Characterization of PVC compounds with torque rheometers

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Introduction

Torque rheometers with laboratory mixers and laboratory extruders are down-scaled production machines, which enable the simulation of production processes in a lab environment. This measuring method allows testing and comparing materials close to production conditions. The Thermo Scientific™ HAAKE™ PolyLab™ OS System is a modular torque rheometer. (Figure 1). The communication between the measuring sensors such as the torque sensor, mass temperature sensors and mass pressure sensors, the drive unit, and the computer takes place via a CANopen Bus.

Laboratory mixers

Laboratory mixers consist of a liquid or electrically heated measuring chamber, mixer rotors, and a feeding device. For a mixer test, the chamber is heated to the chosen test temperature. The rotors are set to a defined constant speed (Figure 2). An exact quantity of the test material is pressed into the empty mixer chamber by a feeding piston. The required torque and the sample temperature are measured and recorded over the mixing time.

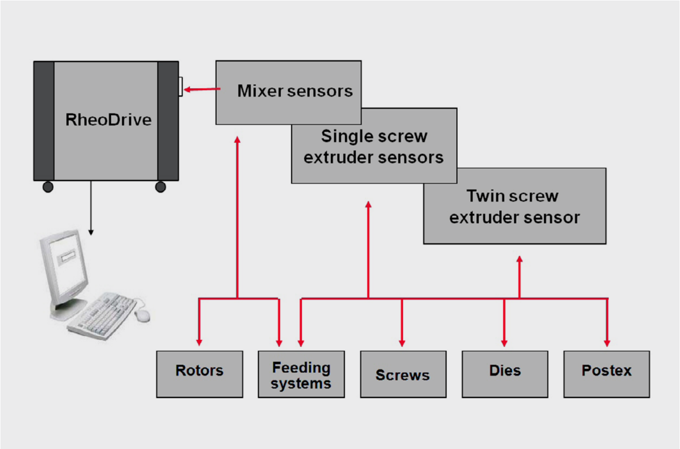

Figure 1. HAAKE PolyLab System includes the HAAKE RheoDrive, mixer and extruder sensors.

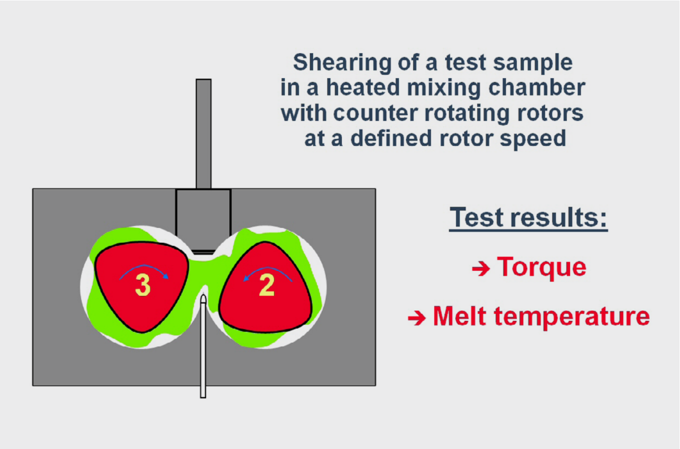

Figure 2. Laboratory mixer—measuring principle.

The measured values are plotted over the mixing time in a mixer rheogram. The mixer rheogram shows information about the melting, viscosity, cross-linking, and degradation behavior of the sample (Figure 3). The graph shows the melting behavior of a PVC dry blend: At the beginning of the test, the loading of the PVC powder into the mixer causes an instantaneous torque increase (loading peak). The powder then distributes in the mixer chamber and some components of the compound (e.g., waxes) melt due to the high mixer temperature. Both effects lead to a drop in torque.

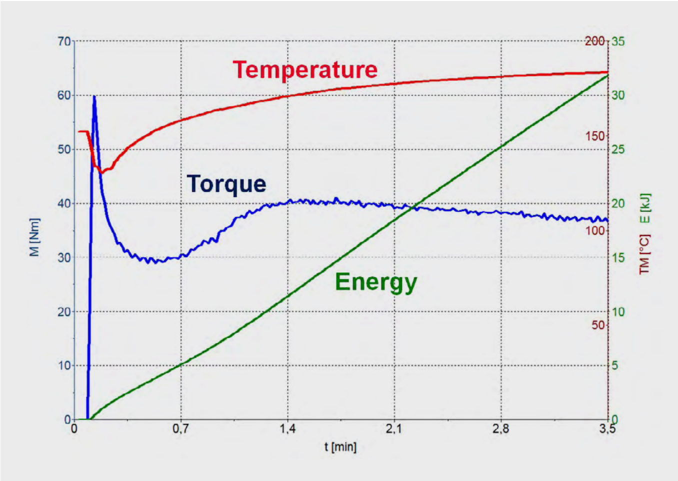

Figure 3. Laboratory mixer rheogram.

Due to the increase in mass temperature and the introduced shear energy, the PVC starts to combine into bigger agglomerates. This causes an increase in viscosity, which leads to an increase in torque. This process results in a second torque maximum. The PVC dry blend forms a homogeneous melt. After this second torque maximum, the torque drops again until it reaches a constant value. A balance between the increase in temperature caused by dissipation and the decrease in temperature caused by heat conduction through the chamber wall is reached. The torque, which is adjusting here, is a relative value for the melt viscosity of the sample.

By comparing different mixer rheograms, the effects of production and quality variations, changes in recipe, and the effect of additives on a product can be tested. The following example shows the effect of different stabilizer proportions on the rheological behavior of a PVC dry blend (Figure 4).

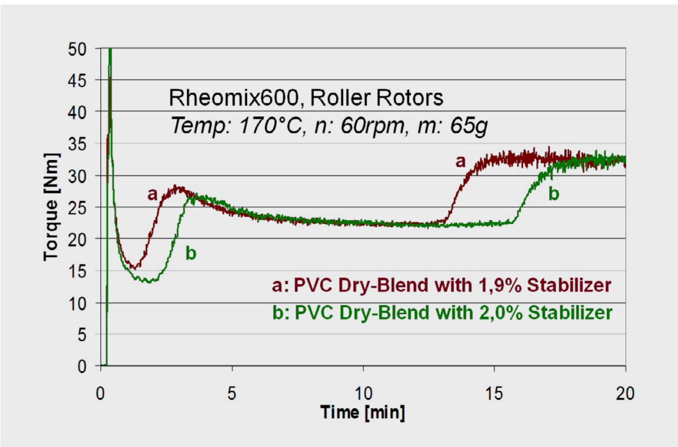

Figure 4. Laboratory mixer rheogram showing the effect of different stabilizer proportions.

The comparison of the two curves shows that the compound with the higher stabilizer content takes longer to melt than the compound with the lower stabilizer proportion. The reason for this is that the stabilizer also has the characteristics of a lubricant. Therefore, less shear energy can be introduced into the higher stabilizer content sample. After both samples completely melt, the two curves run congruently for a longer period of time. This means that the difference in stabilizer has no effect on the melt viscosity. Toward the end of the test, the sample with the lower stabilizer proportion shows an earlier increase in torque. This increase results from the cross-linkage reaction of the PVC sample, which is caused by the degradation of the PVC. The sample with the higher stabilizer proportion shows this torque increase later, therefore it is more stable.

Laboratory extruder

Testing PVC samples can also be achieved with laboratory extruders.

A laboratory extruder can be equipped with pressure sensors along the extruder barrel. By comparing pressure profiles along the extruder, it can be determined whether a sample melts earlier (high-pressure profile) or later (low-pressure profile) (Figure 5).

An advantage of a measuring extruder is that it can be equipped with profile dies and additional units such as tube and tape take-offs. Thus, production can be simulated in small scale. The produced extrudate can be used for additional tests such as mechanical tests, optical investigations, or weathering tests.

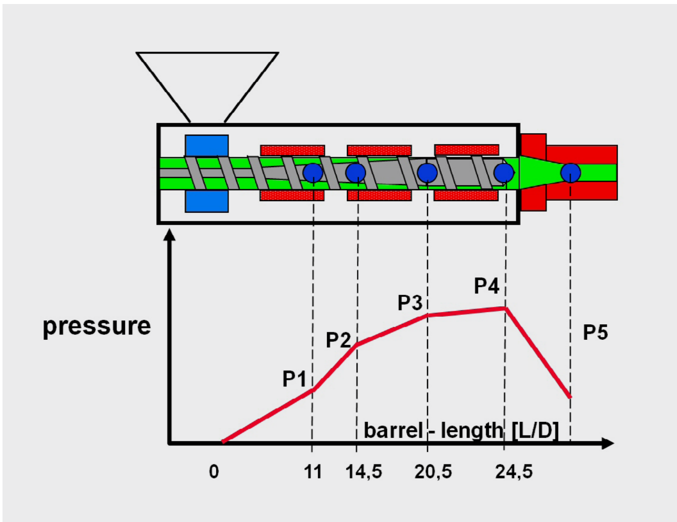

Figure 5. Pressure built-up in a single screw extruder.

Extruder capillary rheology

When the extruder is equipped with rheological measuring dies such as slit or rod-capillary dies and a balance, absolute viscosity data of the sample can be determined. These measuring dies have an exact geometrical defined flow channel (rod or slit geometry), in which the pressure drop is measured (Figure 6). From this pressure drop and the flow channel geometry, the shear stress can be calculated (Figure 7).

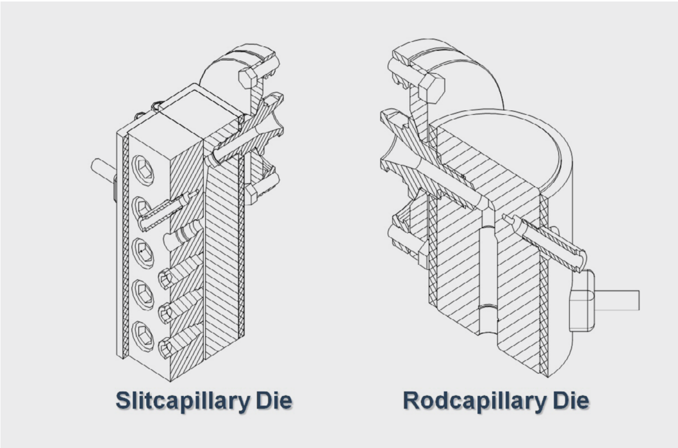

Figure 6. Capillary dies.

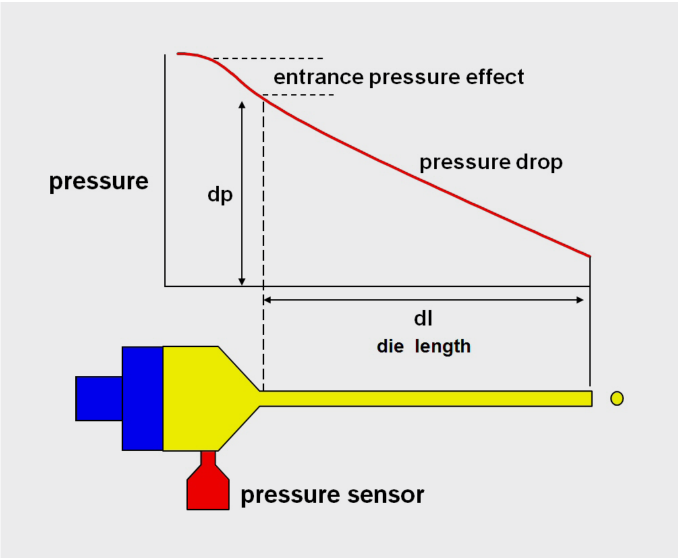

Figure 7. Rod capillary principle.

By means of a balance which is connected to the computer, the output is measured. From the output, the shear rate is calculated. From the shear rate and the shear stress, the viscosity of the melt is then calculated (Figure 8).

Calculations for Newtonian liquids:

Pressure Gradient: $p' = \frac{dp}{dl}$

Shear Rate: $\dot{\gamma} = \frac{4 \cdot Q}{\pi \cdot r^3}$

Volume flow: $Q = \frac{V}{t}$

Viscosity: $\eta = \frac{\tau}{\dot{\gamma}}$

Shear stress: $\tau = \frac{r}{2} \cdot p'$

Figure 8. Rod capillary die calculations.

By increasing the extruder speed stepwise, different shear rates are set. The resulting viscosity curve shows the flow behavior of the sample under different flow conditions (Figure 9).

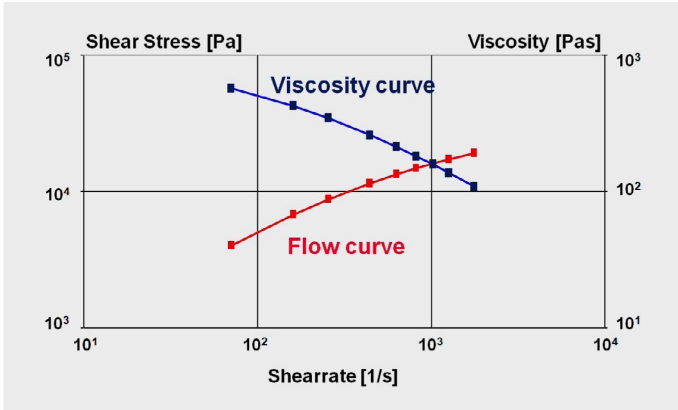

Figure 9. Capillary test measurement results.

By means of the PolySoft Evaluation Software, the course of the curve can be described mathematically. The factors for rheological models, like Ostwald or Carreau, can be calculated by regression analyses (Figure 10). The knowledge of such data is important, e.g., for the modeling of flow channels and molds. Simulation software packages like Moldflow® or Cadmould use, i.e., regression data of the Carreau model for their calculations.

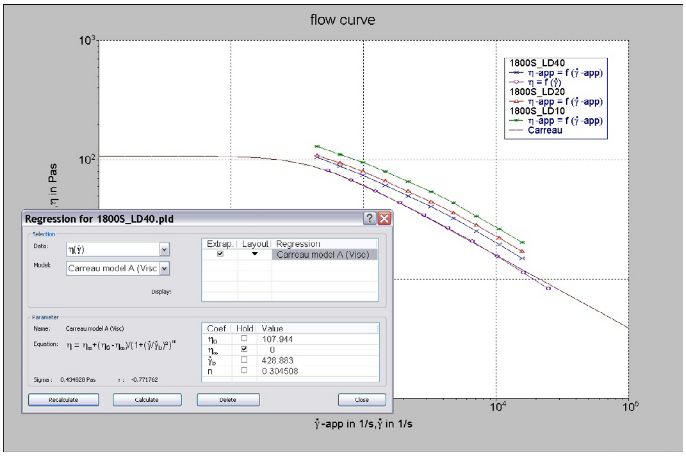

Figure 10. PolySoft Software capillary rheometry regression analysis.

Summary

The HAAKE PolyLab OS Torque Rheometer System is a sensitive and accurate tool for characterizing the processing behavior of PVC dry blends. In combination with laboratory extruders and rheological measuring dies, the flow behavior of PVC melts under production conditions can be determined.

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