DEVELOPMENT AND ANALYSIS OF FEEDSTOCKS TO IMPROVE INJECTION FOR SURGICAL INSTRUMENTS*

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Abstract

Micrometric surgical components typically have high geometric complexity and stringent regulatory specifications. These components need to have excellent mechanical properties and a high demand of products. Besides these factors, it is necessary to maintain a low manufacturing cost, performing essential research on new manufacturing technologies. Metal Injection Molding (MIM) is a process that has been explored , providing an interesting alternative for making medical devices. The research in the production of microcomponents obtained for MIM is gaining momentum and prominence, due to low cost and high productivity of this process. This paper is a study and development of feedstocks with AISI 316L alloy , which is known for its biocompatibility. Four feedstocks with 61%vol. of metal powder were analyzed by capillary rheometry and DSC/TGA.

Keywords: Feedstocks; Metal Injection Molding; AISI 316L alloy; Rheological characterization.

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1 INTRODUCTION

1.1 Powder Injection Molding

The Metal Injection Molding (MIM) process is a type of forming that is often used and highly disseminated in the field of mechanical forming of ceramic and metal materials. The technique consists of mixing a small fraction of polymer components with inorganic powders, where the polymer is a means of conduction that allow the mixture to become fluid and flow into a cavity shaping the component. After the component is injected, it is necessary to remove the polymer by chemical and thermal extraction and then to send this component on to the sintering stage, ensuring the final mechanical characteristics of this component.

1.2 Biocomponents Manufactured by the MIM Process

The market for injection of metal microcomponents has grown significantly in the last 15 years, and is beginning to inject biomedical/biocompatible materials. Stainless steel AISI 316L is becoming an excellent candidate for applications of microcomponents that are used for medical purposes, since this alloy has good mechanical characteristics, excellent corrosion resistance and biocompatibility properties [1,2].

Due to the properties already described above, the AISI 316L alloy was used to develop injectable feedstocks because it has mechanical, thermal, esthetic, sanitary, bacteriological and biocompatible characteristics [3] needed for the use proposed in the present work, since the components made will be used inside a patient's body [4].

1.3 Injectable Mixture

Feedstock is the consequence of the combination of properties of powder (metal) with those of the polymer system (vehicle), a binding agent (surfactant) and the proportion among these three components so that the injectable mass will be homogeneous. Besides the already mentioned factors, the mixing method, the shape and size of the grains are factors that contribute directly to the mixing process [5].

The feedstock morphology provides an adequate feeding flow through the hopper into the injector machine barrel and through a well regulated process good chemical, physical, microstructural and size characteristics are achieved for the part, also ensuring good process productivity [6]. These factors show the importance of the analysis and characterization of the feedstock properties.

1.3.1 - Organic fraction

Regarding structural Concerning structural polymers, it is known that shorter molecular chains ensure less viscosity, which makes it easier to process and fill cavities in the molds, besides reducing the debing time later on [7]. Structural polymers must make flow easier in the injectors with a good response of the shear rate imposed by the screw. This polymer must have good wettability, allowing the incorporation of a greater inorganic load and improving the capillarity effect, making thermal removal easier. Thermal expansion and change of volume during organic fraction fusion have a marked influence on the integrity of biomedical components [8].

The function of the thermoplastic polymer is to make the green part mechanically resistant and to maintain its dimensional integrity during the chemical and thermal extraction stages of the binders [9].

The auxiliary polymers are characterized by their low molecular weight, and their main function is to make the mixing process easier, since it alters the wettability of the interface between the metal particle and the thermoplastic. Besides, it helps reduce the temperature needed for the mixture fusion as a whole [2].

Surfactants are responsible for promoting the interaction of the polar molecules with the non-polar molecules, forming a bridge between the organic fraction and the inorganic fraction. fraction.

1.3.2 Proportionality between powder and binder

The injection mixture needs to be formulated with a correct balance between the inorganic fraction and the organic fraction, since the proportion between the volume of powder and the volume of binder is a determining factor in processing the component. In mixtures where there is a high volume of metal powder, viscosity is high, which may impair the injection stage, causing voids due to the lack of binder and resulting in defects in the later stages. In loads where excess binder is used, this may cause the separation of the organic fraction of the metal particles during the component injection, which may result in heterogeneity in the densification of the part. The optimal load of the metal powder volume used should be slightly smaller than the critical volume. The range where complete filling of the spaces between the particles occurs due to the minimal binder layer, so that the greatest shear occurs during the injection processing and the greater approaching of particles for the later sintering process, is called optimal loading range [10, 11].

Studies performed show that the volume of metal powder has a direct effect on the geometry and dimensions of the components injected during the extraction phase of the organic fraction of the feedstock [11]. Other factors such as size, shape and powder morphology have great influence on the composites, normally on the metal powders with a spherical morphology and smaller size, and tend to generate a better result [12].

The processing capacity of the injectable mixture is closely connected to the choices made in the system formed between the organic fraction and the inorganic load. The binder formed by thermoplastic polymers and waxes will directly influence viscosity, but another factor that strongly influences this parameter is the volume of metal powder used in relation to the binder-polymer system used. In practice the commercially found products have a powder fraction that varies from 50 to 70% of the load volume [13].

1.4 Rheological Assays on Injectable Feedstocks

Viscosity determines the internal resistance of a fluid to flow, and this is the resistance that a material presents to become deformed over the action of shear stress applied [14]. This characteristic is extremely important for injection molding, since it helps characterize the behavior of of the injectable mixture and is taken from the injector machine screw to fill the mold. This process involves high shear rates imposed on injectable mixture I [15]. It is desirable for injectable feedstocks to have low viscosity, so that micro details will be filled in quickly during the injection molding before the raw material is solidified [16, 17].

Newtonian fluids are not affected by shear, independent of the conditions of shear rates imposed on the material, the material will be maintained in constant flow during processing if the other conditions are not changed. The non-newtonian materials are influenced by shear and are divided into two types: dilating and pseudoplastic. The viscosity of the dilating materials increases when they are sheared, so that the material flow is impaired and becomes difficult to process. Materials considered pseudoplastic tend to reduce their viscosity with the growth of the shear rate, thus improving flow and making processing easier [18, 14].

A capillary rheometer, with a schematic shown in Figure 1, was the equipment used to measure material flow. In this assay, the temperature is kept constant during the process, and the higher the pressure used on the piston, the greater will be the flow velocity [18].

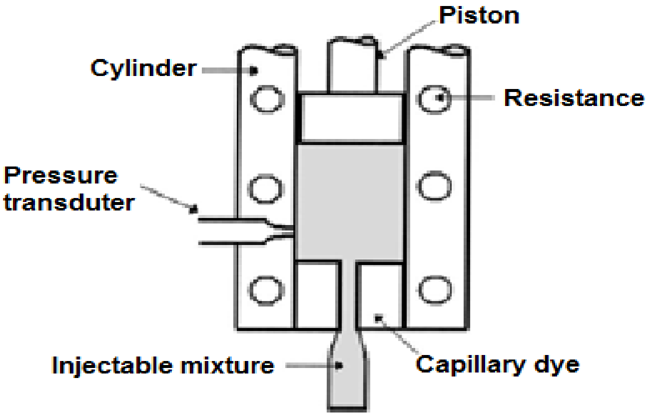


Figure 1 - Capillary Rheometer Schematic [18]

The torque rheometer measures the rheological properties of polymers, but this equipment causes uncertainty because it is difficult to analyze the data obtained. Qualitative indications of the viscosity of the melt, as well as dependency of viscosity on temperature and the degradation temperature are data obtained by the capillary rheometer, but these measures are difficult to interpret and to convert into absolute rheological units [19].

Another way to evaluate the viscosity of injectable masses is by using a torque rheometer, through the torque behavior needed to mix the metal powder and the binder system [20]. The injectable mixtures studied in this article were formulated in the Laboratório de Transformação mecânica (LdTM) at the Universidade Federal do Rio Grande do Sul (UFRGS). Technical articles in which injectable feedstocks were determined that could present the best results for the injection of biomedical components were studied to develop the mixtures.

2 MATERIALS AND METHODS

This article presented four different formulations for the purpose of evaluating the rheological characteristics generated, using the same volumetric percentage of metal powder AISI 316L in all mixtures. All the compositions were mixed in a torque rheometer and Paraffin (PW) was used to help the flow and Stearic Acid (SA) as a binder of the organic and inorganic fraction. The polymers used in the feedstocks were Polypropylene (PP), Low Linear Density Polyethylene (PEBDL), High Density Polyethylene (PEAD) and Polymethyl-Methacrylate (PMMA). The percentages of these materials used in each mixture are described in Table 1. The rheological results obtained in each mixture were compared to the commercial feedstock currently used in injection process.

Table 1. Composition of the samples

Mixture	PP (Vol%)	LLDPE (vol%)	HDPE (vol%)	PMMA (vol%)	SA (vol%)	PW (vol%)
1		**85%**			**2%**	**13%**
2		**70%**		**15%**	**2%**	**13%**
3	**40%**				**5%**	**55%**
4		**40%**	**20%**		**5%**	**35%**

Prior tests on the polymers without any mixture or loading of metal were elaborated to analyze the materials. The results were compared after metal loading to see to what extent the metal powder affects each structural polymer. The assays consisted in rheological analyses in a capillary rheometer and a DSC (Differential Scanning Calorimeter). All formulations contained 61% volume of metal powder and the torque provided by this volume of material when inserted in a torque rheometer was analyzed.
The data sent by the manufacturers of the structural polymers used are shown in Table 2. The data described were considered the most important to choose the materials.

Table 2. Properties of the structural polymers

Polymers	Density g/cm³	Flowability g/10 min	Transition temperature Vicat (°C)
PP	0,905	10.5	90 (194ºF)
LLDPE	0,924	20	94 (201.2ºF)
HDPE	0,964	10	131 (267.8ºF)
PMMA	1,190	8	105 (221ºF)

The information grouped in Table II follows the ISO 1133 and ASTM D 1238 standards. A temperature of 230ºC (446ºF) was used to process PP and PMMA, and for PEBDL and PEAD, the temperature used was 190ºC (374ºF).

The polymers were sent to the Thermal Analysis DSC equipment, model SDT Q600 to verify the fusion and degradation temperatures of the materials.

After the results obtained by the DSC equipment, feedstocks were mixed in a HAAKE POlylab torque rheometer, until the complete homogenization of the polymers, surfactant agents and metal powder was obtained.

After the mixture was ready, the load was sent to the DSC to analyze the final characteristics of the injectable mixture, and the processing point and binder extraction point could be defined.

3 RESULTS

The melting and degradation temperature of the polymers studied, obtained with the DSC equipment, are described in Table 3.

Table 3 - Melting and degradation points of the polymers

Polymer	Melting temperature (°C)	Degradation temperature (°C)
PP	170,15 (338.27ºF)	466,70 (872.06ºF)
LLDPE	125.59 (258.06ºF)	486.88 (908.38ºF)
HDPE	142,61 (288.7ºF)	491,42 (916.56ºF)
PMMA	190 (374ºF)	383,66 (722.59ºF)

The mixtures made and the way to prepare each mixture are shown in Table 4. The mixtures are considered as presented in Table 1.

Table 4. Parameters of mixtures performed in the torque.

Mixture	1st Stage	2nd Stage	3rd Stage	4th Stage
1				
Temperature (°C)	180 (356ºF)	180 (356ºF)	180 (356ºF)	180 (356ºF)
Rotation (rpm)	20	25	40	40
Time (min)	15	15	15	15
Addition	100% polymer and 50% metal powder	No Addition	100% metal powder	No Addition
2				
Temperature (°C)	170 (338ºF)	170 (338ºF)	170 (338ºF)	170 (338ºF)
Rotation (rpm)	25	35	40	50
Time (min)	15	15	15	30
Addition	100% polymer and 50% metal powder	No Addition	100% metal powder	No Addition
3				
Temperature (°C)	150 (302ºF)	150 (302ºF)	150 (302ºF)	150 (302ºF)
Rotation (rpm)	15	25	30	30
Time (min)	15	15	15	15
Addition	100% polymer and 50% metal powder	No Addition	100% metal powder	No Addition
4				
Temperature (°C)	130 (266ºF)	130 (266ºF)	130 (266ºF)	130 (266ºF)
Rotation (rpm)	15	35	50	50
Time (min)	15	15	15	15
Addition	100% polymer and 50% metal powder	No Addition	100% metal powder	No Addition

The results of the mixtures performed in the torque rheometer and expressed in the form of torque x time graphs are demonstrated in Figure 8. The graphs show that all feedstocks can be processed.

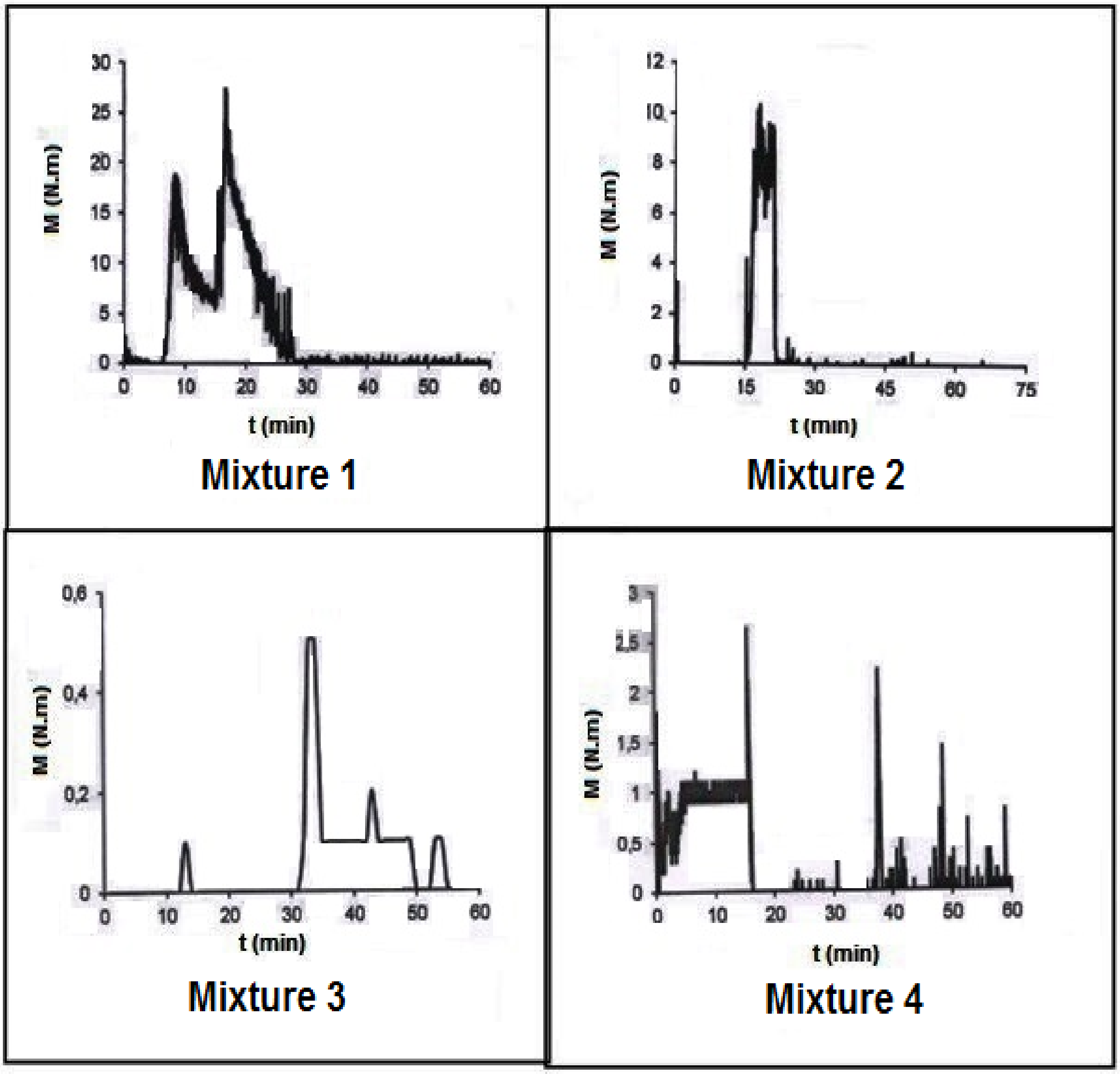
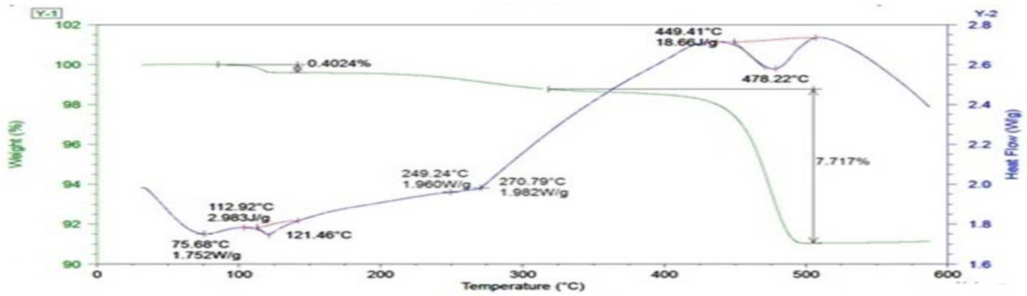
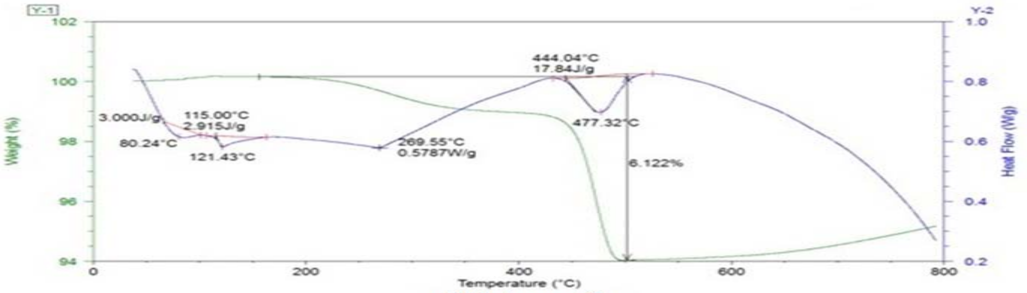


Figure 3 - Torque graphs per time of mixtures performed.

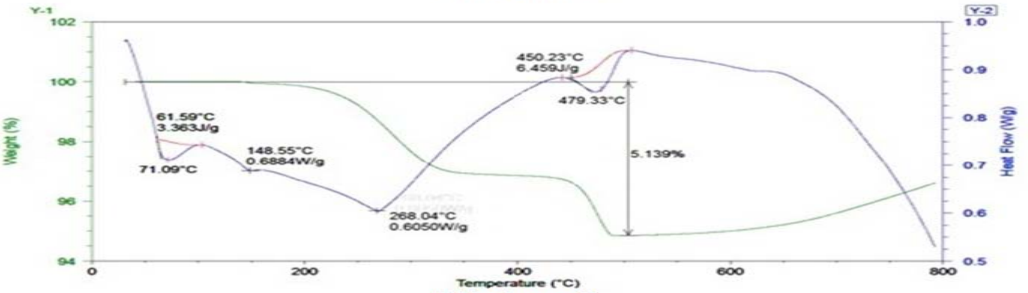
The commercial feedstock and the 4 mixtures formulated were sent to the DSC equipment and the results are shown in Figure 4.



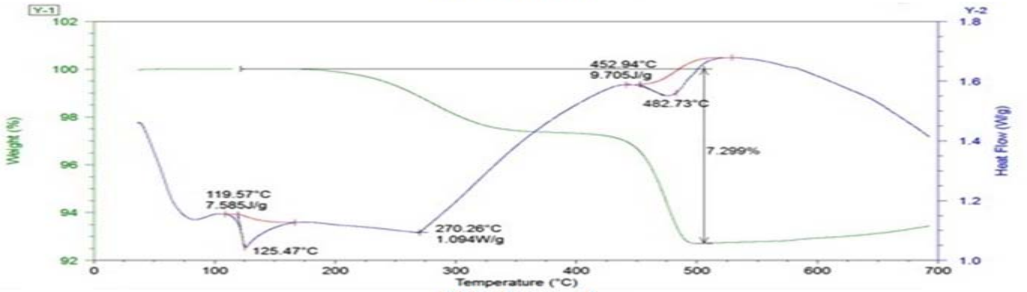
Mixture 1



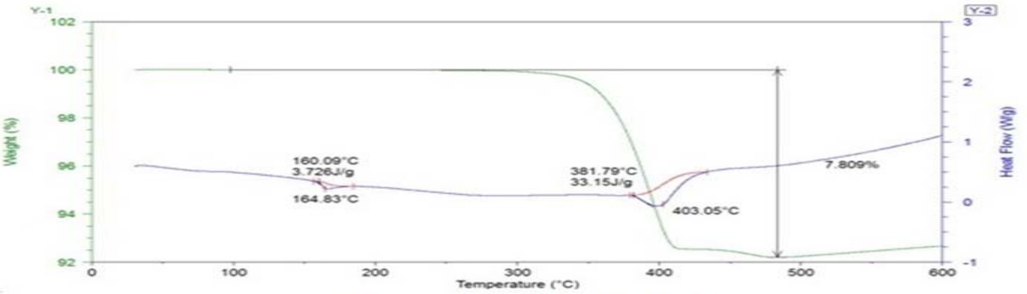
Mixture 2



Mixture 3



Mixture 4



Commercial feedstock

Figure 4 - Melting points of the injected materials studied.

The results of the rheologies done in the capillary rheometer are shown in Figure 5, where it was found that mixture 2 presented the best flow.

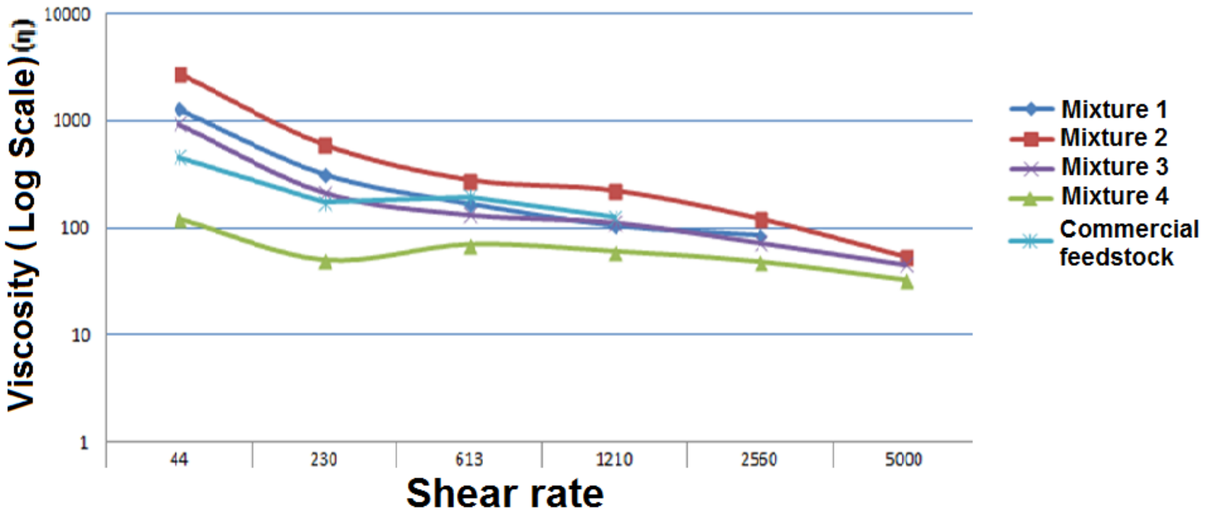


Figure 5 - Graph of the analysis of mixtures in a capillary rheometer.

The results show that the injectable mixtures produced are less viscous when processed at a high shear rate. Mixture 4 has low viscosity and the best processing, compared to the other feedstocks independent of shear rate used. The commercial feedstock shows good processing when it is submitted to low shear rates, but when it begins to be used at high shear rates its viscosity does not diminish so much, as can be seen in mixtures 1 and mixture 3, impairing the processing of the commercial feedstock compared to the other feedstocks.

4 RESULTS AND DISCUSSION

The results obtained in this study show that it is possible to produce feedstocks that are similar to and even greater then the commercial feedstocks. However, it is necessary to be careful because of problems of feedstock homogenization. This was proved by the model of BOUSMINA [21] and can be seen in some of the feedstocks formulated.

It is common knowledge that for every compound there is a proportion of binder for the metal load used. This has already been mentioned by several authors [10, 11, 22]. The DSC results showed that the maximum proportionality obtained in commercial feedstocks is 92.190% in metal powder weight for approximately 7.810% in binder weight. The results obtained in all feedstocks formulated show that all of them obtained a greater amount of inorganic fraction than that of commercially found materials.

The results obtained in the capillary rheometer show that mixture 4 is best processed compared to the other feedstocks, with low viscosity, even if it contains a higher percentage of metal powder, besides having a lower processing temperature. It may be said that this feedstock can be used to inject any component.

5 CONCLUSION

The results discussed in this work show that commercially sold materials may not be appropriate for some processing situations, especially where high shear occurred. This is based on the fact that feedstocks formulated at the LdTM presented better results.
This proves that further study is required in order to obtain injectable feedstocks to produce parts with thin walls, since the commercially found materials generate failures. They do not complete the parts and cause problems in the components, or else they require excessive efforts from the injector equipment to complete a component.

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