PLASTOMETER USE TO EVALUATE RHEOLOGICAL PARAMETERS OF COMMERCIAL POLYETHYLENE

Ilana Oliveira Almeida[1] [a], Joyce Batista Azevedo [a].

[a]Federal University of Bahia, Institute of Science, Technology and Innovation, Camaçari, Bahia.

Abstract: The study of the rheological behavior of polymers is fundamental in the plastics industry. Rheology makes it possible to understand the behavior of polymers under flow and is decisive for material selection depending on the manufacturing process and application. However, equipment for rheological analysis is expensive and difficult to operate. A more accessible alternative, which can be used to visualize the tendency of the rheological behavior of polymers, is the use of a plastometer. By varying the mass of the weight loaded on the plastometer as a function of the cross-sectional area of the piston, it is possible to estimate the shear rate and viscosity of molten polymers, supporting decisions for defining process parameters. In this sense, the rheological behavior of commercial polymers was estimated from plastometer measurements, making it possible to evaluate the type of fluid and their rheological behavior.

Keywords: Rheology, Melt Flow Index, Polyethylene.

UTILIZAÇÃO DE PLASTÔMETRO PARA AVALIAÇÃO DE PARÂMETROS REOLÓGICOS DE POLIETILENOS

Resumo: O estudo do comportamento reológico dos polímeros é fundamental na indústria de plásticos. A reologia permite compreender o comportamento dos polímeros sob fluxo e é determinante para a seleção do material em função do processo de fabricação e a aplicação. No entanto, equipamentos para análises reológicas são caros e de difícil operação. Uma alternativa mais acessível, e que pode ser utilizado para visualizar a tendência do comportamento reológico dos polímeros, é o uso do plastômetro. Através da variação da massa do peso carregado no plastômetro em função da área transversal do pistão, é possível estimar a taxa de cisalhamento e a viscosidade de polímeros fundidos, subsidiando decisões para definição de parâmetros de processos. Neste sentido, o comportamento reológico de polímeros comerciais foi estimado a partir de medições em plastômetro sendo possível avaliar o tipo de fluído e comportamento reológico destes.

Palavras-chave: Reologia, Índice de Fluidez, Polietilenos.

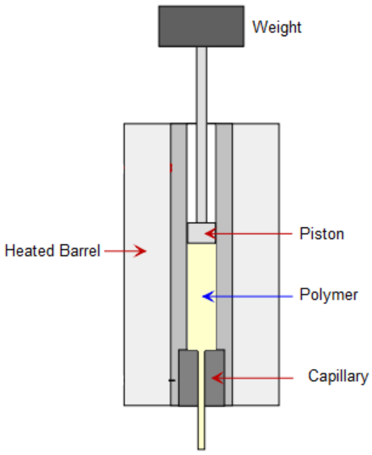
1. INTRODUCTION

Polymers behave, mostly, as viscoelastic materials, that is, under deformation condition, they present a viscous and elastic behavior. Therefore, knowledge of the behavior of polymers under flow is crucial for selecting them for an application and/or process [1]. Rheology is the science that studies the deformation and flow of matter and is used in polymers since in conventional processing operations, such as extrusion and injection molding, these materials are subject to various types of deformations, due to the complexity of the geometry of these devices.

Among the rheological properties, the melt flow index (MFI) is a characteristic of polymers widely used as a raw material quality control, it is a parameter inversely proportional to the viscosity and characterizes the flow properties during processing. Many researchers have investigated and established the relationship of MFI with numerous mechanical, chemical and thermal properties such as yield stress, viscosity, molecular weight distribution and shear rate [2-5].

The determination of the melt flow index consists of the mass flow of the molten polymer through the cylindrical capillary present in the plastometer, equipment used to determine the melt flow index [6-7]. The plastometer consists of a heated barrel with a piston driven by a standard weight and the polymer flows through a capillary that is also standardized (Figure 1). This equipment is used to evaluate thermoplastic polymers in melting states, under controlled pressure and temperature parameters. After heating, the thermoplastic is loaded by a piston and extruded through a matrix, the capillary.

Figure 1. Scheme of a plastometer [8].



Compared to other equipment used for rheological measurements, the plastometer is a more accessible, easy-to-operate device and can be used to visualize the tendency of the rheological behavior of polymers. By measuring the melt flow index with weight variation in the plastometer, it is possible, due to the flow change, to estimate the polymer viscosity as a function of the shear rate [8].

Viscosity is the most important rheological property in polymer processing because flow rates, pressure drops and temperature increases depend on this property [8-9].

In a fluid classified as "ideal", Newtonian fluid, viscosity is a property that remains constant and does not change with the rate or voltage applied to the fluid's movement. In molten polymers, the viscosity depends a lot on the orientation of the macromolecules in the flow direction, which reduces the impediments arising from the interactions between them, changing their behavior and thus classifying the molten polymers in non-Newtonian fluids [6-7].

Through the behavior of the viscosity curve as a function of the shear rate to which the polymer is subjected, it is possible to identify the non-Newtonian phenomena present and classify it as dilatant or pseudoplastic. Being a dilatant polymer, one that has an increase in viscosity with the shear rate and a pseudoplastic polymer when the opposite behavior occurs, that is, a reduction in viscosity with the increase in the shear rate [10].

In this work, considering the above context, the plastometer was used to estimate the rheological behavior of commercial polymers, making it an accessible tool for polymer processing studies.

2. METHODOLOGY

2.1 Materials

To carry out this study, three types of commercial polyethylene, manufactured and supplied by Braskem, were used. Information on the resins described in the technical sheets [11-13] are compiled in Table 1.

Table 1. Polyethylene technical information.

Polymer / Commercial name	Density (g/cm^3)	MFI (190°C/2,16kg)	Applications
High density polyethylene/ JV060U	0.957	7.0 dg/min	Pallets, boxes and bottles
High density polyethylene/ IA59U3	0.960	7.3 dg/min	Boxes and bottles, pallets and covers.
Linear Low Density Polyethylene/ ML3601U	0.939	33.3 dg/min	Water reservoirs, chemical tanks and septic tanks.

2.2 Methods

The melt flow index of the commercial resins was determined using a standardized plastometer at the Materials Development Laboratory at UFBA Camaçari, based on the ISO–1133 standard with a temperature of 190°C and weights varying between 1.2 and 4.91 kg.

The shear rate and melt viscosity can be estimated due to the momentum balance in the fluid flowing in the plastometer capillary [8]. The driving force required for this movement is the pressure difference between the inlet and outlet of the capillary. At steady state, disregarding viscous friction in the barrel and capillary inlet effects, the capillary inlet pressure can be estimated from the mass of the weight loaded in the plastometer and the cross-sectional area of the piston. Therefore, the shear rate (γ_a) in the capillary wall and the melt viscosity (η_a) can be estimated from the melt index, weight mass and the density of the polymer melt according to Equations 1 and 2 [14].

$$\gamma_a = 1.845 \frac{MFR}{\rho} \quad (1)$$

$$\eta_a = 4.860 \frac{M}{MFR} \quad (2)$$

Where:

M is the mass of the plastometer, which in this study varied by: 1.2; 2.16; 3.26, 4.03 and 4.91kg.

ρ is the density of the polymer, considering the density provided in the technical data sheet of the polymer in g/cm³.

MFR is the melt index found at 190°C at 10 g/min.

Eqs.(2)-(3) were obtained taking into account the dimensions of the standardized plastometer and assuming that the melt behaves like a Newtonian fluid. For this reason they are called apparent shear rate and viscosity.

The use of different weights with the same sample allowed a shear rate scan between 5 and 50 s^{-1}. The rheograms of the samples were graphically presented in the form of double-logarithmic plots of *η* versus *γ*.

3. RESULTS AND DISCUSSION

Table 2 shows the mean and uncertainty of the melt index measured in five 60 second “cuts”, as well as the shear rate and apparent viscosity calculated for all samples.

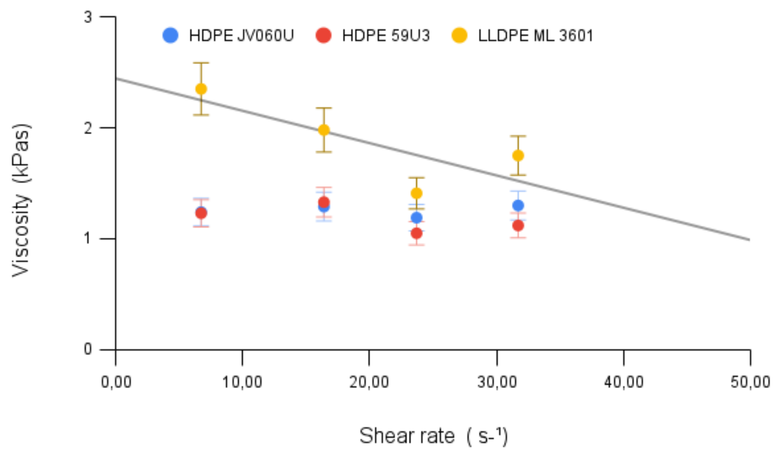
Table 2. Melt flow index (MFR), shear rate (γ) and apparent viscosity (η) calculated for all samples tested with different weights.

Polymer/Grade	M (kg)	MFR (dg/min)	γ (s^{-1})	η (kPas)
HDPE JV060U	1.20	3.47$\pm$ 0.7	6.73$\pm$ 0.97	1.68$\pm$ 0.24
	2.16	8.43$\pm$ 0.7	16.40$\pm$ 0.97	1.24$\pm$ 0.07
	3.26	12.20$\pm$ 2.4	23.70$\pm$ 0.97	1.29$\pm$ 0.05
	4.03	16.33$\pm$ 3.3	31.70$\pm$ 0.97	1.19$\pm$ 0.03
	4.91	22.00 $\pm$ 0.5	42.70$\pm$ 0.97	1.30$\pm$ 0.03
HDPE IA59U3	1.20	4.61 $\pm$ 1.3	8.86$\pm$ 0.5	0.69 $\pm$ 0.04
	2.16	8.50 $\pm$ 0.7	16.34$\pm$ 1	1.23 $\pm$ 0.07
	3.26	11.90 $\pm$ 0.5	22.88$\pm$ 1	1.33 $\pm$ 0.05
	4.03	18.6$\pm$ 0.3	35.70$\pm$ 0.9	1.05 $\pm$ 0.03
	4.91	21.2$\pm$ 4.4	40.88$\pm$ 1.1	1.12 $\pm$ 0.03
LLDPE ML 3601	1.20	2.73 $\pm$ 1.19	5.36$\pm$0.98	2.13$\pm$0.39
	2.16	4.45 $\pm$ 1.34	8.76 $\pm$0.98	2.35$\pm$0.26
	3.26	7.96 $\pm$ 0.8	15.60$\pm$0.99	1.98$\pm$0.12
	4.03	13.86 $\pm$ 0.4	27.20$\pm$ 1.02	1.41$\pm$0.05
	4.91	13.61 $\pm$ 0.4	26.70$\pm$ 1.02	1.75$\pm$0.06

Analyzing the results presented in Table 1, when comparing the result of the melt flow index of the High Density Polyethylene JV060U with the value reported in the technical data sheet of the polymer [11], a difference of 20% can be observed, which can be associated with the difference of some parameters experimental data such as equipment type and operator experience. For these same reasons, there was also a variation between the melt flow index of other polyethylenes with commercial value, being 16.4% for HDPE IA59U [12] and 48.3% for ML 3601 [13] .

As for the rheological properties (Figure 2), there is a more significant variation in viscosity depending on the shear rate calculated for PELBD ML 3601. This behavior is expected for a polymer classified as pseudoplastic, that is, there is a reduction in viscosity with increasing shear rate [7;15].

Figure 2 . Rheogram of commercial polyethylenes



For the HDPEs, a Newtonian plateau was observed, that is, the viscosity was not altered with the shear rates used.

The difference in rheological behavior between the two types of polyethylene is due to the structural difference between them. In HDPE, the chains are more tangled, which makes it difficult to align and slip between the chains, therefore, the viscosity is only reduced with higher shear rates. The presence of small branches in PELDB facilitates chain alignment and flow, which makes this polymer exhibit a pseudoplastic behavior at low shear rates [16].

Studies using the plastometer for analysis of rheological properties is not common. However, through the use of this equipment, through the methodology used, it is verified that it is possible to obtain information about the rheological behavior of polymers in a preliminary way and guide the parameterization of process parameters.

4. CONCLUSION

By varying the flow of commercial polymers in the plastometer capillary, using different weights during the determination of the fluidity index, it was possible to obtain rheological characteristics of different types of polyethylenes through an accessible and conventional technology. The determination, by the methodology used, of the fluidity index, viscosity and shear rate, allowed inferences about the behavior of these materials when subjected to shear forces, which provides a better evaluation of process parameters in the industrial routine.

Acknowledgments

The authors thank CNPq for granting the scientific initiation scholarship, SENAI CIMATEC for donating the polymers and UFBA Camaçari for the opportunity to carry out this work.

5. REFERENCES

1. OSSWALD, Tim A.; HERNÁNDEZ-ORTIZ, Juan P. Polymer processing. Modeling and Simulation. Munich: Hanser, p. 1-651, 2006.
2. DE CARVALHO, Marcelo Sousa; AZEVEDO, Joyce Batista; BARBOSA, Josiane Dantas Viana. Effect of the melt flow index of an HDPE matrix on the properties of composites with wood particles. Polymer Testing, v. 90, p. 106678, 2020.
3. TECHAWINYUTHAM, Laongdaw et al. Recycled LDPE/PETG blends and HDPE/PETG blends: Mechanical, thermal, and rheological properties. Journal of Materials Research and Technology, v. 15, p. 2445-2458, 2021.
4. SALAKHOV, Ildar I. et al. Low-temperature mechanical properties of high-density and low-density polyethylene and their blends. Polymers, v. 13, n. 11, p. 1821, 2021.
5. ARIFF, Zulkifli Mohamad et al. Rheological behaviour of polypropylene through extrusion and capillary rheometry. Polypropylene, p. 29-49, 2012.
6. MANRICH, Silvio. Processamento de Termoplásticos: rosca única, extrusão e matrizes, injeção e moldes. Artliber Ed., 2005.
7. SCHRAMM, Gebhard. Reologia e reometria: fundamentos teóricos e práticos. Artliber Ed., 2006.
8. CANEDO, Eduardo Luís. Fenômenos de transporte. Grupo Gen-LTC, 2000.
9. HAN, Chang Dae. Rheology and processing of polymeric materials: Volume 1: Polymer Rheology. Oxford University Press, 2007.
10. CHEREMISINOFF, Nicholas P. Introduction to polymer rheology and processing. Crc Press, 2018.
11. Data Sheet: High Density Polyethylene JV060U. Braskem, 2018.
12. Data Sheet: High Density Polyethylene IA59U3. Braskem, 2018.
13. Data Sheet: Linear Low Density Polyethylene ML3601U. Braskem, 2018.
14. RAUWENDAAL, Chris. Extrusion. Encyclopedia of Polymer Science and Technology, v. 2, 2002.
15. BECKER, Márcia R.; FORTE, Maria Madalena de C.; BAUMHARDT NETO, Ricardo. Preparação e avaliação térmica e reológica de misturas de PEBD/PELBD. Polímeros, v. 12, p. 85-95, 2002.
16. CRUZ, Sandra A. et al. Avaliação das propriedades reológicas de blendas de PEAD virgem/PEAD reciclado. Polímeros, v. 18, p. 144-151, 2008.