

## **In-process monitoring of the flow properties of polymers for extrusion, compounding and injection moulding**

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### **ABSTRACT**

Two methods of monitoring the flow properties of polymers during processing have been demonstrated. In the first method, a Porpoise Viscometers on-line rheometer was used to monitor extrusion, compounding and film blowing during production. A range of benefits was identified that could not realistically have been met through off-line measurements. As a result, one company has purchased two on-line rheometers and seen a return on the investment in three months; other companies have plans to invest in the technology. In the second method, the injection moulding process was monitored through use of an instrumented nozzle and software that calculated viscosity and automatically identified different parts of the moulding cycle.

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Approved on behalf of Managing Director, NPL  
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## CONTENTS

|           |                                                                            |           |
|-----------|----------------------------------------------------------------------------|-----------|
| <b>1.</b> | <b>INTRODUCTION .....</b>                                                  | <b>1</b>  |
| <b>2.</b> | <b>IN-PROCESS MONITORING FOR EXTRUSION AND COMPOUNDING PROCESSES .....</b> | <b>1</b>  |
|           | 2.1 ON-LINE MEASUREMENT METHOD .....                                       | 1         |
|           | 2.2 PROCESS MONITORING TRIALS AND RESULTS .....                            | 2         |
|           | 2.2.1 Raychem trials .....                                                 | 2         |
|           | 2.2.2 Polypipe Civils trials .....                                         | 3         |
|           | 2.2.3 Reddiplex Group trials .....                                         | 4         |
|           | 2.2.4 UCB Films trials .....                                               | 5         |
|           | 2.3 ON-LINE TRIALS DISCUSSION AND CONCLUSIONS .....                        | 6         |
| <b>3.</b> | <b>IN-PROCESS MONITORING FOR INJECTION MOULDING .....</b>                  | <b>7</b>  |
|           | 3.1 IN-LINE MEASUREMENT METHOD .....                                       | 7         |
|           | 3.2 PROCESS MONITORING TRIALS AND RESULTS .....                            | 7         |
|           | 3.2.1 BPTA trials .....                                                    | 8         |
|           | 3.2.2 Bespak trials .....                                                  | 9         |
|           | 3.3 IN-LINE TRIALS DISCUSSION AND CONCLUSIONS .....                        | 9         |
| <b>4.</b> | <b>CONCLUSIONS .....</b>                                                   | <b>10</b> |
| <b>5.</b> | <b>ACKNOWLEDGEMENTS .....</b>                                              | <b>11</b> |
| <b>6.</b> | <b>REFERENCES .....</b>                                                    | <b>11</b> |

## FIGURES

|                                                                                                                                                                                                                                                                     |    |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----|
| Figure 1. Schematic diagram of the Porpoise P3 on-line rheometer. ....                                                                                                                                                                                              | 11 |
| Figure 2. Porpoise Viscometers Limited's on-line rheometer mounted on a extrusion compounder for trials during production at Raychem Limited. ....                                                                                                                  | 11 |
| Figure 3. Variation measured on-line of the MFI of a filled polyolefin over a 2 day manufacturing run at Raychem. ....                                                                                                                                              | 12 |
| Figure 4. Batch to batch variation of a filled polyolefin compound made on an Intermix extrusion compounder operated by Raychem, (the polymer batches were supplied from different reactors). ....                                                                  | 12 |
| Figure 5. Variation in MFR values over a change over period at approximately 16.75 hours at Polypipe Civils. ....                                                                                                                                                   | 13 |
| Figure 6. Variation of shear viscosity with time for day 1 at three different shear rates (material ref.: HUE000 at 280 °C). ....                                                                                                                                   | 13 |
| Figure 7. Power-law fit to apparent shear viscosity data, day 1 (material ref.: HUE000 at 280 °C). ....                                                                                                                                                             | 14 |
| Figure 8. Comparison of on-line P3 and off-line capillary extrusion rheometer measurements of shear viscosity (material ref.: HUE000 at 280 °C). ....                                                                                                               | 14 |
| Figure 9. Schematic of the nozzle set-up and a picture of the nozzle fitted in an injection moulding machine. ....                                                                                                                                                  | 15 |
| Figure 10. Typical pressure curve as seen at the BPTA trial. ....                                                                                                                                                                                                   | 16 |
| Figure 11. The user panel showing data produced during the BPTA trial. The top right shows the pressure and screw position. On the left the injection phase is highlighted. The bottom shows graphs of a user selection of in-line process measurement points. .... | 16 |
| Figure 12. Results from trials performed on a range of polypropylenes (PP) and one high density polyethylene (HDPE). ....                                                                                                                                           | 7  |
| Figure 13. Typical graph of the viscosity of polypropylene as a function shear rate. The grade of polypropylene shown here is different from the grade used at the trials. ....                                                                                     | 7  |

## 1. INTRODUCTION

The quality of plastics products produced by injection moulding, extrusion or extrusion compounding, for example, is dependent on the magnitude and consistency of the flow properties of the material. These properties affect how the material performs during processing. They are also a measure of the material's structure and thus provide an important indicator of the consistency of the material and its behaviour in service.

These properties have traditionally been measured off-line using either melt flow rate [1] or capillary extrusion rheometry techniques [2-4]. However, the time delay between sampling and off-line measurement can be significant resulting, potentially, in the production of out-of-specification material during that period. Furthermore, off-line measurements of melt flow rate can be excessively time consuming, variable and they cannot always distinguish differences between materials, thus leading to reduced industrial confidence in their use. Off-line measurements by capillary extrusion rheometry can improve on the variability and the ability to distinguish between materials compared with melt flow rate measurements but they can be even more time consuming.

In order to remain competitive, UK companies need to reduce scrap rates and increase consistency and quality. One way of achieving this is to be able to monitor and control the rheological properties of polymers during processing. An important rheological parameter is viscosity - a measure of the ease of flow of the material. On-line techniques can be used to measure the viscosity of polymers during extrusion processing to provide more immediate measures of process consistency and product quality, and also to provide new data to enable the user to optimise materials and processing parameters more effectively. Through continuous monitoring of the processing conditions and the material's properties reduced costs and higher levels of manufacturing efficiency can be achieved, thereby improving industrial competitiveness. More significantly, such measurements can provide data for automated process control. However, the use of on-line methods for the measurement of viscosity is still in its infancy and off-line measurements are often inconsistent and difficult to relate to the processing situation. To overcome these problems the use of an on-line measurement system was demonstrated that could be readily attached to existing commercial extrusion equipment, thus providing data directly relevant to the actual process history. In addition, an in-line method for measuring viscosity during injection moulding was also demonstrated.

On-line rheological measurements are defined for the purposes herein as measurements made on process equipment in which the polymer is taken from the process stream to the rheometer and then either returned to the process stream or sent to waste. In-line measurements are defined as where the measurement is performed on the process stream. This report describes the on-line and in-line techniques used and presents some results of the industrial trials that were carried out. This report is deliverable 12.1 of a "Studio" project entitled "Industrial Assessment of Techniques to Measure Rheological Properties of Polymers during Processing".

## **2. IN-PROCESS MONITORING FOR EXTRUSION AND COMPOUNDING PROCESSES**

### **2.1 ON-LINE MEASUREMENT METHOD**

The approach adopted for these trials was to mount a Porpoise Viscometers Limited's P3 on-line rheometer onto the production line to make measurements of the flow properties of polymer melts during production. The rheometer was mounted using a transfer pipe to a standard pressure transducer port on or near the extruder die head of the processing equipment. Mounting the rheometer by this method resulted in rapid and easy installation with only the transfer pipe being tailor-made to fit the plant. The transfer pipe's function was to convey melt under pressure from the extruder to the rheometer. The melt then encountered a gear pump in the rheometer that controls the rate of flow through the die. A pressure transducer was located just upstream of the die. The rheometer was programmed to test normally at three or four flow rates, referred to as "trickle", "purge", "high" and "low" although fewer or more rates can be used. The objective of the trickle was to minimise the waste generated by the rheometer, and the purge phase was to introduce fresh material from the extruder head to the rheometer so that subsequent measurements at the high and low flow rates were made on fresh material.

From measurements of the pressure above the die, the dimensions of the die and the flow rate the apparent shear stresses and apparent shear rates in the melt can be determined. The apparent shear viscosity of the material, the ratio of the apparent shear stress to the apparent shear rate, can then also be determined. These apparent viscosities were not corrected for either entrance pressure drop or non-Newtonian velocity profile effects. Both of these corrections would result in a decrease in the values obtained. The measured data were also used to automatically determine melt flow rate values that are more widely understood and used by industry for quality control. The results were logged automatically for each test and stored in a single file for each day of the trial.

The die normally used had a length to radius ratio of 7.636, the same as for the MFR test, although the diameter and length can be varied depending on the behaviour and filler, if any, of the polymer. A diameter of 3 mm was typically used thus giving a die length of 11.45 mm. The die had a profiled entry geometry to avoid "dead" regions forming upstream of the die.

A schematic diagram of the equipment is shown in Figure 1 and a photograph of the equipment in use at Raychem's Swindon site is shown in Figure 2.

### **2.2 PROCESS MONITORING TRIALS AND RESULTS**

A number of on-line trials have been carried out using the Porpoise P3 rheometer. Key features of these trials are now presented, highlighting important aspects, findings and outcomes of the work.

Extrusion compounding is one of the major processes used throughout a very competitive plastics industry and it is vital for the industry that the process can be optimised and controlled. By doing so it can reap the benefits of reduced scrap rate and increased product

quality assurance. The quality of materials produced by extrusion compounding is dependent on the magnitude and consistency of the rheological properties of the compound during processing.

### 2.2.1 Raychem trials

Raychem Limited manufacture wire and cable, connectors, heat shrinkable tubing and moulded parts in addition to a number of other products related to electrical distribution and communications systems. These products are primarily based on the use of plastics as the insulating component. The integrity of the insulator is of key importance to the quality of the final product, and thus the quality of the plastics compounds is critical. A number of complex, highly filled materials for these applications, often containing many ingredients, are extrusion compounded in-house by Raychem.

A production compounding extruder at Raychem Limited was targeted for the trial using the on-line Porpoise P3 rheometer. Trials were carried out over a prolonged period, monitoring viscosity and hence melt flow rate during actual production runs covering a wide range of compounds.

The trials clearly demonstrated the ability of the instrument to monitor the consistency of compounds over extensive periods, Figure 3. In this trial the viscosities at two different shear rates and the melt flow rate were each determined every 12 minutes over the 45 hour long run. Thus the technique was able to provide much more frequent and rapid monitoring than was possible using off-line methods, thereby ensuring better control of the process and consequently better quality of the product. Such a frequency of sampling could not possibly be economically or practicably achieved by off-line measurements such as melt flow rate. Even if off-line monitoring at an equivalent frequency were performed the time delay between compounding the material and obtaining data would be significantly greater thereby reducing its value to the operator for process control.

The output of melt flow rate values from the instrument for use in monitoring the process might at first appear to be a regressive step in that such data are determined from two shear viscosity values obtained at two different shear rates. Thus the operator is throwing away one half of the information. From two points one can obtain a magnitude and rate dependence of viscosity but by reducing the information to a single point one can obtain only a magnitude (N.B. both data points can be and were stored for later use). However it was beneficial in terms of the acceptance of the equipment by the operators who, although skilled, were not familiar with shear viscosity values. This will predominantly be the case throughout the industry as the melt flow rate is the standard measure of the flowability of polymer melts. The acceptance of the instrument by the operators was key to the successful adoption of the technology by industry.

The use of the P3 also demonstrated that it was possible to identify differences between nominally the same polymers made by different reactors, Figure 4. These different batches were identifiable as having different melt flow rates. The use of the rheometer also helped identify the time taken to switch compounding to a different formulation which has implications for optimising when to start collecting compounded material of guaranteed quality. These were some of the benefits of using the on-line rheometer that were identified through this trial: they are largely applicable to all the trials and clearly indicate the advantages of on-line rheological monitoring.

Following the trial, Raychem have purchased two on-line Porpoise rheometers, which have provided a return on capital investment in only three months.

### 2.2.2 Polypipe Civils trials

Extrusion compounding type trials were also carried out at Polypipe Civils although this time it involved the use of the on-line rheometer on a compounding line used for producing pellets from plastics recyclate, prior to the pellets being re-used in-house. The recyclate was largely in the form of flake. Polypipe Civils produce a range of extruded drainage products. The objectives of the trial were to assess the potential for using the equipment for quality control purposes to monitor the reprocessing facility and to aid in-house materials selection. The change-over from producing one compounded material to another is shown in Figure 5 between approximately 16.5 and 17 hours. Melt flow rate measurements made off-line on these materials and the corresponding values measured on-line are presented in Table 1.

Table 1: Comparison of on-line and off-line MFR measurements: Polypipe Civils trial.

| Time (hours) | MFR on-line | MFR off-line | % difference between on-line and off-line MFR | % difference of on-line value from mean of on-line values | % difference of off-line value from mean of off-line values |
|--------------|-------------|--------------|-----------------------------------------------|-----------------------------------------------------------|-------------------------------------------------------------|
| 15.75        | 0.230       | 0.211        | 8%                                            | +1                                                        | +4                                                          |
| 16.25        | 0.252       | 0.217        | 14%                                           | +12                                                       | +7                                                          |
| 17           | 0.197       | 0.181        | 8%                                            | -12                                                       | -11                                                         |

These data show the trend of a reduction in the melt flow rate values over the period of the transition from one compound to another. The trend is exhibited by both the on-line and off-line measurements. However the on-line measurements were made every 5 minutes compared with significantly longer periods for the off-line measurements. The on-line measurements do indicate a variation within each compound, for example in the first part of the trace of Figure 5. Thus infrequent selection of just one small sample at a time, as is only practicable for the off-line melt flow rate method, will obviously not provide data that will yield information with the same level of confidence as on-line data sampled more frequently.

### 2.2.3 Reddiplex Group trials

The use of the on-line rheometer to aid a materials evaluation, product and process development programme was carried out at Reddiplex Group plc on an extrusion line. In this sense the trial had different principle objectives compared with the previous two, although in principle the P3 rheometer was still used for assessing the consistency of the material being evaluated. The trial was carried out over a period of four days on one grade of material and on a single product. The process conditions and plant set-up were modified during that period to obtain improved product properties, particularly surface appearance and dimensional accuracy. Additional on-line and off-line measurements of the moisture content of the granular material, the dimensions of the extrudate and the process conditions of

pressure and temperature were also made during the trial. Measurements of the shear viscosity of the material were also made off-line after the trial using a RACER capillary extrusion rheometer at NPL.

Results obtained using the P3 rheometer at three different extrusion rates, equivalent to apparent shear rates of 6.9, 16.4 and 39 s<sup>-1</sup>, indicated that greater scatter was obtained when using lower flow rates although similar trends and features were apparent in all three curves, Figure 6. More importantly there was a variation in the viscosity with time. On the first day the viscosity values gradually increased over the first 80 minutes of testing by approximately 10%, Figure 6. On subsequent days of the trial significant variability in the shear viscosity of the material was observed including significant step changes in viscosity that could not be related to changes in other parameters, either of the process or of the extrudate's properties. Step changes of up to approximately 15% were observed in the viscosity measured at the highest shear rate. Apparent shear viscosity values measured at a single apparent shear rate of 39 s<sup>-1</sup> covered the range  $\approx 2700 - 3300$  Pa.s. The apparent shear rate ( $\dot{\gamma}_{ap}$ ) dependence of apparent shear viscosity ( $\eta_{ap}$ ) is shown in Figure 7 where all data except for points at the start and end of testing shown in Figure 6 are presented. The use of a power-law apparent shear viscosity model ( $\eta_{ap} = k\dot{\gamma}_{ap}^{n-1}$ ) yielded a reasonable fit to the data with  $n = 0.625$  and  $k = 11.1$  kPa.s.

Comparison of apparent shear viscosity results obtained on-line and off-line, the latter measured using a RACER capillary extrusion rheometer, is complicated by the different geometries used, specifically that of the barrel and die diameters and the die lengths. The P3 rheometer determines apparent shear viscosity values that are not corrected for either entrance pressure drop effects or for the effects of a non-Newtonian velocity profile (Rabinowitsch). The effect of applying these corrections would be to reduce the magnitude of the apparent shear viscosity values thus yielding better agreement with the off-line data. Apparent viscosity values obtained using the capillary rheometer with a single die of 4 mm diameter, 60 mm length ( $L/R = 30$ ) and not corrected for entrance pressure drop and non-Newtonian velocity profile have been presented for comparison with values obtained using the P3 rheometer, Figure 8. The aspect ratio  $L/R$  for the P3 on-line rheometer was 7.636. Apparent shear viscosity values obtained using the capillary rheometer were approximately 35% lower than those obtained using the P3: the latter values varied by up to approximately  $\pm 10\%$ . By also using results obtained using a shorter die ( $L/R = 5$ )<sup>1</sup> and calculating, by interpolation, the apparent shear viscosity values that would have been obtained if a single die of  $L/R = 7.636$  had been used, then values were determined for the capillary rheometer that were approximately 25% lower than the P3 values. Reference was made during the trials to the skinning effect of the die by cross-linked material. Analysis of the theory for fluid flow indicates that a reduction in diameter of the P3 die (3 mm) by  $\approx 8\%$ , equivalent to a skin thickness of  $\approx 0.1$  mm, would account for a 35% difference in viscosity values. This skinning effect was more likely to have occurred in the P3 rather than in the RACER as material was flowing through it for a much longer period of time.

The discrepancy between on-line and off-line apparent shear viscosity measurements of  $\approx 35\%$  was therefore considered to be due possibly to material variability ( $\pm 10\%$ ) affecting the

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<sup>1</sup> Due to difficulties encountered in testing this material, this measurement was made using a pressure transducer of a non-optimal range with the consequence that the errors incurred may be larger than permitted by ISO 11443 [4].

off-line capillary rheometry results, the effect of skinning of the wall of the P3 die with cross-linked material, and also due to assumptions in the analysis due to the use of different die geometries in the P3 and capillary extrusion rheometers.

The use of the on-line Porpoise P3 rheometer during the material, product and process development trial identified that material variability was significant and contributed to the problems experienced in establishing acceptable processing conditions for the product.

#### 2.2.4 UCB Films trials

On-line monitoring has applications and benefits for monitoring the production of extrusion processes as well as for compounding and trials development. To demonstrate this two trials using the Porpoise P3 on-line rheometer were carried out at UCB Films. The first was to monitor the consistency of the major core layer used for multi-layer blown film production. The second trial was for monitoring the pelletising of in-house scrap material. With any polymer production process a certain amount of scrap is generated and film blowing is not an exception. Scrap material is generated through, for example, bubble bursts and film that is out of specification. The on-line rheometer was used to monitor the recycling process involving the conversion of flake, film or tube scrap into re-useable pellets.

It was reported that the variability of the melt flow rate was again clearly identified with the P3 on the film blowing plant. Of significance was the trend in MFR value with the stability of the bubble that suggests that by better control of the MFR fewer bubble bursts can be achieved with significant savings in down time and lost productivity. It was also reported that in monitoring the recycling plant there was a possible correlation between the form of the feedstock, e.g. flake, film or thick-walled tube, and the MFR of the reprocessed material. It was suggested that this relationship is due to the energy input required to reprocess the material. The re-use of this recycled material for the core layer of blown film, and its inherent variability due to the form of the scrap, has potentially very significant implications as it is a source of variation in the material and may be responsible for problems experienced with the film production. If the melt flow rate is significantly affected then the effect on the extensional flow properties of the melt may be even greater and it is likely that this is an important key to the processability of the polymer.

### 2.3 ON-LINE TRIALS DISCUSSION AND CONCLUSIONS

It is clear that these on-line trials have benefited the participants in providing a significant improvement in their understanding of their materials and processes, and has either provided or has the potential to provide significant financial benefits in some cases.

The trials carried out by Raychem on heavily filled polymers clearly highlighted the value of the on-line measurements and the benefits of the data obtained. They were able to monitor the consistency of compounds, detect differences between polymers made by different reactors, and identify the time taken to switch compounding to a different formulation. Such differences had not been easily, if at all, identified with off-line measurements. Furthermore, the on-line technique was able to provide more frequent monitoring than was possible using off-line methods, thus ensuring better quality control. The prolonged trials indicated that the rheometer was able to cope with heavily filled polymers and required minimal operator intervention.

The success of the Raychem trials was quickly translated to significant financial savings, even during their first trial. The financial benefits of implementing such on-line technology have been clearly demonstrated by the purchase by Raychem of two instruments to date since the trial. The economics of installing such technology is made favourable in the Raychem case by the added value to the compounded material as is it subsequently processed into its final product form, in this case electrical cables. This added value consideration is applicable to all situations where the item being produced is a component of the final product as consideration to the effects of failure of that component on the final product must be made, for example an engine component in a car.

The potential benefits of implementing the on-line technology have been clearly demonstrated by the Raychem trial and many of the observations made are equally applicable to the other trials. The benefits of a significantly improved understanding of the process have been demonstrated on both extrusion and extrusion compounding plant with implications for improving the process to reduce scrap rates and improving the quality of the product. Through on-line monitoring the confidence in the quality of the product is increased through more frequent measurements. Furthermore there is the additional benefit that through reportedly reduced scatter in measurements compared with off-line melt flow rate measurements the confidence in the data is increased. Consequently the understanding and control of the process is improved as an observed variation is taken to be a true variation in the material rather than possibly being the result of the higher scatter in melt flow rate values measured off-line.

### **3. IN-PROCESS MONITORING FOR INJECTION MOULDING**

#### **3.1 IN-LINE MEASUREMENT METHOD**

In the injection moulding in-line trials the original nozzle on the machine is replaced by an instrumented nozzle. The polymer melt passes through this nozzle, where the viscosity is measured, just prior to entering the mould, Figure 9. The flow through the nozzle generates a pressure drop that is measured by two transducers P1 and P2 positioned along its length. In addition, the position of the screw is measured in real time using an external displacement transducer to determine the flow rate of melt through the nozzle. Since the dimensions of the nozzle channel are known, the viscosity can be determined. No correction was made for the non-Newtonian velocity profile (Rabinowitsch) and no correction was required for entrance effects in the nozzle as measurements were made in a channel of constant cross-section along its length.

The set up used in the trials was tailor-made for each system and assembled at the various trial sites. The nozzle was also instrumented with a thermocouple to measure the temperature of the polymer material. A heating jacket on the nozzle provided better thermal control with a second thermocouple connected to the heating control unit. The piezoelectric transducers used were produced by Kistler. These transducers were chosen because of their small size as they are required to measure pressure in a channel of small diameter. The transducers were designed to operate at a maximum pressure of 3000 bar and up to 350 °C. Data acquisition was performed using a National Instruments data acquisition board driven by LabVIEW based software developed at NPL.

In the trial cases, the injection phase takes typically about one second out of a total cycle time ranging from 20 - 40 seconds. The data acquisition software determined the different stages in the moulding cycle from the pressure and screw position values and also determined during what time frame the pressure drop was sufficiently stable to calculate the viscosity.

In this technique the viscosity is measured at the shear rates occurring during the formation of the product: thus viscosity data is determined at a single shear rate. The value of the shear rate ranged from  $700 \text{ s}^{-1}$  to  $4000 \text{ s}^{-1}$  and was dependent on the injection moulding machine and moulding conditions used. This is higher than typical values from off-line laboratory measurements.

### 3.2 PROCESS MONITORING TRIALS AND RESULTS

In this report the focus will be on two trials. One of the trials was conducted at the BPTA in Telford and one at Bepak PLC in King's Lynn. Some key features of the trials will be discussed in the following sub-sections (some details have been omitted to protect partner companies' commercial information).

Data acquisition software was developed during the project to process the pressure and screw displacement traces of the current injection cycle. This program was called Dimlog (Data injection moulding machine logging).

The following tasks are performed by Dimlog:

- 1) Acquisition of in-line and product data.
- 2) Handling raw in-line data to determine the in-line process measurement points.
- 3) Provide a user interface.

From the pressure and screw position data the following in-line process measurement points were detected:

- a) Position of screw at the beginning of the injection phase.
- b) Melt pressure in the nozzle at the end of the filling phase.
- c) Position of the screw at the end of the filling phase.
- d) Melt pressure in the nozzle at the end of the packing phase.
- e) Screw position at the maximum pressure.
- f) Maximum pressure difference in the nozzle during the cycle.
- g) Melt pressure integral during a cycle [from beginning of fill to end of fill].
- h) Position of the screw at the end of the holding phase.

#### 3.2.1 BPTA trials

The first trial was performed at the British Polymer Training Association (BPTA) in Telford. The BPTA provides training and has a wide range of processing equipment. The injection machine used was an Engel ES 650/100HL producing a set of four spoons in the mould. The nozzle manufactured at NPL had a minimum internal radius of 3 mm and the distance between the pressure transducers was 77 mm. Most of the materials used were polypropylene: one batch of high density polyethylene was also used. A typical injection pressure curve is shown in Figure 10. During the injection phase higher pressures can be

observed and a pressure drop  $P1 - P2$  indicates the flow of polymer into the mould. Once the mould is filled the pressure drop becomes negligible. The bottom curve shows the screw position.

The software calculates a viscosity value from the pressure data collected. The user interface panel, Figure 11, shows the pressure data on the right during the moulding cycle. The injection cycle is magnified on the left side. The bottom four graphs show the change in time of selected parameters (viscosity, pressure values, temperature etc.) that can be used for quality control.

The shear rate is set by the production parameters and therefore the viscosity can be measured only at that shear rate. A typical shear rate at the wall during the injection phase for these trials was  $4 \times 10^3 \text{ s}^{-1}$ . The viscosity results for a range of polypropylenes (PP) and for one high density polyethylene (HDPE) are shown in Figure 12. The viscosities were calculated without applying entrance or Rabinowitsch correction factors. As expected, because of shear thinning, the actual viscosities measured here are 1 to 2 orders of magnitude lower than the laboratory measurements or on-line measurements at lower shear rates. As Figure 13 shows, polypropylene is strongly shear thinning.

In Figure 12, it can be seen that the first batch of PP (N) shows a viscosity value of about 47 Pa.s. The effect of recycling the material on its processability was then investigated. The spoons produced in this trial were ground up and reintroduced into the injection moulding machine. A first regrind (rg1) of PP (N) shows more or less the same value of viscosity as the virgin material (N). The second regrind (rg2) shows a distinct difference in viscosity compared with the virgin (N) and first regrind (rg1): the decrease in viscosity with time for rg2 is indicative of flushing out of the previous material, as was evident from a change in appearance of the product on changing over from coloured (c2) to uncoloured (rg2) material. Finally, a sharp rise in viscosity can be observed as HDPE, used in the last run, flushes out the lower viscosity coloured PP batch (c3).

### 3.2.2 Bepak trials

The second trial discussed here was performed at Bepak, a large injection moulding company in King's Lynn. The injection moulding machine used was a Demag Ergotech 125-320 to which a MTN/IED100 external displacement transducer was fitted. The instrumented nozzle was manufactured at Bepak and designed at NPL. It had a minimum internal radius of 3 mm and the distance between transducers was 100 mm. The material used was ABS. The parts produced in this trial were intended to be sold.

Unfortunately, the pressure transducers failed during the trial and could not be replaced in the time scale available. However, one series of data was obtained prior to this failure. The results were shown during the trial on the front panel of the Dimlog program as in the BPTA trial. A small increase in screw velocity was observed during the start-up period which could be explained by the warming up of the injection moulding machine: the run was performed immediately after starting up the machine. The work has identified potential need for further investigation of characteristic features. For instance, the variations in pressure in the nozzle during the injection phase resulted in substantial variations in the viscosity. However, the magnitude of these fluctuations might be too large to be caused by pressure variations alone.

Details of the cycle times and pressures used commercially by Bepak are regarded as confidential and have not been identified in this report.

### 3.3 IN-LINE TRIALS DISCUSSION AND CONCLUSIONS

In these in-line trials the viscosity was determined at relatively high shear rates ranging from  $700 \text{ s}^{-1}$  to  $4000 \text{ s}^{-1}$ . In order to compare them with typical off-line laboratory experiments an extrapolation of off-line data is necessary. The quoted shear rates are averages from a narrow band of shear rates that are determined by the injection screw speed. An effect can be seen due to the acceleration and deceleration of the screw including when the injection is stepped in speed. The viscosity has been related to an average value of shear rate because of the delay in the pressure drop response to changes in the screw velocity. A change in screw speed and associated increase in pressure takes a finite time to impact on the pressure drop between transducers in the nozzle because the change is buffered by the compressibility of the polymer melt. Taking into consideration the rather small injection time of typically 1 s, this effect cannot be neglected. The time period of the relatively stable pressure drop between the pressure transducers used for the calculation is only a fraction of the total injection time. The whole process of calculating the viscosity from pressure data obtained from an injection moulding machine puts great demands on the acquisition software.

In the Bepak trial, a strong influence of pressure on viscosity was observed. The pressure increased during injection to 250 MPa leading to a significant increase in viscosity. This effect needs to be further investigated.

This in-line technique is a powerful way of measuring the viscosity under the conditions of the moulding process. This and the other in-line measurements form a powerful set of information gathering tools for 100% quality control, problem detection, optimisation and troubleshooting.

## 4. CONCLUSIONS

Collaboration between the National Physical Laboratory, Porpoise Viscometers Limited and compounders and convertors has demonstrated the value of in-process measurement techniques for extrusion, compounding and injection moulding. These techniques address some of the issues that render off-line methods unfavourable, thereby increasing industrial confidence in the use of such in-process measurement methods.

The application of in-process monitoring to plastics processing can offer considerable benefits in terms of improved product quality control and processing efficiency. The use of an instrument mounted on extruders and compounding extruders and an instrumented nozzle on injection moulding machines have demonstrated the benefits of:

- near immediate process and material consistency measures,
- data that can be used for process control and material quality control,
- significantly higher sampling rates than practicably possible by off-line methods,

- data obtained with relatively low levels of operator input and cost compared with off-line methods.

The collaboration has produced:

- a clear demonstration of the benefits of measuring the viscosity of the polymer during processing, such that Raychem achieved a payback on their investment in 3 months,
- a better understanding of the on-line measurement of rheological properties and their use for process monitoring,
- a demonstration of the potential of measuring viscosity in-line during injection moulding.

## **5. ACKNOWLEDGEMENTS**

This report was prepared as part of a project on the measurement of materials properties related to processing carried out in a programme of research financed by the Engineering Industries Directorate of the UK Department of Trade and Industry.

The NPL are pleased to acknowledge the substantial contributions made by BPTA, Bepak Europe Ltd, Polypipe Civils Limited, Porpoise Viscometer Limited, Raychem Limited, Reddiplex Group plc and UCB Films in carrying out this work.

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2. Rheology: Principles, Measurements, and Applications, C.W. Macosko, 1994, VCH Publishers (UK) Ltd, Cambridge, England.
3. Melt rheology and its role in plastics processing: theory and applications, J.M. Dealy and K.F. Wissburn, 1995, Chapman and Hall, England.
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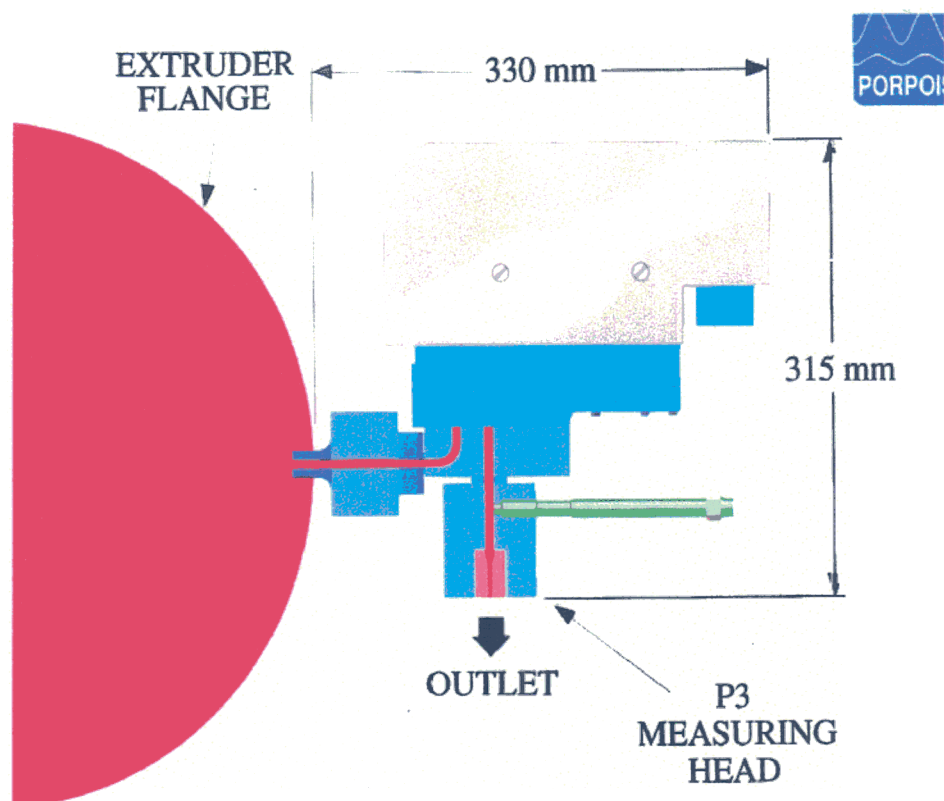


**Figure 1. Schematic diagram of the Porpoise P3 on-line rheometer.**

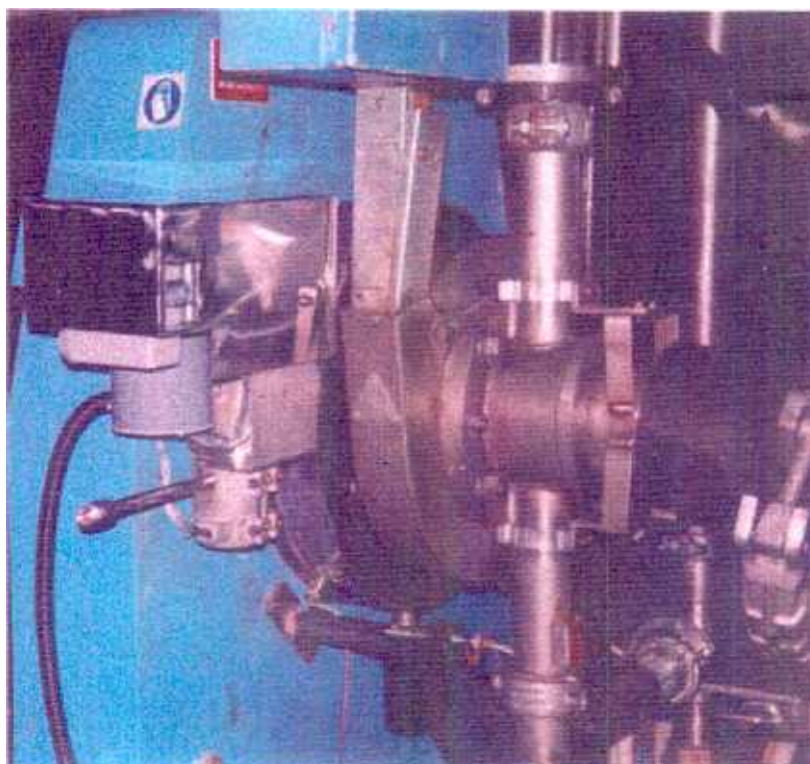


**Figure 2. Porpoise Viscometers Limited's on-line rheometer mounted on an extrusion compounder for trials during production at Raychem Limited.**

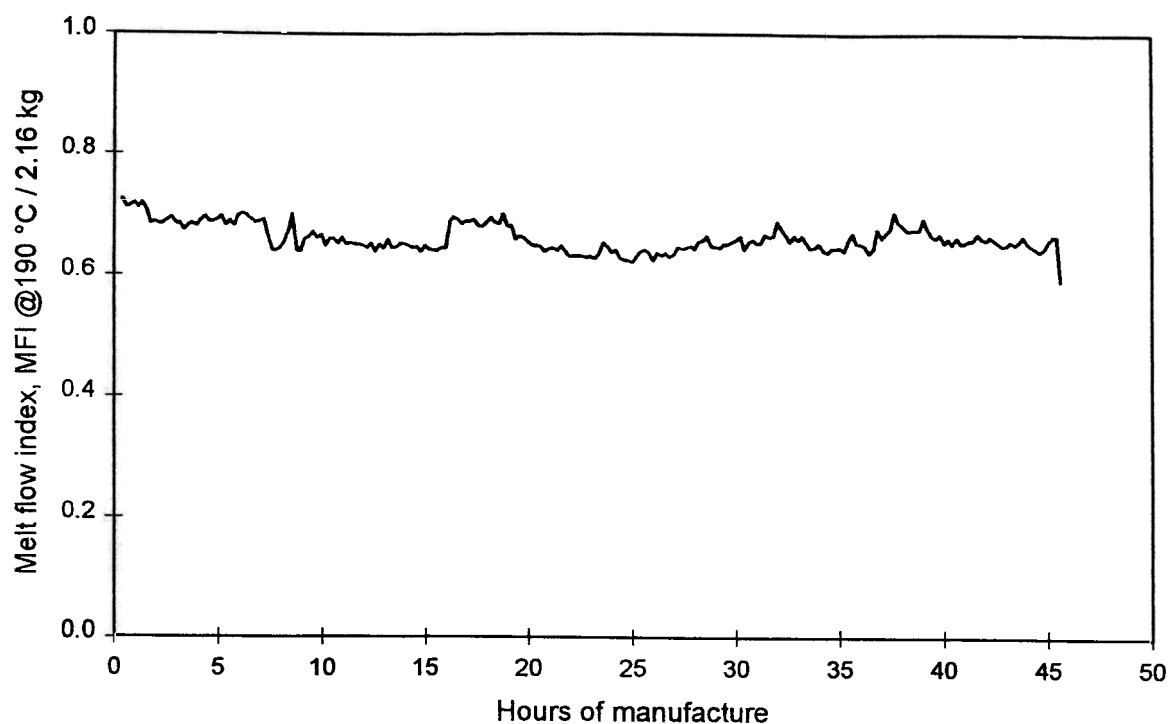


**Figure 3. Variation measured on-line of the MFI of a filled polyolefin over a 2 day manufacturing run at Raychem.**

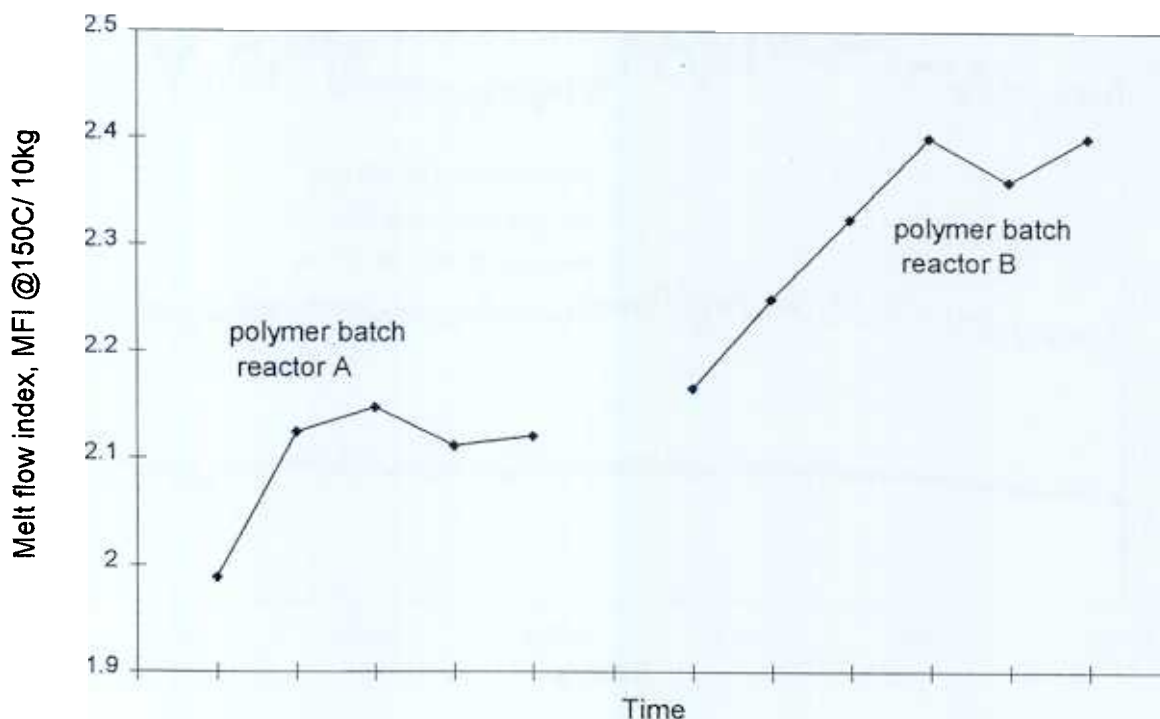


**Figure 4. Batch to batch variation of a filled polyolefin compound made on an Intermix extrusion compounder operated by Raychem (the polymer batches were supplied from different reactors).**

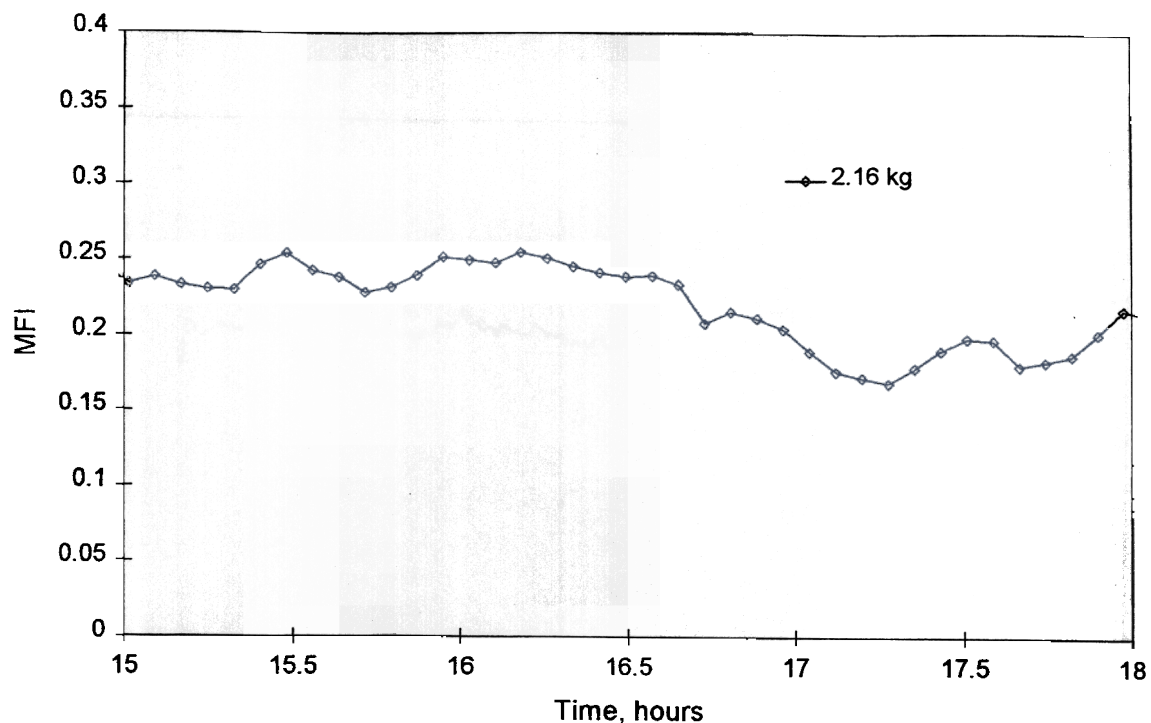


**Figure 5. Variation in MFI values over a change over period at approximately 16.75 hours at Polypipe Civils.**

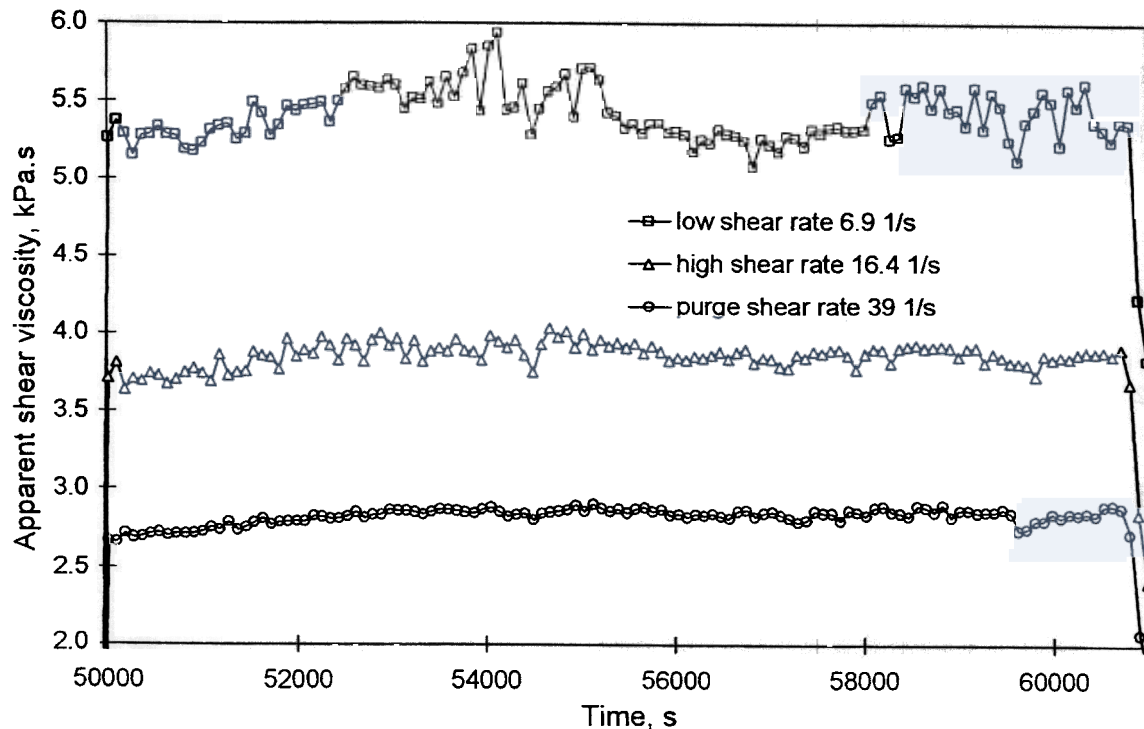


**Figure 6. Variation of shear viscosity with time for day 1 at three different shear rates (material ref.: HUE000 at 280 °C).**

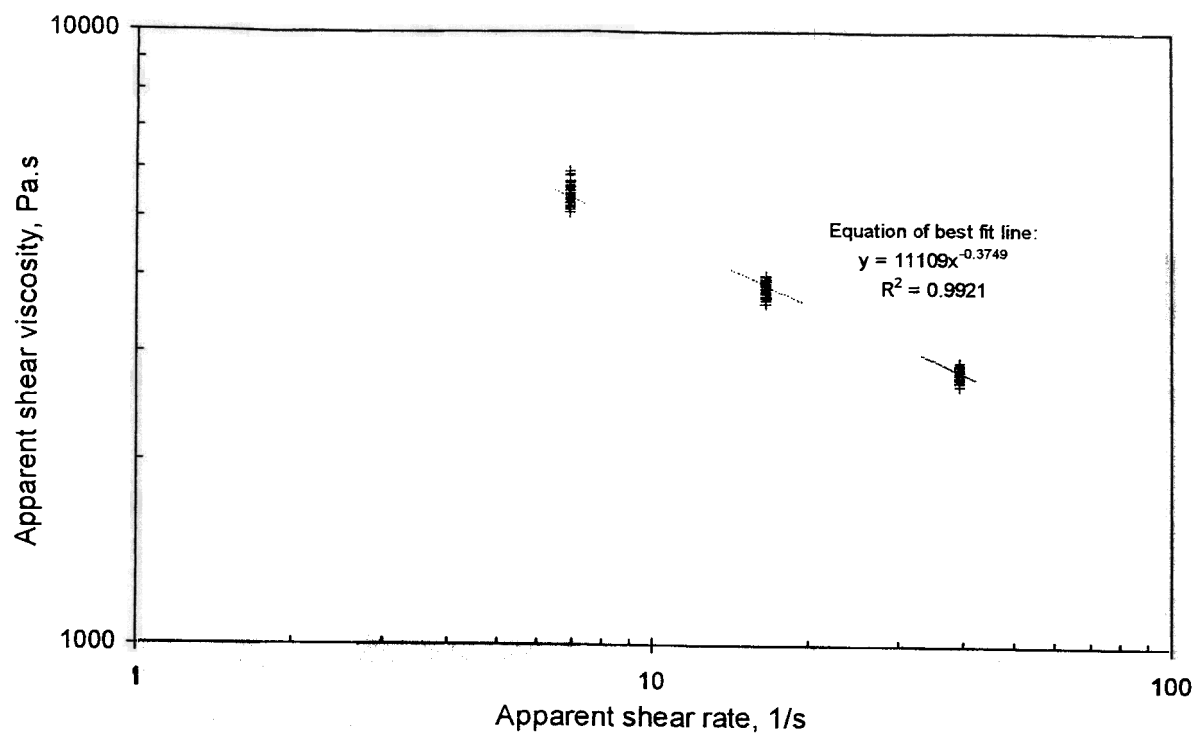


**Figure 7. Power-law fit to apparent shear viscosity data, day 1 (material ref.: HUE000 at 280 °C).**

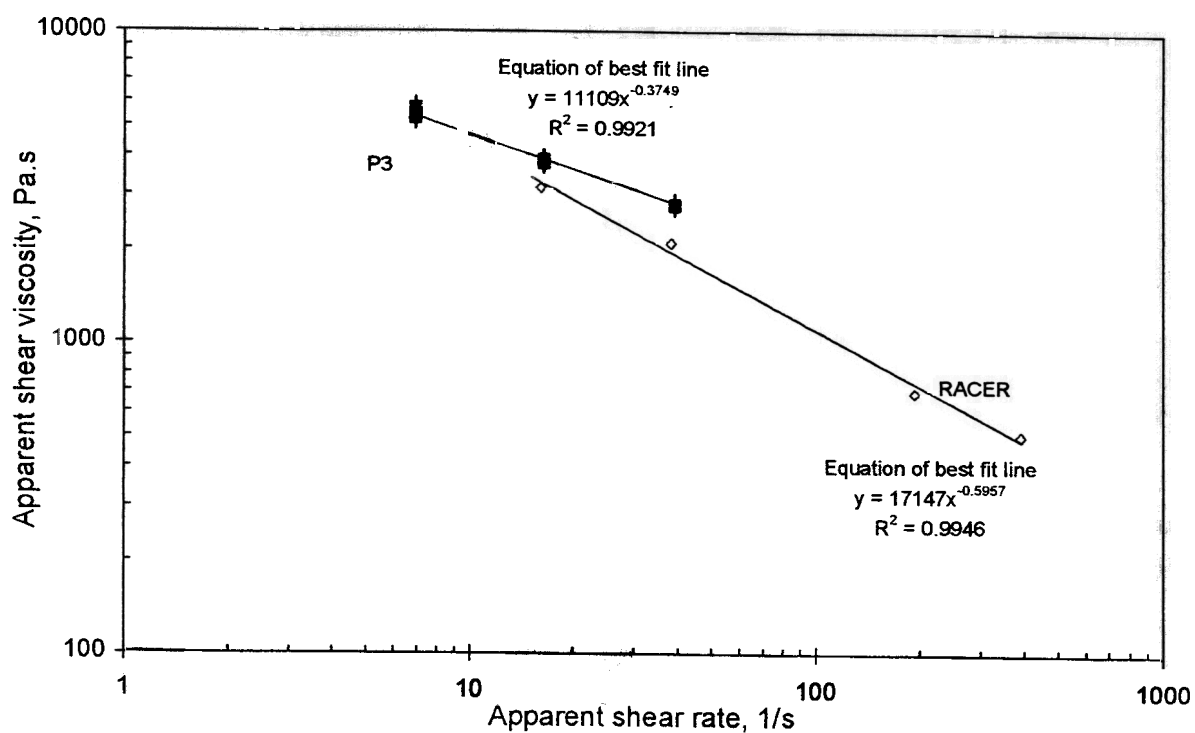


**Figure 8. Comparison of on-line P3 and off-line capillary extrusion rheometer measurements of shear viscosity (material ref.: HUE000 at 280 °C).**

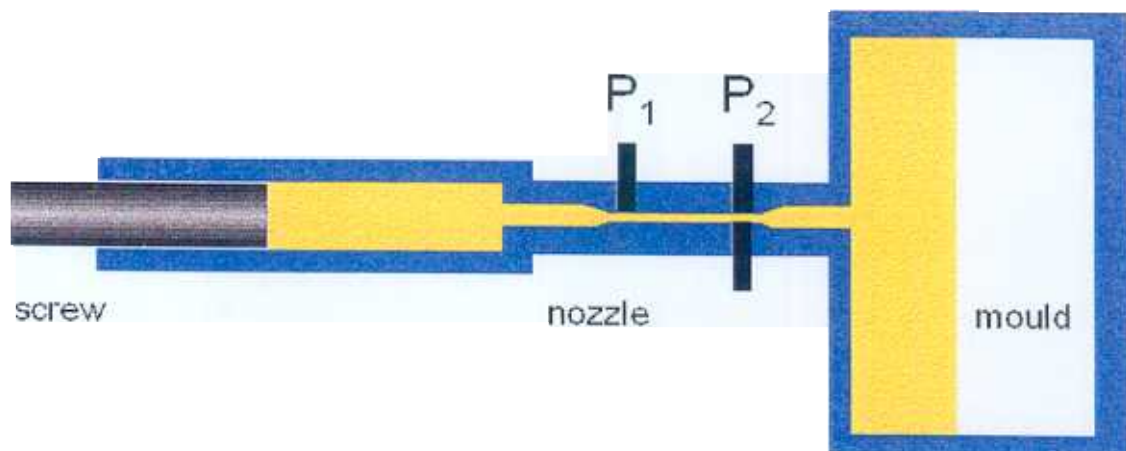


**Figure 9. Schematic diagram of the nozzle set-up and a picture of the nozzle fitted on an injection moulding machine.**

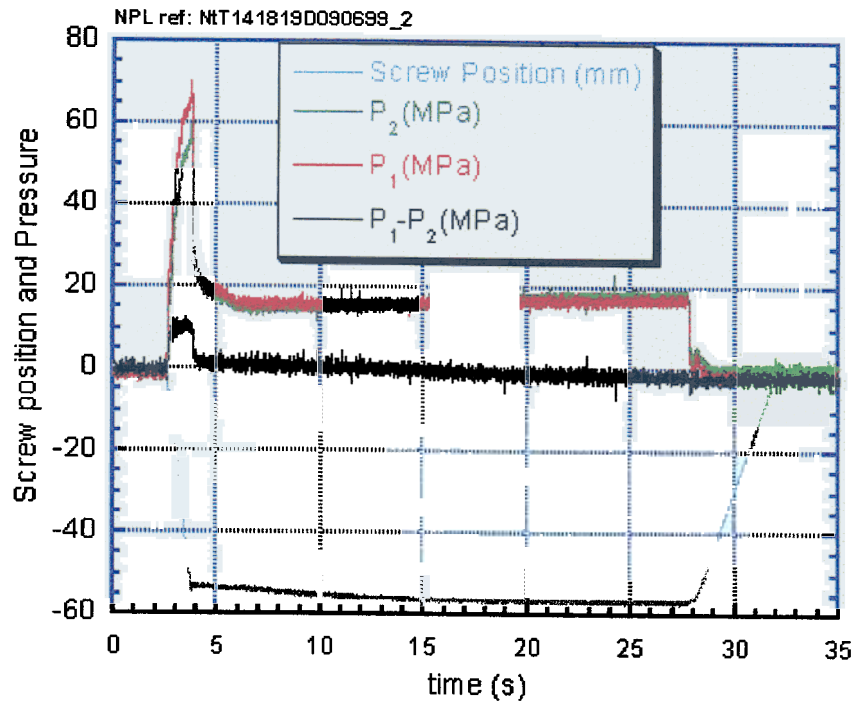


Figure 10. Typical pressure curves as seen at the BPTA trial.

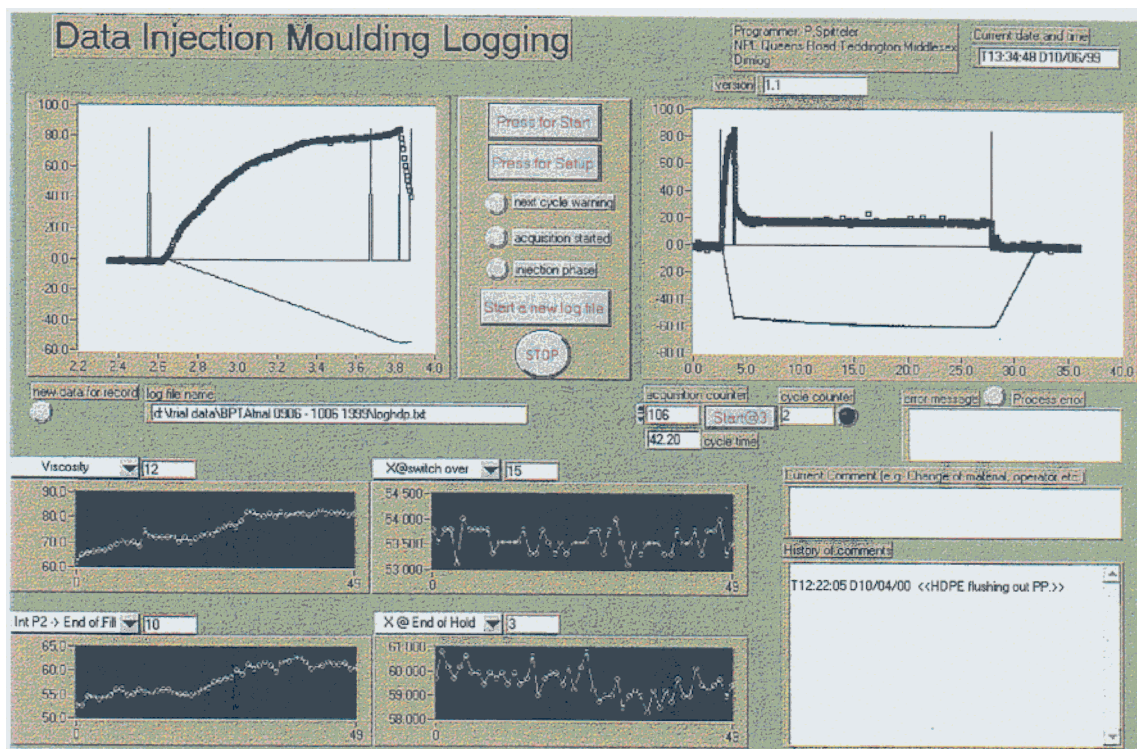


Figure 11. The user panel showing data produced during the BPTA trial. The top right shows the pressure and screw position. On the left the injection phase is highlighted. The bottom shows graphs of a user selection of in-line process measurement points.

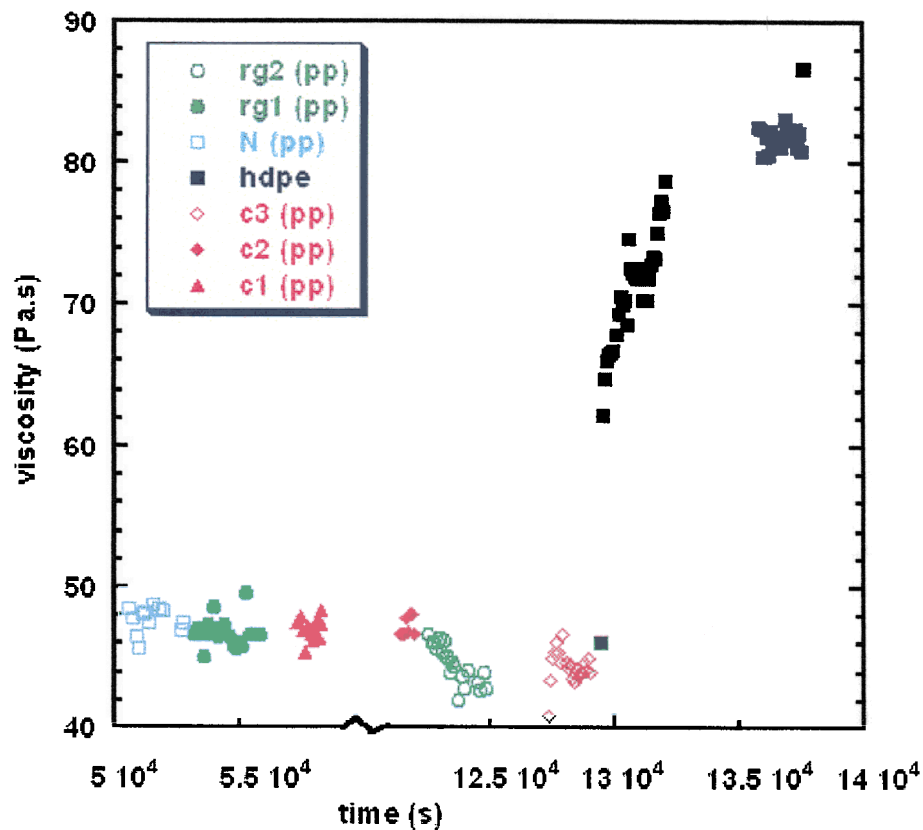


Figure 12. Results from trials performed on a range of polypropylenes (PP) and one high density polyethylene (HDPE).

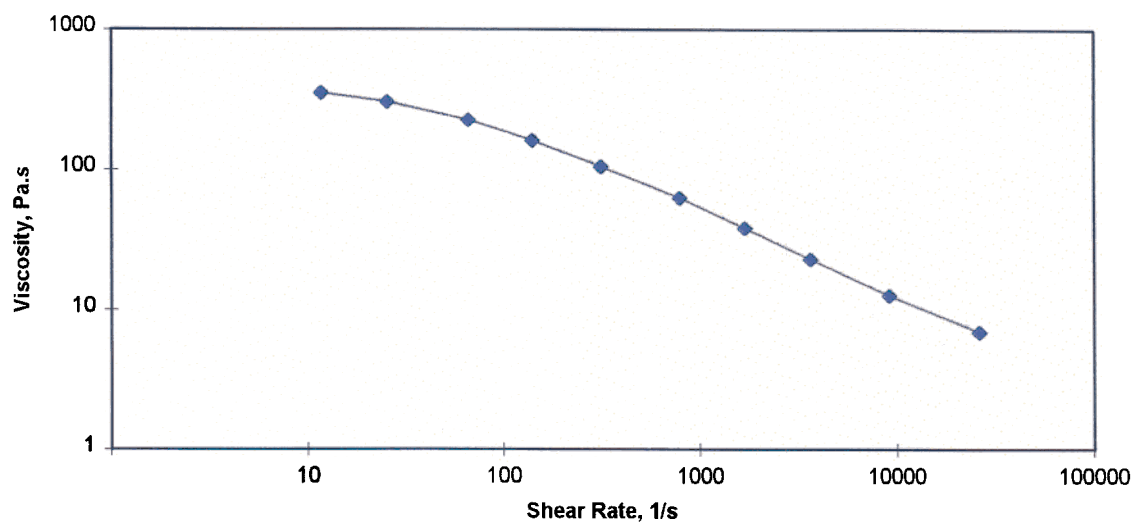


Figure 13. Typical graph of the viscosity of polypropylene as a function of shear rate (the grade of polypropylene shown here is different from the grade used in the trials).