Effect Of Injection Molding Temperature And Powder Loading On SS 316L-CNT Green Compact

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Abstract
This research delves into the examination of how temperature fluctuations and powder loading levels influence the Metal Injection Moulding (MIM) green component. The feedstock employed in this study combines stainless steel (SS316L-CNT) powders with a composite binder system comprising High Density Poly Ethylene (HDPE), Paraffin Wax (PW), and Stearic Acid (SA). This combination of powders results in a dual-peaked particle size distribution, featuring fine and coarse SS316L-CNT powders with weight proportions of 32% and 68% respectively. These powders are combined at a powder loading of 66% and 67% by volume. The manufacturing process employs the Battenfeld BA 250 CDC injection-moulding machine to craft a MIMA tensile specimen. For determining the density of the as-moulded part, the Archimedes water immersion method, following MPIF Standard 42, is employed. During the experimental process, the injection temperature is modified within the range of 110 to 140°C, while maintaining a consistent injection pressure of 360 bars. To assess green strength, a three-point bending test is performed using the INSTRON 5567 machine, adhering to the MPIF Standard 15 guidelines. The conclusions drawn from the study highlight the heightened sensitivity of the bi-modal feedstock, particularly with increased powder loading, to temperature fluctuations. Furthermore, the study underscores that the influence of injection temperature on the resultant strength and density of the as-moulded part is relatively subdued..

Keywords: Metal injection moulding, CNT, Binder system, density, green strength, powder loading.

Metal Injection Moulding (MIM) has emerged as a highly efficient method for producing intricate and small metallic components on a considerable scale. This innovative technique offers the capability to manufacture a large volume of components with exceptional mechanical properties and precise geometrical characteristics, all at a lower cost compared to conventional methods (Barriere et al., 2003). The fundamental fusion of powder and binder is termed as the feedstock. This concept entails a delicate coating of each powder particle within the feedstock blend with a fine binder film, establishing intimate connections with neighboring particles. This arrangement effectively fills the interstitial spaces between the powder particles, as the binder ensures comprehensive distribution. The ratio of solid powder concerning the overall volume formed by the mixture of powder and binder is defined as the powder loading. Generally, a substantial powder loading is expected in MIM feedstock. However, an excessive binder quantity can lead to binder separation during the molding phase, potentially causing defects or non-uniformities in the final molded components. Moreover, an overly excessive binder surplus can contribute to compact slumping during debinding, as the grip on particles diminishes with the removal of the binder (German and Bose, 1997). An increased powder loading correlates with reduced volume shrinkage within the compact and precise control of dimensional accuracy, a vital aspect for mass-producing intricate MIM parts. Nevertheless, excessively elevated powder loading can result in excessively high feedstock viscosities, posing a risk to the effectiveness of the injection molding process (Li et al., 2007).

Table1. Chemical composition of SS 316L stain less steel powder.

Elements	**wt%**
Carbon	0.03
Silicon	0.75
Manganese	2
Phosphorous	0.045
Sulphur	0.03
Chromium	0.46
Nickel	0.02
Molybdenum	1.77

Table2. Particle size distributions.

	D10	D50	D90	SW
Coarse	9.463	19.406	40.158	4.259
Fine	5.680	11.325	19.640	4.773

The pursuit of minimizing the fraction of binder presents an opportunity to reduce debinding durations and mitigate shrinkage rates. Past endeavors in this direction have emphasized the significance of maintaining a minimal binder quantity to prevent particle agglomeration, while simultaneously ensuring appropriate viscosity levels for effective injection molding. Another avenue for improvement involves particle distribution. The application of bi- or tri-modal particle size distributions emerges as a viable strategy to enhance quality, allowing for increased powder loading without compromising viscosity thresholds. Furthermore, the incorporation of multi-modal size distributions assists in alleviating concerns related to rheological dilatancy in concentrated suspensions (Resende et al., 2001).

This study embarks on an investigation that delves into the effects of both moulding temperature and powder loading within the injection moulding process. This investigation specifically employs SS316L-CNT powders, distinguished by their bi-modal particle size distributions. The chosen binder system is a composition of High Density Poly Ethylene (HDPE) and Paraffin Wax (PW), enriched with the addition of Stearic acid (SA) as a surfactant. Notably, this binder configuration has exhibited effectiveness in molding various types of metal (Chuankrerkkul et al., 2008a, b; Omar et al., 2003) and ceramic (Chuankrerkkul and Nilpairach, 2011) powders. The combination of HDPE and PW with stainless steel leads to the creation of a resilient and robust compact during the molding process. Moreover, the inclusion of PMMA content enhances the compact's rigidity (Chuankrerkkul et al., 2007). Additionally, HDPE's water-soluble nature expedites the debinding process by introducing supplementary pores within the compact via water-leaching. This feature facilitates the capillary-driven removal of PMMA binders during thermal debinding (Ibrahim et al., 2011; Jamaludin et al., 2009a, b).

The purpose of this investigation is to thoroughly examine the injection moulding process utilizing a feedstock that showcases a bi-modal particle size distribution. This feedstock is anchored by High Density Poly Ethylene (HDPE), Paraffin Wax (PW), and Stearic Acid (SA) as essential binder components. The study initiates by assessing the rheological properties of the feedstock. Subsequently, it delves into an analysis of any observed moulding defects, while also evaluating the as-moulded flexural strength and theoretical density percentages. These assessments are conducted across a range of injection temperatures to provide a comprehensive understanding of the process.

MATERIALS AND METHODS

The metal powder of choice is SS 316L combined with Carbon Nano Tubes, encompassing gas atomized stainless steel powders with a pycnometer density of 7.93 g/cm3. The detailed chemical composition of this metal powder is provided in Table 1. Additionally, HDPE showcases densities of 1.2963 and 1.1919 g/cm3, correspondingly. A binder system, formulated to comprise 73% HDPE, 25% PW, and 2% SA by weight, is established. To enhance the moldability of the binder system, 2% SA by weight is introduced as a lubricant. SA, characterized by a density of 0.847 g/cm3, serves this purpose. The particle size distribution is outlined in Table 2. To differentiate between SS316L-CNT volume fractions of 65% and 66%, the feedstock is denoted as B1_65 and B1_66, respectively. The process of blending SS316L-CNT powders with binders occurs within a custom-made mixing equipment for a duration of 95 minutes at a temperature of 70°C. Following the mixing process, the paste undergoes granulation using a robust crusher.

To investigate the rheological characteristics, a Shimadzu 500-D capillary rheometer is utilized. The injection molding of the MIMA tensile specimen, depicted in Figure 1, is conducted using a conventional plastic injection-molding machine. To assess the temperature's influence, the injection pressure is maintained at a consistent 350 bars, while the injection temperature is methodically adjusted at increments of 10°C, covering the spectrum from 120 to 150°C.

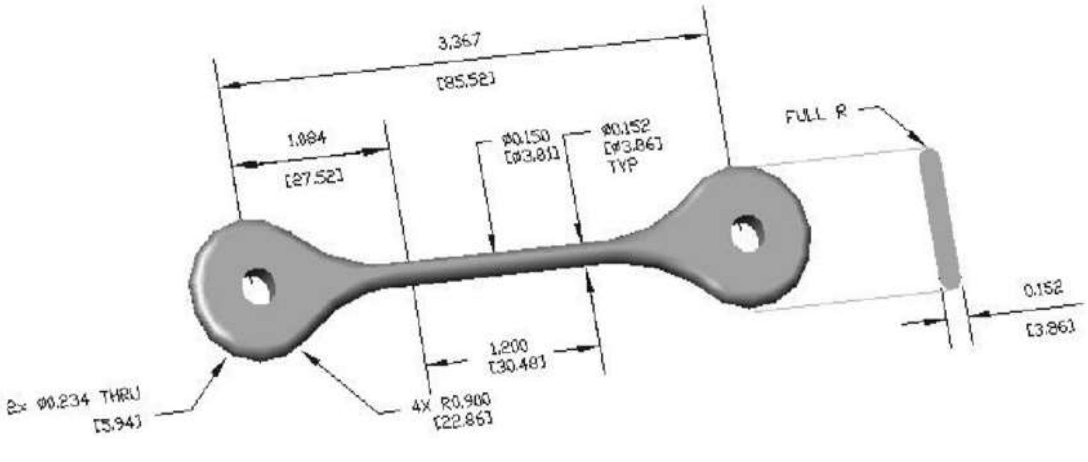


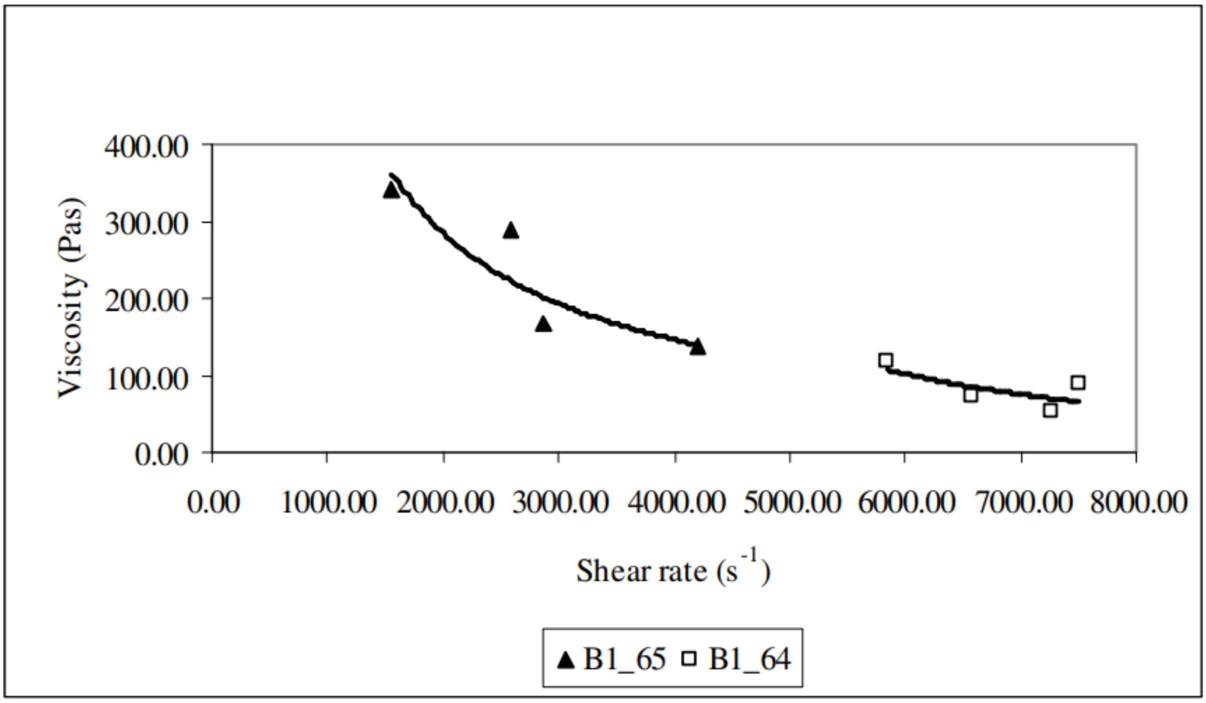
Figure1. Injection molded specimen.

RESULTS AND DISCUSSION

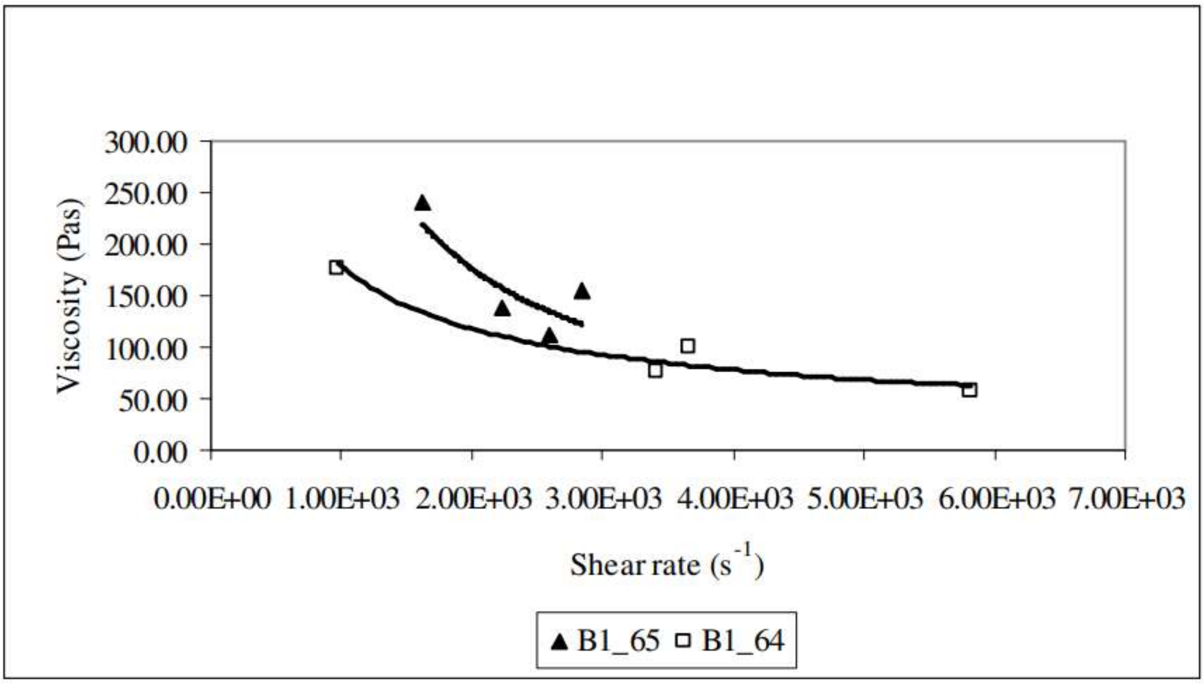
Rheological properties

In the domain of the MIM process, the rheological properties of the feedstock play a crucial role in ensuring consistent flow and uniform filling within the mold cavity. The evaluation of feedstock rheological attributes entails an examination of its viscosity, responsiveness to shear forces, and sensitivity to temperature variations (Khakbiz et al., 2005). As depicted in Figure 2, for both B1_65 and B1_66 at injection temperatures of 120°C, 130°C, and 140°C, the apparent viscosity displays a decrease with increasing shear rates and temperatures.

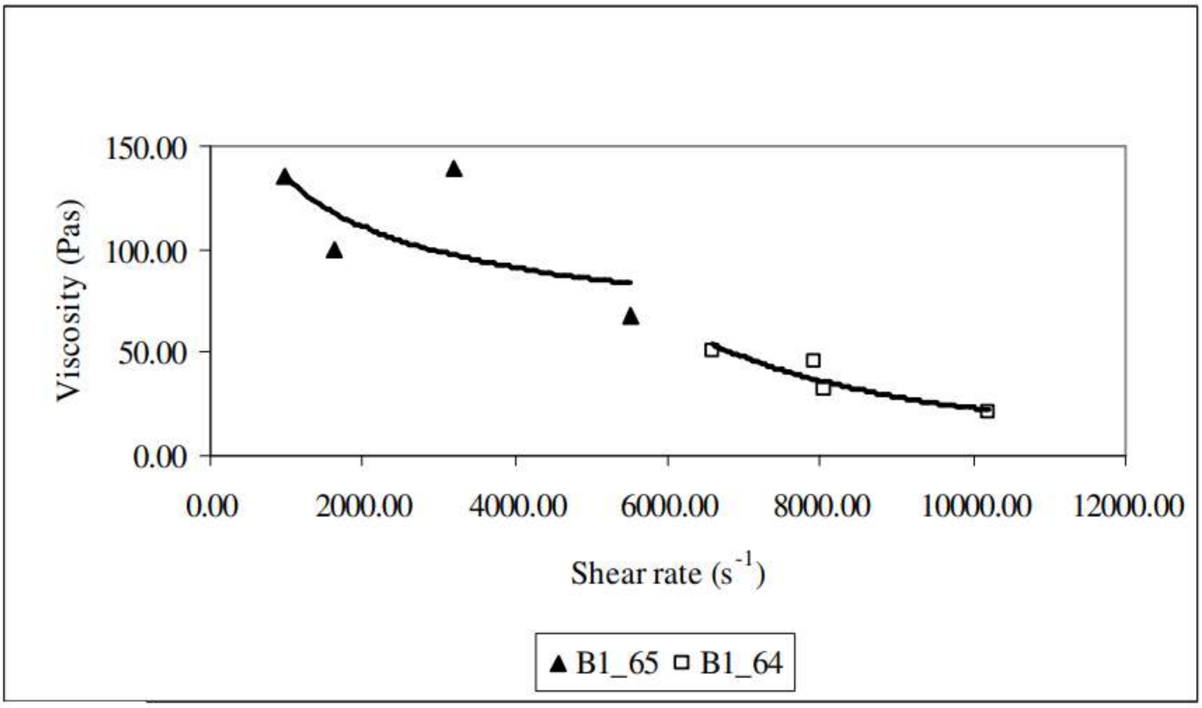
The data presented in Figure 2 highlights a distinct contrast between B1_65 and B1_66. Specifically, B1_65 exhibits a higher shear rate and lower viscosity compared to B1_66. Furthermore, as outlined in Table 3, the activation energy (E) associated with B1_66 surpasses that of B1_65. This elevated activation energy implies a heightened susceptibility to temperature variations during the injection molding process. This susceptibility can potentially lead to accelerated solidification within the mold (Jamaludin et al., 2008; Yimin et al., 1999). The interplay between temperature sensitivity (activation energy) and pressure sensitivity (flow behavior index) offers valuable insights into the stability and resilience of the feedstock throughout the injection molding procedure.



At 120^0



At 130^0



At 140^0

Figure2. Apparentv is cosity as function of shear rate.

Table 3 captures the flow behavior index (n), which encapsulates the observed pseudo-plastic behavior in the feedstocks. This index reflects how the feedstock responds to shear stress, while the flow activation energy assesses its sensitivity to temperature changes. Both feedstocks display the characteristic shear-thinning effect intrinsic to polymers, where higher shear rates correspond to lower apparent viscosity. Smaller flow behavior index values (n) signify greater shear sensitivity and more pronounced pseudo-plastic behavior. Importantly, specific molding defects, such as jetting, are often associated with low flow behavior index values (Yang et al., 2002).

Moreover, the moldability index confirms the superior performance of B1_65 compared to B1_66. This difference can be attributed to the reduced inter-particle friction within the B1_65 feedstock. Enhanced moldability is typically expected in feedstocks with lower powder loadings, although it correlates with the ultimate product's shrinkage. Within Table 3, both B1_65 and B1_66 demonstrate optimal moldability index values at 130°C.

The study conducted by Resende et al. (2001) concentrated on bi-modal mixtures of iron spherical powder (with particle sizes of 1 and 7 µm). Their findings revealed that these bi-modal distributions exhibited lower flow behavior index values at the same powder loading compared to mono-modal distributions. The presence of bi-modal distributions resulted in a decreased impact of powder loading on the flow behavior index. Additionally, the study indicated that mono-modal distributions displayed an increase in the flow behavior index with higher powder loading, while bi-modal distributions showcased the opposite trend. These conclusions correlate with the outcomes presented in Table 3, particularly for the 130°C condition.

Further insights are gleaned from the research by Li et al. (2007), who examined 17-4PH stainless steel powder in mono-modal distributions across varying powder loadings. Their work demonstrated that the shear sensitivity of the feedstock, within a powder loading range of 60 to 68% by volume, increased at an injection molding temperature of 135°C. However, the opposite trend was observed at a powder loading of 72% by volume. The phenomenon of shear thinning within MIM feedstock, as shear rates increase, arises from the alignment of powder particles and the orientation of binder molecules in line with the flow direction.

The rheological characteristics of feedstock within the MIM process have a substantial impact on achieving consistent flow and uniform filling within the mold cavity. The evaluation of these characteristics involves considering viscosity, shear responsiveness, and temperature sensitivity (Khakbiz et al., 2005). Figure 2 illustrates that the apparent viscosity decreases with increasing shear rates and temperatures for both B1_65 and B1_66. This effect is observed across injection temperatures of 120°C, 130°C, and 140°C.

The data in Figure 2 highlights that B1_65 exhibits a higher shear rate coupled with lower viscosity compared to B1_66. Moreover, as shown in Table 3, the activation energy (E) associated with B1_66 is higher than that of B1_65. This elevated activation energy indicates a greater susceptibility to temperature changes during the injection molding process, potentially leading to rapid solidification within the mold (Jamaludin et al., 2008; Yimin et al., 1999). The interaction between temperature sensitivity (activation energy) and pressure sensitivity (flow behavior index) provides valuable insights into the feedstock's stability and resilience throughout the injection molding procedure.

Table 3 provides insights into the flow behavior index (n), which captures the pseudo-plastic behavior evident in the feedstocks. This index indicates how the feedstock responds to shear stress, while the flow activation energy measures its sensitivity to temperature variations. Both B1_65 and B1_66 exhibit the characteristic shear-thinning effect intrinsic to polymers, where higher shear rates result in lower apparent viscosity. In alignment with this behavior, smaller flow behavior index values (n) indicate heightened shear sensitivity and more pronounced pseudo-plastic characteristics. It's noteworthy that specific molding defects, such as jetting, are often associated with low flow behavior index values (Yang et al., 2002).

Moreover, the moldability index reinforces the superior performance of B1_65 over B1_66. This distinction can be attributed to the reduced inter-particle friction within the B1_65 feedstock. While enhanced moldability is typically anticipated in feedstocks with lower powder loadings, its extent is proportional to the eventual product's shrinkage. Notably, both B1_65 and B1_66 exhibit optimal moldability index values at 130°C, as indicated within Table 3.

The study conducted by Resende et al. (2001) focused on bi-modal mixtures of iron spherical powder (with particle sizes of 1 and 7 μm), revealing lower flow behavior index values for the same powder loading compared to mono-modal distributions. Bi-modal distributions exhibit a diminished impact of powder loading on the flow behavior index. Additionally, the same study demonstrated that mono-modal distributions experience an increase in the flow behavior index with higher powder loading, while bi-modal distributions display the opposite trend. These findings harmonize with the outcomes presented in Table 3, particularly evident at the 130°C condition.

Further insights are gained from the work of Li et al. (2007), who explored 17-4PH stainless steel powder in mono-modal distributions across varying powder loadings. Their findings indicated that the shear sensitivity of the feedstock, within a powder loading range of 60 to 68% by volume, increased at an injection molding temperature of 135°C. Conversely, the reverse trend was observed at a powder loading of 72% by volume. The phenomenon of shear thinning within MIM feedstock, as shear rates intensify, is a result of the alignment of powder particles and the orientation of binder molecules in synchronization with the flow direction.

Table3. Rheological properties.

Flow activation energy,E(kJ/mole)	**B1_65(36.54)**		**B1_66(159.90)**	
Temperature	Flow behavior index(n)	Moldability index	Flow behavior index(n)	Moldability index
120	-0.949	156	0.043	90
130	0.4058	761	-0.0411	149
140	-1.055	171	0.7166	109

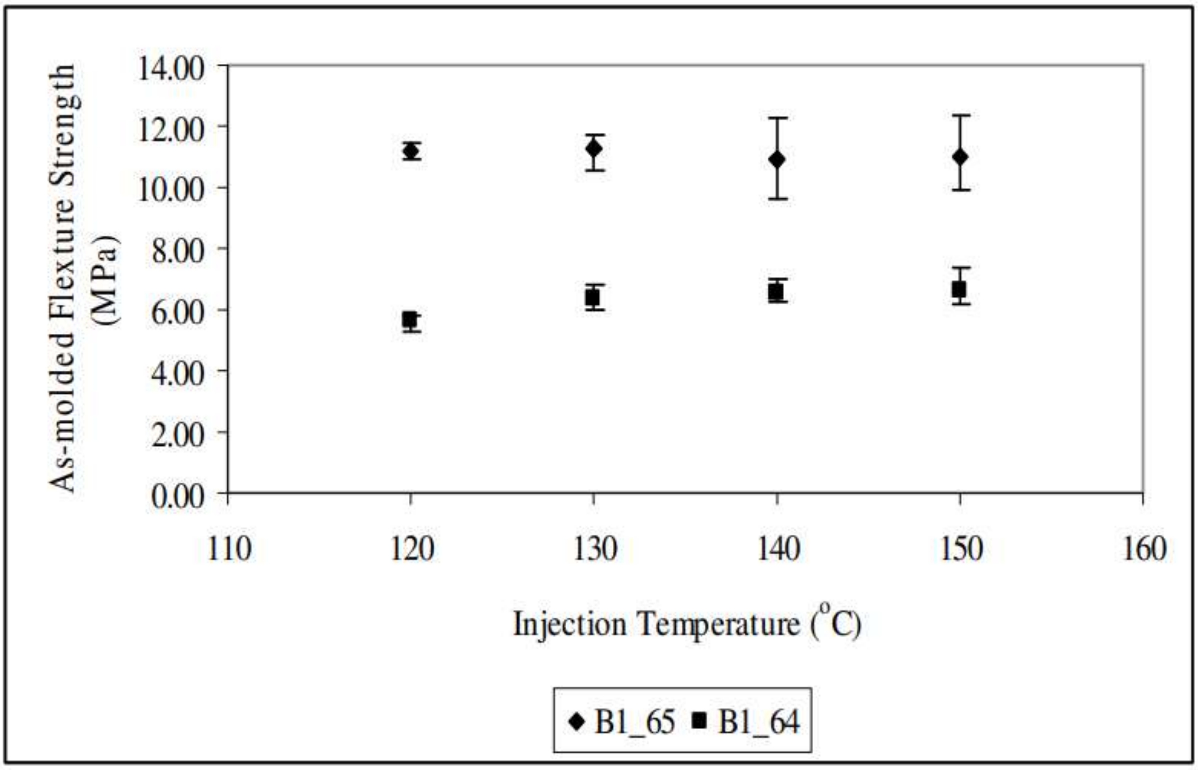


Figure3. Molded flexure strength.

Certainly, the study conducted by Li et al. (2007) brought to light that the activation energy exhibited an increase at a powder loading of 72% by volume. This finding suggests that at this specific powder loading, the feedstock demonstrated greater susceptibility to temperature fluctuations while showing diminished responsiveness to shear stress. This behavior stands in contrast to observations made at powder loadings below 68% by volume.

Furthermore, the insights gleaned from Table 3 offer additional depth. At a powder loading of 65% by volume, the feedstock's temperature sensitivity displayed an escalation, aligning with an increase in shear sensitivity at the same powder loading (130°C). However, this trend underwent a reversal at 130°C, as shear sensitivity decreased.
In the realm of highly filled materials, a conventional phenomenon unfolds wherein, beyond the yield point and low shear rates, particles tend to align themselves in the direction of flow. This alignment facilitates inter-particle movement, leading to a higher maximum packing fraction and reduced viscosity. The presence of pressure counteracts this effect, resulting in heightened viscosity. At elevated shear rates, particle layers cannot effectively form, causing particles to slide over each other, which prompts volume expansion. The concept of rheological dilatancy is associated with the increase in powder loading within the binder suspension, as further elucidated by Haunsnerova et al. (2006).

Properties of the moulded specimens

Following ejection from the mold cavity, the evaluation of green strength for each compact involved a three-point bending test utilizing the INSTRON 5567 machine, in accordance with MPIF Standard 15. Concurrently, density measurements were conducted using the Archimedes water immersion method, following MPIF Standard 42. The outcomes are visually depicted in Figures 3 and 4.
Figure 3 illustrates that the as-molded flexural strength of the high powder loading (B1_66) surpasses that of the low powder loading (B1_65). This distinction can be attributed to the interlocking mechanism formed among powder particles, creating a strong matrix binding. However, injection temperature exhibits minimal influence on the as-molded strength. Despite the elevated flexural strength associated with higher powder loading, the increased inter-particle friction challenges the moldability of feedstock with high powder loading. Omar (1999) aimed to enhance the moldability of such feedstock by increasing the stearic acid content in the binder composition, albeit requiring a prolonged period for green compact solidification prior to ejection.
Furthermore, Figure 4 emphasizes that B1_66, characterized by an elevated volume fraction, achieves a superior as-molded density compared to B1_65. This investigation confirms that the injection temperature's impact on the as-molded flexural strength of either feedstock remains insignificant.
However, the success of the molding process significantly hinges on the feedstock's rheological properties at different injection temperatures. As shown in Table 4, B1_66 faces challenges in fully filling the mold cavity at 120°C and 130°C, in contrast to B1_65, which achieves successful injection molding even at the lower temperature of 120°C.
The efficiency of the injection molding process is intricately linked to the rheological attributes outlined in Table 3. Notably, B1_66 exhibits a substantially higher activation energy (E) of 159.90 kJ/mol, surpassing B1_65's value of 36.54 kJ/mol. This elevated activation energy renders B1_66 susceptible to premature solidification within the mold cavity, often occurring before reaching the downstream regions within the cavity, especially under low injection temperatures.

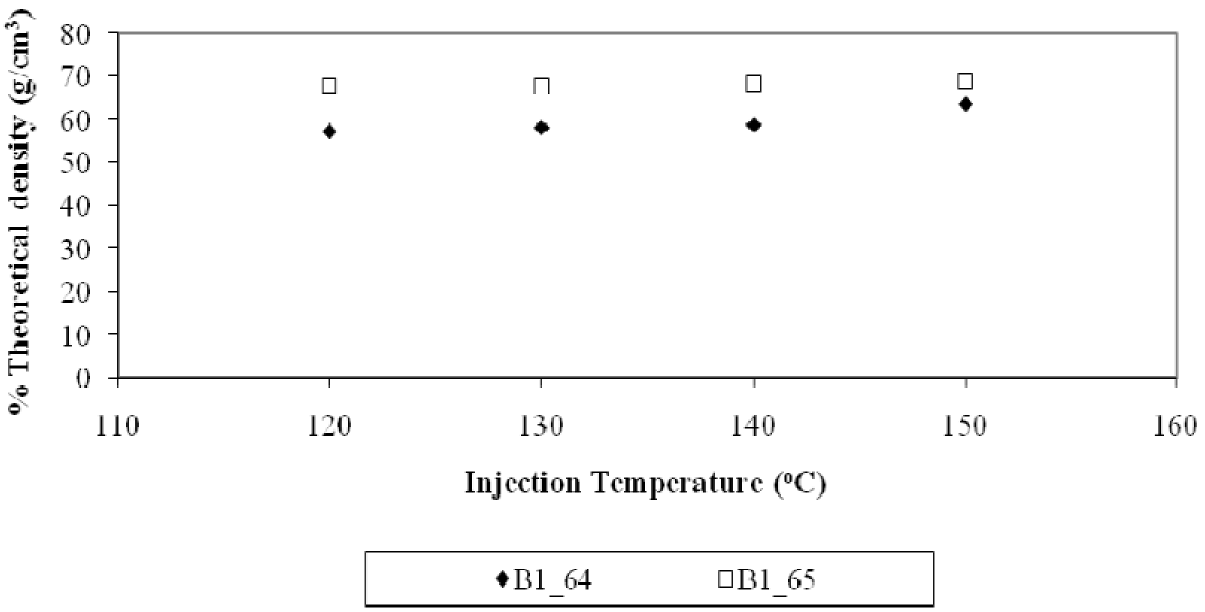


Figure4. As-moldeddensity.

Table4. Remarks for injection molding ofB1_66.

T	**Remarks**
120	Incomplete filling. Only the up stream and middle part filled
130	Incomplete filling. Only the up stream and middle part filled
140	Complete part produced with weldline at the downstream
150	Complete part produced with weldline at the downstream

CONCLUSIONS

The achievement of effective injection moulding for MIM feedstock hinges upon the inherent rheological characteristics of the feedstock itself. Notably, when dealing with a bi-modal powder distribution feedstock featuring heightened powder loading, temperature variations tend to exert a more pronounced influence compared to alterations in the powder quantity. In the context of the resulting product, the flexural strength and density of the as-moulded component derived from the bi-modal powder distribution feedstock exhibit a direct correlation with the amount of powder that is loaded into the process.

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