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Construction of a plastomer for the analysis of polypropylene fluidity under different temperatures and use of additives

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Abstract. A low-cost plastomer was built and analyzed the main factors that can improve its fluidity, such as the use of different specifications and additives. Thermoplastics are materials currently used everywhere, from simple applications such as making toys to aerospace applications. Among the usual thermoplastics in our daily lives we have polypropylene, widely used as raw material for the manufacture of various plastic materials. One of the factors that most interferes with the quality of PP in industrial use is its fluidity index, measured by the use of plastomer, an extremely expensive equipment. From the analysis of various temperatures and percentages of additives it was possible to identify factors that improve its fluidity, improving industrial applicability. It is concluded that there is a direct relationship between the increase in the melt index (fluidity index) of the melt and the increase in temperature and additives by up to 1%, emphasizing that with 2% worse results can be obtained from the IF. This increase in the bottom IF can be explained by the theory of free volume, where the higher the temperature, the greater the free volume between the molecules and the lower their viscosity, that is, the easier their flow will be.

Keywords. Polypropylene. Injection. Fluidity. Temperature. Additive.

1. Introduction

Polymeric materials are materials used since ancient times, when natural materials were used as raw material for the manufacture of objects (such as rubber). Polymers are widely used in the most diverse industrial fields and are gaining more and more market share due to their new applications to replace ceramic and metallic materials.

Among polymers, we can focus on polypropylene (PP), which can be used in new applications due to increased production capacity and reduced costs. Polypropylene has characteristics that make it suitable for injection molding, extrusion, thermoforming, rotational molding and blow molding processes, but emphasizes the importance of injection molding for the most diverse types and shapes of parts.

The injection process, even though it is widely used, is susceptible to failures when the Fluidity Index of the material is not reached at the level requested for the elaboration of such part, thus being one of the main factors to be analyzed regarding the origin of the raw material.

Therefore, in the processing of polymers, it may be necessary to add additives to improve fluidity, as well as to analyze its factory fluidity index. Finally, to process simple polymer materials (such as polypropylene) to obtain higher quality products, it is necessary to understand the initial flow index through the plastomer and add additives to improve the index.

1.1. Structure

Injection is one of the most used processes for polymers, from the manufacture of simple parts to complex parts, so the purpose of this work is to design and build a low cost plastomer for the analysis of polypropylene fluidity, as well as to analyze the improvement of its fluidity with the use of additives and different injection temperatures.

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2. Literature revision

2.1. Polymers

The word polymer originates from the Greek poly (many) and mere (repetition units). Thus, a polymer is a macromolecule composed of many (tens of thousands) of repeating units called mere, linked by covalent bond. The raw material for the production of a polymer is the monomer, that is, a molecule with one (mono) repeating unit. Depending on the type of monomer (chemical structure), the average number of mere per chain and the type of covalent bond, we can divide the polymers into three major classes: Plastics, Rubbers and Fibers. [1]

Many physical properties depend on the length of the molecule, which is its molar mass. As polymers generally involve a wide range of molar mass values, a wide variety of changes in their properties can be expected.

Changes in the size of the molecule, when it is small, cause major changes in its physical properties. These changes tend to be smaller with the increase in the size of the molecule, and for polymers the differences still exist, but they are small. This is advantageously used, commercially producing various types (grades) of polymers, to meet the particular needs of a given application or processing technique. [1]

The response of polymers to mechanical forces at high temperatures is related to their main molecular structure. In fact, a classification scheme was developed based on the behavior of these materials in the face of increased temperatures. Thermoplastics (or thermoplastic polymers) and thermosetting materials (or thermosetting polymers) are two subdivisions. Thermoplastics soften when heated (finally liquefy) and harden when cooled - these processes are completely reversible and can be repeated. On the molecular scale, as the temperature increases, the secondary bond strength decreases (due to the greater movement of the molecule), facilitating the relative movement of the adjacent chains when tension is applied.

Irreversible degradation occurs when the temperature of a molten thermoplastic polymer is raised excessively. In addition, thermoplastics are relatively soft. Most linear polymers and those that have some branched structures with flexible chains are thermoplastic. These materials are normally manufactured with simultaneous application of heat and pressure.

Examples of common thermoplastic polymers include polyethylene (PE), polystyrene (PS), poly (ethylene terephthalate) (PET) and polyvinyl chloride (PVC). [2]

In Figure 1 we have an example of a polymerization reaction.

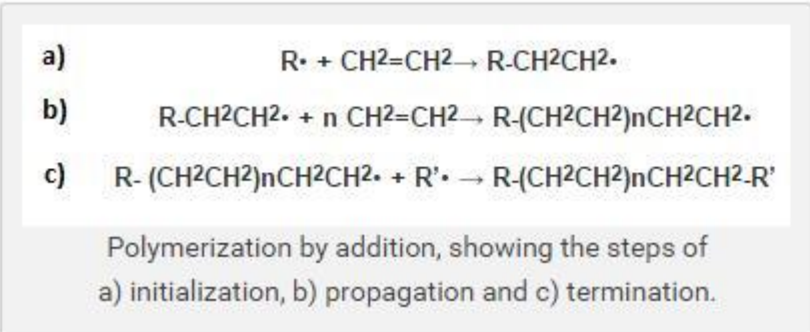

Fig.1 Polymerazation by addition

The mechanical, optical and chemical properties of these materials depend on the size, composition, chemical structure and other factors, directly related to their applications, so, for example, if a material has chemical resistance, it can be used in environments where there is constant exposure to some chemical or similar product, such as Polyethylene (PE) used in packaging for chemical products, cleaning products such as bleach, alcohol, etc., without being attacked. Another example may be Polycarbonate (PC), which has excellent impact resistance and is a transparent material, therefore, it is used in police shields, lenses for glasses, tiles, headlights for motor vehicles, etc. [3]

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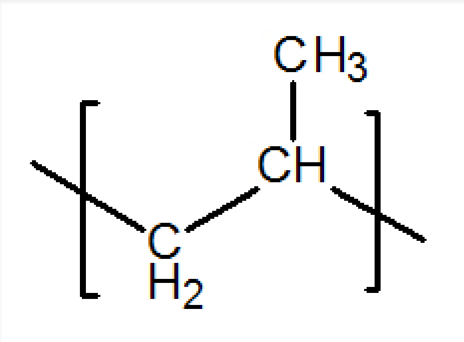
2.2. Polypropylene(PP)

Polypropylene (PP) is one of the most used thermoplastic polymers in the industry, with a variety of applications that include rigid and flexible packaging, disposables, tubes and injected products for the most varied uses. The petrochemical industry offers several types of polypropylenes, such as: homopolymer PP, heterophasic copolymer PP and random copolymer PP. The homopolymer PP contains only the propylene monomer in its molecular chain and, being predominantly of isostatic configuration, it can reach a degree of crystallinity of up to 70%. [4]

Polypropylene (PP) is a homopolymer (Fig 2), of the polyolefin class, thermoplastic, recyclable and endowed with great industrial importance. Its main characteristics are low density (0.905 g / cm3), with glass transition temperature (Tg) of -20 ° C and crystalline fusion temperature (Tm) of 165 ° C, low cost and high chemical resistance to solvents. In Figure 2 we have the configuration of its polymeric chain. [5]



Molecular structure of PP

Fig.2 Molecular structure

Source –[5]

Polypropylene is produced from the polymerization of propene, a gaseous by-product of oil refining, in the presence of a catalyst and under controlled conditions of pressure and temperature. [6] Polypropylene has excellent electrical and insulating properties, chemical inertia and resistance to moisture typical of non-polar hydrocarbon polymers.

It is resistant to a variety of chemicals at relatively high temperatures and practically insoluble in all organic solvents at room temperature. Absorption of solvents by polypropylene increases with increasing temperature and decreasing polarity. The high crystallinity of the polypropylene gives the polymer a high tensile strength, rigidity and hardness. Polypropylene is practically free from breaking under environmental stress. However, it is intrinsically less stable than polyethylene in relation to thermal degradation and oxidative degradation. Therefore, for satisfactory processing, polypropylene must be stabilized by incorporating thermal stabilizers, UV absorbers and antioxidants. [7]

2.3. Injection, Flow rate and Density

The fluidity index is the measured value of the extrusion rate of a molten material, under specific conditions of temperature and pressure, through a capillary of specific diameter and length, according to ASTM D 1238 (Fig 3).

During the production of injected using thermoplastics, the only parameter of polymeric flow that the producer has easy access to is the fluidity index. [8]

The fluidity index measurement equipment is called a plastometer (Figure 3), consisting of a capillary with defined dimensions, electrically heated and a piston responsible for the extrusion of the thermoplastic.

Measuring the flow index of a thermoplastic is a simple process. Once the plastometer reaches the indicated temperature, a certain mass of thermoplastic is placed inside its capillary. After a previous heating time of 6 to 8 min, the extrusion of the molten thermoplastic is initiated through the capillary orifice - standard matrix of circular shape. The gravimetric flow (amount of mass of the extruded thermoplastic in 10 min) is the fluidity index of the tested thermoplastic.

The fluidity index of a thermoplastic is defined for a specific load (piston mass) and temperature, as indicated in Tables 1 and 2. The fluidity index test is a particularly useful method in quality control tests applied to thermoplastics , its application being indicated for the measurement of melt flow rates in the range of 0.15 to 50 grams for 10 minutes (manual method); and in the range of 0.50 to 300 grams for 10 minutes (automatic method). The manual method differs from the automatic method in that the extruded thermoplastic is extracted, respectively, through interference from the operator or the equipment itself [9]

The fluidity index is influenced by the molecular structure of a thermoplastic (average molar mass, polymeric branches) and also by the conditions of measurement of the extruded flow [9]

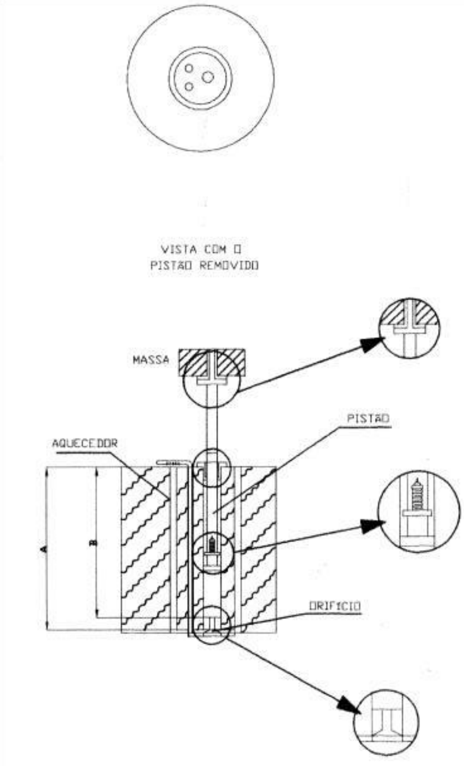


Fig. 3 Plastometer ASTM D 1238

3. Materials and Methods

3.1. Materials

The materials used are the recycled PP supplied by a local company named Oeste Plásticos in the form of pellets, and according to the supplier it is a polypropylene with an average fluidity index indicated for the injection process. It has an excellent balance of rigidity / impact properties, excellent surface finish and good processability. Its main applications are in plastic boxes, buckets and tubes.

Another material used is talc, a phyllosilicate mineral, with a chemical composition $Mg_3Si_4O_1(OH)_2$, used here as an additive to increase the fluidity of polypropylene.

3.2. Methods

In the first part of the study, ASTM (Standard Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer) D 1238 - 04 was analyzed, an international guideline that gives the standards for the construction and validation of plastomers. Based on this, the construction of the plastomer was designed in the most pragmatic and least costly way possible, following all the rules of ASTM D 1238.

The initial processing of the polypropylene was carried out in the plastomer, without the use of additives, using a temperature of 230ºC, the official working range used for the polypropylene, seeking to get as close as possible to an official analysis process.

About 0.600 kg were used as the starting material of polypropylene to be processed in the plastomer. The procedure for determining the Average Fluidity Index is described by the ASTM D1238 standard. It concerns the measurement of the flow rate of a polymer through an orifice of specified dimensions, under pre-stipulated conditions of load, temperature and position of a piston in the plastomer. The weight of the extruded polymer in ten minutes of the experiment is the flow rate of the polymer, its unit being g / 10min. 10min [1].

Example: Fluidity 12 consists of 12g / 10min.

The standard ASTM D1238 for the case of PP, normalizes the temperature of 230 ° C and load of 2.16 kg and time of 1 minute for sample removal. The identification of the time for sample removal was carried out as follows, the capillary matrix was inserted in the plastomer cylinder, the temperature was adjusted for the studied polymer, 230 ° C for PP, the cylinder was loaded with the amount pre-determined material, the material was compacted for the removal of air and then the weight of 2.16 kg for the PP was placed on the piston, and the extrudate collection began.

Ten samples were taken from the extrudate, which were weighed and their fluidity index was calculated in g / 10min.

Using a simple rule of three, we obtained the fluidity index value for pure PP, which has a unit of g / 10 minutes.

$$0{,}5768g - - - - - - - - 60s$$
$$x - - - - - - - - - - 600s$$
$$x = 5{,}768g/10min$$

Triplicates of each sample were made to determine the fluidity index, and of each sample their averages were determined thus obtaining the PP fluidity index values after multiple processing (Fig 4).

In the second stage of the study, polypropylene (PP) was processed with the addition of the additive (talc), 0.5, 1 and 1.5% after each processing, and the same methodology as the first stage of the study was used.

The third stage, the temperature was modified, evading ASTM D1238 on purpose, to analyze whether the fluidity index was changed. With the application of the three stages, the following table was built:

Nº amostra	Talco (%)	Temperatura (ºC)	IF (g/10min)
1	0	230	4,4
2		250	5,8
3		280	11,3
4	1	230	5,7
5		250	7,0
6		280	11,3
7	2	230	4,2
8		250	6,9
9		280	11,9

Parameters used in the IF analysis.

Fig.4 IF x Temperature

For each temperature condition and additives, five samples (cuts) were obtained which were weighed (to calculate the IF). Afterwards, an average was taken at each point, thus providing an evaluation of the results

4. Results and discussion

Figure 6 shows the results of the IF of the fused PP under the tested conditions shown in Table 1. First, the data related to the ASTM D-1238 standard are observed, that is, to verify the accuracy of the equipment.

At a temperature of 230ºC and without the use of additives, the IF measured was 4.45g / 10min, showing that the PP used in the experiment is a raw material with low fluidity, confirming these results with the company that made it available.

Analyzing the graph, now with different temperatures and additives, there is a significant increase in the IF as the temperature increases, also with the addition of 1% talc, however, the addition of 2% talc did not show significant improvements in the IF , and may even impair fluidity as seen in the graph with a temperature of 230ºC (Fig 5).

Considering only the temperature of 280ºC, the additive does not seem to interfere significantly.

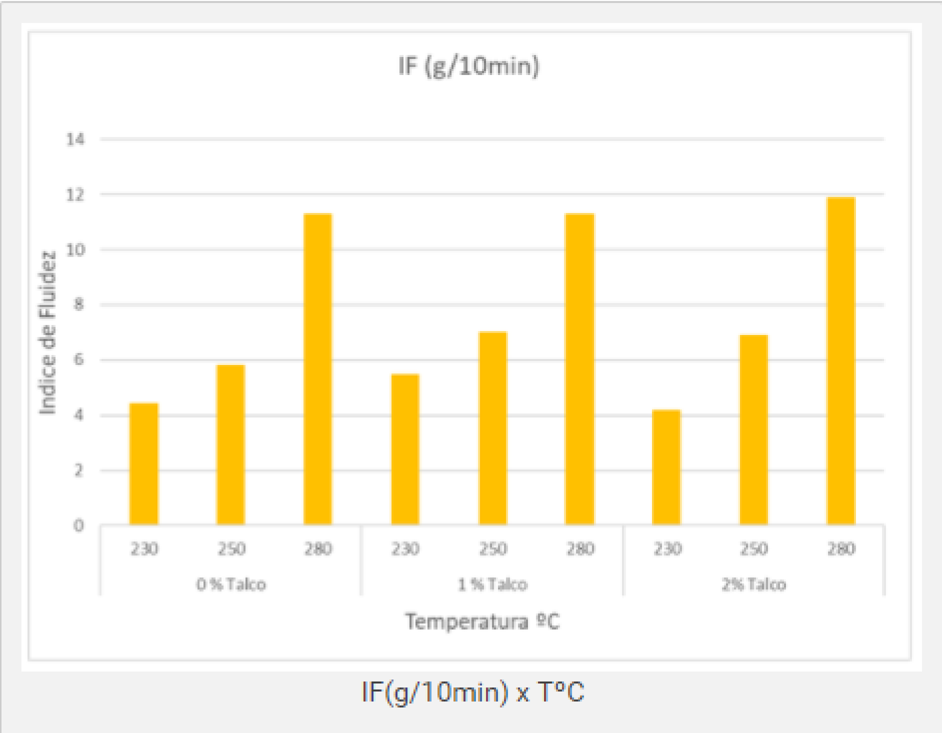


Fig. 5 Results

This increase in melt IF can be explained by the theory of free volume, where the higher the temperature, the greater the free volume between the molecules and the lower their viscosity, that is, the easier their flow will be.

5. Conclusion

Through the results of this study, the influence of the evaluated parameters (temperature and additives) on the behavior of the PP was verified. It is concluded that there is a direct relationship between the increase in the melt IF and the increase in temperature and additives by up to 1%, noting that with 2% worse results can be obtained for FI.

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The plastomer was shown to be calibrated when following the ASTM D-1238 standards when compared to the report shown by the company that made it available, thus showing itself fully capable of being used for real tests in companies and universities, at a much lower manufacturing cost. when compared to brand new equipment.

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