Application Note
LR-38

Production of a blend of two different concentrations by a parallel double screw extruder and a further processing through a melt pump and following measurement of the rheological characteristics in slot and rod capillary dies

Rheology Application Notes

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Introduction

Plastic melts and melts of blends are no Newtonian, but viscoelastic liquids, which means their flow behavior is to a high degree dependent on the shear rate that affects them. Generally such melts show intrinsically viscous behavior, where the viscosity decreases at increasing shear rate and the elastic characteristics become more evident.
The aim of this test was the production of different blends in a parallel double screw extruder and their rheological characterization in a capillary die. In the process the shear rate was regulated by conducting the material through a melt pump whose speed was variable, and the shear stress was measured. In addition this technology should be used to compare the blends with the original components of the blends.

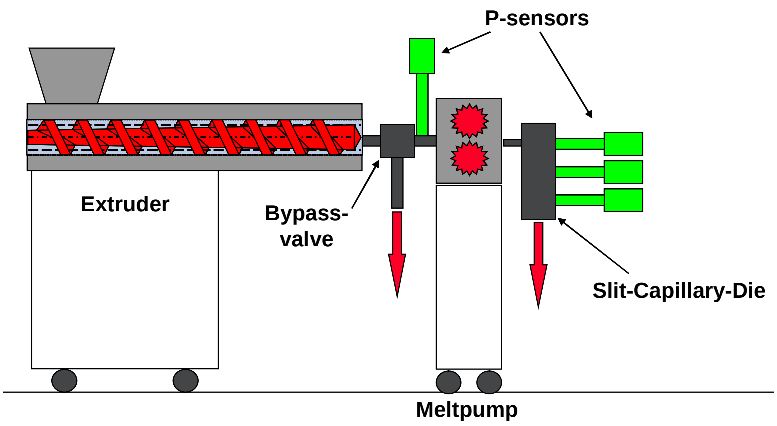
Experimental set-up

For producing and testing the blends and their original components the following experimental set-up was chosen:

The extruder was driven by a *PolyLab OS* of Thermo Electron. A constant speed (100/min) was set and the torque was measured online.
For the extrusion and the production of the two original components and/or the two blends the parallel double screw extruder *Rheomex PTW* was used. The extruder has got a screw diameter of 24 mm. With the help of different screw configurations which can be built up from different screw segments, the extruder can be adapted to the different requirements of extruding.
A standard screw configuration which is especially suitable for producing blends was selected. A temperature profile from 200°C to 260°C towards the extruder output was set.

The blend compounding and/or the filling in of the two original components was controlled by two single screw dosing devices, the so-called *Meetering Feeders*. Here the mass flow was preset by the speed setting of the *Meetering Feeders* after a gravimetric calibration.

In the extruder the homogeneous blend was produced and transported continually by the *melt pump*. Because the volume flow transport out of the extruder did not vary due to the constant extruder speed, the pressure required for the capillary measurement was built up by varying the speed of the melt pump (from 5/min to 60/min).
To let off the exceed material, the melt pump has got a bypass, which was controlled manually in this application.



The measurement of the flow variables took place in *slot* and in *rod capillaries* to guarantee the covering of a broad shear rate range.

The evaluation of the test as well as the control and the coordination of each application unit was done by a special software, *PolySoft*.

Bases of the evaluation

The measurement of the pressure as basis of the following calculation of the shear stress and the viscosity takes place after the extrusion in the PTW 24p and the further transport through the melt pump to slot and rod capillaries, as shown in Fig. 2.

For this a stationary speed ramp is run via the melt pump speed. This means that the melt pump speed is changed in fixed steps from a minimum to a maximum value by the software.

By inquiring the slope of the measuring variable Pressure p over the time the software tests the stationary measuring condition of the respective melt pump speed. If a stationary measuring condition is reached, the volume flow Q is determined from the geometry of the melt pump and the speed. From the pressure gradient p' in the measuring die, which was recorded at the same time, the apparent shear stress τ_{app}, the adjacent apparent shear rate $\dot{\gamma}_{app}$ and the apparent viscosity η_{app} are calculated.

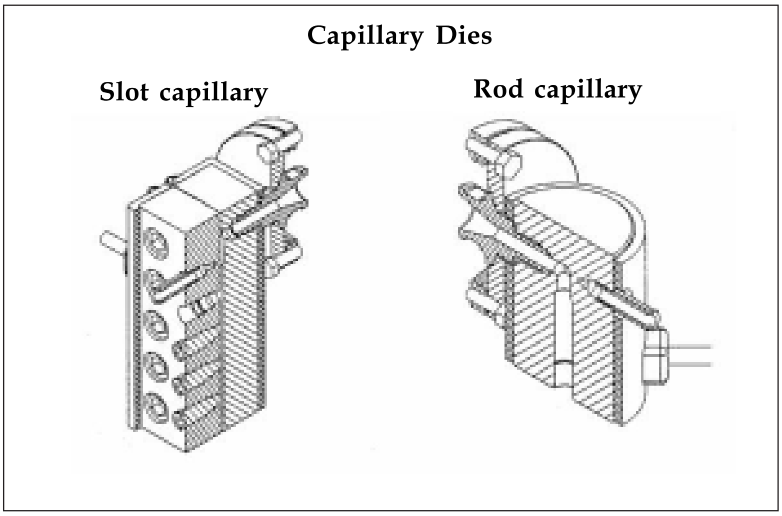


Fig. 2: Slot and Rod Capillary Die

After that the software independently triggers the next speed step.

Extrusion processes generally have low shear rates (between 10 and 1000 1/s). For those shear rate ranges slot capillary dies are usually used. For Newtonian liquids in a slot capillary of the width W and the height H and a measuring length dl the flow variables τ and $\dot{\gamma}$ are calculated according to the formulas:

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

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

Shear stress: $\tau_{true} = \frac{H}{2} \cdot p'$

Apparent shear rate:

$$\dot{\gamma}_{app} = \frac{6 \cdot Q}{W \cdot H^2}$$

Apparent viscosity:

$$\eta_{app} = \frac{\tau}{\dot{\gamma}_{app}}$$

To take the intrinsically viscous flow behavior into account, the so-called Weißenberg-Rabinowitsch correction is run by the software. By this Weißenberg-Rabinowitsch correction, the apparent shear rate and the apparent shear stress are converted into the true shear rate $\dot{\gamma}_{true}$ and the true shear stress τ_{true} by logarithmic differentiation (according to Fig. 3).
A polynomial is lain through the points determined according to Newton.
By a slope correction method according to

$$\dot{\gamma}_{true} = \frac{1}{3}\dot{\gamma}_{app}\left(2 + \frac{\partial \lg \dot{\gamma}_{app}}{\partial \lg \tau_{true}}\right)$$

the true values for $\dot{\gamma}_{true}$ are then determined.

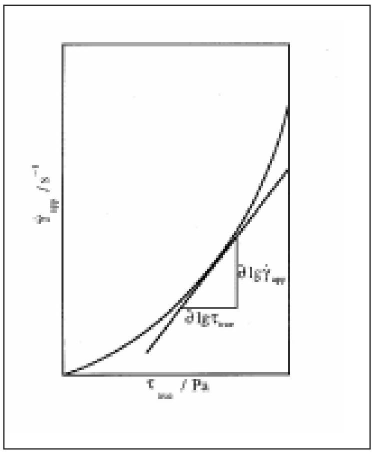


Fig. 3: Weißenberg-Rabinowittsch Correction

High shear rates, (>1000 1/s), as they occur in injection moulding, are usually measured with the help of rod capillary dies. The theoretical correlation for the accompanying flow variables for a die diameter r and a die length dl can be seen in the following formulas:

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

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

Apparent shear stress:

$$\tau_{app} = \frac{r}{2} \cdot p'$$

Apparent shear rate:

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

Apparent viscosity:

$$\eta_{app} = \frac{\tau}{\dot{\gamma}}$$

Due to the small diameters, the pressure sensor cannot directly be built into the capillary of rod capillary dies. Thus the installation is done before the die mouth. Because the pressure measurement is done before a diameter contraction in this case, measuring errors occur due to inflow pressure losses which you have to take into consideration. For this reason the so-called Bagley correction is applied.
To do this, three measurements with capillaries of the same diameter but of different lengths are necessary to run.
Through the measured points, a compensating function is lain.
After the calculation of the compensating function, any value pair of pressure and shear rate for any $\dot{\gamma}$ can be determined of each measurement.

The L/D ratio of the measuring capillary is lain off as abscissa and the pressure before a capillary measured at a special shear rate is lain off as ordinate. An extrapolation of the L/D ratio = 0 is the inflow pressure loss that occurs at the measured shear rate. The pressure values required for the calculation of the shear stress then have to be reduced by the calculated inflow pressure loss (before the calculation of the shear stress). The result is a Bagley – corrected shear stress τ_B.
You have to consider that the inflow pressure loss changes when the shear rate changes, and that it is dependent on the geometry of the die inflow.
After that, a Rabinowitsch-Weißenberg correction is done for the slot capillary dies. Because of the geometry of the rod capillary die the formal correlation for the slope correction method is:

$$\dot{\gamma}_{true} = \frac{1}{4}\dot{\gamma}_{app}\left(3 + \frac{\partial \lg \dot{\gamma}_{app}}{\partial \lg \tau_B}\right)$$

After the Rabinowitsch-Weißenberg correction the true viscosity function is given by

$$\eta_{true} = \frac{\tau_{true}}{\dot{\gamma}_{true}}$$

The capillary rheology software of Thermo Electron runs this correction automatically. In addition to the automatic correction of the shear rate, all processes are also shown graphically.

Identification of the used materials

The following materials were used for producing the blend:

Luran 358 N:
Styrol-Acrylnitril Copolymer (SAN) of the company BASF

Lexan 161:
Polycarbonat (PC) of the company GE

Blend 1:
composed of 25% Luran 358 N and 75% Lexan 161

Blend 2:
composed of Z 40% Luran 358 N and 60% Lexan 161

Luran is processed in products of the plastic industry in different applications. For example, transparent parts of food processors, lighters and battery boxes are made of Luran. Many different products are also made of Lexan. The product mix reaches from helmets and visors for motorcyclists to basic material for compact discs. The company BASF offers the blend of these two materials under the name Terblend-S. The features of this blend are a higher thermal form stability and a significantly higher viscosity compared to Luran. For example, motor vehicle rear light housings are made of Terblend-S.

Presentation and discussion of the results

First the flow variables of Luran and Lexan were determined after the extrusion in slot and rod capillaries. After that the two blends with the defined compositions were produced in the extruder. Their flow variables were also determined. The viscosities of the two blends and their original components which were corrected according to Bagley and/or Rabinowitsch-Weißenberg are lain off over the shear rate in Fig. 4.
Figure 7 shows the significant difference in viscous behavior between the purely Lexan and Luran samples. The viscosity of Lexan is much higher than the viscosity of Luran. As expected, the viscosities of the two blends are between the viscosities of the pure original components.

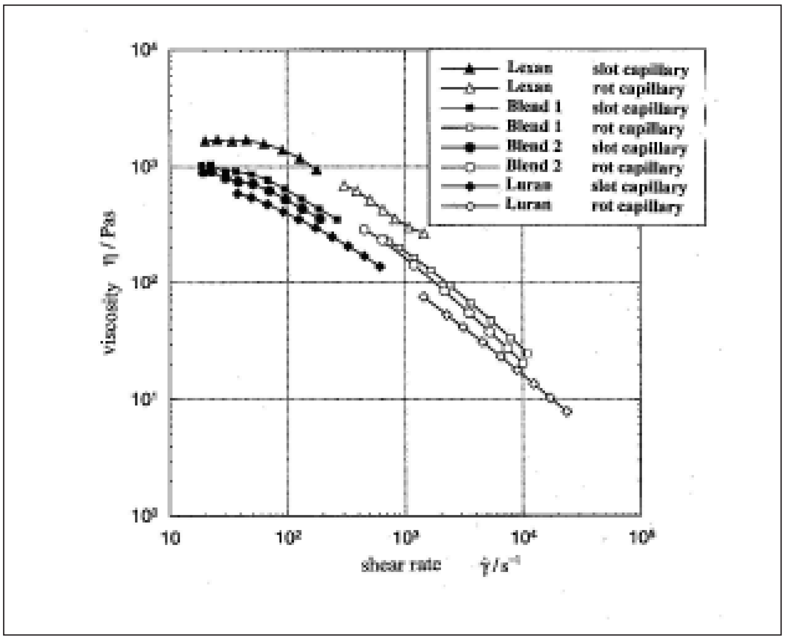


Fig. 4: Viscosity functions of Loran and Lexan and their two blends at 260 °C

The viscosity function of blend 2 is only slightly higher than the function of blend 1. This fact can originate in the non-linear ratio of components which means that in case of additional adding of a low-viscous material the viscosity does not decreasing according to the ratio of components. The reason for this is that the viscosity of Lexan is even reduced by adding a small amount of the low-viscous Luran.
From this test, the importance of a process-oriented measurement of the flow behavior at the production of a blend, as it was done in this work, gets evident.

Summary

In these tests, two blends were produced in a parallel double screw extruder. The blends were transferred through a melt pump whose speed was variable and then tested rheologically in slot and rod capillary dies. The flow variables of the original components of the blends were determined in the same way. Then they were compared with the variables of the blends.
It was shown that the capillary-rheological measuring method at the output of a melt pump had been developed to a practical technology. The measuring results that were corrected according to Bagley and Rabinowitsch-Weißenberg show that the viscosity functions of slot and rod capillary measurements turn into each other.

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