Application Note
LR-43

HAAKE MiniLab - Compounder and Reactor

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Application Report

Abstract

The use of a conical twin screw extruder with backflow channel combines aspects of mixing and extrusion in a batch process. Tests and results of the new HAAKE MiniLab micro compounder are discussed in the following paper. With a total filling volume of 7 ml and a built in slit capillary die the applications focus on compounding and reactions of small amounts of polymers in molten stage.

Introduction

The HAAKE MiniLab combines two great areas of application: mixing and rheological recording of melt characteristics. In this application report mixing processes as blending, compounding, and adding of additives are described in detail. As conical twin-screw extruder with a back flow channel the HAAKE MiniLab can be operated as circulation reactor and thus uses the advantages of both extruder and mixer. The filling volume is an important quantity because the total filling amount has to be known to rate a formulation. It is shown how the filling volume can be determined and set as low as 7 ml. Co-rotating as well as counter-rotating pairs of screws can be used. In addition to the usual setting of the speed a particular torque can be set as well. The speed is controlled that the pre-set torque value is reached.

Tests are reported which can be run by the basic version of the unit using the standard control panel. Standard polymers can be tested without problems. Powder and liquids fillers can be used if the filling speed and the loading procedure are optimized in a pre test. Monitoring chemical reactions is also possible. The increase of the pressure signal was chosen as the most sensitive value. A comparison via a relative viscosity is also possible.

Figure 1: HAAKE MiniLab

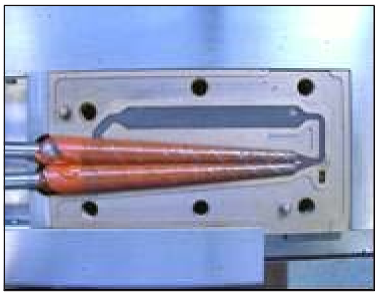
Mini-Filling volume

The following table (1) has been drawn up with the help of the average values of different tests. Co-rotating and counter-rotating screws differ only slightly in the filling volume. In addition to the total volume, the volume of two other characteristic points has been determined: The available volume after the feeding zone and the volume of the circulation loop. The back flow channel itself has a volume of 1.50 ccm. The whole amount of the material can't be extruded as rod or fiber. The strip that remains in the back flow channel can also be used as a test specimen for further investigation.

Selecting the screw types: Co- or Counter rotating?

In order to open up an even wider spectrum of applications for the customer, the MiniLab works as co-rotating or as counter-rotating twin-screw extruder. This results in different types of screws. Simply exchanging two gear wheels in the gearbox changes the rotating direction of the screws.

Figure 2: Co-rotating screws
a) PE melt



b) Asaclean (PS cleaning compound) + Remafin orange duration of the test t=90 s, test condition like in Figure 4

Figure 2 shows the co-rotating pair of screws. It also shows a faster compounding of the master batch in comparison to Figure 4. In the following table (2) further characteristics of the used screws are summarized.

Extrusion ratio

The values for the extrusion ratio (extrusion mass /total mass) are summarized in Table 3, here at the

Screw	Volume total	From feeding zone	Circulation loop
Counter-rotator	7.16 ml	5.84 ml	5.15 ml
Co-rotator	7.00 ml	5.75 ml	5.02 ml
	Volume back flow channel: 1.5 ml		

Table 1: Filling volume

example of LDPE 1800S at 190 °C and 90 rpm. In addition to a complete batch filling, the case of a smaller filling amount (70%) that has been also selected.

Direction of rotation:	co	counter
Resident time distribution	wide	narrow
Forced extrusion	-	+
Cleaning	+	-
Extruder amount	+	0
Blending of sensitive products	++	0
High shear rates, dispersing	-	++
Rheological measurements	0	++
Required duration of blending	++	+

Table 2: Characteristics of the screw types

LDPE1800S Screws	Filling rate 100%	Filling rate 70%
Counter-rotator	49.5 %	39.0 %
Co-rotator	58.5 %	58.5 %

Table 3: Extrusion ratio

Blend EVA LDPE1800S Screws	Filling rate 100%	Filling rate 70%
Counter-rotator		
10 % EVA	49.9 %	36.5 %
20 % EVA	50.0 %	44.2 %
Co-rotator		
10 % EVA	53.7 %	45.1 %

Table 4: Extrusion ratio PE-EVA Blends

Due to the smaller number of screw flights, less material sticks on the co-rotating screws. About 9% more material can be extruded. If the unit is not filled completely, the amount which can be extruded drops as expected, because the amount of sample in the circulation loop (about 21%) is constant.

In Table 4 the results or extrusion tests for polymer blends of LDPE and EVA are listed. Especially the counter-rotating screws are not sensitive to different materials. In the case of a complete filling the output is about 50%. This shows an advantage of the counter-rotating screws with sticky materials, as EVA Blends.

Comparison of the operating mode "constant speed" and "constant torque"

We want to point out the use of a "constant torque mode" for this batch process. In addition to the usual setting of the speed a particular torque can be set as well. The speed is controlled that the preset torque value is reached. Of course the most important application is the setting of the maximum value of M (alarm value). If M = max is reached, the speed is reduced so that the test is continued with the maximum torque allowed.

Another application is described in Figure 3. When extruding via the open bypass at constant speed results in an asymptotic function. The amount of extruded material is not constant over the time.

Extrusion at constant torque results in a linear rise of the extruded mass. The advantage is an extrusion of the material in a more even rod. The amount of extruded material is not constant over about 90 sec. this about 90% of the accessible output.

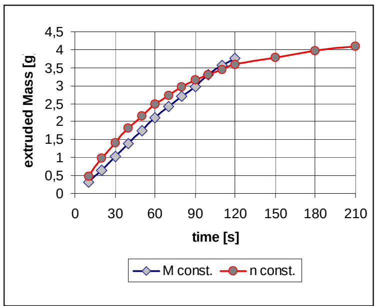


Figure 3: Comparison of extruded mass at constant speed n and at constant torque M (co-rotator).

Polymer Alloying

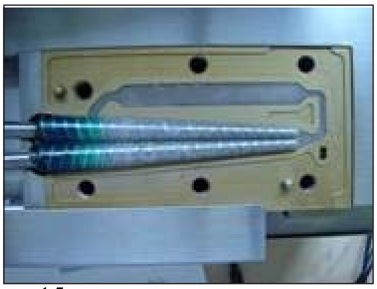
A central task of the HAAKE MiniLab is mixing different polymers (blending/alloying) or compounding of additives (com-pounding). PE/EVA blends have already been discussed at the extrusion tests. The principle of blending and compounding is shown clearly by means of a series of Pictures with colored master batch (Figure 4 from the left to the right). After the HAAKE MiniLab is completely filled, two pellets of master batch have been added. After 15 sec the back flow channel is reached. The color trace moves forward and has entered the back flow channel after four minutes (middle left). A minimal brightening has disappeared after seven minutes; the material is blended homogeneously. This is valid for the total area of the screw.

Compounding

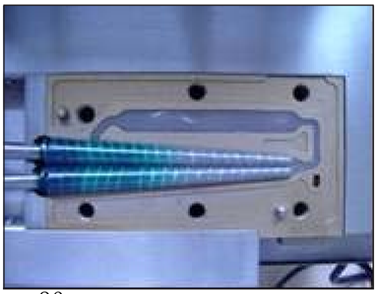
The results of different tests have been summarized. Of course for all extruder and mixer tests the test parameters have to be adjusted to the material system. Exact "recipes" cannot be given. However, here are some examples and general hints.

PE talcum	Rubber blend
PE strengthened (TIO2, chalk, talcum)	EPDM+oil+3% finely dispersed filler,
PE + 1.5% powder	TPU/Chewing Gum 95/5, 80/20
Polylactides with active agent	
Cellulose with active agent	Oil-Bariumferrit paste

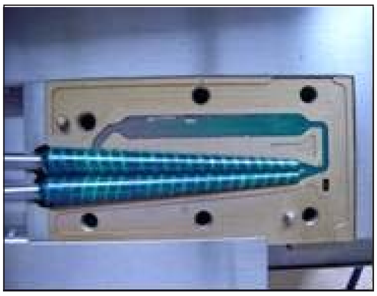
- **Polymer pellets:**
  Easy to handle, the temperature window also gives the processing temperature (e.g. from tables, data banks) for the HAAKE MiniLab. Fill in two charges, filling speed between 20 and 50 rpm.
- **Polymer pellets with fillers/ addictives:**
  At low contents of fillers you can premix. Higher contents of fillers can lead to a separation in the funnel. Powder blocks the threads of the screws before the pellets melt. Here the pellets can be filled in first, and then powder and pellets can be filled in alternately.
- **Fine dispersed powders:**
  In this case it is important to move the filling piston slowly downward. This can be adjusted at the filling device if necessary. After the end of the test the filling device has to be cleaned very thoroughly.
- **Rubber:**
  The filling speed has to be reduced due to high development of torque.
- **Low viscous substances:**
  Generally it is recommended to swivel out the pneumatic feeding device and feed fluid direct onto the rotating screws (n = 20 - 50 rpm). To make a paste supplementary substances are added gradually at low speed at n < 10 rpm into the feeding device. The same procedure can be used for melting substances.
- **Adhesives, low viscous polymeric melts:**
  Clean the feeding device very thoroughly at the end of the test. Clean the piston with suitable solvent, if possible.



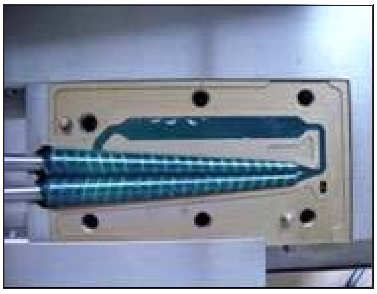
t = 15 sec



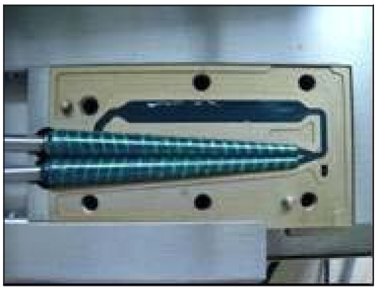
t = 90 sec



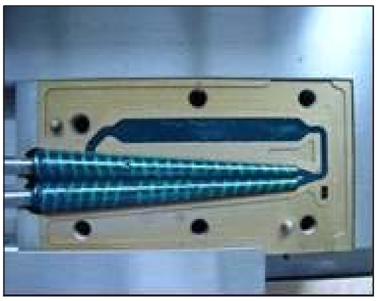
t = 4 min



t = 5 min



t = 7 min



t = 10 min

Figure 4: Asaclean 6.96 g Remafin green MB, T = 220 °C, 50 rpm

The HAAKE MiniLab as reactor

Monitoring a reaction over time with the HAAKE MiniLab is explained in a concrete example here. Polymers of organic molecules such as lactic acid can be processed as strong and adaptable implants today.
The required long reaction times can only be reached with difficulties in conventional extruders. By the possibility of running the reaction mixture (monomer and catalyst) in a circulation loop, the required reaction times can be set. At the end of the test the polymer is extruded as rod. This rod can be used in sample specimens for decomposition tests in different media.
Fig. 5 shows the graph of the HAAKE PolyLab monitor software, which has been used for running the test. Due to the low viscosity of the educt no pressure signal can be detected.
The reaction starts at 7.5 min with a pressure signal at pressure transducer 1 (p-D1). An increasing torque and pressure signal at the second pressure transducer (p-D2) can be noticed (the back flow channel is now filled completely with polymer).
After 20 minutes the constant pressures p-D1 and p-D2 show the reaction is finished. The pressure drop between the sensors p-D1 and p-D2 of 15 bar correlates to with increased viscosity of the melt in the back flow channel.
Figure 5 shows the sensitivity of the instrument. Although torque signal is only about 3% of the whole measuring range (maximum: 5.5 Nm) and is less suitable for evaluation, the reaction can be monitored with the more sensitive pressure signals.
In contrast to conventional glass reactors, the temperature (TM) of the highly viscous melt can also be controlled exactly, because of the large relation of the metal extruder block compared to the small amount of sample. It acts like a heat sink and ensures isothermal test condition.

The HAAKE MiniLab as relative rheometer

The backflow channel of the HAAKE MiniLab is designed as a slit capillary. With the two pressure transducers in the backflow channel the pressure drop in the defined flow channel can be measured. From this pressure drop a shear stress is calculated. To determine the shear rate, a mass flow in the flow channel is correlated with the screw speed of the HAAKE MiniLab. To get a flow curve, different screw speeds are set and the pressure drop at the screw speeds is then measured.
Limitations:
It is not yet possible to get an absolute correlation between the screw speed and the mass flow. So the calculated shear rate and therefore also the calculated viscosity can only be a relative value.
In the following example a PP was tested for the influence of the residence time on the flow characteristic (Figure 6).
The PP sample was filled into the HAAKE MiniLab and immediately after the loading process was finished, a capillary test at ten different screw speeds was made.
After this residence time a second capillary test (similar to the first one) was done.
Figure 6 shows that the residence time of 30 minutes has caused a significant drop of the melt viscosity over the whole shear range. We assume this drop is caused by degradation of the polymer.

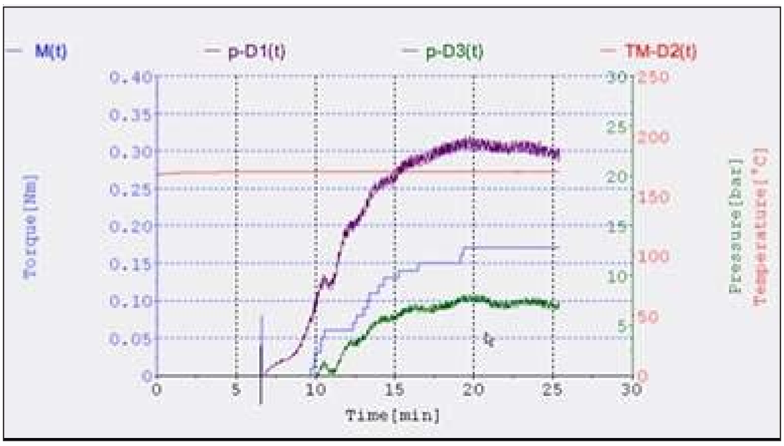


Figure 5: Monitoring the reaction time online, polymerization of L-lactid to biodegradable polymers

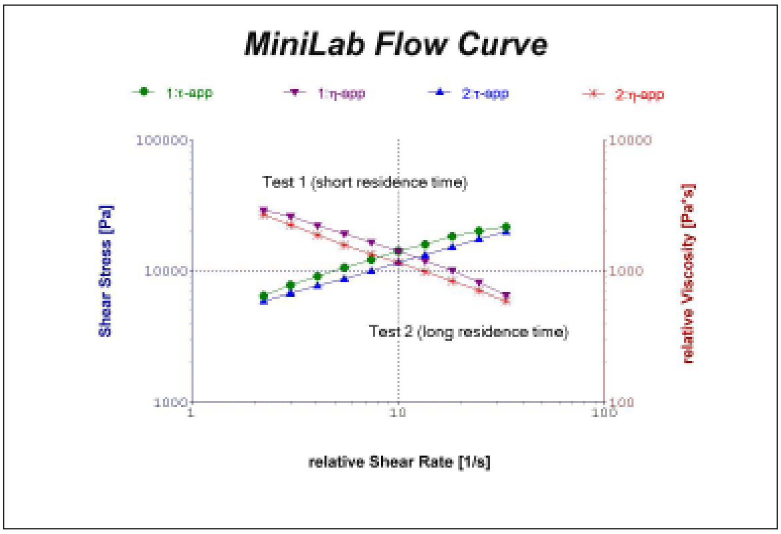


Figure 6: Influence of the residence time on the flow behavior of a PP sample.

Conclusion

In this application note tests are reported which can be run by the basic version of the unit using the standard control panel. Standard polymers can be tested without problems. Powdery fillers and liquids can be used if the filling speed and the loading procedure are optimized in a pre test.
Monitoring chemical reactions is also possible. The increase of the pressure signal was found as the most sensitive value. A comparison via a relative viscosity is also possible. We hope to have given helpful hints, tips and ideas for further work with the HAAKE MiniLab.

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