

DETERMINING MELT FLOW INDEX ON-LINE USING A HELICAL BARREL RHEOMETER

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Abstract

A new approach and a technique has been proposed and developed to measure the melt flow index (MFI) using a Helical Barrel Rheometer (HBR), the portable on-line viscosity measuring instrument. The HBR can be fed on-line from a melt slip stream, or off-line with pellets. The viscosity is calculated from the pressure measurement and rotor speed, without measurement of the torque or the flow rate. The measured viscosities are representative of flows within the processing equipment, from which not only an MFI value can be calculated and monitored but also a spectrum of viscosity data over a wide range of shear rates can be obtained in a semi-continuous manner.

Introduction

Originated in the laboratories of Imperial Chemical Industries (ICI), the MFI is widely used and specified as a single point rheological quality control test in the ASTM, BS, DIN, ISO and JIS in plastics manufacturing for over 50 years. Many product specifications were written based on this simple test. The MFI is basically defined as the weight of the polymer extruded in 10 minutes through a capillary of specific diameter (2.0955 mm) and length (8.000 mm) by pressure applied through dead weight under prescribed temperature conditions. ASTM D1238 specifies the details of the test conditions for commonly used polymers. For example, the test conditions for polypropylene (PP) is are 230°C with a combined weight of 2.16kg on the 9.472 mm diameter piston. The amount of PP that flows in 10 minutes is defined as the MFI of that particular PP. ASTM D1238 gives the accuracy of the MFI value obtainable from a single measurement as carried out by different operators at different locations to be in the range of ±9% to ±15%, depending on the magnitude of the MFI. The possible error sources come from poor packing, dirty barrel, worn die and uneven test temperature and some other factors [1].

The extrusion rate in the MFI test is usually quite slow, much slower than normal extrusion. Due to its very short flow channel (the L/D ratio is around 4), the

entrance and exit pressure drops may considerably affect the measurements. Since the MFI represents only one single point of the viscosity curve, it does not reflect the real rheological properties of the test resins. Other limitations affecting its use as a process control device are the difficulty of automatic operation and the length of time required between samples. Recently, attention has been given to some other techniques, especially on-line measurement, which offers great time saving during the manufacturing. In addition, the viscosity data collected by such means may cover a wide shear rate range, especially in the high shear rate region [2-3].

In this work, a new technique has been developed to use a HBR as an on-line rheometer to generate an MFI value together with a spectrum of viscosity over a wide range of shear rates. The technique has particular value for monitoring the vis-breaking reactive extrusion process of polypropylene.

Experimental

Materials

Polypropylenes with different MFI values were provided by EXXONMOBIL. Table 1 lists their grades and the MFI values accordingly.

Viscosity measurement using HBR

The Helical Barrel Rheometer (HBR) was connected to a 32 mm diameter, 40 L/D long Killion segmented single screw extruder, as shown in figure 1. This rheometer is based on classical extruder screw equations for drag and pressure drop terms. However, the helical channel is in the barrel, rather than as part of the screw, so that pressure and temperature readings may be made uninfluenced by the passage of the screw flight past the sensors. At closed discharge, equating drag and pressure flows results in:

$$\Delta P = 6 \left(\frac{\pi D}{h} \right)^2 N \eta \quad (1)$$

where h = channel depth
D = rotor diameter

ΔP = pressure difference over one flight
 N = rotor speed

By rearrangement,

$$\eta = \frac{1}{6} \left[\frac{h}{\pi D} \right]^2 \left(\frac{\Delta P}{N} \right) \quad (2)$$

For the particular dimension of the HBR used in this work ($D=23\text{mm}$, $h=1\text{mm}$):

$$\eta = \frac{\Delta P}{31326N} \quad (3)$$

For a power law fluid (exponent n), the wall shear rate $\dot{\gamma}_w$ is increased by a factor of $(n+1)/2n$, analogous to the Rabinowitsch corrections for capillary and slit flow:

$$\dot{\gamma}_H = 4 \left(\frac{\pi D}{h} \right) N \left(\frac{n+1}{2n} \right) = 291N \left(\frac{n+1}{2n} \right) \quad (4)$$

Results and Discussion

This particular application of using the HBR to determine the MFI value on-line is for vis-breaking reactive extrusion process of polypropylene since it is faster and can be installed on line with the reactive extruder. The shear rates measured via HBR were matchable to the conditions in the running extruder and the data can be easily fed back directly to the control box through proper instrumentation design.

Figure 2 shows the shear viscosity of PPs versus shear rate at 230°C obtained with the on-line HBR rheometer. The shear rate ranges from 10 to 1000 1/s, which is typical of processing conditions within an extruder.

Several authors [1,5,6,7] have shown that a master curve can be generated especially for many individual types of polymers by plotting $\eta \times \text{MFI}$ versus $\dot{\gamma} / \text{MFI}$ on a log-log scale. They also introduced a modified WLF type equation to try to generate the similar master curve at different temperatures. The data of Figure 2 have been converted in this fashion and re-plotted as Figure 3.

Once the master curve M is generated by calibration of known MFI PPs, it can be used to convert HBR data into MFI data. First define the master curve in logarithm as:

$$Y_M = f(X_M) \quad (5)$$

Where $Y_M = \log(\eta \times \text{MFI})$

$$X_M = \log(\dot{\gamma} / \text{MFI})$$

The MFI of an unknown PP can be determined by calculating the shift in X and Y axes required to superimpose raw viscosity data onto the master curve, M.

From this master curve, it is useful to define the equation of a -45° line A going through the Y intercept at $X=1$, i.e., at $\dot{\gamma} / \text{MFI} = 10$:

$$Y_a = A - X_a \quad (6)$$

For data in figure 3, $A=3.86$, then Equation 6 becomes:

$$Y_a = 3.86 - X_a \quad (7)$$

To determine the MFI of an unknown PP sample B, the raw viscosity (as determined by the HBR) is plotted against shear rate as shown in Figure 4. These data could be represented by a best fit equation based on known flow models such as Power law, Carreau or Cross, or simply by a best fit polynomial equation determined in the data acquisition program:

$$Y_B = f(X_B) \quad (8)$$

Also superimposed on Figure 4 is the master curve M from Figure 3, and the equation for the -45° line A from the Equation 7.

The intersection of the -45° line A with the best fit line B through the data for the unknown PP sample B locates coordinates (X, Y) which are unique to the MFI of the sample. The coordinates could be read off of the graph, or more accurately calculated by the simultaneous algebraic solution of Equations 7 and 8.

From the algebraic solution, $Y_B = 2.84$ at $X_B = 2.02$ at the intersection of line B with line A. The MFI for the unknown sample B is readily determined by the transposition of the Y axis or X axis to obtain superposition of curve B with the master curve M:

$$\text{Log MFI} = Y_a - Y_B = 3.86 - 2.84 = 1.02$$

$$\text{Log MFI} = X_B - X_a = 2.02 - 1 = 1.02$$

From the above algorithm, the MFI of the unknown PP sample can be calculated, which is 10.43. Subsequently, it was revealed that sample B was EXXON PP 1024E4 with a nominal MFI of 12; and which we then confirmed as 10.5 using a regular melt flow indexer. The agreement between the HBR determined MFI and the conventional standard measured MFI is excellent.

Conclusions

It has been shown that the HBR can be used as a versatile on-line rheometer for measuring viscosity over a range of shear rates, and that the data so obtained can be used to determine a Melt Flow Index which is equal to the MFI measured via the standard ASTM procedures.

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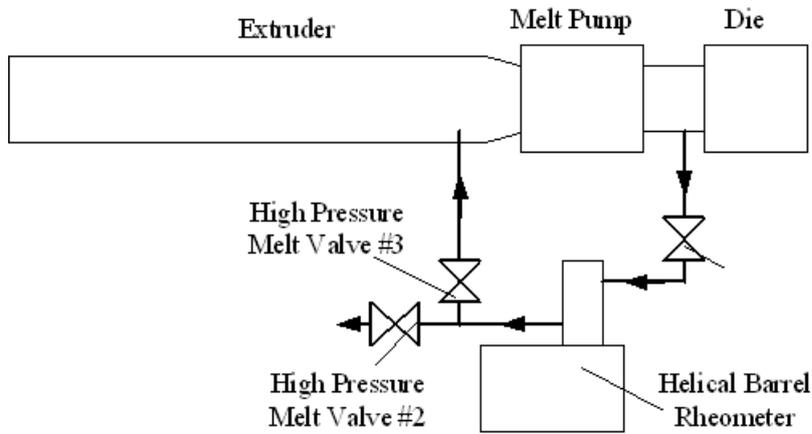


Figure 1. Schematic Drawing for connecting of HBR to Extrusion line

Table 1. Grade of PPs used in the experiment

Grade	PP5341	PP1042	PP1052	PP1044	PP1105E
MFI (g/10mins)	0.8	2	5	20	35
MFI _M (By Melt flow indexer) (g/10mins)	0.82	2.19	5.52	18.7	37.1

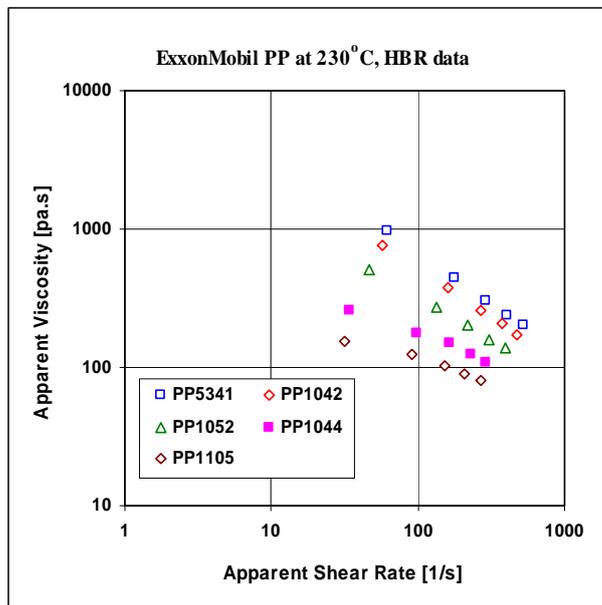


Figure 2. Viscosity curve of polypropylene at 230°C obtained through HBR

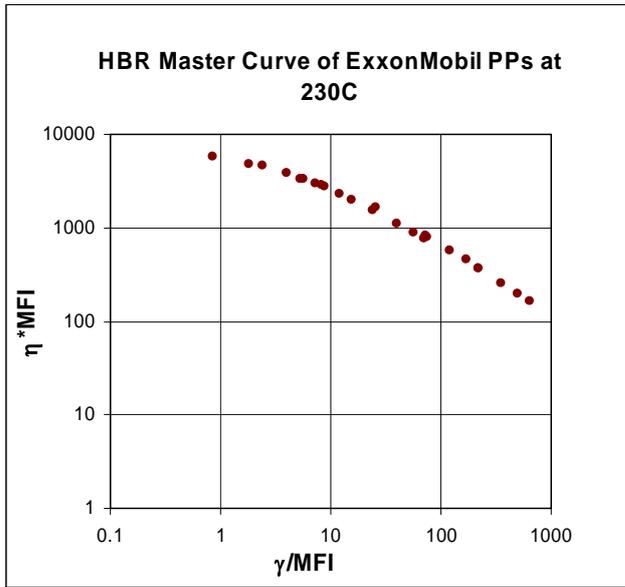


Figure 3. Master curve of polypropylene at 230°C obtained through HBR

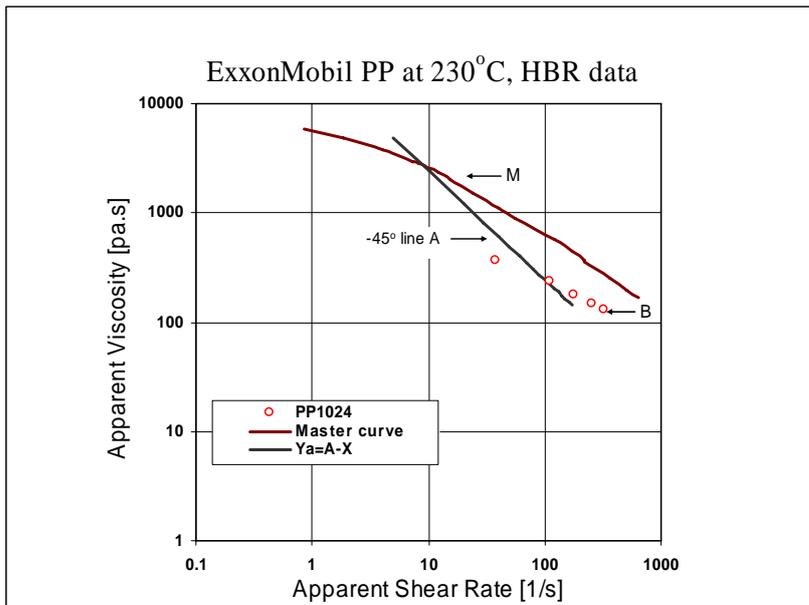


Figure 4. Using master curve and original HBR data to obtain MFI

Key words: Melt flow index, Polypropylene, Viscosity, Helical Barrel Rheometer