MiniLab - Compounder and Reactor

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Product-Info

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 MINLAB mircocompounder 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.

Figure 1: MiniLab.

INTRODUCTION

The 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 double-screw extruder with a back flow channel 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.

MINI-FILLING VOLUME

The following table 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 1.5 cm^3. The whole amount of the material cannot 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 double-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 shows the co-rotating pair of screws. It also shows a faster compounding of the

Table 1 (above): Filling volume.

Table 2 (below): Characteristics of the screw types.

Screw	Volume total	From feeding zone	Circulation loop
Counter-rotator	7.16 ml	5.84 ml	5.15 ml
Co-rotator	7 ml	5.75 ml	5.02 ml

Volume back flow channel: 1.5 ml

	Co-rotation	Counter-rotation
Resident time distribution	wide	narrow
Forced extrusion	-	+
Cleaning	+	-
Extruded amount	+	o
Blending of sensitive products	++	o
High shear rates, dispersing	-	++
Rheological measurements	o	++
Required duration of blending	++	+

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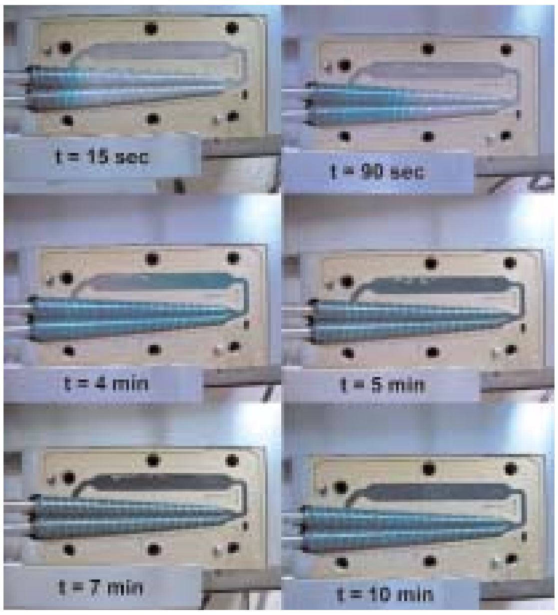

Figure 4: Asaclean 6.96 g Remafin green MB (T = 220°C, 50 rpm).

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, some examples of tested applications are given in Table 5 and general hints are listed hereafter.

- Polymer pellets: Easy to handle, the temperature window also gives the processing temperature (e. g. from tables, data banks) for the MiniLab. Fill in two charges, filling speed between 20 and 50 rpm.
- Polymer pellets with fillers/ additives: 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.

PE Talcum	Rubber Blend
PE strengthend (TiO_2, 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

Table 5: Typical compounding applications as tested on the MiniLab.

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.

MINILAB AS REACTOR

Monitoring a reaction over time with the 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.

Figure 5 shows the graph of the 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.

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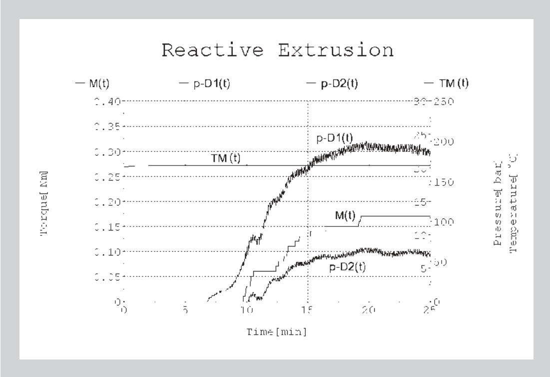
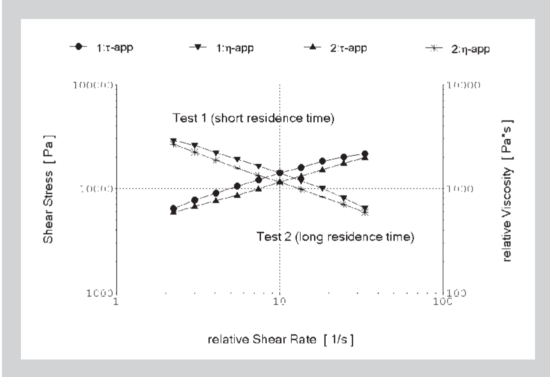
Figure 5 (left): Monitoring the reaction time online, polymerization of L-lactid to biodegradable polymers.

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





MINILAB AS RELATIVE RHEOMETER

The MiniLab is not aimed to be considered as a rheometer, but some rheological information, in real time, is a valuable additional information during the ongoing processes. Therefor, the backflow channel of the 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 MiniLab. The back flow on the screws makes it difficult to get an absolute correlation between the screw speed and the mass flow for different materials, so the calculated shear rate and therefore also the calculated viscosity can only be a relative value. However for a defined application, the relative values are quite relevant, and using different screw speeds allows to get a flow curve.

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 MiniLab and immediately after the loading process was finished, a capillary test at ten different screw speeds was made.Then the sample run in a loop for 30 minutes at a constant speed of 100 rpm. After this residence time a second capillary test (similar to the first one) was done. It showed a significant drop of the melt viscosity over the whole shear rate range caused by the degradation of the polymer.

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 MiniLab.

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