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AN ANALYSIS OF RHEOLOGICAL PROPERTIES OF INCONEL 625 SUPERALLOY FEEDSTOCKS FORMULATED WITH BACKBONE BINDER POLYPROPYLENE SYSTEM FOR POWDER INJECTION MOLDING

Binder formula is one of the most significant factors which has a considerable influence on powder injection molding (PIM) processes. In the study, rheological behaviors and properties of different binder systems containing PIM feedstocks, Inconel 625 powder commonly used in space industry, were investigated. The feedstocks were prepared 59%-69% (volume) powder loading ratios with three diversified binder systems by use of Polypropylene as backbone binder. The average particle size of the Inconel 625 powder used was 12.86 microns. Components used in the binder were mixed for 30 minutes as dry in three dimensional mixing to prepare binder systems. Rheological features of the feedstock were characterized by using a capillary rheometer. Viscosities of the feedstocks were calculated within the range of 37.996-1900 Pa.s based on the shear rate, shear stress, binder formula and temperature. “*n*” parameters for PIM feedstocks were determined to be less than 1. Influences of temperature on the viscosities of the feedstocks were also studied and “*Ea*” under various shear stresses were determined within the range of 24.41-70.89 kJ/mol.

Keywords: Powder Injection Molding, Feedstock, Rheological properties, Inconel 625

1. Introduction

Powder Injection Molding (PIM) is an advanced manufacturing technology for the production of complex, high volume, and net-shape components [1]. PIM is referred as Metal Injection Molding (MIM) when dealing with metal or alloy powders and as Ceramic Injection Molding (CIM) when ceramics are processed [2].

PIM is a new process, which combines traditional powder metallurgy (PM) and plastic injection molding technologies [3]. PIM technology has been successfully applied in twenty years to a large number of metallic materials, rising in numbers every year. Nickel base superalloys seem to be good candidates to be processed by this technique, providing cost savings when compared to other techniques, such as investment casting [4]. Superalloys are a specific type of heat resisting alloys based on cobalt, iron or nickel, displaying a heat corrosion resistance, high strength at increased temperatures and satisfying oxidation resistance. These specific technical features make them usable for various applications in the petrochemical industries, aerospace, medical and automotive; on the other hand, their high toughness and strength make them difficult to shape through forging or machining. In today's industry, casting is frequently utilized to produce superalloys. Nevertheless, their products generally have the characteristics of element segregation and

low dimensional tolerance [5]. Nickel superalloys, such as Inconel 625, were developed to withstand the intense conditions present in gas turbine engines while preserving relatively high mechanical properties. High performance metallic components made of this alloy can be fabricated using a wide variety of industrial processes and are typically found in the combustion chamber, fuel injection system, and gas generator assemblies [6,7]. Costly manufacturing processes, such as machining, investment casting, and hot isostatic pressing, have been used for the fabrication of small complex shaped parts so far [7]. Advances in technology have allowed the production of high performance materials like superalloys with high melting points. PIM is an alternative production method for Inconel 625 which is a specific material group [8]. Complex geometry design, minimum material loss, quick scale-up response time, and significant cost savings on moderate or large production volumes can be enabled by this production method [9]. PIM process begins with the preparation of feedstock. Feedstock preparation and binder removal are critical processing steps and play a central role in PIM part production. Therefore the development of feedstocks for PIM is the area of the technology with the greatest improvement potential [10]. Mouldability is defined as gauge of rate and ease of shaping of feedstock for a given characteristic [11]. Binder is a key component, which provides the powder with the flowability and formability neces-

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sary for PIM [12]. The rheological properties are significant in the PIM step, since they involve the flow of the feedstock during that step [13]. Rheological analysis can be made to quantify the stability of the feedstock for accomplishing a successful manufacturing process [14]. Therefore, parameters such as viscosity, activation energy, and flow behavior index are important in the rheological investigations.

No study about flow behavior of feedstock produced with Inconel 625 alloy was found in the literature. In this study, three different binding systems containing polyethylene glycol (PEG), paraffin wax (PW), carnauba wax (CW), and stearic acid (SA), were prepared using polypropylene (PP) as backbone binder. These binder systems mixed with nickel base Inconel 625 superalloy powder to obtain feedstocks with different powder loading rates (59%-69% by volume). Rheological properties, such as viscosity, shear rate, shear stress, flow behavior index, and activation energy of obtained feedstocks, were investigated depending on temperature and pressure.

2. Materials and method

2.1. Powder and binder characteristic

The metal powder used in these experiments was gas atomized Inconel 625 superalloy with a pycnometer density of 8.44 g/cm^3 . The fine graduated metal powder was provided from SANDVIK OSPREY Corporation. Fig. 1 shows the powder particles, which are usually in spherical shape. Chemical composition of the utilized Inconel 625 Superalloy powder is given in Table 1. The size distribution of these powders was measured using a laser light scattering machine (Malvern Master Sizer-E) and the measurement results are given in Fig. 2.

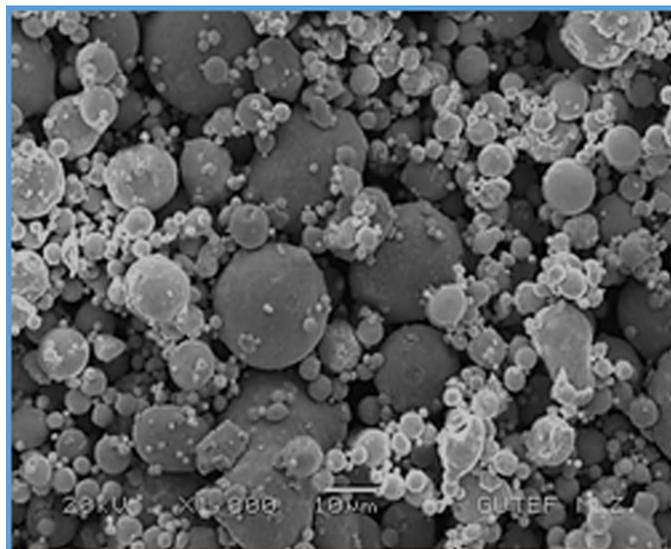


Fig. 1. SEM image of gas atomized Inconel 625 Superalloy powder

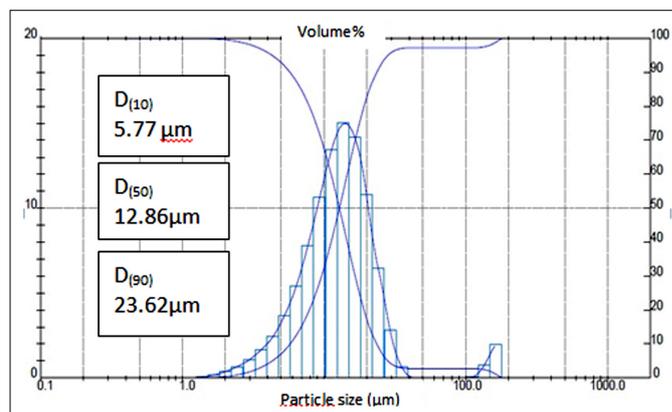


Fig. 2. Particle size distribution graphic of Inconel 625 powder

TABLE 1

Chemical composition of Inconel 625 Superalloy used in the experiments

Powder		C	Mn	Si	P	Cr	Mo	Fe	Nb	Co	Al	Ti	S	Ni
Inconel 625	Min	—	—	—	—	20.0	8.0	—	3.15	—	—	—	—	B
	Max	0.10	0.50	0.50	0.015	23.0	10	5.0	4.15	1.0	0.40	0.40	0.015	B

Using the system with multiple binding components has been reported to minimize the errors that can occur in the binder removal step [15]. Therefore, three different binder formulas have been prepared for experimental studies. The binders that are used in this experiment contain several components: polypropylene (PP) backbone polymer, polyethylene glycol (PEG10000-8000), paraffin wax (PW), carnauba wax (CW) filler, and stearic acid (SA) lubricant. The filler is utilized to fill the clearances among the powder particles, and thus it decreases the viscosity of the feedstock, simplifying PIM. The lubricant corroborates the adhesion among powder and binder, reducing the agglomeration of the powder. The backbone polymer supply the basis durability of the green parts [16]. Table 2 presents the material features of

the components of the binders. Table 3 presents the formulas of the binders in feedstocks F1, F2, and F3.

TABLE 2

Some properties of binder components

Component	Density (g/cm^3)	Melting point ($^{\circ}\text{C}$)
Polypropylene	0.85	189
PEG 10000	1.200	58-63
PEG 8000	1.204	60-63
Paraffin Wax	0.90	90
Carnauba Wax	0.97	98-112
Stearic Acid	0.84	67-69

TABLE 3

Compositions of binder formulations

Binder formulation	Composition	Content (wt. %)
F1	PEG 8.000	68
	Polypropylene	27
	Stearic Acid	5
F2	Paraffin Wax	67
	Carnauba Wax	12
	Polypropylene	20
	Stearic Acid	1
F3	PEG 10.000	25
	Paraffin Wax	33
	Carnauba Wax	11
	Polypropylene	30
	Stearic Acid	1

2.2. Preparation of feedstocks

Feedstock consists of two subsequent steps, namely, dry mixing and heat mixing. Binder formulations given in Table 3 were prepared by blending as dry in a mixer (Turbula) for 30 min. in the first step. The viscosity of the prepared binder formulas was determined to range from 10 to 30 Pa.s. Feedstock preparation for PIM is a critical stage since shortcomings in the quality of the feedstock cannot be improved by ensuing processing regulating. Thus, it is crucial that the feedstock is homogeneous and free of binder – powder dispersion or particle segregation. According to Supati et al., failure to separate the powder inappropriate or uniformly rheological behavior of the feedstock will trigger molding defects, such as warping, fractures, or pores, that will cause to unequal shrinkage or distortion in the sintered samples [17]. In the second step: the binder was heated at 190°C in the heat stirring system. At this temperature PP was completely melted and in stirring with thermoplastic which provided an appropriate condition for being stirred with metal powder. Thereafter metal powder was stepwise supplemented to obtain a feedstock with 59%-69% vol. powder loading. The stirring process sustained for 30 min to achieve homogeneous feedstocks.

2.3. Thermal analysis

Differential thermal analysis (DTA) and thermogravimetric analysis (TGA) are utilized to analyze the qualification of the feedstock. DTA values of feedstock yields the melt temperatures of the binders, which are utilized as references in setting the mold and barrel temperatures. The thermogravimetric profile provides knowledge on the decomposition temperature range of the binder components [18] and the degree of weight loss [16]. DTA and TGA thermograms of the feedstock specimens were noted using Exstar S11 7300 Model Synchronous TGA/DTA analyser in the existence of argon atmosphere up to 500°C at a heating rate of 10°C/min.

2.4. Rheological measurements

Rheological behavior of the prepared feedstock was calculated by using a Capillary Rheometer (ASTM D 1238 and TS EN ISO 1133). Experiments were performed in which the specimens were extruded throughout a die with 8 mm length and 2.095 mm diameter. The piston speed was diversified to get varied shear rates and the corresponding pressure fall measurements throughout the length of the die were used to calculate the shear stress. Viscosities were determined depending on the changing shear stress and shear rate. Characteristically, shear rate in PIM process varies from 100 to 100.000 s⁻¹. In this shear rate range, viscosity of a covetable feedstock at molding temperature must be lower than 1000 Pa.s [19]. In the present study, rheology of the feedstocks was carried out among the range of 110-190°C temperature and 10.8-135.0 kPa shear stresses.

3. Results and discussion

3.1. TG and DTA analysis of the binder systems

DTA and TG analyses of the binders F1, F2, and F3 heating curves are represented graphically in Figs. 3 and 4. Fig. 3 depicts the locations of the peaks that come up to these three binders are permanently indicating that each component in the binders is consistent. A few endothermic peaks are displayed for each heating curve for F1, F2 and F3 in Fig. 3. The melt temperatures of PEG 10000, PEG 8000, SA, PW, CW, and PP are 59, 63, 65, 89, 93, and 187°C, respectively. However, the corresponding melt temperatures are presented in Table 2. Distinctive melting temperatures of the binder systems ensure that when one binder component (wax) has melted, the remainder binder component (polymer) act as a backbone support, hold the shape of the moulded part [20].

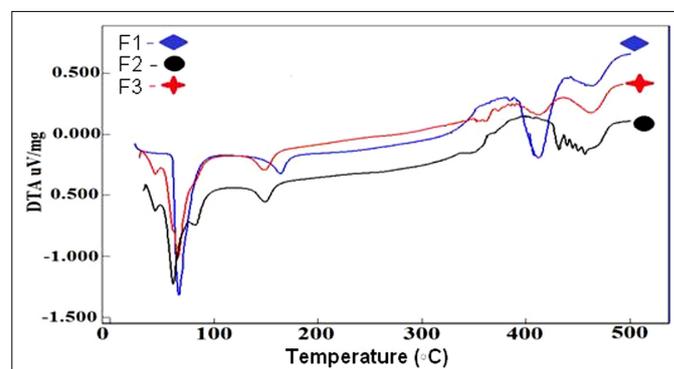


Fig. 3. DTA curves of the binders F1, F2 and F3 (heating rate: 10°C/min; atmosphere: Ar)

TG results of the binders are shown in Fig. 4. TG analysis was carried out on feedstocks for the prediction of thermal decay of binder components being subjected to higher temperatures. The binders degrade at 240-450°C. This knowledge is beneficial

in setting the thermal debinding parameters [16]. Consequences of the TG demonstrate that the percentage weight loss of the binder is too close to the rate before mixing.

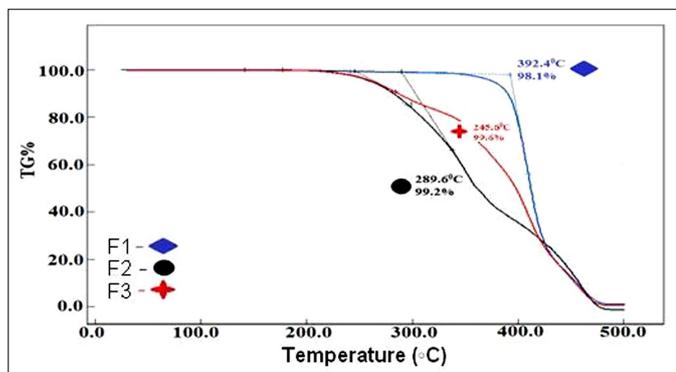


Fig. 4. TG results of binders F1, F2 and F3 (heating rate: 10°C/min; atmosphere: Ar)

When the TGA curve of the novel binding system F3 is analyzed, it is obtained that the components left this novel system step by step similar to the F1 and F2 systems. It should be taken into account that the amount of backbone binder PP in F3 is more than that of F1 and F2 (Table 2). The amount of components used as backbone binder should be higher to carry the green part without distortion until sintering stage in the PIM process [21]. From this point of view, F3 binder system should have a higher green strength compared to F1 and F2 after the molding process. Rheological processes are studied between 110 and 190°C according to the results of the TG and DTA analyses.

3.2. Effect of temperature on viscosity

The effect of temperature changes was examined on the viscosity of the feedstock. The correlation of viscosity with the temperature is considerable in PIM process, as well [22]. The viscosity was decreased depending on the increased temperature [23]. If the viscosity is very susceptible to the temperature alteration, a little undulation of temperature through molding will production viscosity variations, making stress concentration in the molded part, eventuating in distortion and fracture [24].

The effect of temperature on the viscosity (η) of feedstocks was also investigated and results obtained are given in Figs. 5-7. From the slopes of these curved lines by use of the Arrhenius equation, flow activation energies (E_a) of the feedstocks were calculated and submitted in Table 4.

The effect of temperature on viscosity can be denoted by the Arrhenius kind equation [23]:

$$\log \eta = Ea/R(1/T) \quad (1)$$

E_a is the flow activation energy; η is the viscosity; T is the temperature; R is the gas constant. The value of E_a denotes the influence of temperature on the viscosity of the feedstock. Providing that the value of E_a is low, the viscosity is not so

susceptible to temperature change. Thus, a little fluctuation of temperature through molding will not result in instantaneous viscosity alteration [24]. A sudden viscosity alteration could give rise to undue stress concentrations in molded parts, eventuating in distortion and fracture [25].

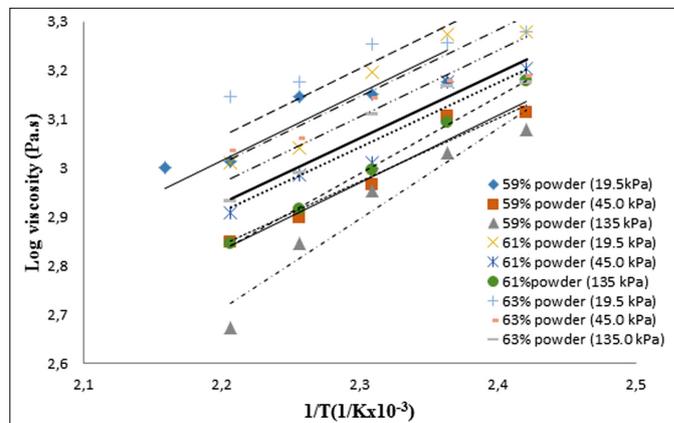


Fig. 5. Change of viscosity depending on the temperature of the different powder loading rates and shear stress for F1 formula

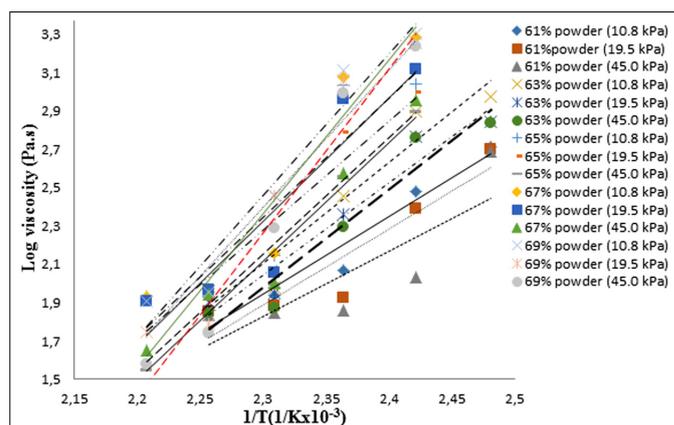


Fig. 6. Change of viscosity depending on the temperature of the different powder loading rates and shear stress for F2 formula

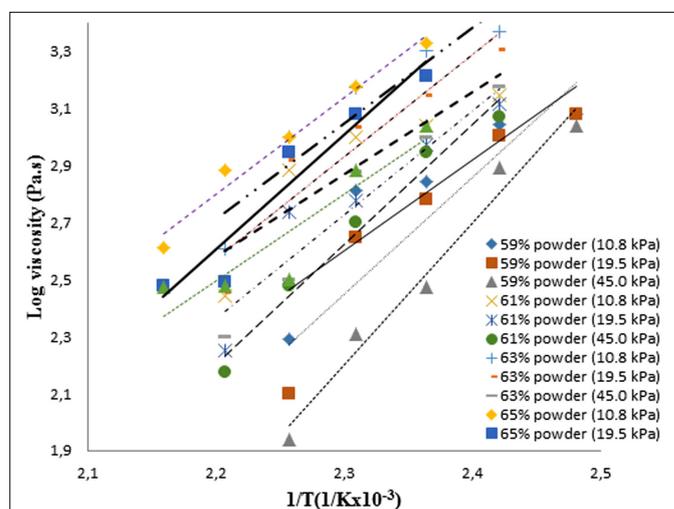


Fig. 7. Change of viscosity depending on the temperature of the different powder loading rates and shear stress for F3 formula

As clearly seen in Figs 5-7, viscosity decreased with increasing temperature. Viscosity change has been reported depending on the applied temperature and shear stress by [26].

Flow could not be obtained in feedstocks prepared with F1 between 110 and 190°C when the powder load ratio is more than 63%. Dependent change of E_a value with changing powder load ratio and shear stress is determined when the flow activation values given in Table 4 are analyzed. As shown in Table 4, the E_a values of the feedstocks variation between 29.7 and 70.8 kJ/mol. The flow activation energy is at the lowest value of 24.4 kJ/mol for the F3 containing feedstock with 65% powder loading (τ : 19.5 kPa). The flow activation energy rises with the powder loading increased from 63% to 69%. Thus, powder loading may be favored into the range from 61% to 65%. Karatas et al. avowed that low viscosity with low activation energy is a fundamental requirement for a good PIM [27].

TABLE 4

Change of E_a depending on the binder formula and powder load rate

	Powder load ratio (% vol.)	E_a (kJ/mol)	
		τ : 19.5 kPa	τ : 45.0 kPa
F1	61	52.39	52.68
	63	45.48	46.86
F2	61	32.90	28.39
	63	40.15	42.38
	65	47.36	53.18
	67	53.35	50.60
F3	69	66.56	70.89
	61	33.24	34.99
	63	30.70	34.79
	65	24.41	25.17

F3 containing feedstocks show the lowest E_a values. These consequences are also in accordance with the flow behavior index (n) as seen in Table 4, which is due to the carnauba and paraffin wax content of F3. It has been stated by Sotomayor that, for feedstocks with several powder loadings, a decrement of E_a value is determined as metallic content in the blend rises, which could be closely associated to a better thermal conductivity of the specimens. Hence, the feedstock with maximum powder content is the best preference from the viewpoint of temperature susceptible for PIM applications [24]. As reflected in Table 4, the E_a values of F3 containing feedstocks change from 34.9 to 24.4 kJ/mol and this range indicates the lowest values given in Table 4.

3.3. Flow Behavior Index

Flow behavior index (n) was calculated in the range between 110 and 180°C of feedstocks containing powder values in varying proportions (59%-69%). “ n ” values were determined in the range between 0:50 and 1:10. Rheological characteristics of feedstocks containing 63% powder were evaluated in the

range of 130-180°C and the results are represented in Fig. 8-10. We determined n values from the slope of the logarithmic plots of shear stress against shear rate. The consequences for varied feedstocks in the range of 130-180°C are presented in Table 5.

The whole of feedstocks prepared follow a pseudoplastic behavior. The basis feature of a pseudoplastic fluid is that viscosity diminishes with the rise of shear rate. This type of fluids adapts excellent to the power law:

$$\tau = K\dot{\gamma}^n \quad (2)$$

Where $\dot{\gamma}$ is the shear rate, k is an invariant, τ is the shear stress and n is the flow behavior index [26]. In the event of a pseudoplastic behavior, $n < 1$. This equivalence indicates shear dependence of viscosity. Contrary to Newtonian fluids, viscosity of non-Newtonian fluids varies with the increase or decrement of shear rate. Dilatant fluids ($n > 1$) display a rise of viscosity on meeting increased shear rate. In this case, powder and binder separate under a high pressure. Meanwhile, in pseudoplastic substances ($n < 1$), the viscosity decreases with the rise of shear rate [19].

The value of n demonstrates the rate of susceptible of viscosity versus shear rate. The lower the value of n is, the more susceptible the viscosity to shear rate gets. During the PIM process, a pseudoplastic behavior is desirable and, thus, a decrement in viscosity with an increase of shear rate take place [24]. Smaller n of feedstock determines a higher shear susceptible and more pseudoplastic behavior of the feedstocks [28].

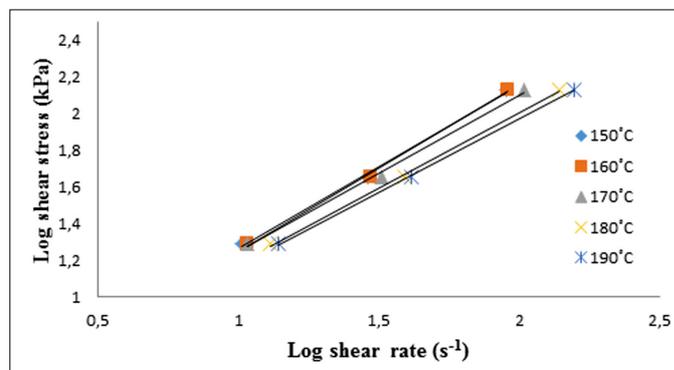


Fig. 8. The diagrams of Log Shear rate-shear stress for powder loading ratio at 63% and 37% F1

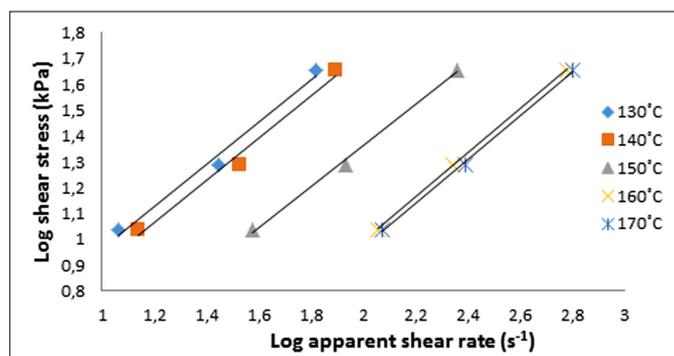


Fig. 9. The diagrams of Log Shear rate-shear stress for powder loading ratio at 63% and 37% F2

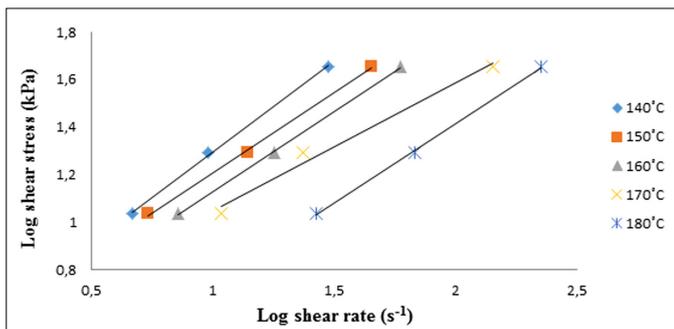


Fig. 10. The diagrams of Log Shear rate-shear stress for powder loading ratio at 63% and 37% F3

The powder loading or binder content in feedstock is one of the most crucial factors that have a significant effect onto PIM process. Viscosity of feedstock is very susceptible to the solid content. Concordantly, we can highlight that viscosity increases as the powder loading rises as it was expected. When rising the powder loading, the feedstock shows a more pseudoplastic behavior [24].

TABLE 5

Flow behavior index consequences of feedstocks

Binder formulation	Temperature (°C)	<i>n</i> , (63% Powder load)
F1	150	0.892
	160	0.907
	170	0.855
	180	0.819
	190	0.799
F2	130	0.821
	140	0.816
	150	0.793
	160	0.856
	170	0.852
F3	140	0.761
	150	0.674
	160	0.679
	170	0.537
	180	0.671

“*n*” parameters were indicated for feedstocks to be less than 1, which shows that both suitability and a pseudoplastic behavior for PIM. From the shear rate-shear stress curves, and it was obvious that all the feedstocks were fundamentally pseudo-plastic, but the flow behavior index values are different. F3 containing feedstock has an “*n*” values of 0.5, less than the values of 0.852 for F2 containing feedstock and 0.855 for F1 containing feedstock (170°C). Thus, F3 containing feedstock demonstrates greater pseudo-plasticity compared with F2 and F1. Hence, F3 containing feedstock was the best feedstock for PIM, since it has better rheological stability and enormous pseudoplasticity. High quality products with externally deficiencies or less defects arising from temperature incline and shear rate variation in the injection molding cavity could be anticipated to be able to be produced.

3.4. Fluidity

The evaluation of the feedstock rheological properties is depended on the viscosity and its temperature susceptibility and shear susceptibility. Fig. 11 and 12 show the variation of viscosity as increasing shear rate at temperatures 170°C and 180°C at 63% and 65% powder loading, respectively. Consequences display that all the feedstocks exhibit a pseudoplastic behavior or shear thinning, which is desirable for PIM.

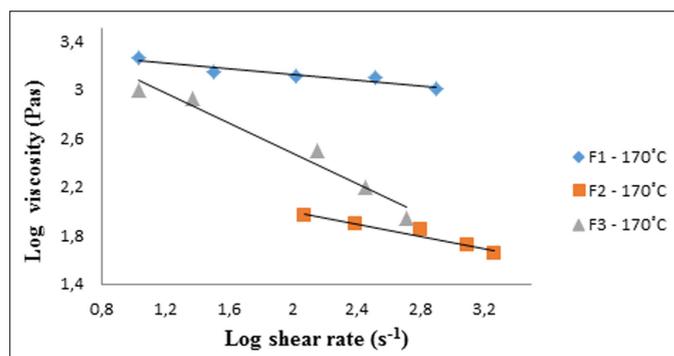


Fig. 11. The diagrams of Log shear rate-log viscosity for F1, F2, F3 containing feedstocks and constant powder rate-temperature (63% powder – 170°C)

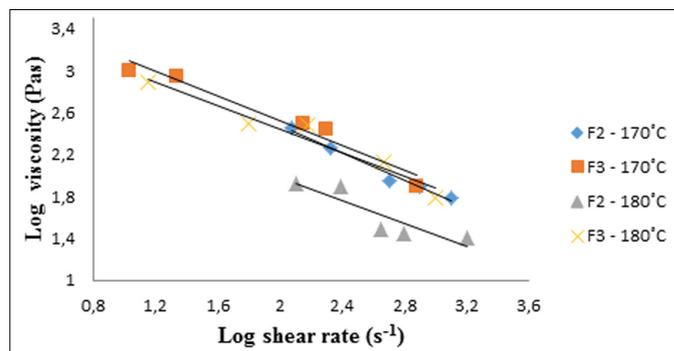


Fig. 12. The diagrams of Log shear rate-log viscosity for F2, F3 containing feedstocks and constant powder rate and different temperature (65% powder – 170, 180°C)

Injection molding bases on the melt viscous flow into the die cavity, and this requires specific rheological characteristic features. As shown in Figs. 11 and 12, the viscosity increased with diminishing temperature at the low shear rates. This situation was caused by the components in the binder formulas. Furthermore, the powder loading of the feedstock had visible influence in the viscosity [29]. Raza et al. suggested that for successful metal injection molding the shear rate ranges between 102 and 105 s⁻¹ and the maximum suitable viscosity for feedstock is 103 Pa at the molding temperature [30]. Considering the viscosity values recommended for PIM process, less than 1000 Pa s in the shear rate range between 100 and 1000 s⁻¹ [31]. So as to inject the feedstock into injection molding machine, the viscosity and shear rate should be 1000 Pa.s and in the range of 100 to 10000 s⁻¹ respectively [32]. From the results, it is clear

that the feedstocks for F1, F2 and F3 formulation are suitable for Inconel 625 injection molding.

The minimum shear rate values ($13.228\text{--}150.51\text{ s}^{-1}$) are found in feedstocks prepared with F1; conversely, the maximum values ($16.871\text{--}643.078\text{ s}^{-1}$) are obtained from F2 used feedstocks. The viscosity values for F1 and F2 containing feedstocks vary in the range of $898.741\text{--}1474.304$ and $70.121\text{--}641.940\text{ Pa s}$, respectively. High amounts of PW and CW in F2 and F3 are thought to cause high flowability.

Hidalgo et al. suggested that the carnauba wax allows solid load to improve values over 70%. This fact is due to the molecular constituents of the carnauba wax that makes this wax behave as a surfactant in the mixing process and improving the wettability binder-powder [33].

Viscosity values of the novel binder system F3 vary between 109.996 and 1499.777 Pa.s . Feedstocks which contain F3 have viscosity values between the viscosity values of F1 and F2 containing feedstocks (see Fig. 11). This situation is thought to be related with the effectiveness of PW and PP in the binding system. When used as a backbone binder in a binding system, it is known that PP increases the green density after molding; on the other hand, it decreases the flowability [23].

The superiority of feedstock's flowability produced with F3 system over F1 and F2 and high PP content make F3 system a good candidate for a perfect injection molding process. The change of flowability according to binding system is shown in Table 6 at constant powder load ratio (63% by volume) and constant temperature (170°C). While the flowability value of feedstock prepared with F2 is the highest, it is the lowest for F1 as seen in Table 6. It is thought that the high amount of PW and CW in F2 is the reason for high flowability. PW enables higher flowability for feedstocks in PIM process [27]. Flowability values of feedstocks prepared with F3 are close to the values of F2 feedstocks.

TABLE 6

Fluidity values of feedstocks depending on the binder formulas

Binder + powder, (170°C)	$1/\eta$, ($\times 10^{-3}\text{ Pa}^{-1}\text{ s}^{-1}$)
F1 + (63% powder)	1.23 (γ : 138.4)
F2 + (63% powder)	5.07 (γ : 228.8)
F3 + (63% powder)	3.16 (γ : 142.6)

4. Conclusions

PP derivative components used as backbone binder take on a critic task on the process of powder injection moulding. It provides green density which is necessary until sintering process after powder injection moulding and debinding processes. F3 binder improved within the scope of this study is a binder formula which has the highest PP rate and it is identified that it provides the green density which is essential for samples acquired from the studies of rheology. In anticipation of gathering superior characteristics of F1 and F2 binder systems, F3 binder

system is enhanced as genuine binder formula. The findings are summarized as noted below:

- All feedstocks exhibit a pseudo-plastic or shear thinning behavior and viscosity of feedstocks was decreased depending on the increased temperature.
- The increase of powder load rate increases the viscosity of Inconel 625 feedstock at low shear rates for F1, F2, and F3. As the shear rate and temperature increase, viscosity is diminished.
- The use of a PP backbone polymer consequences in a suitable flow behavior of the PIM feedstock and thereby enhances the quality of the injection molding. Also, a PP backbone polymer stabilizes spiral flow and hence improves the quality of green parts.
- Data propounded that the formulated binder system F3 (consisted of 25 wt.% of PEG 10000, 11 wt.% of Carnauba wax, 30 wt.% of Polypropylene, 33 wt.% of paraffin wax, and 1 wt.% of SA) procures a perfect wax-based binder system.
- This study has revealed that binder content (especially backbone polymer) strongly influences rheological properties of the Inconel 625 feedstocks.

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