Online Rheometer - Increased selectivity through continuous measurement of the extensional viscosity

Introduction

Online process monitoring of rheological properties in polymerization and pilot plants is essential for fast and effective process control of polymer production. In addition to the pure polymer production, customized polymer-compounding to specific material properties is becoming increasingly important.
So far, the most common task of Online Capillary Rheometry has been continuously determination of Melt Flow Rate (MFR) and Melt Volume Rate (MVR) in order to adjust the production slots to the individual polymer specification more quickly.
To characterize the process ability of a polymer, beside determination of the Melt Flow Rate (MFR) also further rheological parameters such as shear and elongation viscosity is of high interest. In this paper we will illustrate how to carry out shear and elongational measurements by using a GÖTTFERT Online Rheometer.

Setup for measuring extensional viscosity by using a GÖTTFERT Online Rheometer (RTS)

RTS is an Online Die Rheometer with two capillaries measuring simultaneously. This arrangement is similar to the laboratory measurement by using a twin-die capillary rheometer, which is shown in Figure 1.
The measurement with the capillary rheometer is carried out with a long and a short capillary. The pressure loss of each capillary is plotted over length/diameter (L/D) ratio of the die and extrapolated to L/D of zero (Bagley correction [1] to determine entrance pressure loss) and corrected for the shape of the velocity profile by Rabinowitsch-Weissenberg [2]. Using Cogswell model [3], elongation viscosity is calculated from the entrance pressure loss and the apparent shear stress and shear rate of the long capillary.

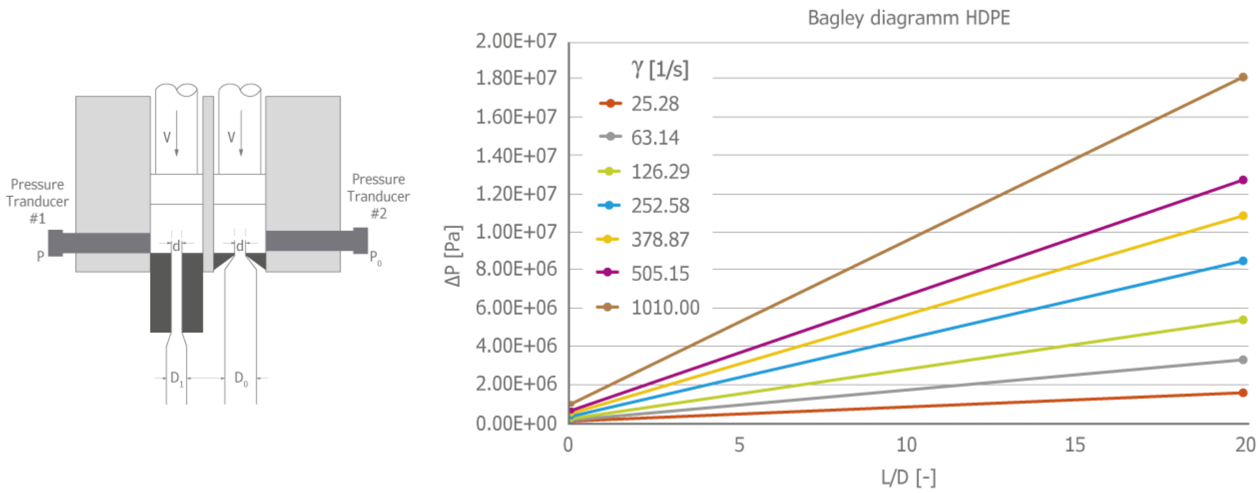


Figure 1: Twin Die capillary rheometer with Bagley correction

Figure 2 shows the basic arrangement of the online capillary rheometer RTS. The setup is similar to the capillary rheometer in the laboratory equipped with a long and a short capillary (zero length die). The pressure is determined in the RTS before and after the die.

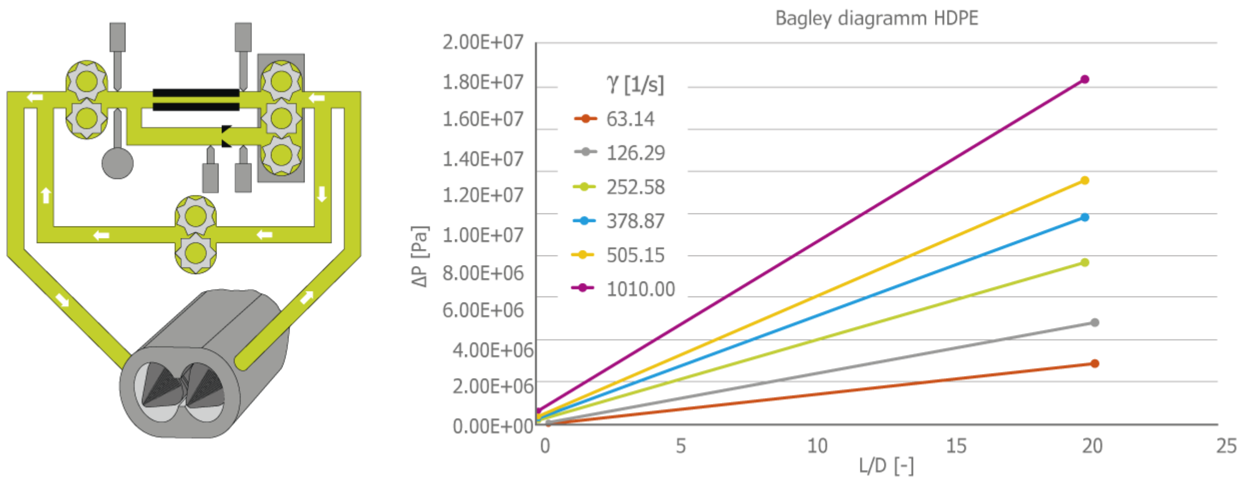


Figure 2: RTS with long and short die and Bagley Correction

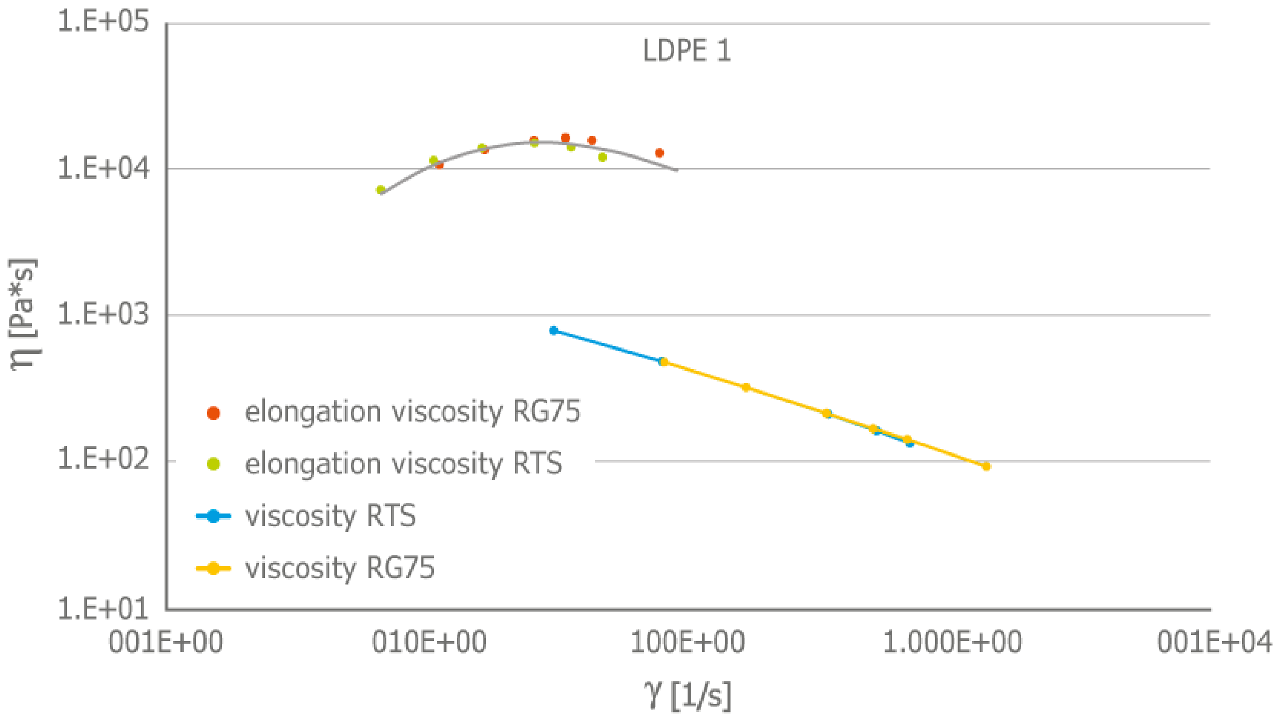
The great advantage of this arrangement with long die and zero length die in the RTS is that existing bores on existing devices can be used to retrofit or convert to this test method. The described arrangement in the RTS, which is also possible in the MBR-TD, opens up the unique possibility of measuring melt index in addition to measuring the shear and elongation viscosity.

Comparison of online and laboratory measurements

To check the arrangement, comparative measurements with the RG75 capillary rheometer (laboratory device) with the capillary configuration 40/2 and 0/2 mm length / diameter and the RTS (online device) with the capillary configuration 60/2 and 0/2 mm are used. The examined materials two LDPE.

The investigation is carried out in an average shear rate range of approx. 20-1000 1 / s. The viscosity is corrected in each case using Bagley (entrance pressure loss) and Rabinowitsch Weissenberg. Elongation viscosity and elongation rate can be calculated using the Cogswell model from entrance pressure loss, which is determined by the Bagley correction, and apparent rheological data. Elongation viscosity function determined in this way provides a simple description of the visco-elastic processes in cross-sectional transitions of flow channels.

The curves for 2 LDPE in Figure 4 measured at 190 °C show the characteristic curve of the strain hardening for the elongation viscosity due to the polymer branches in the test material. Both materials have the same MFR of 4 g/10 min, so they cannot be differentiated using the MFR method. In the slope of the viscosity function, is a similar slope with only relatively minor differences. Elongation viscosity, however, differs significantly. Elongation viscosity of material 2 shows a much more flexible course of the elongation hardening with also higher values.



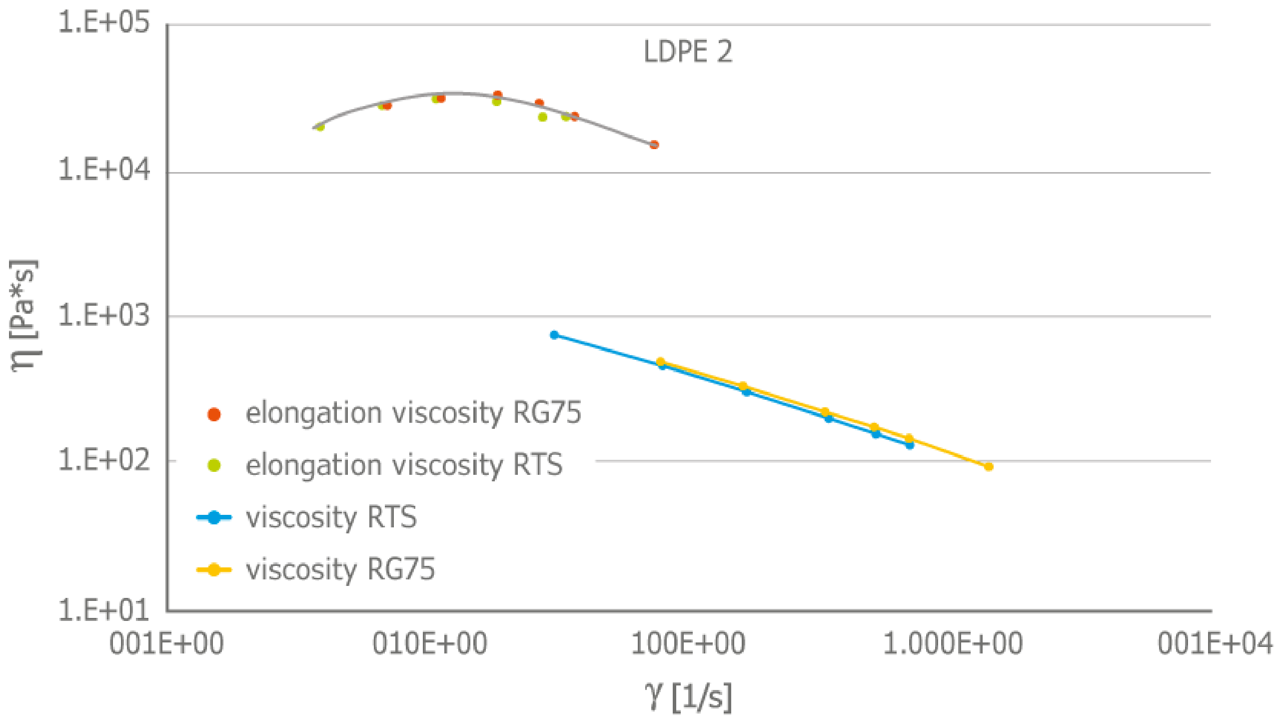


Figure 3: Comparison of shear and elongation viscosity between online and laboratory measurements of two LDPE

The analysis of the numbers now provides an even clearer picture than the graph. While there is no selectivity for the melt index, extension viscosity shows a very clear difference of over 60 % as a result of the different branching, whereas the viscosity gives an evaluation in the reverse order. Shear viscosity correlates with average molecular weight, which, however, differs only slightly here.

Material	LDPE1	LDPE2
Melt Index [g/10 min]	4	4
Difference [%]	-	0
Shear Viscosity [Pas] at $\dot{\gamma}$=1/s	5611	5006
Difference [%]	-	-10.8
Elongation viscosity maximum [Pas]	18600	30500
Difference [%]	-	+64 %

Table 1: Comparison of the selectivity of melt index, viscosity and elongation viscosity for 2 LDPE

This example shows that a clear material differentiation with different molecular weight distribution or branching is possible via the elongation viscosity, which cannot be detected via the methods frequently used up to now, such as the determination of the melt index.

Conclusion

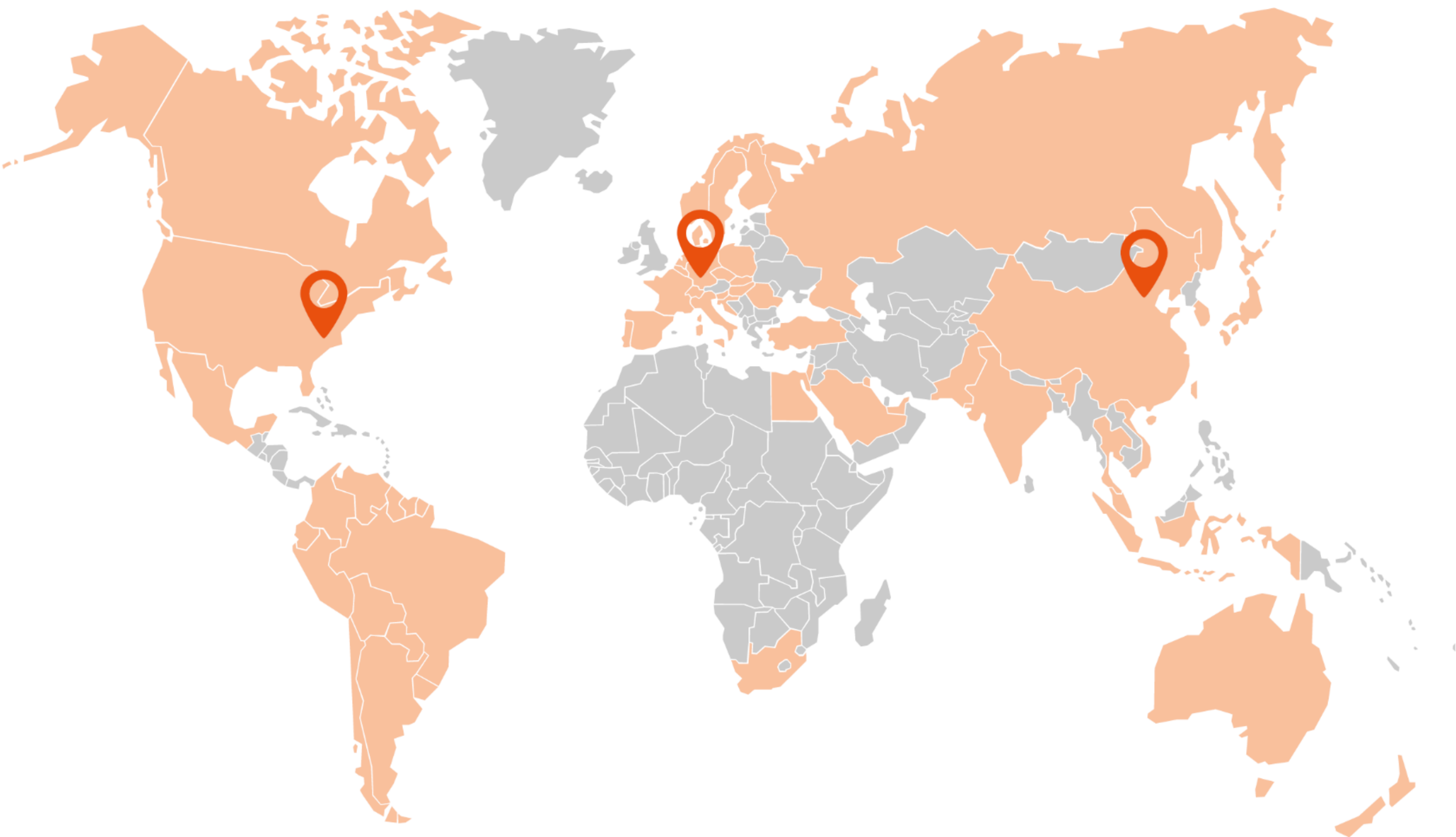
The determination of the elongation viscosity by using a GÖTTFERT Online Rheometer RTS via the zero-length die is a very effective option to increase the measuring range and the application of the Online Rheometer.

A comparison test with a high-pressure capillary rheometer shows a very good correlation between the data obtained in the laboratory and the data obtained online.

The extension in regard to the determination of the elongation viscosity function provides a high level of selectivity for different material structures such as branching and molecular weight distribution that cannot be achieved with a conventional measuring arrangement.

The Add-on provides a determination of MVR / MFR, apparent and corrected viscosity and elongation viscosity in an alternating mode of operation, which is only possible with GÖTTFERT Online Rheometers.

THIS IS RHEOLOGY



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