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

Lab 5, Cone and Plate Viscometry
October 20/21 and 27/28, 2008

Objective: The goal of this lab is to use a viscometer to measure the shear rate and temperature dependent viscosity of several samples. Week 1 will focus on standards and well-defined samples and will use the controls available directly on the viscometer. Week 2 will focus on temperature dependence and will use the computer controls to access a more broad array of flow conditions.

Safety: The main risk in this experiment comes from the direct exposure between the sample and the environment. (The instrument uses approximately 0.7 mL of sample during the measurement.) We will mitigate that risk by using samples that are safe. Do wash your hands afterwards (but being careful about the water, apparently) in order to protect yourself from exposure to the samples. In some experiments, the sample temperature could be raised. In those cases it is important to be careful of hot surfaces.

Instrument Preservation: The viscometer is described by the manufacturer as fairly robust. However, don't push parts together too hard against each other. If something isn't moving, don't force it. If you feel a large amount of resistance, then you may be putting something together wrong.

Procedure to perform Viscometery

The viscometer is located in the unit operations lab Chemical Engineering, 103 Crawford Hall. A URI Standard Operating Procedure (or SOP) for how the viscometer is used has not yet been written (as of 10/20/08), since the instrument is brand new. A prototype for an SOP is provided by the instructions in the manual (see Chapter 3), and these should be followed.

Prepare the instrument

1. Follow the prototype standard operating procedure to install the cone into the instrument. Adjust the gap between the cone and plate to 0.15 mm.

Prepare the sample

2. Loosen the viscometer clamp, raise the viscometer, and place about 0.7 mL of the sample on the plate.
3. Lower the viscometer and cone back down into the sample. Trim the sample so it resembles the picture(s) in the manual. Practice lowering the viscometer a few times so you get a good idea about how to return to the correct gap.
4. Tighten the viscometer clamp.
5. Use the speed and start buttons to get the viscometer going.
6. Record the steady state torque, shear stress, shear rate, and viscosity.

Experiment 1: Water

Obtain some distilled water. Measure its viscosity at all 8 speeds.

Experiment 2: Standard polydimethylsiloxane #1

Obtain the viscosity of a standard polydimethylsiloxane at all 8 speeds, at room temperature. Note if shear rate has an effect on viscosity. Also note any observations you make about the sample and the experiment.

Experiment 3: Temperature and shear rate dependence of honey

Set the gap (if necessary). Pour approximately the right amount of honey onto the sample holder, and use a toothpick to trim the sample size to the proper amount.

First run a viscosity experiment at room temperature and the slowest speed. Note the temperature from the thermocouple display. Then use the software to run viscosity experiments (again at room temperature) across a range of shear rates from 10 to 1000 s^{-1}. (Check with Prof. Greenfield that you are choosing enough shear rates.) How does the result for the viscosity at the lowest shear rate compare when performed without and with the software?

Next use the temperature controller (water bath) to raise the temperature. The minimum temperature that can be controlled without cooling is 35°C, so start there. Run the same range of shear rates that you used at room temperature.

Continue the experiment at a number of higher temperatures. Viscosity can change quite a lot with a small change in temperature, so don't change it by much! Be sure to provide enough time for the bath and plate temperatures to equilibrate. Record an estimate of the plate temperature using the water bath thermometer.

The objective will be to interpret your results using time temperature superposition, so having some overlaps in viscosity between temperatures is very useful. Sampling long enough to obtain good data is also useful.

Experiment 4: see Prof. Greenfield

Software procedure

Start the "Bohlin" software after the viscometer is turned on. Log in as username "che" and password "polymer". Several tests have been defined already. You can run one of these tests, or you can edit the test to change the shear rates, sampling times, etc.

You will be encouraged to modify the experiment protocols.