Application Note
LR-35

Characterization of PVC compounds

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Characterizazion of PVC compounds with torque rheometers

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 PolyLab-System is a modular torque-rheometer of the latest generation (Fig. 1). The communication between the measuring sensors like torque sensor, mass temperature sensors and mass pressure sensors, the drive unit and the computer takes place via a CANopen bus.

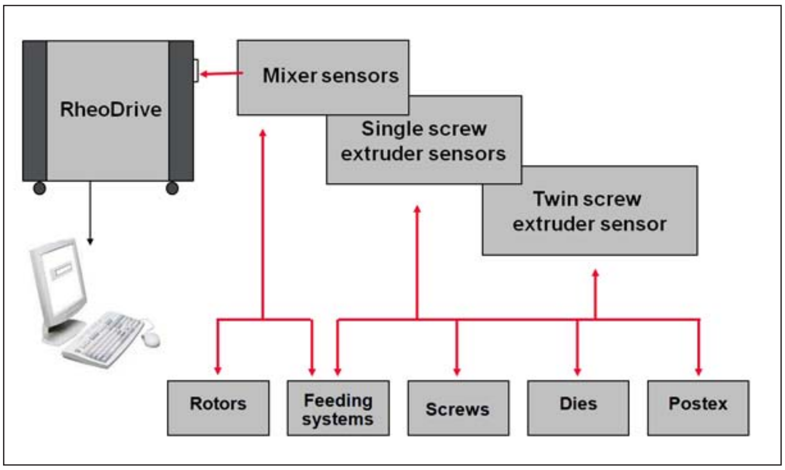

Fig. 1: RheoDrive+Mixer/Extruder-Sensor = PolyLab System

1.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 (Fig. 2).
An exact quantity of the test material is pressed into the empty mixer chamber by means of the piston of a feeding device. As measured values the torque, required for driving the rotors and the sample temperature are recorded.

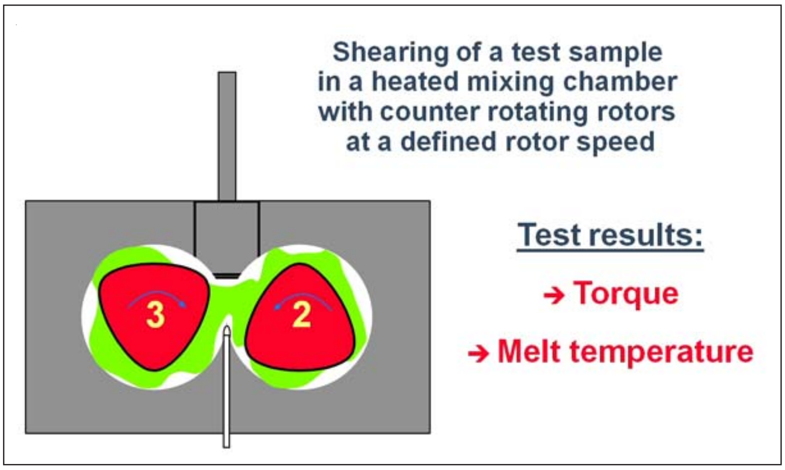

Fig. 2: Laboratory Mixer - Measuring Principle

The measured values are plotted over the mixing time in a so called mixer rheogram.
This mixer rheogram gives information about the melting, the viscosity, the cross-linking and the degradation behavior of the sample (Fig. 3).
The following 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 increase of the torque (loading peak).
After that, the powder distributes in the mixer chamber and some parts of the compound (e.g. waxes) melt due to the high mixer temperature. Both effects lead to a drop of the torque.

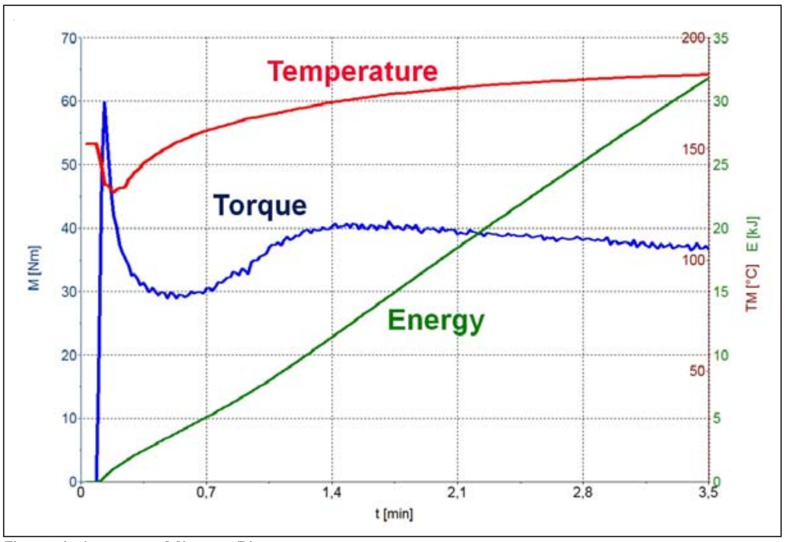

Fig. 3: Laboratory Mixer - Rheogram

Due to the increase of mass temperature and the introduced shear energy, the PVC starts to combine to bigger agglomerates. This causes an increase of viscosity, which leads to an increase of torque. This process results in a second torque maximum. The PVC Dry Blend forms a homogeneous melt.

Due to additional increase of the sample temperature the torque drops again until it comes to a constant torque after a period of time. A balance between the increase of temperature caused by dissipation and decrease of 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 of 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 (Fig. 4).
The comparison of the two curves shows that the compound with the higher stabilizer content melts later than the compound with the lower stabilizer proportion. The reason for this is that the stabilizer has also the characteristics of a lubricant. This is why less shear energy can be recorded at the sample with the higher stabilizer proportion.
After melting completely, the two curves run congruently for a longer period of time. This means that the difference in stabilizer has no effects on the melt viscosity.
Reaching the end of the measurement, the measuring curve of the sample with the lower stabilizer proportion shows an earlier increase of the 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 increase of torque later, therefore it is more stable.

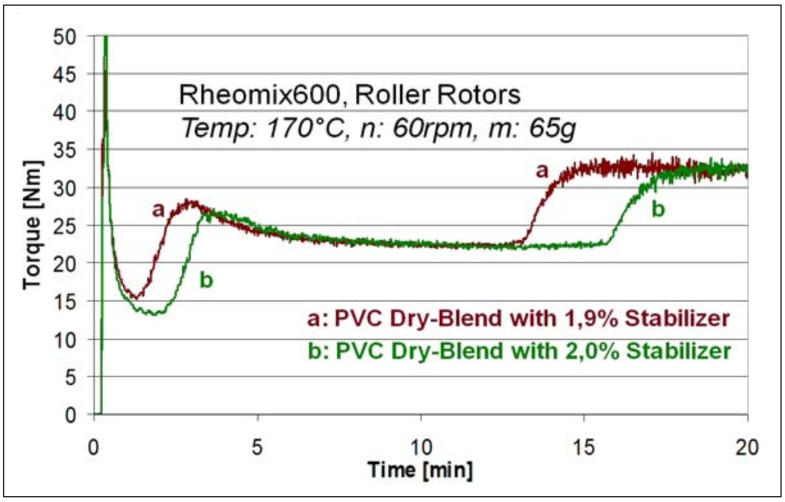

Fig. 4: Laboratory Mixer - Rheogram

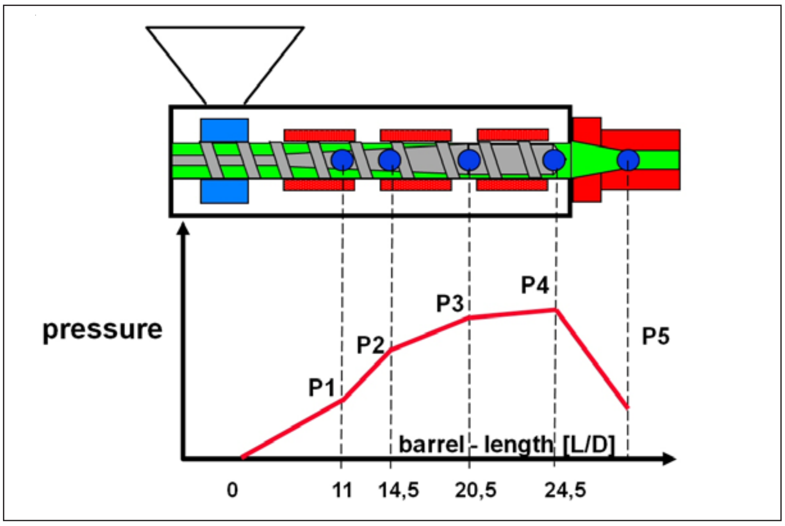

Fig. 5: Pressure built-up in a single screw extruder

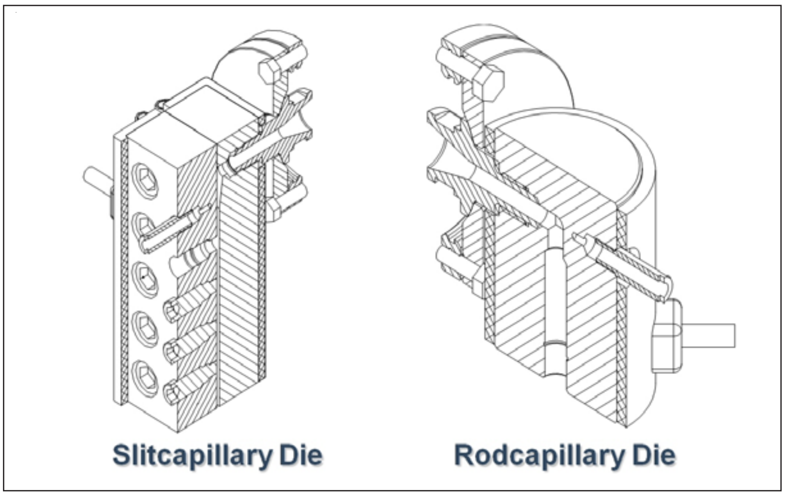

Fig. 6: Capillary Dies

Laboratory extruder

Another possibility of testing PVC samples are tests 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) (Fig. 5).
An advantage of a measuring extruder is that it can be equipped with profile dies and further units like tube and tape take-off. Thus a production can be simulated in small scale.
The produced extrudate can be used for further tests like mechanical tests, optical investigations, or weathering tests.

2. Extruder Capillary Rheology

When the extruder is equipped with rheological measuring dies like slit- or rod-capillary dies and a balance, absolute viscosity data of the sample can be determined.
These measuring dies have a geometrically exactly defined flow channel (rod or slit geometry), in which the pressure-drop is measured (Fig. 6).
From this pressure-drop and the flow channel geometry, the shear stress can be calculated (Fig. 7).
By means of a balance which is connected to the computer, the output is measured.
From the output, the material density and the capillary geometry the shear rate is calculated.

From the shear rate and the shear stress the viscosity of the melt is then calculated (Fig. 8).
By stepwise increasing the extruder speed, different shear rates are set. The resulting viscosity curve shows the flow behavior of the sample under different flow conditions (Fig. 9).

By means of the 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 (Fig. 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.

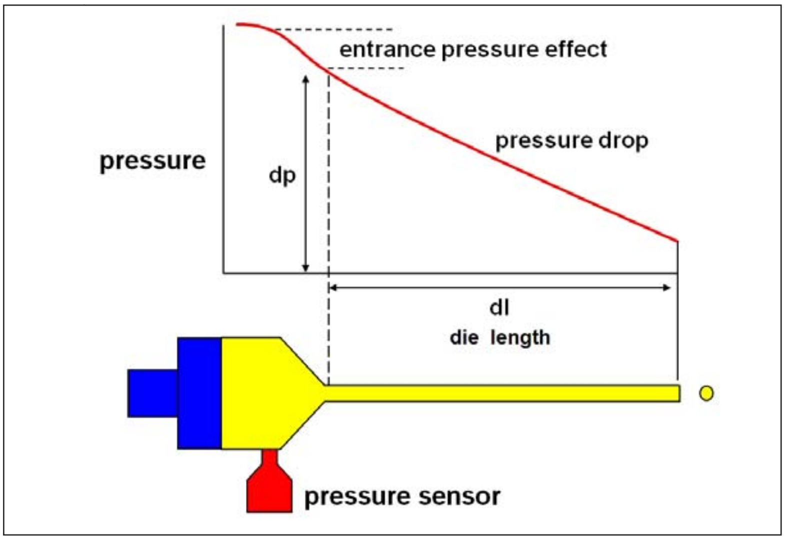

Fig. 7: Rod Capillary Principle

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: $\tau = \frac{r}{2} \bullet p'$

Fig. 8: Rod Capillary Die

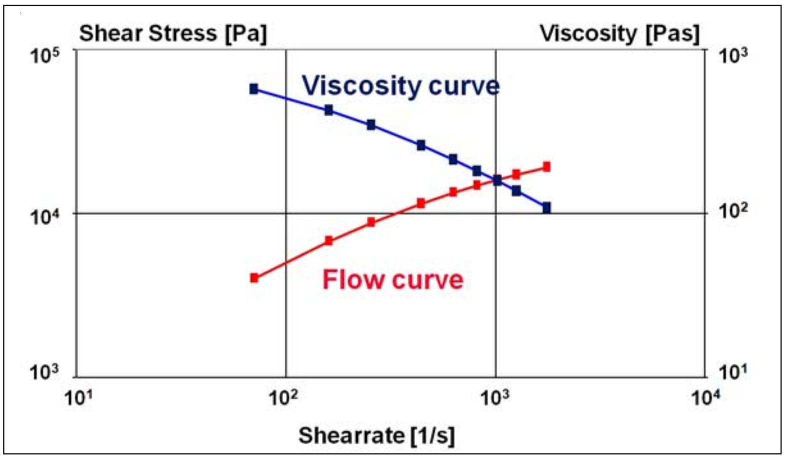

Fig. 9: Capillary Test - Measurement Results

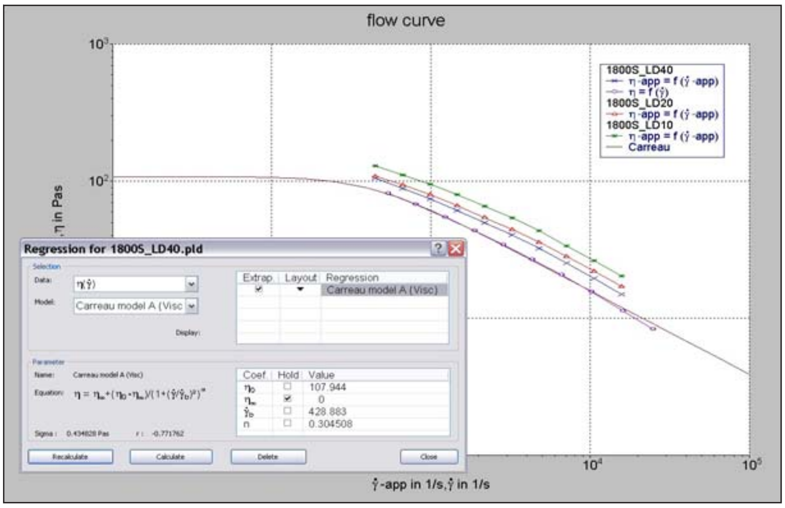

Fig. 10: PolySoft Capillary Rheometry - Regression Analysis

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