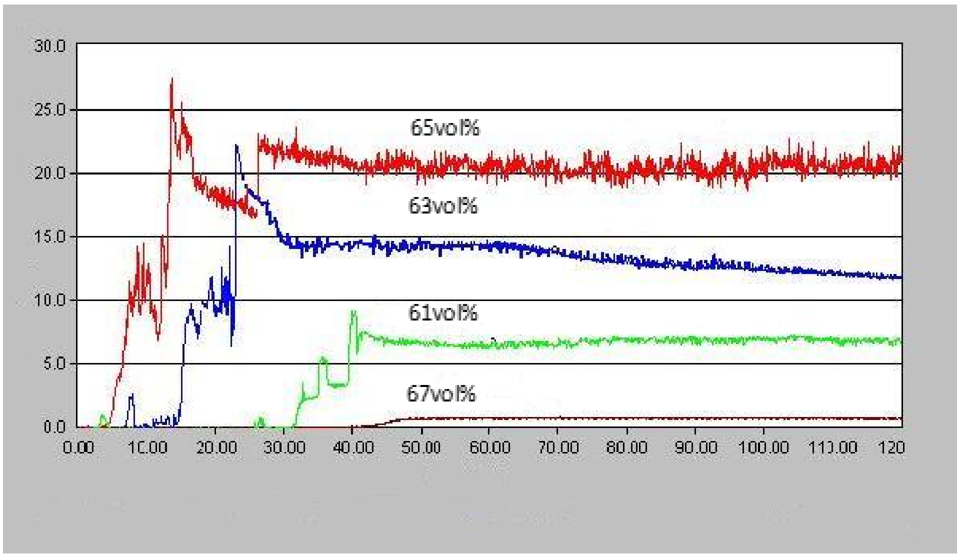


Figure 1: Torque levels of feedstock compounding at different powder loading.

In the case of 67 vol % powder loading, it seems that there is no mixing registered between powder and binder addition due to very minimum torque value which is near zero reflecting an excessive content of powder. These were believed to be due to powder-binder separation during mixing and possibly of instability phenomenon which indicates that the ratio of powder to binder was too high and not sufficient to provide flowability of the particles.

3.2 Rheological Behaviour

The rheological analyses have been carried out at various temperatures and different pressure. MIM feedstock is generally considered to be pseudoplastic fluid, which indicates a decrease of viscosity with increase on shear rate and temperature. The viscosities of TPNR backbone polymer based feedstock decreasing with shear rate are shown clearly in Figure 2.

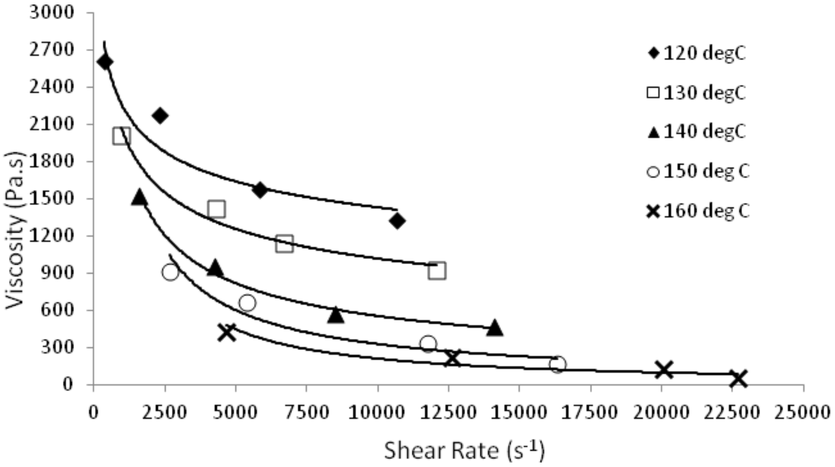


Figure 2: Effect of shear rate on viscosity of feedstock.

Pseudoplastic fluids are preferred for MIM feedstock. 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 moulding usually ranges between 100 and 10,000 s^{-1} and maximum viscosity for moulding is 1000 Pa.s at the moulding temperature.[1,5,6]

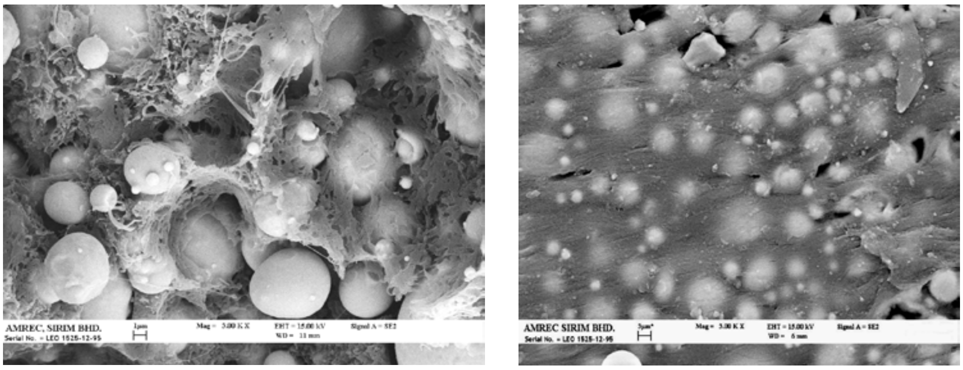
3.3 Injection Moulding of the Feedstock

As several trial been done, the injection moulding parameters had summarised in Table 1. According to the observation during injection moulding process by using the vertical injection moulding machine, the parameter setting is not much different between the feedstock compounded at different formulation.

Table 1: Summaries of injection parameters obtained from the vertical injection moulding machine which had produced good part.

Model	MCP HEK-GMBH injection moulding (vertical injection moulding)
Temperature	Nozzle: 190°C–200°C
	Mould: Room temperature
Injection pressure	30 MPa (300 bar)
Cycle time	10–20 sec

At the end of moulding, the binder provides a mechanical interlocking to the particles which gives the compact shape and the necessary handling strength. Figure 3 clearly shows the SEM of the green specimens at two different regions; at the fracture surface and on the outer surface for four different binder systems.



(a) (b)

Figure 3: SEM micrograph of green parts (a) fracture and (b) outer surface.

It can be seen that, the binder fills practically all the interstitial spaces between the powder particles. The outer surface is filled with more binder than those of fracture surface. This is due to a greater contact of feedstock flow to the cavity wall and also that was the area to solidify first.

3.4 Debinding Process

Figure 4 shows the graph of percentage main binder (PW) removed against time at different temperature for feedstock with PW/TPNR/SA binder system.

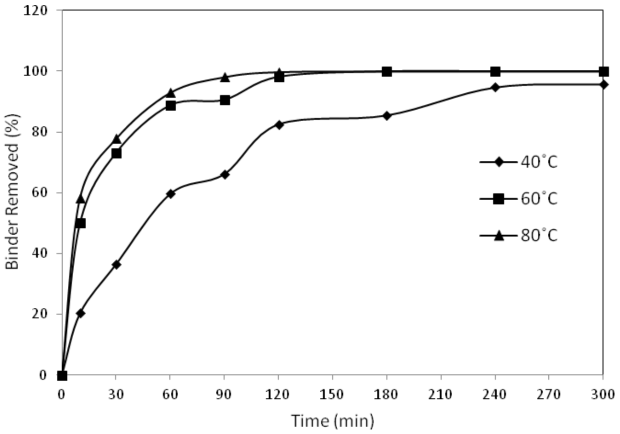


Figure 4: Kinetic solvent extraction of feedstock with PS/TPNR/SA binder system at different temperature.

It was carried out at temperature 40°C, 60°C, and 80°C and eight time intervals. For temperature 40°C, the percentage of PW removed is increase slowly as the time increase. From the graph observation; for 40°C, it shows that till the end of 300 min the percentage of Paraffin wax removed was just about 95.7%. But, for the 60°C and 80°C graph, the percentage of paraffin wax removed achieved 100% by the time of 180 min. At temperature 80°C, the percentage of paraffin wax removed from the green body increase faster at 10 min to 60 min and achieved almost 100% at 120 min. However at the temperature of 80°C, the specimen undergo swollen backbone polymer at longer time. This is because at higher temperature and longer time of solvent extraction process, the backbone polymer becomes soften as the penetration of higher temperature of solvent which also contribute to the extension of pore channels at the inner region.

3.5 Sintering Process

The theoretical density of the 316 L stainless steel is 7.90 g/cm^3. As the sintering temperature increases, the density of specimen also increases. For example, a change of temperature from 1320°C to 1340°C (part PW/TPNR/SA–1) causes the density to increase by 0.1%, from 7.674 to 7.679 g/cm^3. The increasing in the density from 1320°C to 1340°C indicates that the progress of the first stage sintering (the formation of inter-particle necks) at 1320°C to the second stage at about 1340°C results in significant transport among the particles. The sintering temperature of 1320°C is only at the intermediate stage of sintering where pores begin to close up and densification starts. The relative density is 97.14%.

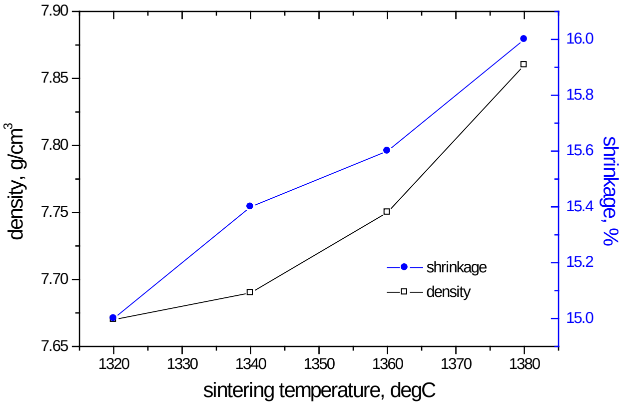


Figure 5: Density and shrinkage for sintered part at various sintering temperature in vacuum atmosphere.

At 1360°C, the part shows a smaller increase of 1% in density, from 7.679 to 7.751 g/cm^3. At a density of 7.751 g/cm^3, it has achieved 98.11% of the theoretical density. Further sintering at 1380°C the density of the sintered specimen increased to 7.858 g/cm^3 and the relative density reached 99.47% that is really closed to theoretical density. Results obtained were comparable with others worked as they reported densities of 316L SS sintered part are in ranges of 95%–99%.

The Figure 6 shows the result of tensile and elongation conducted on the samples. Some results are comparable to MPIF Standard 35 (2000) for metal injection moulded part. Standard 35 states that the minimum ultimate tensile strength (UTS) and per cent elongation are 450 MPa and 40% elongation.

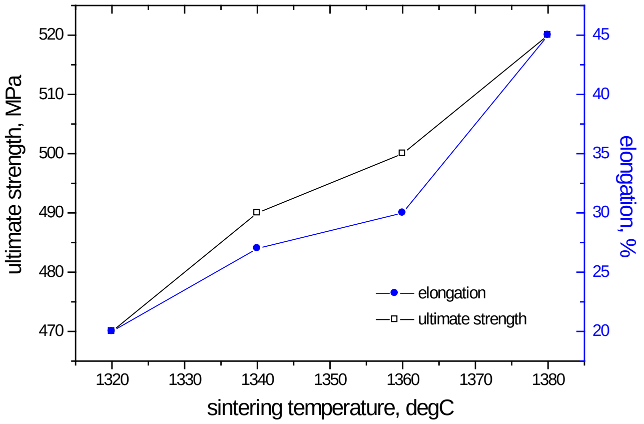
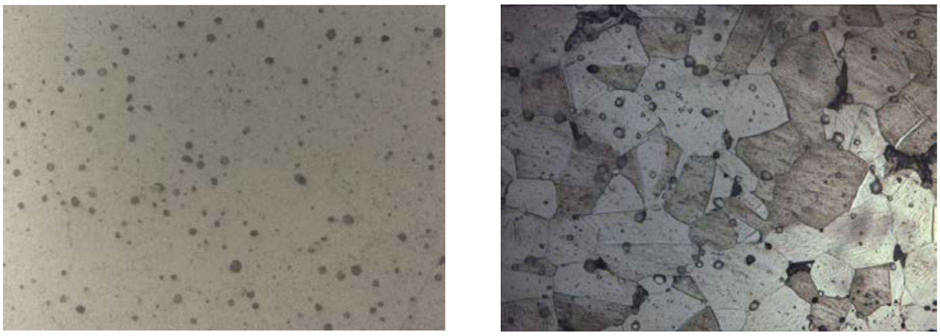


Figure 6: Ultimate tensile strength and elongation for sintered part at various sintering temperature in vacuum atmosphere.

High value of tensile strength for the sintered specimen was result in a subsequent increase in elongation percentage. From the graph plotted it is clearly shows that the percentage of elongation increases with increasing in sintering temperature.

The optical micrographs of sintered specimen in Figure 7 shows the typical pores structures (unetched) and typical microstructure (etched), which display the austenitic grain and pores under vacuum sintering conditions at 1380°C at 500× magnification.

The pores structure and microstructures of the sintered specimen are relatively influenced by the densification of specimen. It could be seen that the sintered part at the optimised sintering condition has the least porosity; the pores are rounded, small and appeared to be isolated in their distribution as the sintering temperature was increased. An increase in temperature promotes neck growth and change in pore morphology. The particle boundaries become thicker and darker indicating that increased sintering temperature resulted in more active changes at the particle boundaries during sintering.



Unetched structure Etched structure

Figure 7: Pores structure and microstructures of sintered specimens at 1380°C under vacuum atmosphere. Magnifications of all specimens at 500×.

The optical micrographs show the typical structure of stainless steel with clear austenite grain, grain boundaries and twinning structures. These microstructures affirmed an austenite stainless steel material. The present of different colours were due to the different plane of austenite phases.

4. CONCLUSION

The new develop feedstock based on TPNR rheological behaviour has been investigated. The feedstock showed pseudo-plastic behaviour which is indicated by a decrease in viscosity with shear rate. The variation in viscosity was correlated with the phase change of NR from a dispersed phase to a continuous phase. It was concluded that, binder formulations and MIM feedstock were shown a pseudo-plastic flow behaviour and suitable for metal injection moulding process. The used of TPNR backbone polymer results in favourable flow behaviour of the MIM feedstock and thus improves the quality of the injection moulding. The mechanical properties of sintered specimen shows that the parts comply with the international standard MPIF 35 MIM when the specimens sintered at 1360°C and 1380°C with the ultimate tensile strength ranges from 470 to 520 MPa and the elongation ranging from 44% to 68%.

5. REFERENCES

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