“YIELD STRESS” AS IMPORTANT AS “VISCOSITY”?

viscosity relates to yield stress

“Yield Stress” could be a term that applies to our daily lives. When under pressure, if the stress is too great, you start to “change” in a figurative sense. In the world of science and engineering, fluids and semi-solid materials that are subjected to a strong-enough force will start to move. Basically they “change” from their original shape and move in response to the applied force, remaining in motion as long as the force is great enough.

Materials in motion can be measured for resistance to flow. “Viscosity” is the parameter of interest. Quality Control departments throughout the pharmaceutical industry use viscometers to evaluate test samples and verify that the measured viscosity falls between established limits. This measured value can actually relate to how the material is processed in manufacturing or to what the end user may sense when the material is used.

The pertinent question is how “Viscosity” relates to “Yield Stress”? Should R&D, Manufacturing, and Quality Control all be concerned about Yield Stress? R&D’s job is to characterize the flow properties of new formulations. This includes measuring physical properties like viscosity and yield stress. QC test methods handed down from R&D most often include a viscosity test, but seldom one for yield stress. This is not so much an oversight as a pragmatic decision to limit the amount of work required by QC to certify product acceptability.

Note that yield stress relates to how consumers evaluate creams and ointments when they squeeze a tube to discharge the product. The squeezing force that initiates flow is the yield stress value of the ointment. A liquid, which has ingredients suspended within, fails if the suspension settles out prematurely. A fluid’s ability to hold the particles in suspension is characterized by its “yield stress”. The start-up torque required to pump a material is directly proportional to the yield stress of the material being processed.

There are many more examples that emphasize the importance of knowing the yield stress of pharmaceutical liquids and semi-solid materials, so is it appropriate to reconsider measurement of this parameter in QC? Significant changes in the world of test instrumentation may give impetus for making a change. Select bench top instruments that measure viscosity can also measure yield stress.

The controlled stress/rate rheometer in Figure 1 provides the most effective capability because the required sample size is small, temperature equilibrium is quickly achieved, and the test algorithms can execute quickly and automatically. In the first example “Controlled Stress” is a method of operation that applies increasing torque to the spindle until

Figure 1: Example of Controlled Stress Rheometer with Cone/Plate Spindle Geometry.

rotational movement is detected. The onset of rotation equates with the yield stress of the test sample. As shown in Figure 2, the rubbing ointment has a yield stress slightly below 1000 Pascals when spindle movement is first detected. Note that the viscosity then decreases dramatically with small subsequent increase in shear rate.

In the second algorithm using "Controlled Rate" method, viscosity is measured by rotating the spindle at different rotational speeds to establish the "flow curve" for the material. As shown in Figure 3, the viscosity of cough syrup reduces from above 1000cP to less than 10cP at a shear rate of 100 reciprocal seconds. This shear rate is typical of swallowing action. The viscosity flow curve gives assurance that this formulation of cough syrup will perform acceptably when taken by an adult.

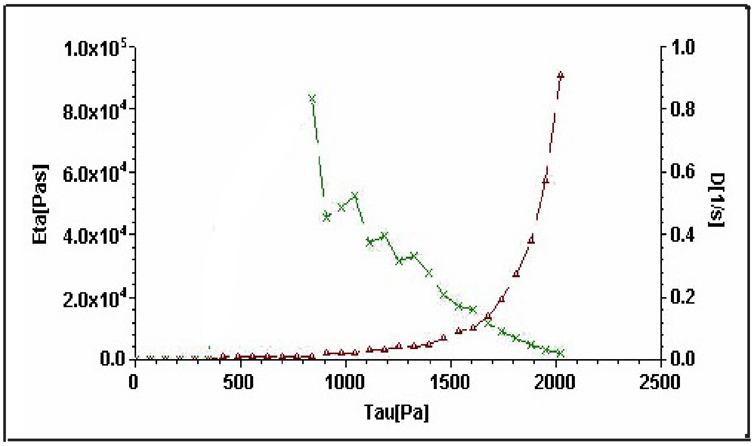

Figure 2: Yield Stress Curve Using Controlled Stress Test Method

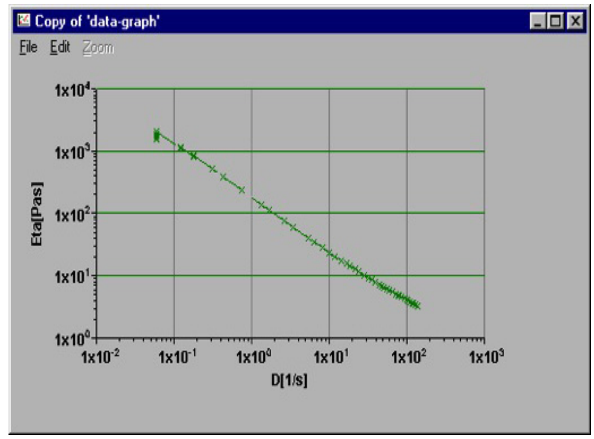

Figure 3: Viscosity Flow Curve Using Controlled Rate Test Method

Automated operation of the rheometer in both of the above examples allows the test to complete without the need for a technician to attend the instrument. Although the time required for each of these tests may be relatively short, longer term tests can be set up just as easily and run without operator involvement.

What now makes the difference in today's world of laboratory instrumentation is the intelligence that is built into stand alone devices like the controlled stress/rate rheometer. There is no requirement for a PC to run these types of tests. R&D may choose to use the PC when performing characterization work because they want to save files and conduct comparative analysis on multiple data sets. Once the test methodology is established, the algorithm for running the QC test is stored in instrument memory and stand alone operation commences.

Data values saved from each test can include yield stress and the "flow index". The latter is the best-fit line through the viscosity data obtained by rotating the spindle at multiple speeds and plotting the values on log-log scale. The flow index characterizes the "flowability" of the material and illustrates how the material will move in response to different shearing actions. The "flow index" more completely defines flow behavior compared to a single viscosity value measured at a specific speed.

In summary, the significant win-win for pharmaceutical manufacturers is that QC now has an automated tool, the controlled stress/rate rheometer, that provides more complete rheological data, yield stress and flow index, for assuring product acceptability.

Author: Robert G. McGregor - General Manager
Global Marketing and High-End Lab Instrument Sales
Brookfield Engineering Laboratories, Inc., 11 Commerce Blvd., Middleboro, MA 02346
Tel: (508) 946-6200 ext. 7143 Fax: (508) 946-6262
Email: r_mcgregor@brookfieldengineering.com
Website: http://www.brookfieldengineering.com
VertMarkets - Pharmaceutical Online - Quarterly Column - Reprint Permission - November 2013