

Improved melt flow rate measurements: an industrial case study

Summary

The melt flow rate method is widely used in the plastics industry and is likely to remain a dominant tool for quality control and assurance. However, it is widely accepted within the industry that the melt flow rate method has limitations. To address these limitations various developments to the melt flow rate method have been undertaken in recent work at NPL. These developments encompassed: the use of multi-load testing to enable melt flow rate data to be extrapolated more reliably to higher shear rates, the use of alternative die geometries to separate the material's shear and extensional flow behaviours, and the measurement of extrudate swell and draw-down also to determine the material's extensional flow behaviour.



As part of this work a number of industrial case studies were undertaken to demonstrate the relevance of the new measurement methods. The case study with Synbra Polymers Ltd is reported here. The results clearly demonstrate that through the use of these new methods, using equipment that is widely available, significantly more information on the flow behaviour of the materials can be obtained. This has enabled Synbra Polymers to clearly differentiate between their materials and has provided measures that correlate well with how they behave in processing.

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INTRODUCTION

Two samples of polypropylene (PP), a virgin homopolymer grade (HHH100) and a recycled grade (HHG000), were investigated using the multi-rate and extensional flow techniques developed for the melt flow rate instrument. The objective was to assess the potential of the newly developed methods in their ability to differentiate between the two materials that processed differently on their production line, yet were similar in behaviour when tested using the standard melt flow rate method.

METHOD

All measurements were made using a standard Ray-Ran Test Equipment Ltd 5MPCA Advanced Melt Flow System Instrument, the only additional requirements being the use of a short die and a hand-held micrometer.

This instrument has a linear displacement transducer that allows the region over which the measurements are taken to be split into many segments. Thus in performing the multi-rate measurements reported herein 20 separate determinations of melt flow rate per barrel charge were made. For a single barrel charge, four different loads (0.325 kg, 2.16 kg, 5.0 kg and 12.5 kg, all identified in ISO 1133) could be applied to enable the flow behaviour to be determined over a range of melt flow rates. So for each load in a multi-load test approximately five separate determinations of melt flow rate were obtained. The loads were applied in increasing value to enable better operator control to be exerted over the progress of the test.

To determine the extensional flow behaviour a short die was used that was 1.05 mm in diameter and 0.24 mm in length. Its dimensions were chosen in order to produce melt volume flow rate (MVR) values similar to those obtained using the standard die. The short die can be used to determine entrance pressure drop data that has successfully been correlated qualitatively with extensional viscosity values [2]. Using both the standard and short dies, it is possible to obtain shear viscosity data corrected for entrance effects for the materials.

As an alternative approach to characterising the extensional flow behaviour, extrudate swell measurements were made in-situ using a non-

contacting laser scanning micrometer mounted beneath the die exit, and also with a hand-held micrometer on the cooled extrudate after the melt flow rate test had been completed. The extensional flow behaviour of the materials was determined from these measurements as indicated by Rides [1]: extensional viscosity is related to how the extrudate draws down during the test under its own weight. For these tests a standard die, a load of 2.16 kg and a temperature of 230 °C were used.

RESULTS

Multi-load and short die testing

The MVR of the materials was measured using the standard die and also the short die, Figures 1 and 2 respectively. In using the standard die, Figure 1, there is little difference between the two materials, particularly at the lower loads (2.16 kg would be the standard load [ISO 1133] to use for PP). Thus the standard melt volume or melt mass-flow rate test would be unable to differentiate between the materials. At higher loads the recycle (HHG000) had slightly higher MVR values – the difference being approximately 20%. However, when using the short die there was a significant difference in the MVR values of the two materials for all loads, Figure 2. The recycle (HHG000) had higher values, by a factor of approximately $\times 2$, compared with the virgin material. Again, the difference between the materials increased with increasing load indicating that the standard conditions [ISO 1133] are perhaps not best suited for this specific application. It is noted however that at the high loads the melt volume flow rate values are high and that manually operated MFR testers may not have sufficient accuracy to realise the differences between such materials.

Extrudate swell and draw down behaviour: single load, standard die results

Each sample was extruded under the same conditions, using a load of 2.16 kg. The extrudate diameter was measured during extrusion using a laser scanning micrometer and also subsequently using a hand-held micrometer on the solidified extrudate. The results clearly showed a significant difference between the two materials, Figure 3. The recycle (HHG000) exhibited less swelling and also a slightly greater draw-down of the extrudate. The reduced swelling suggests a less

“elastic” flow behaviour. This difference in extrudate swell magnitude was also observed in results obtained using the laser scanning micrometer.

The gradient of the hand-held micrometer data, Figure 3, i.e. extrudate diameter versus distance, gives an indication of the relative extensional viscosities of the materials: for example the larger the negative gradient the lower the extensional viscosity of the material [the actual diameter also has an effect on the calculated values]. From these data it is possible to calculate approximate extensional viscosity values as a function of strain rate [1], Figure 4. It can be seen that the virgin material (HHH100) exhibited higher extensional viscosity values than the recycle (HHG000) at equivalent strain rates, by a factor of approximately x2.

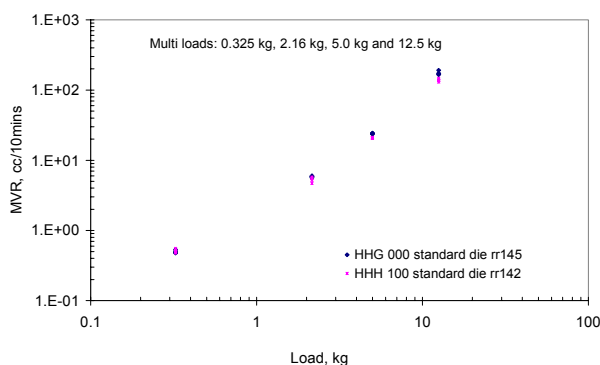


Figure 1: Comparison of the melt volume flow rates measured using a standard die of the virgin (HHH100) and recycle (HHG000) polypropylenes at 230 °C.

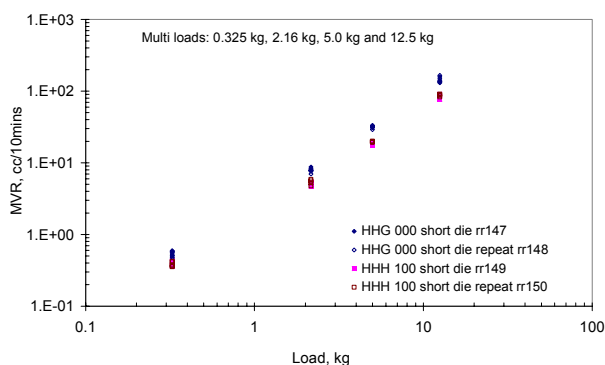


Figure 2: Comparison of the melt volume flow rates measured using a short die of the virgin (HHH100) and recycle (HHG000) polypropylenes at 230 °C.

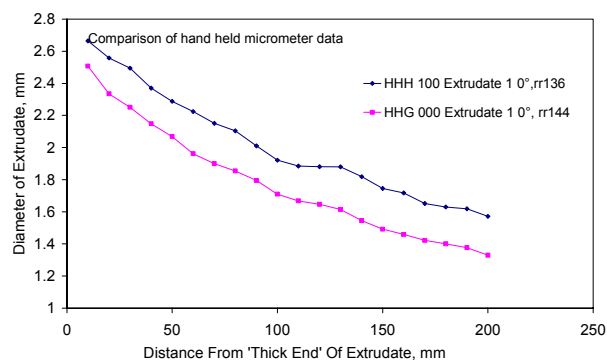


Figure 3: Comparison of the extrudate swell behaviour of the virgin (HHH100) and recycle (HHG000) polypropylenes measured using a hand-held micrometer.

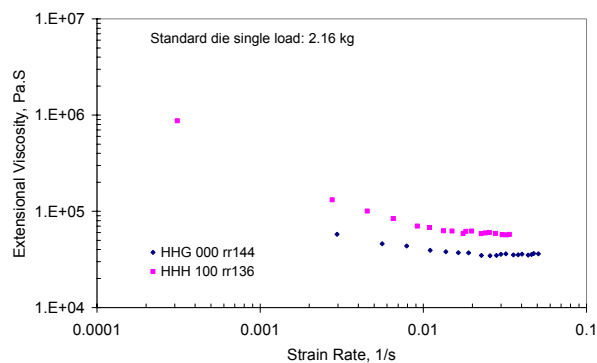


Figure 4: Comparison of the estimated extensional viscosities of the virgin (HHH100) and recycle (HHG000) polypropylenes determined from extrudate swell measurements using a hand-held micrometer.

Conclusions

By using the short die it is possible to observe differences in the MVR behaviour of the materials, particularly at high loads. These differences were not apparent when using the standard testing conditions.

The measurement of the extrudate swell also allowed clear distinction of the behaviour of the two materials. The virgin material had a larger extrudate diameter indicating a more “elastic” flow through the die. These differences were observed in results obtained using both the laser scanning micrometer and the hand-held micrometer.

Quantitative extensional viscosities of the materials were determined from the measurement of extrudate diameter using the hand-held micrometer. The reduction in diameter of the extrudate (draw-down) as the test progresses is an indication of the extensional viscosity of the material. A greater draw down indicates a lower extensional viscosity. The virgin PP (HHH100) had significantly higher extensional viscosity values than the recycle (HHG000).

In conclusion, differences in the batches of material have been identified using the multi-load short die test procedure, through measurement of the extrudate swell, and through determination of the extensional viscosity via extrudate draw-down.

The differences observed in the processability of these materials can be explained by the observed differences in the extensional flow behaviour, characterised either by the short die measurements or by the approximate extensional viscosities obtained from extrudate swell measurements.

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