Injection molding machine with height-adjustable slit die for rheological measurements of polymer melts under processing conditions

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ABSTRACT

Rheological properties of two grades of polypropylene and two grades of polystyrene melts were studied using a slit die designed to be attached to an injection molding machine or extruder. The die has three exchangeable inserts to vary the slit height. The experiments were performed using the slit die connected to an injection molding machine and the results were compared with off-line experiments by capillary, plate-plate, and cone-plate rheometer. The selected polymers had essentially the same results with different slit heights, verifying the absence of wall slip. The agreement with the conventional off-line rheometer results was very good for all polymers under the study. This suggests that a simple slit die construction can be used as an inexpensive rheometer to measure rheological properties of polymer melts for processing purposes. The shear history of the investigated melt in the in-line slit die experiments resembles that in actual melt processing and therefore determination of rheological properties under true processing conditions is possible.

INTRODUCTION

The rheological properties of polymers are closely related to their processing behavior and hence to the end-product quality. Even though polymer melts are known to exhibit both viscous and elastic response, the key rheological property in most practical processing situations is the shear viscosity and its dependence on shear rate, temperature and (possibly) pressure. Conventionally, the shear viscosity is measured by means of a rotational or capillary rheometer. With the rotational rheometer, either a cone-plate or plate-plate configuration is used for polymer melts. In general, the rotational rheometer is capable of providing reliable shear viscosity data only at relatively low shear rates (say, below 10 s^{-1}) due to the onset of edge fracture. The capillary rheometer, on the other hand, is most appropriate for high shear rates. With this instrument, a round-hole die is generally chosen, but a slit die may also be used.

Alternatively, the shear viscosity measurements can be made using a die that is connected to the real processing equipment, either an extruder or injection molding machine. Such experiments are often called in-line measurements. If the volumetric flow rate through the die and the pressure gradient in the fully developed region are known, the shear viscosity can be determined relatively straightforwardly, as in the case of a capillary rheometer. An obvious advantage of using the processing equipment as a rheometer is the fact that the thermo-mechanical history experienced by the test material is fairly similar to that in the actual processing operation. By contrast, in the conventional capillary rheometer the melting of the polymer takes place by

means of heat conduction only and consequently there is practically no mixing and homogenization before the entry into the die.

In the past years, several developments have been directed toward performing rheological measurements in conjunction with injection molding machine[1,2,3]. and extruder[4]. In addition to in-line shear viscosity measurements, also extensional viscosity has been evaluated through contraction flow analysis[3,5]. Use of an injection molding machine provides an advantage in that the volumetric flow rate can be directly determined from the axial screw speed during the injection, provided that the leakage flow over the non-return check valve is negligibly small. In the case of the extruder, one needs to collect the out-flowing material for a certain period of time and weigh it to determine the mass flow rate, which when divided by the melt density will yield the volumetric flow rate. Moreover, the flow rate from the extruder typically exhibits some fluctuations. As in the capillary rheometer, both the round-hole and slit dies can in principle be used in these measurements. The structure of the slit die enables the direct measurement of the pressure gradient via two (or more) pressure transducers mounted along the die in the region where the flow is fully developed, which eliminates the need to correct for the entrance pressure drop.

In this study a tailor-made slit die was developed and tested with an injection molding machine for measuring shear viscosity of two grades of polypropylene and two grades of polystyrene. The purpose of the work was to evaluate the opportunities to use the special modular die design for easy access rheological data useful for processing purposes. The different slit sizes were also used to verify the absence of wall slip.

SLIT DIE THEORY

When a fluid flows in a wide slit, the edges of the slit can often be assumed to have negligible effect on the flow field. As a rule, this holds when the slit width $w \geq 10h$ (h = slit height). When a slit die is connected to an injection molding machine, the flow rate in the die can be calculated from the radius of the screw R_s and the injection rate V_{inj}.

$$Q = \pi R_s^2 V_{inj} \tag{1}$$

Shear stress at the die wall is

$$\tau_w = \frac{h}{2}\frac{\Delta p}{l} \tag{2}$$

where Δp is the pressure gradient over the flow length l. Shear rate for a Newtonian fluid (apparent shear rate) in a rectangular slit can be calculated from the flow rate Q

$$\dot{\gamma}_{wa} = \frac{6Q}{h^2 w} \tag{3}$$

For shear-thinning polymers the non-parabolic velocity profile must be taken into account and true shear rate at the wall be determined. This can be made by Rabinowitsch correction, but a simple, yet reasonably accurate alternative for accounting for the non-Newtonian velocity profile has been proposed by Schümmer and co-workers[6,7] and developed further by Giesekus and Langer[8]. This procedure is based on estimating the shear rate and viscosity at vertical distance y from the slit centerline, where the apparent shear rate equals the true shear rate. Hence, considering that under fully developed conditions the apparent shear rate, as well as the shear stress, varies linearly with radial position, one obtains

$$\eta(x^*\dot{\gamma}_{wa}) = \eta_a(\dot{\gamma}_{wa}) \tag{4}$$

where the factor $x^* = 2y/h$ and h is the slit height. Apparently, when the power-law coefficient $n = 0.36 - 1.2$, x^* varies only slightly, so that a representative value of x^* may be chosen for most materials with very little loss in accuracy. For a rectangular slit geometry this approximation is given as

$$x^* = \left(\frac{2n+1}{3n}\right)^{n/(n-1)} \approx 0.79 \qquad (5)$$

Schümmer approximation shifts data only horizontally (to the left) and can be applied to single points.

EXPERIMENTAL

Materials

All the experiments were performed with two grades of polypropylene homopolymers (PP); Moplen HP501H and Moplen HP501L (LyondellBasell) and two grades of polystyrene (PS); Polystyrol 143E and Polystyrol 158K (BASF). Table 1. lists their densities and melt flow rates (MFR) or melt volume flow rates (MVR), provided by the supplier.

Table 1. Properties of tested polymers (data provided by suppliers)

Polymer/ Grade	Density (kg/m^3)	MFR (g/10 min) / MVR* (cm^3/10 min)
PP-1/ Moplen HP501L	900	6.0 (230 °C, 2.16 kg)
PP-2/ Moplen HP501H	900	2.1 (230 °C, 2.16 kg)
PS-1/ Polystyrol 143E	1040	10* (200 °C, 5.0 kg)
PS-2/ Polystyrol 158K	1050	3.0* (200 °C, 5.0 kg)

Measurements with an adjustable slit die

The shear viscosity measurements were made with an injection molding machine turned into a rheometer by modifying it with a specially manufactured slit die (Fig. 1a) having an adjustable height. The slit height adjustment was accomplished by means of three exchangeable inserts that produce heights of 0.75, 1.0, and 1.5 mm (Fig. 1b). All the slits have the width of 15mm, thus they fulfill the requirement of "infinitely wide" ($w \geq 10h$) slit. Using this kind of design has several advantages. The die can be attached to different injection molding or extrusion machines using a simple fitting piece. The structure enables an easy and fast modification of the slit height to suit polymer melts with various flow properties. Using different slit sizes also allows for the detection of the possible wall-slip. Common problem with slit dies is their difficult cleaning: melt residues in the die can burn in the narrow corners and distort the flow cross-section. With the present design the cleaning is significantly facilitated by the detachable inserts and this problem is eliminated.

A high-precision, all-electric Fanuc Roboshot α-30C injection molding machine (IMM) was used in these tests. It has a maximum injection pressure of 200 MPa, injection rate of 300 mm/s and clamping force of 300 kN. The diameter of the plasticizing screw was 20 mm. The slit die was fixed to the stationary mounting plate of the machine (Fig. 2a) and heated with a band heater. The temperature was controlled by a Pt100 resistance temperature detector (RTD) sensor for maintaining the set temperature precisely over the entire die geometry. Pressure was recorded at two points, P1 and P2 by Dynisco pressure transducers (type: MDT 462H-1/2-5C-15/46) with the output voltage of 0-10 V and pressure range of 0-50 MPa. The distance between the two pressure sensors was 55 mm. Pressures in the slit during the injection cycle were recorded using an Agilent 34970A data acquisition unit (Fig. 2b) connected to a PC enabling data collection directly in a spreadsheet program.

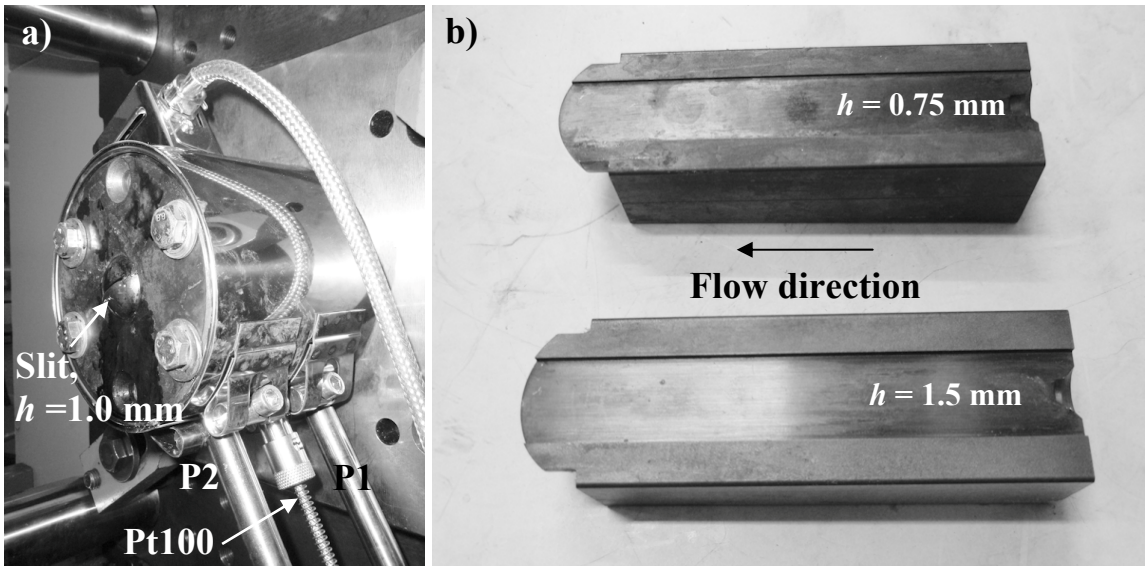

Figure 1. a) The die mounted with 1 mm slit insert, pressure sensors P1 and P2, band heating and temperature regulation with Pt100 sensor. b) 0.75 mm and 1.5 mm exchangeable slit inserts

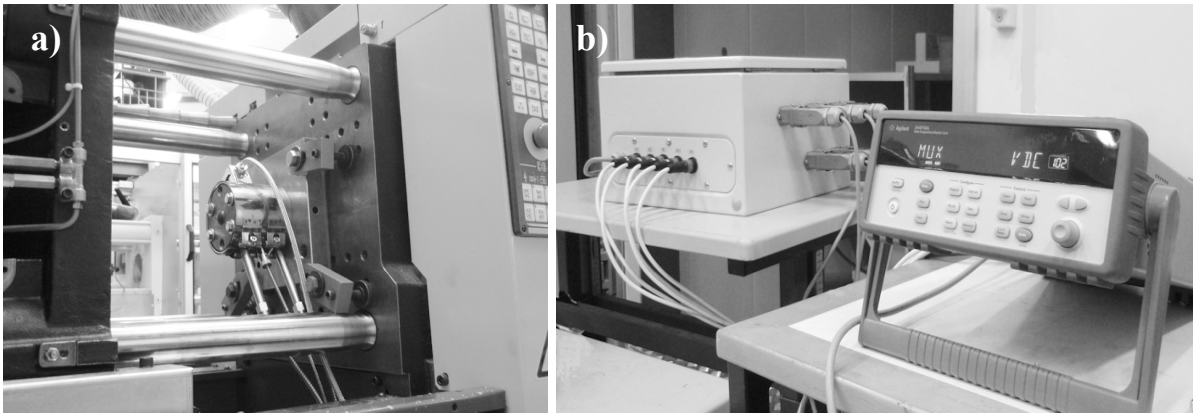

Figure 2. a) Slit die fixed to the mounting plate of the injection molding machine and b) data acquisition equipment

The tests were carried out at two temperatures, 200 and 230 °C. Injection rates up to 130 mm/s were used, and the maximum shear rate was achieved with the smallest slit height. The Schümmer approximation with $x^* = 0.79$ was applied in order to take into account the plug-like velocity profile in the die. The injection rates and corresponding volume flow rates and apparent shear rates are shown in Table 2. For PP-2 and PS-2 the injection pressure limit (200 MPa) of the machine, as well as the maximum pressure sensor capacity (50 MPa) were approached in the measurements at 200 °C with the smallest slit (h = 0.75 mm) and highest injection rates. Therefore in these test conditions V_{inj} = 10 – 50 mm/s were used for PP-2 and PS-2. At 230 °C V_{inj} = 20 – 90 mm/s were used for PP-2 and PS-2, as well as for PP-1 and PS-1 at both test temperatures.

As the pressure is recorded inside the slit, no correction for entrance pressure drop is necessary. For each injection rate a minimum of five parallel tests were made, and the pressure at each rate was calculated as an average of the measured points in the area where the pressure reached the steady value. An example of recorded slit pressures is given in Fig. 3 where pressure curves obtained in separate tests are plotted together in order demonstrate the time scale at different injection rates.

The assumption in the fluid mechanics is that the fluid velocity at the wall is zero, meaning that the fluid adheres to the wall. The assumption in the fluid mechanics is that the fluid velocity at the wall is zero, meaning that the fluid adheres to the wall. This is not true in all flow situations: especially highly entangled linear polymers have been reported to exhibit wall slip. In case of slipping, the viscosity values obtained with different slit heights will not collapse on the same curve: in the presence of slip the shear rate at wall is smaller than assumed in the situation where melt adheres to the wall. The true wall shear rate is then found if the slip velocity is known and subtracted from apparent shear rate[9,10]. Polypropylene and polystyrene do not typically exhibit wall slip, which should be seen as a good superposition of the results with all the slit heights used here.

Table 2: Injection rates, corresponding volume flow rates, and apparent wall shear rates

Slit h [mm]	V_{inj} [mm/s]	Q [mm^3/s]	$\dot{\gamma}_{wa}$ [s^{-1}]
0.75	10	3142	2234
	20	6283	4468
	30	9425	6702
	40	12566	8936
	50	15708	11170
	70	21991	15638
	90	28274	20106
1.00	10	3142	1257
	30	9425	3770
	50	15708	6283
	70	21991	8796
	90	28274	11310
1.50	10	3142	559
	40	12566	2234
	70	21991	3910
	100	31416	5585
	130	40841	7261

Fig. 3 demonstrates the limitation of a small-size injection molding machine as a rheometer: The screw stroke of the machine is 70 mm, and with 20 mm diameter screw the maximum shot volume is then approximately 22 cm^3. Thus at injection rate of 10 mm/s the stroke lasts approximately 7 seconds whereas at injection rate of 90 mm/s it takes less than 0.8 s. This may deteriorate the measurement accuracy, and especially with high injection rates several parallel tests are needed in order to verify the results. In our experiments the parallel tests gave – also at highest injection rates – very consistent pressure readings thus indicating reliability and good reproducibility of the measurement system.

Comparative measurements with conventional rheometers

Capillary rheometer measurements were performed using Göttfert Reograph 6000 capillary rheometer and a 20 mm long round-hole die with 1mm diameter. The entrance pressure drop was measured with an orifice die, and subtracted from the pressure measured with L/D=20 die.

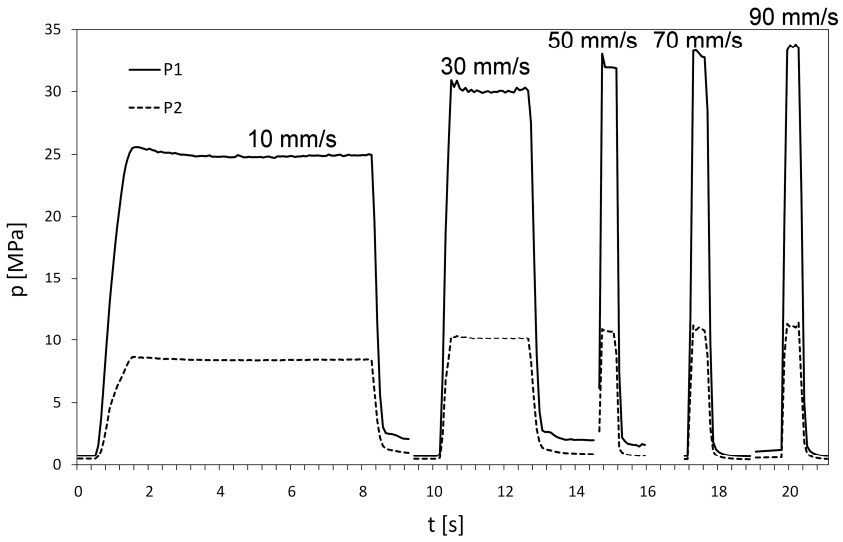

Figure 3. Recorded pressure curves with slit h = 1 mm for PP-2 at 200 °C and injection rates 10, 30, 50, 70 and 90 mm/s. The curves are from separate tests but plotted together to demonstrate the time scale at various injection rates

Due to the conically expanding outlet of the die, a small additional pressure drop develops in the expansion area if the melt adheres to its wall filling it completely. This was corrected by dividing the measured Δp_e by 1.5, based on an empirical procedure suggested in our earlier study[11]. Shear rate was corrected using a Schümmer approximation analogously to slit die data. For a round-hole die the correction factor $x^* = 0.83$.

Low-shear data was measured in steady-shear and dynamic oscillatory shear experiments, assuming that Cox-Merz rule[12] can be applied. Experiments were done using Anton Paar MCR301 rheometer with CTD600 convection oven. For steady shear experiments, a cone-plate geometry with 25 mm diameter and 2° cone angle, and for oscillatory shear a parallel-plate system with 25 mm diameter, were used. All the experiments were carried out under nitrogen atmosphere.

RESULTS

Viscosity curves constructed from steady shear, dynamic oscillation, capillary rheometer and IMM/ slit die experiments are presented in Figs. 4 a-c for all four materials at 200 and 230°C. For all the tested materials the comparison with capillary rheometer data shows very good agreement. Generally the trend observed in in-line experiments compared to capillary rheometry is somewhat lower viscosity, which has been attributed to stronger pre-shearing by plasticizing screw; in capillary rheometer the melt is pushed through the die by a piston and experiences a lot less shearing[13].

Good agreement between the capillary rheometer and slit die data confirms the starting point of the study: the flow rate can be calculated with a sufficient accuracy from the screw speed and dimensions, that is, the leakage flow through the non-return valve seems to be insignificant.

The excellent superposition of the viscosity curves obtained with different slit heights verifies the absence of wall slip as expected.

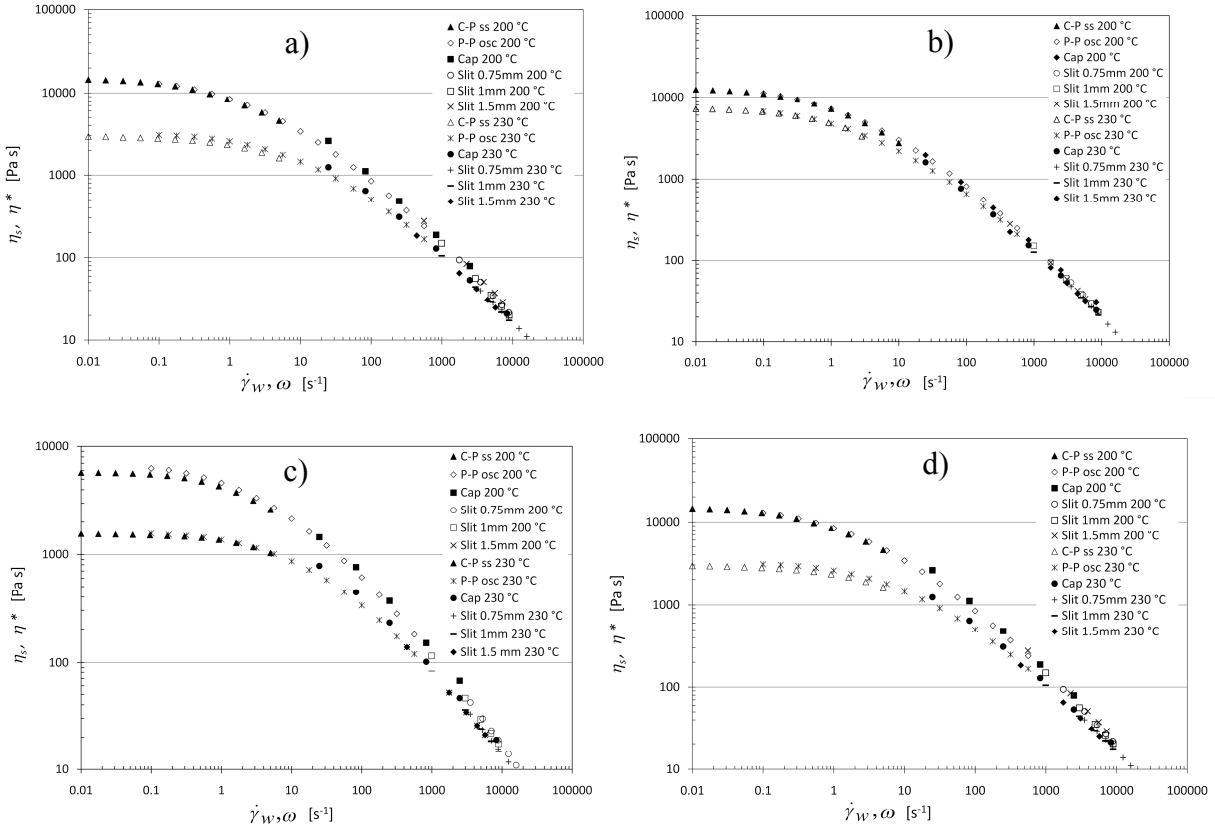

Figure 4. Viscosity curves at 200 and 230 °C a) PP-1, b) PP-2, c) PS-1, d) PS-2

CONCLUSIONS

Viscosity measurements for two polystyrenes and two polypropylenes were made using a tailor-made slit die with variable gap height of 0.75, 1.0 and 1.5 mm. Viscosity values calculated from the pressure recordings with all three dies showed a good superposition, which verifies the assumption of no-slip at the wall. For all the materials the results were also comparable with the ones obtained by capillary rheometer and rotational rheometer.

This study showed that a relatively simple, tailor-made slit die can offer an attractive alternative as a rheological tool for small polymer processing companies that cannot invest in proper stand-alone rheometers. The experimental procedure is straightforward and fast, and use of three different slit heights enables evaluation of occurrence of wall slip for the tested polymer melts. Moreover, the thermo-mechanical history of the melt in the measurements is similar to the processing methods where screw plasticizing is used and therefore viscosity measurements simulate the true processing conditions. Detachable slit modules enable easy cleaning thus eliminating one of the downsides usually associated with slit dies.

The die construction offers also various possibilities for modification: One of them would be building a constriction valve downstream of the flow to increase the back pressure in the die and so to evaluate the pressure dependence of viscosity. The shear rate and pressure range in this study was limited by the capacity of the molding machine used: In order to obtain higher shear rates and to measure viscosity under elevated pressure, machine with larger shot

volume and injection pressure capacity is needed.

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