Rheological measurements with the new HAAKE MiniLab 3 micro-compounder, and their correlation with dynamic oscillation data

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Introduction

The Thermo Scientific™ HAAKE™ MiniLab Series micro-compounder is well known for being the ideal equipment, when it comes to compounding and processing of very small sample quantities of about 7 ml.
The HAAKE MiniLab 3 is a small conical twin-screw extruder equipped with a back flow channel and a by-pass valve, which allows users to control the residence time of the sample in the compounder.
The patented design of the back flow channel has a slit-capillary flow channel and two pressure transducers, which are used to measure the pressure drop in the capillary. From the pressure drop and the geometry of the slit capillary, a shear stress can be calculated. From the selected screw speed and the measured back pressure, a shear rate is correlated. The shear stress and the shear rate values are used to calculate the relative sample viscosity at different screw speeds.
This lab report shows the correlation between the relative viscosity measurements done on the new HAAKE MiniLab 3 micro-compounder, with results of absolute rheological measurements done with a high end rheometer the Thermo Scientific™ HAAKE™ MARS™ 60 rotational rheometer.

Materials & Sample Preparation

For this study two different LDPE grades (Lupolen 1800H and Lupolen 1800S from LyondellBasell) have been mixed in different ratios.
To have consistent sample preparation and a homogenous product, all samples have been pre-compounded on a Thermo Scientific™ Process 11 Twin-screw Extruder.

Table 1: Compounding of the samples

No.	Sample	Compounding
Sample 1	"LDPE1800 H"	100% Lupolen 1800H
Sample 2	"LDPE1800 H2S1"	66% Lupolen 1800H + 34% Lupolen 1800S
Sample 3	"LDPE1800 H1S1"	50% Lupolen 1800H + 50% Lupolen 1800S
Sample 4	"LDPE1800 H1S2"	34% Lupolen 1800H + 66% Lupolen 1800S
Sample 5	"LDPE1800 S"	100% Lupolen 1800S

Testing Equipment

a) HAAKE MiniLab 3 micro-compounder with pneumatic ram feeder

Fig. 1: The HAAKE MiniLab 3 micro -compounder.

Set of co-rotating screws
Software: Thermo Scientific™ HAAKE™ PolySoft OS Software for MiniLab
N_2 purge

b) HAAKE MARS 60 rotational rheometer
20 mm parallel plates measuring geometry
Controlled test chamber (CTC) for temperature control
N_2 purge

Test Conditions & Test Procedure

Extruder
Sample weight: 6.5 g
Testing temperature: 190°C
Inert gas: N_2 purge
Feeding speed: 50 rpm
Testing speed: Speed program from 50 rpm to 350 rpm (controlled via the HAAKE PolySoft OS Software)
Rheometer
Testing temperature: 190 °C
Testing mode: Frequency sweep in controlled deformation (CD) mode
Frequency range: 0.1 - 628 rad/s
Deformation: 1%

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After a preheating time of about 10 minutes, the pressure transducers of the MiniLab micro-compounder have to be calibrated at the selected measuring temperature, to avoid any temperature effect on the pressure measurement.
The pneumatic feeding piston and the extruder barrel are constantly purged by a constant nitrogen flow to avoid the presence of oxygen during the tests, to prevent degradation of the LDPE samples.
The sample is then fed into the running extruder by means of the pneumatic feeding piston.
After 1-2 minutes, the extrusion pressure is equilibrated, which indicates that the sample is properly molten and ready for the rheological test.
The rheological test itself is done and controlled by the HAAKE PolySoft OS Software. The software runs the pre-programmed measuring steps (Fig. 2), checks when steady state conditions are reached, and then measures the pressure drop between the pressure transducers in the slit capillary channel to calculate the shear stress.

Results of Rheological and Extruder Tests

The results of two rheological tests with Sample 1 ("LDPE1800 H") can be seen in Fig. 3.
Each measuring point in the flow curve corresponds to one screw speed on the HAAKE MiniLab micro-compounder.
With increasing shear rate the viscosity decreases, due to the typical shear-thinning behavior of polymer melts.
In addition, the viscosity curves of the two independent tests are nearly identical, which proves the very good repeatability of the test method.

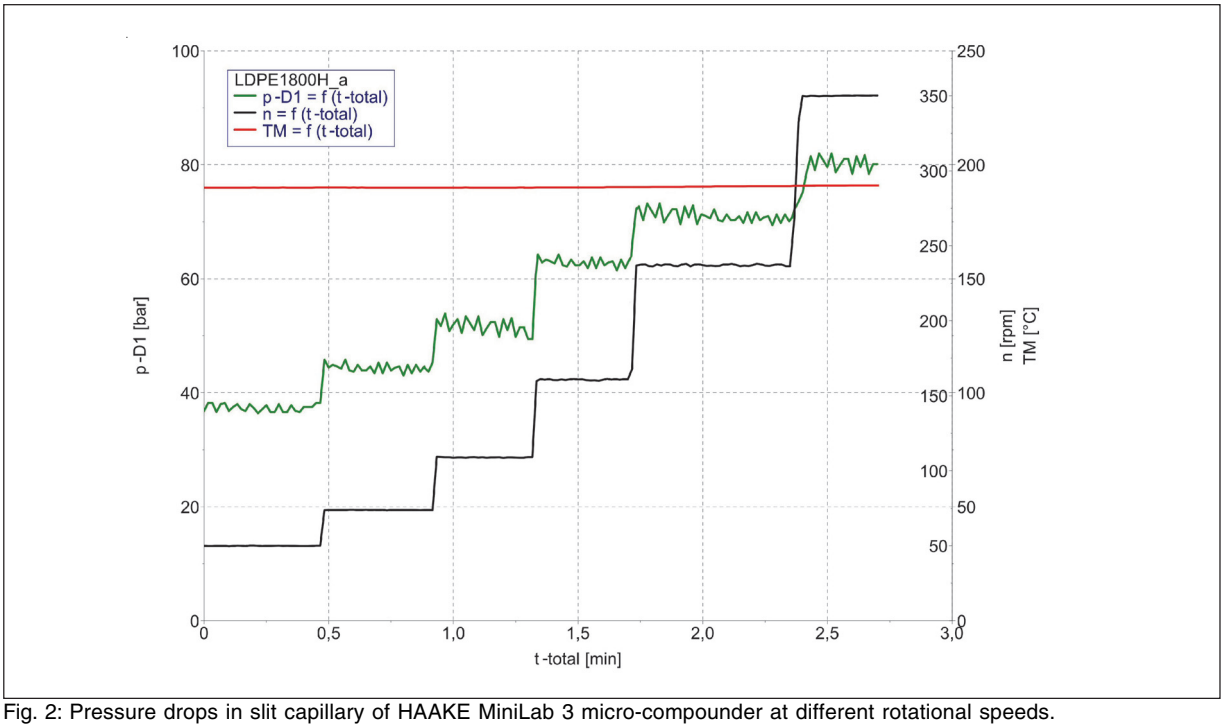

Fig. 2: Pressure drops in slit capillary of HAAKE MiniLab 3 micro-compounder at different rotational speeds.

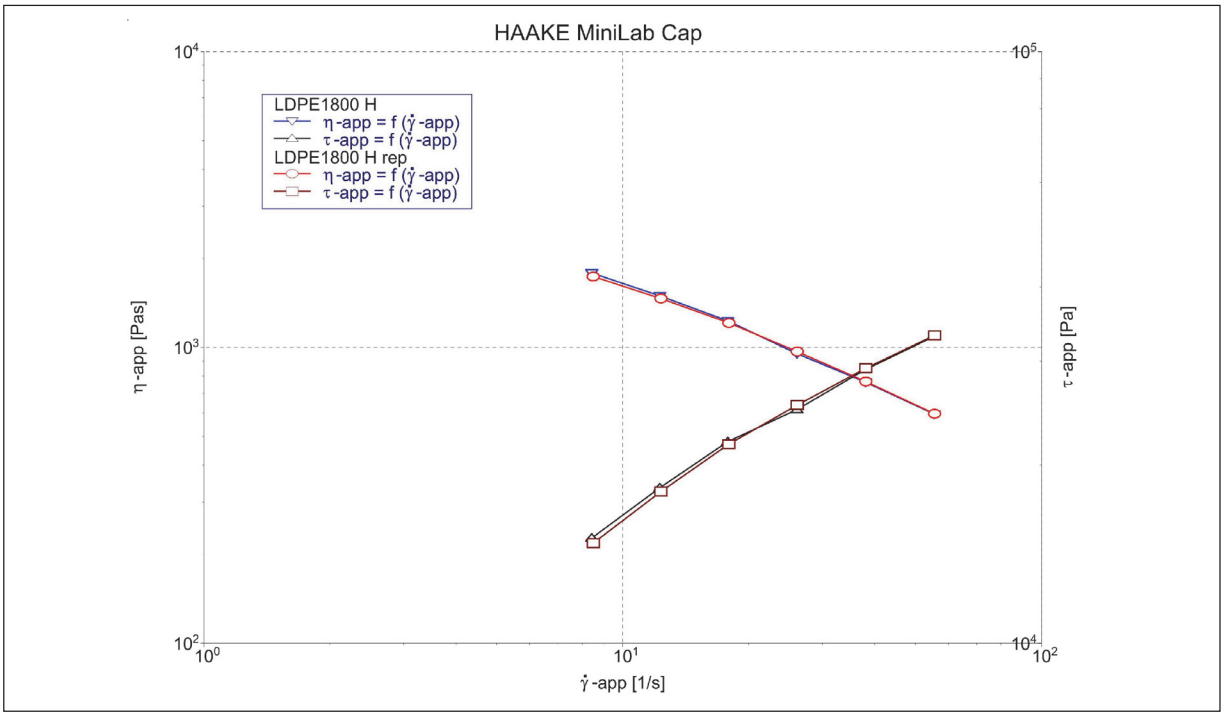

Fig. 3: Apparent viscosity $\eta_{\dot{\gamma}app}$ and apparent shear stress $\tau_{\dot{\gamma}app}$ as a function of the apparent shear rate for low density polyethylene. The results of two independent runs with the same polymer are presented.

Fig. 4 shows the results of the rheological measurements done with all five compounded samples in one diagram. It is easy to see how the sample viscosity drops with the increase of LDPE1800S and the decreasing amount of LDPE1800H in the compound.

Relative Measurement vs. Absolute Measurement

It is interesting how the relative tests results generated on the HAAKE MiniLab 3 correlate to the results done on an absolute Rheometer.

To check this, one of the compounds (Sample 3 "LDPE1800 H1S1") was tested in an oscillatory frequency sweep experiment on a HAAKE MARS 60 rheometer.

For the comparison of the tests with the HAAKE MiniLab micro-compounder and HAAKE MARS rheometer the rule of Cox-Merz relation is applied.

Empirically the two scientists who gave the Cox-Merz relation their names found that the steady-shear viscosity measured as a function of shear rate could be directly compared to the dynamic complex viscosity measured as a function of angular velocity:

$$|\eta^*|(\omega) = \eta(\dot{\gamma}) \text{ -> } \omega = \dot{\gamma}$$

This relationship was found to be valid for many polymer melts and polymer solutions, but it rarely gives reasonable results for suspensions. The advantage of this Cox-Merz relation is that it is technically simpler to work with frequencies than with shear rates. Most of the time polymer melts cannot be measured at shear rates lower than 50 1/s in a rotational rheometer in open sensor systems such as cone & plate or parallel plate due to the elastic effects encountered. Thus instead of measuring a flow curve in steady-state shear, one can more easily use the complex viscosity of dynamic testing [1].

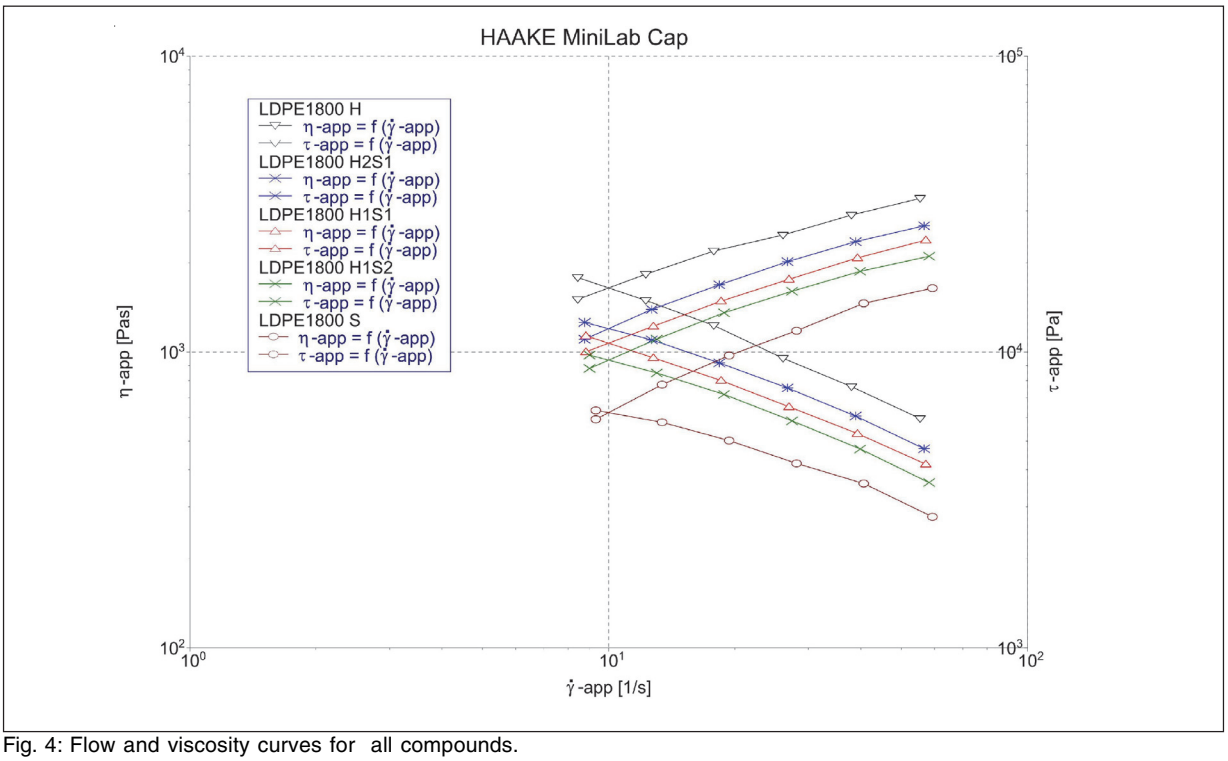

Fig. 4: Flow and viscosity curves for all compounds.

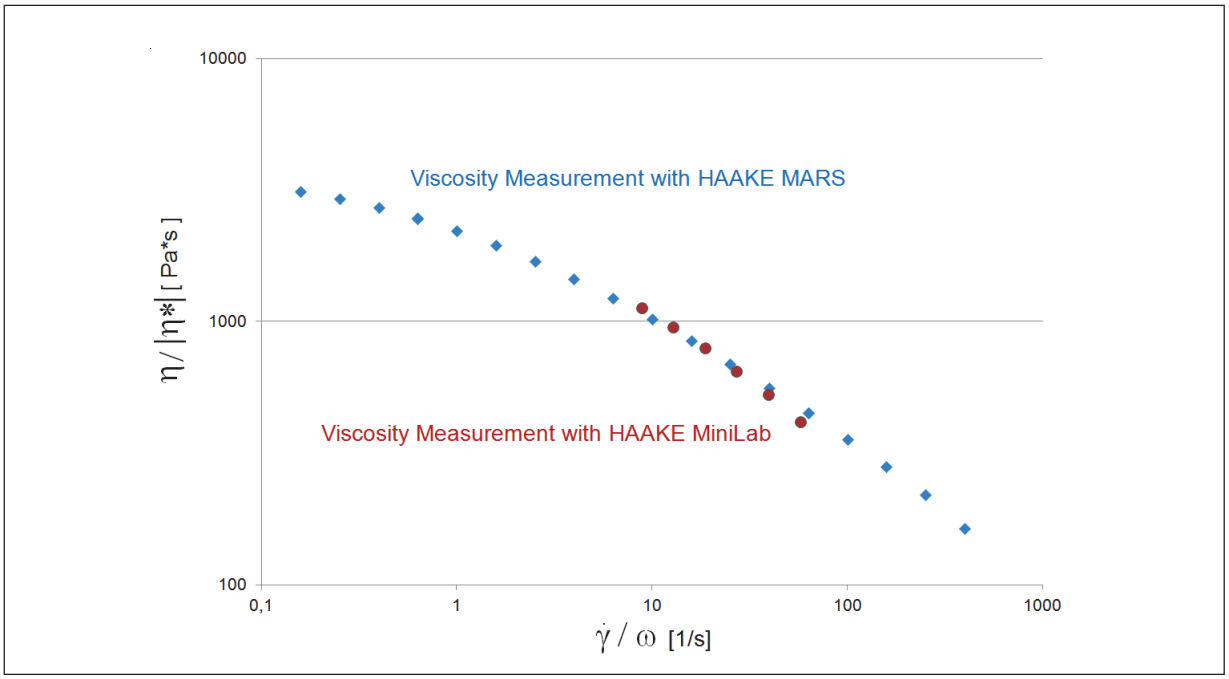

Fig. 5: Comparison of viscosity data obtained from measurement with HAAKE MiniLab 3 extruder and HAAKE MARS rheometer.

Conclusion

Fig. 5 shows nicely how the relative viscosity measurement performed on a HAAKE MiniLab micro-compounder correlates with the absolute data obtained from a high end rotational rheometer.

The experiment proves that measuring the change in flow behavior using pressure sensors in the slit capillary channel eliminates possible influences from screw forces and thus delivers reliable rheological information.

Measuring the rheological data directly during compounding has a two-fold advantage to the researcher. First of all it saves time when the measuring takes place directly in the extruder, and sample preparation can be neglected. But also structural changes within the sample that occur during compounding can be observed directly, and valuable process information can be delivered on the spot.

The HAAKE MiniLab 3 micro-compounder collects the rheological data under process conditions. When a broader measuring range is required, the HAAKE MARS rheometer is a perfect complementary extension of the experimental setup.

Fig. 6: The HAAKE MiniLab 3 micro-compounder.

Literature

[1] Schramm, Gebhard / A Practical Approach to Rheology and Rheometry, 2nd Edition, Karlsruhe 2004, p. 124.

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