Gradus Vol 4, No 2 (2017) 323-328
ISSN 2064-8014
GRADUS
GRADUS.KEFO.HU

EFFECT OF RECYCLING PROCESS ON PET MELT FOR SHEAR VISCOSITY

*Attila Bata *[1], Gergely Tóth [1], Dorottya Nagy [1] and Károly Belina [1]*

[1] Department of Materials Technology, GAMF Faculty of Engineering and Computer Science, John von Neumann University, Hungary

Keywords:
PET,
regrind,
oscillation rheology,
capillary viscosity,
recycling,

Article history:
Beérkezett: 13 September 2017
Átdolgozva: 21 September 2017
Elfogadva: 20 October 2017

Abstract

Recycled PET is already used in production, but only in small amount. Our final aim is to increase the regrind percentage in injection molded products. This research is focused on the characterization of original and regrind PET materials. Inherent viscosities were determined to characterize the materials. Flow curves of pure and regrind materials were measured at different temperatures. It was determined that the viscosity of the recycled material is lower than the value of the original material at each temperature. The molecules of the recycled material were supposed to shorten during the grinding process. If the temperature increases the shear viscosity decreases, but there was no linear correlation between these results. The structure viscous behavior of the polymer melt was revealed, while the viscosity decreases with increasing shear rate.

1. Introduction

Nowadays polyethylene terephthalate (PET) is one of the most common plastics, also because of its wide spread use. For the PET bottles there are certain places where recycled PET is already used and produced. In this case, only about 10-25% recycled PET is used. Our goal is to increase the quantity of the recycled PET in production. With the recycled materials new bottles, products can be made. This requires a development path, and a more sophisticated technology, as not easy to determine the antecedents of once used bottle: what was stored in it, how it was used previously [1]. PET is a hygroscopic thermoplastic polymer, therefore drying process is very important, since it affects the rheological properties of the material, the subsequent processing, and also the product characteristics [2].

The rheological analysis mentioned in this paper was performed with polyethylene terephthalate (PET) material, in which the structural, elastic and viscoelastic behavior of a specific base material was investigated. The study was carried out by ARES-G2 type oscillation rheometer, and Goettfert Rheograph 25 capillary rheometer, so the materials was examined at low and high shear rate. The original granulate was analyzed between 270-290°C. Then, the original base material was processed with injection molding machine, and then it was granulated again. The granulated material was measured on the same temperatures. The measuring values are presented on diagrams and from these conclusions were made.

* Corresponding author. Tel.:
E-mail address: bata.attila@gamf.uni-neumann.hu

2. Materials

RAMAPET N180 was used as the base material. It is a general purpose, non-reheat PET resin for bottles, film, thermoformed containers and other transparent applications. It has good optical properties, the mechanical properties are representative for a PET resin.

PET is hygroscopic, and since moisture adversely affects the inherent viscosity (IV) during melt processing of the polymer, it must be dried prior to molding. Undried PET pellets can contain up to 0.2% (2000 ppm) of moisture. After drying the moisture content typically is below 50 ppm [3].

PET has good surface hardness, stiffness, and dimensionally stability that absorbs relatively low volume of moisture. It has good gas barrier properties and good chemical resistance except for alkalis (which hydrolyse it). Its structure varies from amorphous to fairly high crystalline; it can be highly transparent and colorless, but thicker sections are usually opaque and off-white. PET material is heat-resistant until 180°C, does not decrease in strength, its melting temperature is around 260°C. Above 250°C the crystallite section is continuously disappearing, so the melting process of the material could be observed. Usually the material is processed in the melt state, so only above this temperature point it is worth to study the rheological properties [4]. The measurements were performed at 270°C, 280°C and 290°C.

3. Method

3.1. Rotational viscometry

ARES G2 rotational rheometer was used for our measurements, which means a shear viscosity investigation method in low shear rate range and temperature steps near the processing temperature. During the measurements the shear rate was changed between 0,05 – 20 1/s. With the rotational viscometry torsion flow could be measured in polymer melt with cone-plate geometry. The measuring technique conceptual layout is shown in Figure 1. The CMT means combined motor and transducer concept [8].

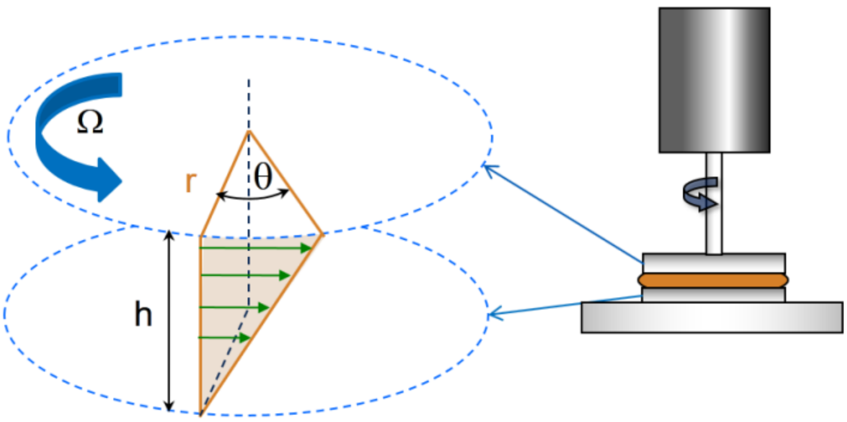

Figure 1. Rotational viscometry CMT conceptual layout and marking system [5] [6]

3.2. Capillary rheometer

The capillary rheometer is a measuring device in which the shear viscosity of thermoplastics can be measured. Goettfert Rheograph 25 capillary rheometer was used with100-10 000 ^{1}/s shear rate. The measuring temperatures were near to the processing temperature [7]. The measuring method is based on continuous melt flow in known geometry capillary(s), while the pressure drops were measured between capillary inlet side and atmospheric pressure (Figure 2.).

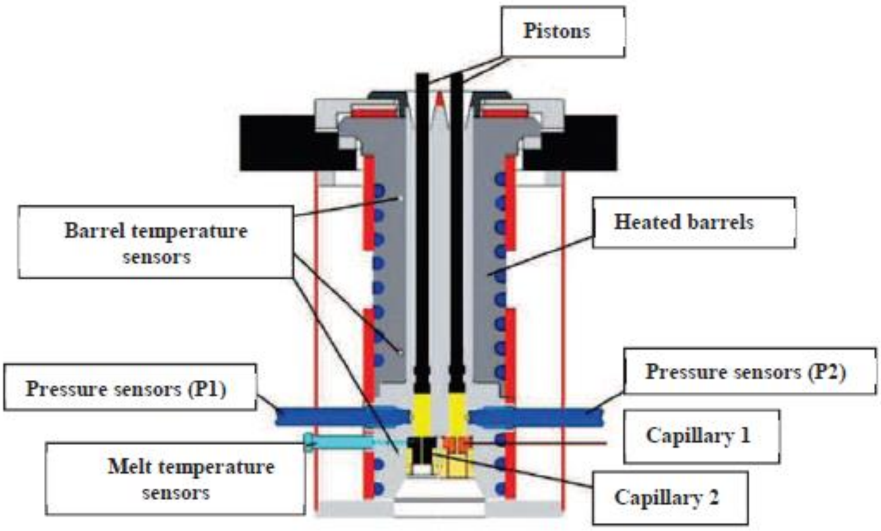

Figure 2. Capillary rheometer conceptual layout [7]

The variable parameters are the barrel temperature and piston speed (parallel with the deformation rate). In this case, the shear viscosity depends on temperature and deformation (shear) rate. In our tests the shear rate changed between 100-10.000 ^{1}/s and the barrel temperatures changed in three steps around the material supplier recommended process temperature. The measuring method uses two other geometry capillaries because of the entrance and exit effects (pressure drop). With this equipment in one step we can do the Bagley correction (correct the pressure drop), which gives the real shear viscosity [8].

4. Results and Discussions

4.1. Rotational viscometry

The viscosity curves were determined in function of shear rate. The measurements were performed with original material and with recycled material. The viscosity of the original material is shown at different temperatures in Figure 3.

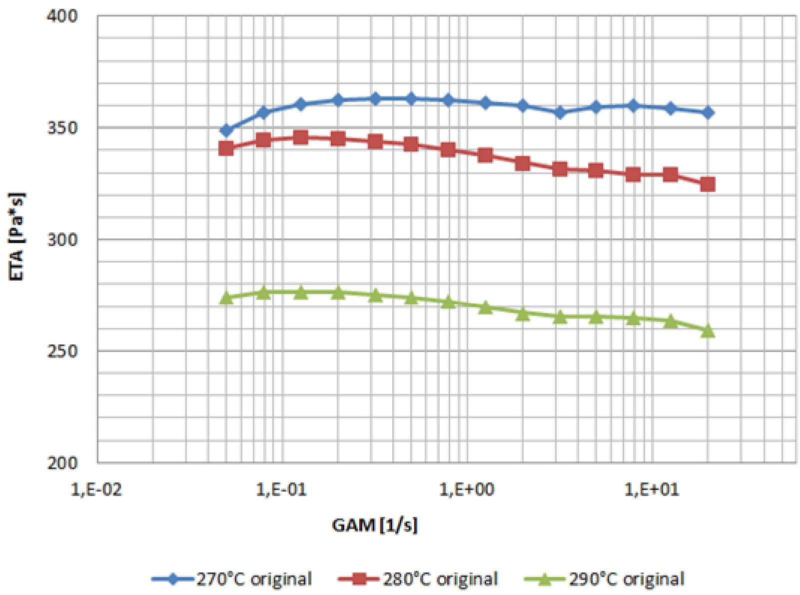

Figure 3. Shear viscosity curves of original PET

The curves at different temperatures are not the same. It can be observed that the viscosity is the highest at 270°C. The higher is the temperature, the lower is the viscosity.

From the differences of the curves, the transformation of the molecular structure can be determined. The viscosity of the recycled materials is shown in Figure 4.

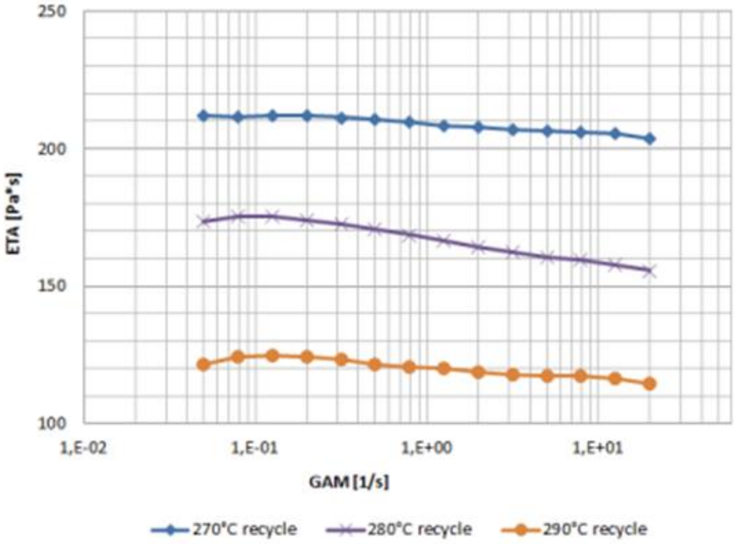

Figure 4. Shear viscosity curves of recycled PET

The recycled material shows the same tendency. Increasing the temperature the viscosity decreases. During the recycle and grinding processes, the molecules in the materials can easily degrade. It is because of the rising thermal and mechanical strain during the processes. The degradation makes the molecular weight lower, so the molecular density dispersion broadens. The polymer with shorter chains can move easier. The continuously decreasing molecular weight is the impact of the recycling. The lower molecular weight means easier molecular movement. Above the glass transition temperature the segments of the polymer move increasingly. The viscosity of the original and recycled materials is shown in Figure 5.

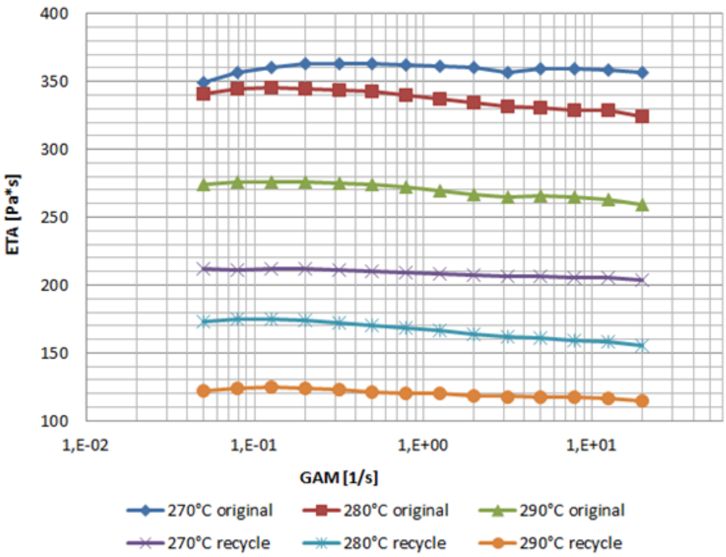

Figure 5. Shear viscosity curves of original and recycled PET

In each diagram the viscosity curves show a typical form. In the curves of Newtonian fluids it could be recognized linear curves which do not depend on the shear rate. In our measurement the viscosity curves approach to horizontal, but they decrease in a small extent. It was confirmed that these materials behave like non Newtonian fluids. At constant temperature the difference between the original and the recycled materials approaches 150 Pa*s.

4.2. Capillary rheometer

The measurements were performed with original material and with recycled material. The viscosity of the original and recycled materials is shown in Figure 6.

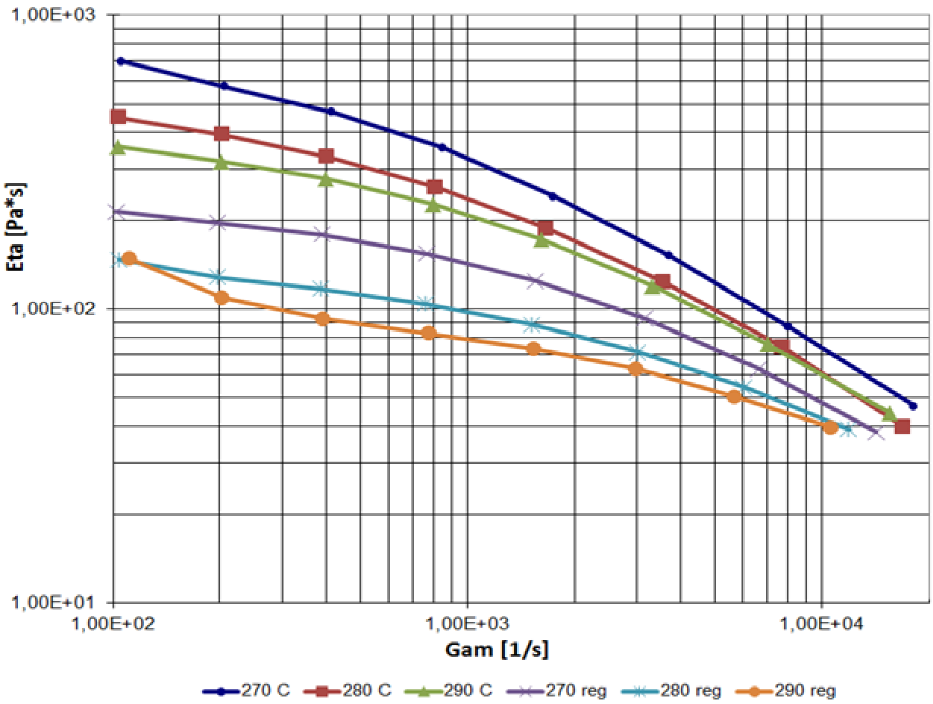

Figure 6. Original and recycled PET shear viscosity curves

Comparing the data to the 270°C viscosity curve of the base material in the lower shear range (100-1000 1/s): There is a ~30 - 35% reduction at 280 °C, and we can see a 40 - 50% reduction at 290°C. As we approach the larger shear ranges (10000 - 18000 1 / s) the differences are reduced: at 280 °C ~15 - 18%, while at 290°C ~7 – 13%.

In the initial shear rate range the recycled material shows a 70% reduction at 270°C, while in the higher shear range this value shows a 15% - 30% reduction. We could observe viscosity reduction in the results: at 280 ° C, ~ 65% and at 290 ° C ~ 50%. Although the initial viscosity values at 290 ° C show a different tendency compared to the curve we measured at the previous two temperatures. This can be attributed to a measurement error with great certainty.

From these values we can conclude that we have to consider viscosity change of the material during the recycling, because with such a degree of deviation we could assume that the material will not be able to withstand the requisitions, which are required by the design.

5. Conclusion

In our investigation one type of material was measured, but at different temperatures. An original and a recycled material were examined. The measurements were performed by an ARES G2 rotational rheometer and a Goettfert Rheograph 25 capillary rheometer at 270°C, 280°C and 290°C. The shear rate was changed from 0.05 to 20 ^{1}/s and 100-10 000 ^{1}/s.

It was determined that the viscosity of the recycled material is lower than the value of the original material at each temperature. The molecules of the recycled material were supposed to shorten during the grinding process. If the temperature increases the shear viscosity decreases, but there was no linear correlation between these results. The structure viscous behavior of the polymer melt was revealed, while the viscosity decreases with increasing shear rate. There is a lot of possibility in this searching area so we would like to make further investigations.

Acknowledgement

This research is supported by EFOP-3.6.1-16-2016-00006 "The development and enhancement of the research potential at John von Neumann University" project. The Project is supported by the Hungarian Government and co-financed by the European Social Fund.

References

[1] Ronkay Ferenc: PET palackok anyagának fizikai újrahasznosítása 2006
[2] Czvikovszky Tibor, Nagy Péter, Gaál János: A polimertechnika alapjai, Műegyetemi kiadó, Budapest, 2000 http://www.tankonyvtar.hu/hu/tartalom/tkt/polimertechnika-alapjai/adatok.html
[3] http://plasticker.de/recybase/docs/27055_1463559873.pdf
[4] F.Ronkay, T.Czigány: Properties modifying of recycled PET with additives.
[5] General Rheo Training_2015_v1 with questions
[6] TA instruments factory Ares G2 rotational viscometer equipment education material – Study of
[7] rheology theory and applications; http://www.tainstruments.com
[8] A Szűcs 2010 Műanyag ömledékek nagysebességű áramlásának tanulmányozása, PhD thesis,
[9] University of Miskolc (in Hungarian).
[10] http://www.kerpely.uni-miskolc.hu/downloads/docs/cikkek/szucsandras-ertekezes.pdf
[11] G Toth, A Bata and K Belina: Polycarbonate melt shear viscosity in wide range shear rate
[12] IOP CONFERENCE SERIES: MATERIALS SCIENCE AND ENGINEERING 175:(1) Paper 012056. (2017) 4th International Conference on Competitive Materials and Technology Processes (IC-CMTP4). Miskolc, Magyarország: 2016.03.03 -2016.10.07.