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POLYMER MELT VISCOSITY MEASURING BY AN INJECTION MACHINE

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Abstract
In my article I would like to present a special measurement procedure of polymer melt viscosity measurement. The general idea is based on the capillary viscometer measuring technique, but It is closer to the production process' variable conditions, for example the change in melt cross section, melt temperature increase with the shear stress and the melt pressures. An injection machine is able to measure and regulate the filling process pressure, speed and temperatures so, everything is given for a viscosity measurement. Of course It is not a standard measuring process, but it may be very interesting to compare the results with the standard measuring tool's results. In the end I illustrate the results of the standard and non standard procedures in one diagram. The flow curves show a wide range of shear rate, temperature, and shear stress.

1 Introduction

A significant number of plastic products are produced using injection moulding. In the injection moulding method the plastic raw material is formed as a melt. During the production process it has a great importance knowing and controling the raw material's flow properties. The science of this area is rheology.

For plastic melt viscosity measurements the simplest and the most common testing equipment and measuring instrument is the MFI technique. However, an MFI measurement gives very little information of what really happens with the melt during processing. Well-equipped testing laboratories can have acces to rotational and oscillation viscometers which provide accurate results, but ignore the changing condition variables during the processing. The processing conditions are better taken into account by a capillary rheometer, which is closer to the injection molding shear rate range (10-20E 1/s) may also examine the material behavior. Any of these measuring instruments are expensive and the users need to know the rheological properties of the thermoplastic polymers.

In the article we would like to present a measurement method of polymer melt viscosity measurement. The target was to measure viscosity in line the production taking into consideration the process variables. Nowadays with an injection machine we can control and measure all of the process variables, for example the pressures, temperatures and speeds. After building the measurement system and working out the proper data processing and measurement optimization, the measurement of polymer rheology becomes viable. The flow curves and viscosity curves measured by the injection machine are closer to the actual questioned properties thant the ones measured by standard measuring techniques. This article describes the measurement and mathematical formulas I used in the evaluation process.

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2 Method

2.1 The theoretical background of viscosity measurement [1]

Figure 1. shows a piece of a liquid moving at a strain rate $\dot{\gamma}$ under an applied shear stress of τ . The viscosity of the liquid is the ratio of the applied shear stress to the resulting strain rate (or equivalently, the ratio of the shear stress required to move the solution at a fixed strain rate to that strain rate). The shear strain in Fig. 1. is:

$$\gamma = \frac{du}{dy} \tag{1}$$

where u is displacement in the x direction. The strain rate is therefore:

$$\dot{\gamma} = \frac{d}{dt}\frac{du}{dy} = \frac{d}{dy}\frac{du}{dt} = \frac{dv_x}{dy} \tag{2}$$

where v_x is velocity in the x direction. The relations between viscosity (**η**), shear stress (τ), and shear rate ($\dot{\gamma}$) are

$$\tau = \eta\dot{\gamma} \qquad \text{or} \qquad \dot{\gamma} = \frac{\tau}{\eta} \qquad \text{or} \qquad \eta = \frac{\tau}{\dot{\gamma}} \tag{3}$$
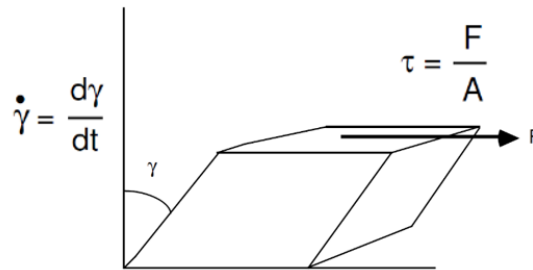

Figure 1. A piece of a liquid moving at shear rate $\dot{\gamma}$ under an applied shear stress of τ .[1]

A Newtonian fluid is one in which the viscosity is independent of the shear rate. In other words a plot of shear stress versus shear strain rate is linear with slope **η**. In Newtonian fluids all the energy goes into sliding molecules on each other. In non-Newtonian fluids, the shear stress/strain rate relation is not linear.
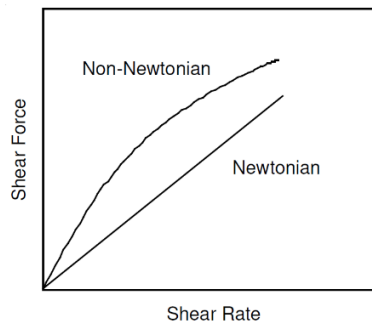

Figure 2. Schematic plots of shear force vs. shear rate for Newtonian and non-Newtonian fluids. [1]

Typically the viscosity drops at high shear rates — a phenomenon known as shear thinning. Although the following development will not discuss shear rate effects in detail, the possibility of experimental results being affected by the shear rate of the measurement should be kept in mind. Plots of shear force vs. shear rate for Newtonian and non-Newtonian fluids are given in *Figure 2*.

2.2 Calculation with round hole capillary

Using the notation in the *Figure 3* and equations (4), (5) a round hole capillary viscosimetric interpretation becomes clear [3].
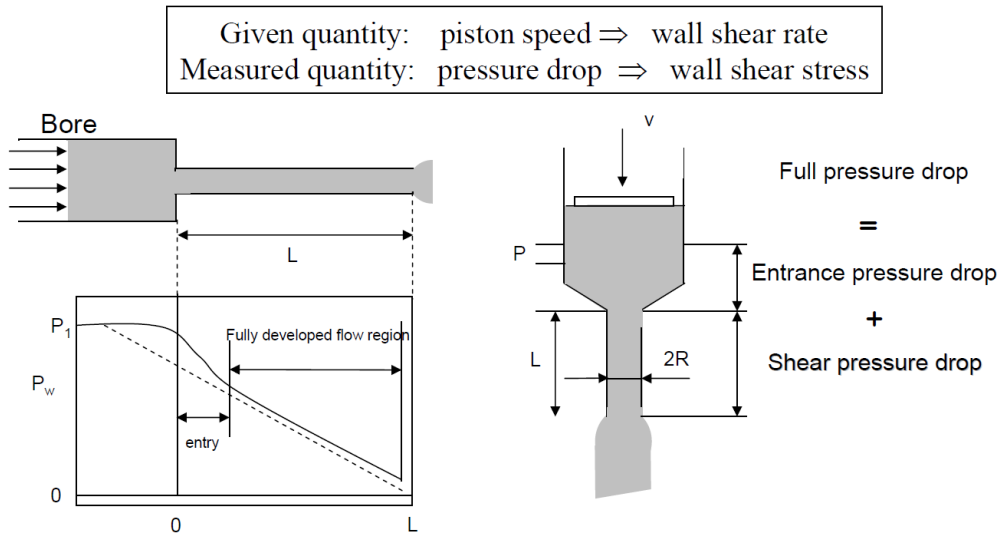
Given quantity: piston speed $\Rightarrow$ wall shear rate
Measured quantity: pressure drop $\Rightarrow$ wall shear stress

Figure 3. Round hole capillar viscosimetria interpretation [3]

The apparent shear rate and shear stress are possible to calculate with the following equations (4), (5). The results of these equations need to be corrected with a factor mentioned later.

$$\dot{\gamma}_{app} = \frac{4 \cdot Q}{\pi R^3}$$

(4)

Where Q is the volumetric flow and R is the capillary radius.

$$\tau_{app} = \frac{R \cdot \Delta P}{2 \cdot L}$$

(5)

Where Δp mean the pressure drop in the capillary and L is the capillary length.

2.3 Viscosity measurement with an injection molding machine

Hereinafter we deal with the non-Newtonian polymer melt viscosity measurement with an injection molding machine. The polymer type what we tested was polycarbonate (DOW Calibre 303 EP). It is a thermoplastic water clear polycondensation type plastic.

We used an ENGEL Victory 1050/300 injection molding machine and two special geometry capillary nozzles. In this case the injection machine was used as a capillary rheometer. With the injection molding machine repeatedly plasticizing and injecting with varied injection settings through of the nozzles into the open air. Variables adjusted were for example the injection speed and the temperatures of the barrel zones. With this method the effect of temperature and injection speed on the viscosity can be shown.

The capillaries used were very important in terms of measurement because this gave us the possibility to calculate the corrected viscosity parameters. The main difference between the two capillaries were the holes length and diameter ratio. The diameter is unified (2mm) and the length was changed, one is very short just 2mm and another 20mm.

This difference is necessary for applying the following Bagley correction. During capillary rheometer tests, pressure is measured above the die inlet. The true pressure drop along the capillary is therefore 'hidden' by an additional pressure drop at the entrance of the die, where the flowing material goes from a wide reservoir (the main cylinder or barrel) to a narrow capillary, possibly also creating turbulences. Assuming that the same additional pressure drop takes place with different

capillary lengths (but keeping constant the barrel and capillary diameters, and inlet shapes), it's possible to correct the pressure reading and estimate much more accurately the true pressure drop. This is called the Bagley correction (named after Edward B. Bagley, American scientist) [2].

3 Results

3.1 Primary results

One of the primary results shown the Figure 4. what can be interpreted as a the temperature effect on viscosity and pressure difference next to constant injection speed. Every line represents a three shot average value. On the time axis between two and four seconds the pressure value is constant so the sampling can be performed. At the constant section of the pressure lines we only need one point from one temperature set for the measurement. So, in this measurement we can log pressure differences (measured), volumetric flow (from the injection speed and barrel diameter), and capillary geometry.
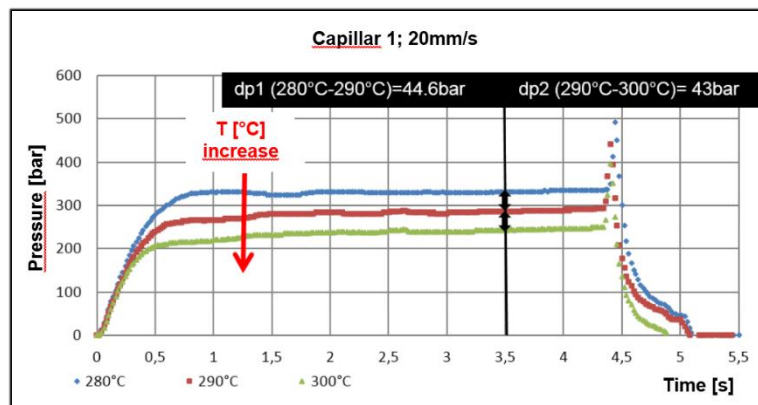
Figure 4. Primary result with capillary 1

First showing the viscosity curves which were calculated from the non corrected data (*Figure 5*). To test our method we take a control measuremen with a capillary viscometer. The reference curves are shown in *Figure 5*.
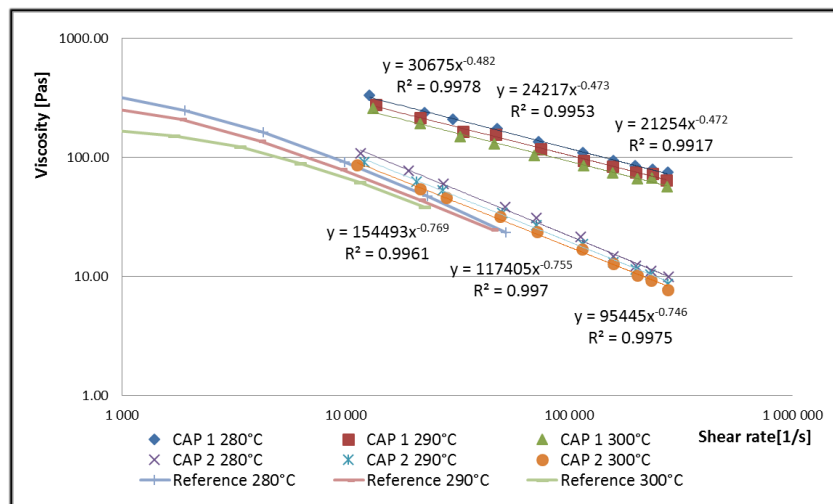

Figure 5. Apparent viscosity curves Capillar 1 vs. Capillar 2

3.2 Correction

First showing the Bagley correction method during the practical process. If we correlate the measured pressure points to the capillaries' lenght and diameter ratio we get *Figure 6*. Every two point measured with the same injection speed (shear rate) but with a different capillary. The two

points can be connected with a line. The line's equation shows us where the line and the vertical axis (pressure axis) intersects.
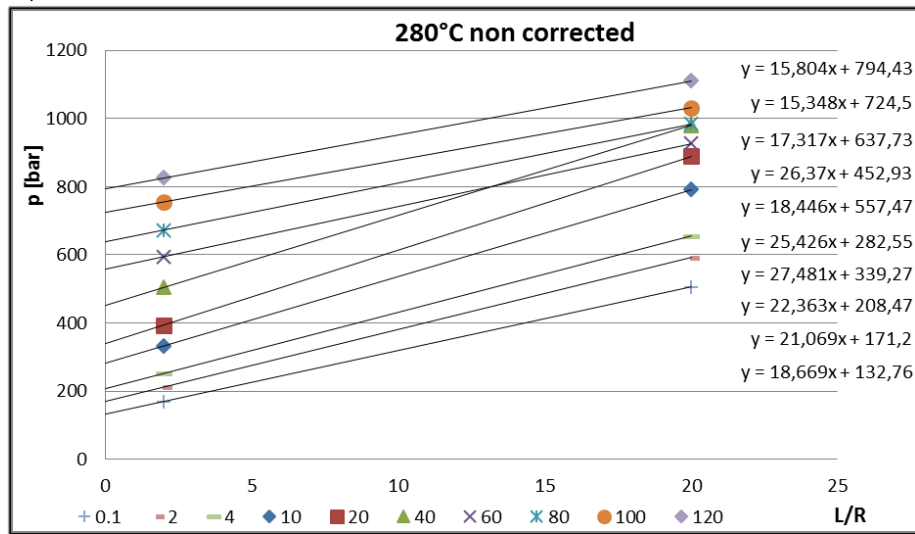

Figure 6. Capillaries L/D and pressure connection (280°C)

If we subtract the points pressure value with the intersection's value the lines' starting point is moved to the origo (*Figure 7*). With this process the Bagley correction is taken. The real pressures are the decreased values.
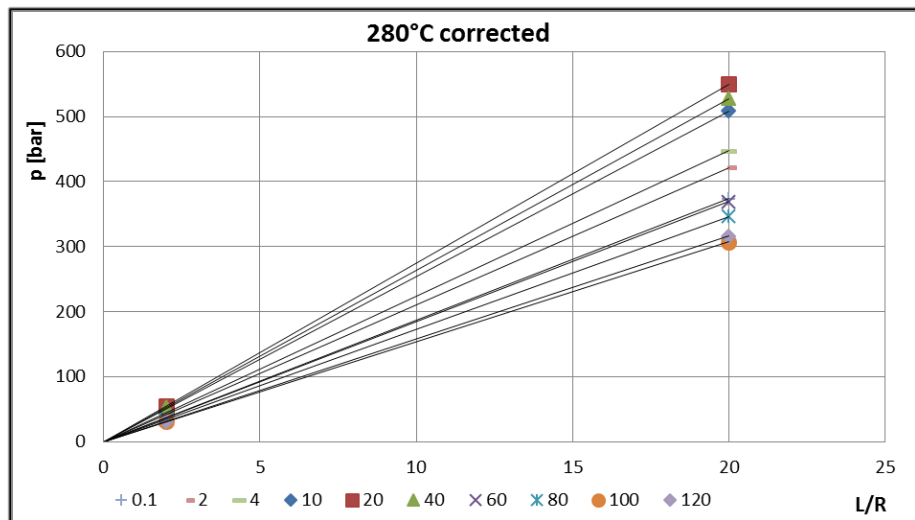

Figure 7. Capillaries L/D and corrected pressure connection (280°C)

4 Discussion

After the corrections *Figure 8.* shows the corrected viscosity curves. It can bee seen that our measurements are close to the reference values and we can increase the shear rate range higher than the reference capillary viscosimeter is able to.
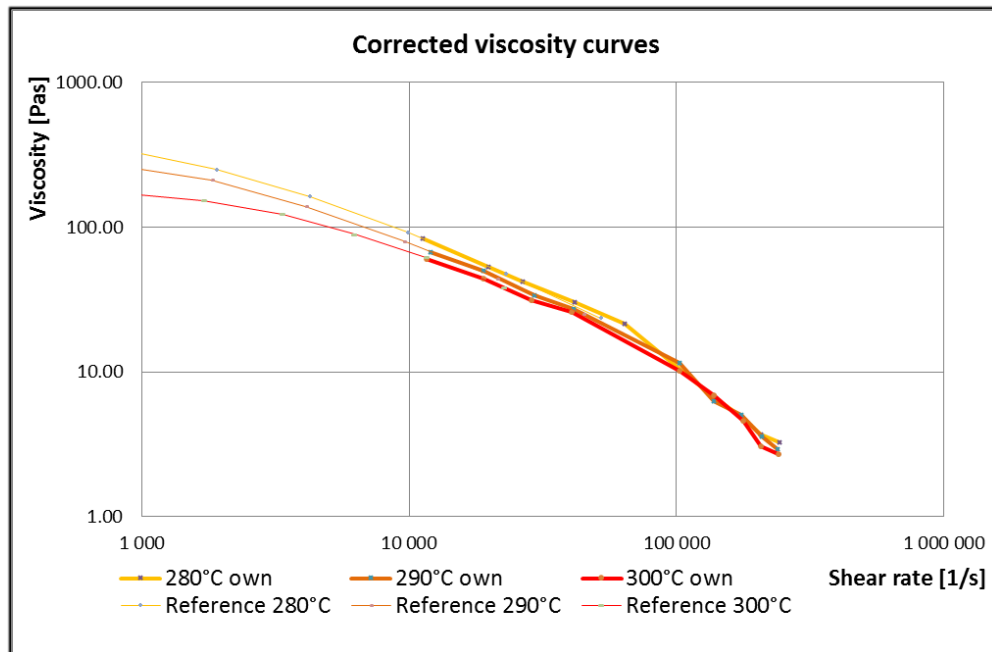
Figure 8. Corrected viscosity curves compared to reference

5 Conclusions

In our work we present a novel measurement method of polymer melt viscosity measuriement using an injection machine. Our target was met as the flow curves and viscosity curves measured by the injection machine show good correlation to the properties what were measured by standard capillary measurement techniques. Currently this way of viscosity measurment too slow but after further optimization this method can be quicker and simpler. The most important thing is althoug that we only use an injection machine and two capillaries for the measurements which are more accessible for industrial entities. In the future we would like to upgrade the method and further improve the procedure. In our plans after working out the upgrade, to have acces to this method of measurement one will just need a new software and two capillaries and one can measure viscosity anywhere and any time with an injection machine during the production process.

Acknowledgment

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