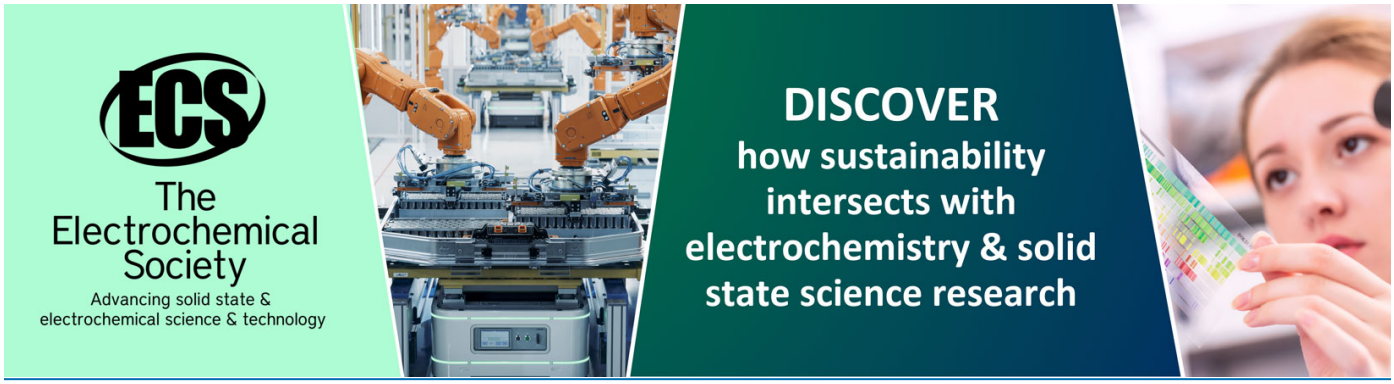
Melt shear viscosity of original and recycled PET in wide range shear rate



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Melt shear viscosity of original and recycled PET in wide range shear rate

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Abstract. Among the plastic materials, the PET is the most often used one. The collecting of the material to be recycled is partially solved. This way we have great quantity of reground PET. During the regrinding the structure of molecules changes and these properties can be measured by rheology methods. Polymer melts are -Newtonian fluids and the flow curves of the materials can be used to characterize the molecular weight and the molecular weight distribution. This research is focused on the characterization of original and ground PET materials. Inherent viscosities were determined to characterize the materials. Flow curves of pure and blended materials were measured at different temperatures.

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, about 10-25 % of the recycled PET is used. Our goal is to increase the quantity of the recycled PET. From the recycled materials the new bottles, products can be made. This requires a development path and a more sophisticated technology since it is not easy to determine for 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 the product characteristics [2].

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

2. Experimental

2.1 Material

RAMAPET N180 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.

The PET is hygroscopic, and since moisture adversely affects the IV during the 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 is typically below 50 ppm [3].

The PET has good surface hardness, stiffness, and dimensional stability that absorbs low volume of moisture. The PET material is heat-resistant until 180 °C, it 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 molten state, so it is worth to study the rheological properties only above this temperature point [4]. The measurements were performed at 270 °C, 280 °C and 290 °C.

3. Method

3.1 The rotational viscometry

Our measurements were performed by a shear viscosity investigation method; that means a low range of shear rate and temperature steps near the processing temperature. This was the reason why the rotational viscometry was measured in the low shear rate range (0.05 – 20 1/s). With the rotational viscometry we could measure torsion flow in a polymer melt between parallel plates. The measuring technique conceptual layout is shown in Fig. 1. The CMT means combined motor and transducer concept [5].

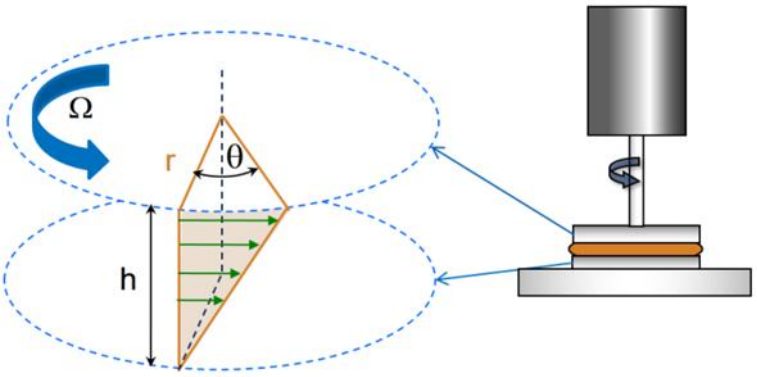

Figure 1. Rotation viscometry CMT conceptual layout and marking system [5]

From the marking system the stress, deformation and deformation rate components can be calculated with the equations described below:

$$\sigma = \frac{2}{\pi r^3} \times \mathrm{M} \tag{1}$$

$$\gamma = \frac{r}{h} \times \theta \tag{2}$$

$$\dot{\gamma} = \frac{r}{h} \times \Omega \tag{3}$$

where r is the circle plate radius; h is the gap between the plates; is the driving engine rotation (rad); is the driving engine angular speed (rad/s) and M is the torque (Nm) [5].

The stress and deformation rate can be calculated from expressions (1) to (3) and the shear viscosity can be defined. It is a frequent measuring technique to determine shear viscosity for small molecular liquids, or oils and thermoplasts at low shear rate. With this method we can measure at higher temperature (to 600 °C) and monitor the degradation process. For our testing we used the SMT (Separate motor and transducer) conceptual TA Ares G2 type rotation viscometer with 25 mm parallel circle plates and 0.4 mm gap. In this type of rotation viscometer the drive engine and the transductor are separated [6].

3.2 The capillary rheometer

The capillary rheometer is a measuring device in which we are able to measure the shear viscosity of thermoplastics. The shear rate ranges between 100 and 10 000 1/s near to the processing temperature [7].

The GoettfertRheograph 25 capillary rheometer was used. The measuring method is based on continuous melt flow in known geometry capillary(ies), while the pressure drops is measured between the capillary inlet side and the atmospheric pressure (Fig. 2.).

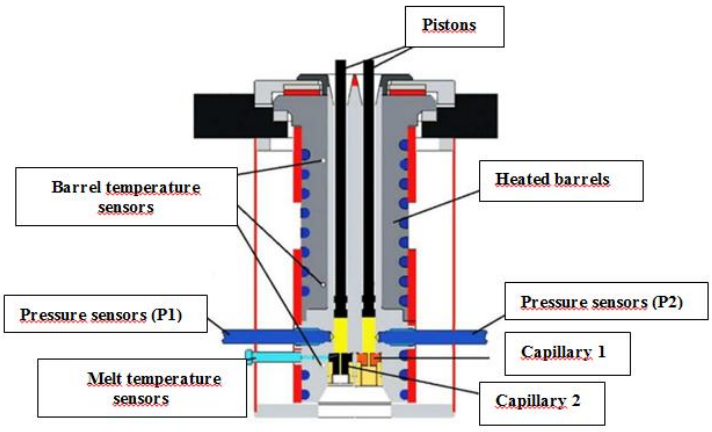

Figure 2. Capillary rheometer conceptual layout [7]

The variable parameters are the barrel temperature and the piston speed (parallel to the deformation rate). In this case, the shear viscosity depend 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

4.1 The capillary rheometer

The viscosity curves were determined as functions of the shear rate. The measurements were performed with original material and with recycled material. The viscosity of the original material is shown in Fig. 3.

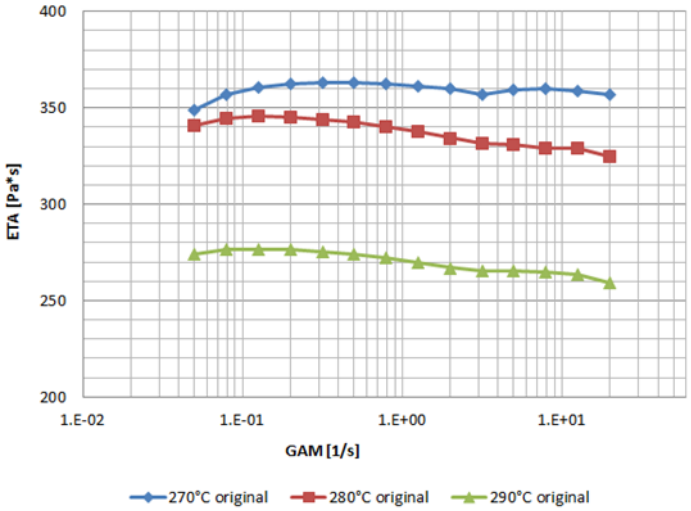

Figure 3. Original PET shear viscosity curves

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 Fig. 4.

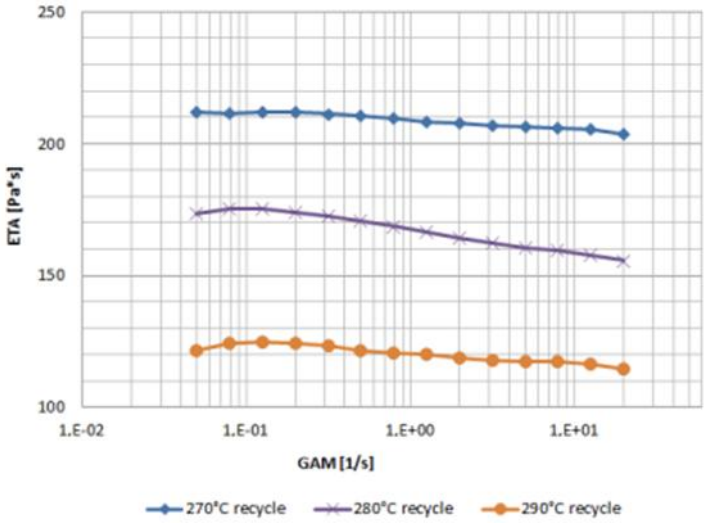

Figure 4. Recycled PET shear viscosity curves

The recycled material shows the same tendency (Fig. 4). Increasing the temperature the viscosity decreases. During the recycling and grinding processes, the molecules in the materials can easily degrade. This is caused by the rising thermal and mechanical strain during the processes. The degradation makes the molecular weight lower, so the molecular density dispersion widens. The polymer with shorter chains can move easier.Above the glass transition temperature the segments of the polymer move increasingly.The viscosities of the original and recycled materials are shown in Fig. 5.

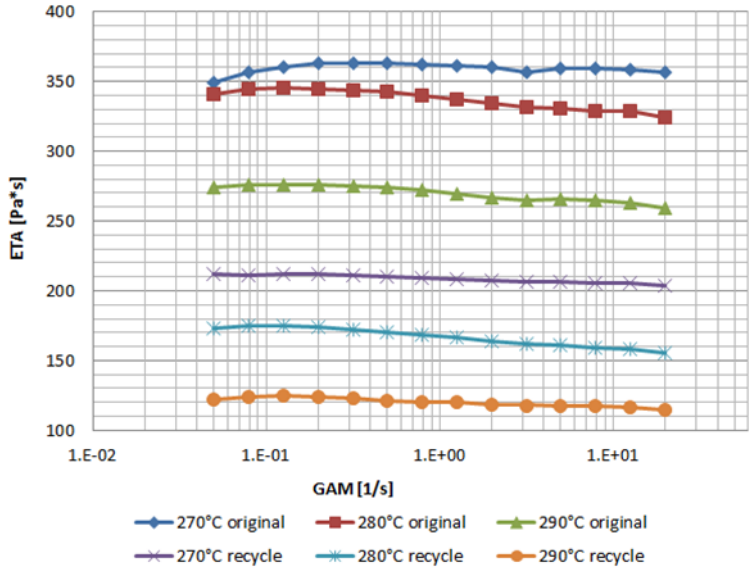

Figure 5. Original and recycled PET shear viscosity curves

In each diagram the viscosity curves show a typical form (Fig. 5). In the curves of newtonian fluids it could be recognised that the curves are almost linear and not dependent on the shear rate. In our measurements, the viscosity curves approach the horizontal line, but they do decrease within a small range. It was confirmed that these materials behave like non newtonian fluid. At a constant temperature, the difference between the original and the recycled materials approaches 150 Pa*s. The impact of the recycling is the continuously decreasing molecular weight. The lower molecular weight means easier molecular movement.

4.2The capillary rheometer

The measurements were performed with original material and with recycled material. The viscosities of the original and recycled materials are shown in Fig. 6.

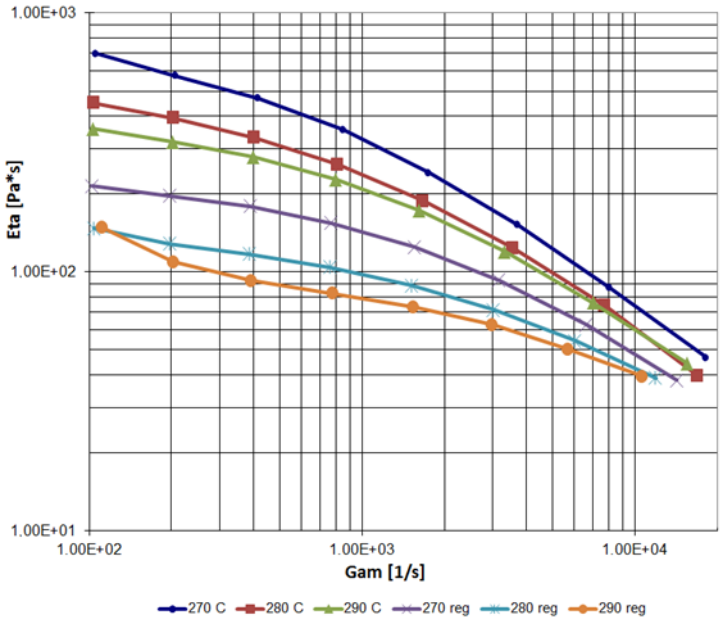

Figure 6. Original and recycled PET shear viscosity curves

Comparing the data to the 270 °C viscosity curve in the lower shear range: There is a ~30 – 35 % reduction at 280 °C, and at 290 °C we can see a 40 – 50 % reduction. 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 speed 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 the following viscosity reductions 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 a great certainty. From these values we can conclude that we have to consider the 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 requirements, which are set by design.

5. Conclusion

In our investigation we measured one type of material, but the temperature of the process was different. An original and a recycled material were examined. The measurements were performed by an ARES G2 rotational rheometer and 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 two variables. The structure viscous behavior of the polymer melt was revealed. This area offers lot of possibilities for future research, so we hope to continue with further investigations.

6. Acknowledgement

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