ChE 529 Polymer Experimental Methods University of Rhode Island
Professor Greenfield Fall 2008

Lab 5 part 3, Polymer Melt Rheology via Melt Index
November 3 and ?, 2008

Objective: The goal of this lab is to use an extrusion plastometer to measure the shear rate dependence of viscosity for melt polypropylene.

Safety: The main risk in this experiment comes from the high temperatures involved, i.e. 190°C. Do NOT touch the hot surfaces with your bare skin. **Remember that polymer extruded from the sample is initially this hot too!** Use heat-resistant gloves if necessary, and preferably use a tool to touch the hot parts of the instrument and sample instead. **Safety glasses are required** since there are high temperatures and pressures involved within the instrument.

Instrument Preservation: The inner cylinder of the instrument needs to be cleaned, but it also must remain scratch-free. Use the cleaner provided with the instrument to scrape polymer out of the cylinder. Clean out the capillary while the polymer is warm, so it doesn't plug up (but be careful not to hurt yourself on the warm cylinder). Solidified polymer may need to be scraped off the piston rod assembly and/or orifice. Use care.

Introduction: The objective of the experiment is to measure the relationship between shear stress and shear rate for polypropylene. Normally an **extrusion plastometer** instrument is used for measuring the *melt index*, which corresponds to the mass of polymer that flows in 10 min. Instead, we will use it as a surrogate for a capillary rheometer. The shear stress will be varied by altering the total mass used to force polymer through the (short) capillary, and the shear rate will be inferred via the total flow rate. The ratio between the capillary length (approx 8mm) and diameter (0.0825 in = 2.0955 mm) is smaller than is found in a typical capillary rheometer.

Procedure to perform a melt index experiment

The extrusion plastometer is on loan from the Industrial and Systems Engineering Department and is currently located in the unit operations lab Chemical Engineering, 103 Crawford Hall. A URI Standard Operating Procedure (or SOP) for how the extrusion plastometer is used has not yet been written (as of 11/2/08), since the instrument has not been operated since being donated to URI. A prototype for an SOP is provided by the instructions in the manual and by this handout, and these should be followed together. Terms in **boldface** are displayed on the picture of the instrument tools.

Prepare the instrument

1. Follow the prototype standard operating procedure to clean the cylinder barrel of any residual polymer from an earlier group. The **cleaning rod** is useful for this.
2. Ensure that the **orifice** capillary is clean as well. A bent paper clip of an appropriate size is useful for cleaning the capillary (and is available at the instrument).
3. Drop the orifice into the cylinder barrel. Ensure it goes all the way down. Practice removing the orifice while the instrument is cool, using the **removal tool**. With the orifice in place, add the piston rod assembly.
4. Preweigh some weighing papers, so they are calibrated for use in determining the mass of polymer that flows.

5. Follow the prototype standard operating procedure to turn on the instrument and to begin a manual program test (i.e. type "A"). Choose a temperature of 190°C. (Note that this is Celsius, not Fahrenheit. We are high above the boiling point of water.)

Load the instrument

6. After the instrument comes up to temperature, use the non-plastic funnel to load the instrument with polypropylene beads. Try not to load pink colorant pellets. Use the **loading tool** to pack in the beads. Load plenty of sample.

7. Once enough sample is loaded, place the **piston rod assembly** back into the top of the cylinder. Press *start* on the keypad.

Conduct the measurement

8. After the release time (i.e. warm up or waiting time) elapses, use the **cutoff tool** to remove any sample that extruded out already, and then simultaneously place the weight(s) on the piston rod assembly and press the start button.

9. After the cutoff time (i.e. flowing time) has elapsed, cut off the sample using the **cutoff tool** and press start to restart the timer. Add a weight when you press start if it is appropriate (see step 11).

10. Set the cutoff sample aside so it can cool. After it cools, measure its mass. *Record the mass and the amount of time it took to extrude the sample out of the plastometer.*

11. Continue as necessary for the different weights. If you run out of sample (only 6.5 cm between the weight and the heater assembly), then you will need to load more sample into the instrument.

Experiment 4: Polypropylene melt viscosity

(Experiment counting continues from the cone-and-plate viscometry labs.)

The objective of the experiment is to measure the relationship between shear stress and shear rate for melt polypropylene. Both shear stress and shear rate will be varied by changing the total weight used to force the polymer through the orifice. The weight with a relatively small slit will sit atop the piston rod assembly. Other weights can be placed on top of it. Each group will choose which ranges of weights they use. Enough weights should be chosen to obtain a range of shear rates and shear stresses. Measurements should be repeated with the same weights, especially initially, to obtain a measure of the reproducibility.