Are Yield Stress and Creep Similar?

rheological terms

Yield Stress is a rheological term that defines how much force is required to cause a liquid or semi-solid material to begin moving. Thick cough syrups, for example, pour slowly out of the bottle; gravity alone appears to provide sufficient force to discharge the fluid. The customer patiently waits until a sufficient quantity comes out and fills the spoon or cup with the recommended dose. Consumers evaluate creams and ointments in part by the ease or difficulty of squeezing a tube to get the product out. Marketing gives input to R&D for the yield stress performance parameter by subjectively describing how the product should flow when used by the customer.

Creep is the steady deformation of a material that is placed under a constant load. When gel caps crack and start to ooze the liquid filler, the force that pushes the material out the crack must exceed the yield stress of the liquid. Gravity alone may be sufficient to cause the seepage. For thicker formulations, there may be other forces in play that assist gravity. Packages of gel caps stacked on top of each other can sometimes experience this problem. The overall weight of the stacked packages can cause those on the bottom to slowly lose their contents over time as product oozes out. This is an example of creep behavior; the consequence can result in partial or total loss of the shipment, depending on the damage caused by the escaping liquid.

Creep is related to yield stress in the sense that the material starts to flow because the yield stress has been exceeded. The onset of creep behavior occurs only when the yield stress threshold has been crossed. So in this sense, the two parameters are clearly related to one another.

How do pharmaceutical manufacturers anticipate the flow problems described above? The science of rheology clearly defines these two parameters and provides R&D with simple tests that can predict flow behaviors related to the above issues. Controlled stress rheometers with cone/plate geometry have the flexibility to measure a broad range of flow parameters, ranging from yield stress to viscosity to creep. They can be used on both easy flowing liquids as well as sticky paste-like ointments, so the advantage of this type instrument is the variety of product types that can be measured and characterized.

Figure 1: Brookfield RST Cone/Plate Rheometer

Figure 1 shows the cone and plate rheometer, which is the preferred tool for most R&D Departments in the pharmaceutical world. The test sample is placed on the temperature controlled plate. The spindle's bottom surface, which is angled slightly relative to horizontal, is brought down on top of the sample, which distributes itself to fill the empty space, also called "gap", between cone spindle and plate. A controlled torque ramp is gradually applied to the spindle, but movement does not take place as long as the yield stress in the sample exceeds the force that is applied. As torque increases, the spindle gradually begins to rotate because the yield stress is overcome. The approximate torque value where rotation commences is called the "yield stress" value for the material.

The type of graph that shows yield stress behavior is shown in Figure 2 for a couple of different materials. Shear rate appears on the y-axis and shear stress is on the x-axis. The curve for each test hugs the x-axis until the applied stress value becomes high enough to allow the spindle to start rotating. In the case of one product, the shear rate gradually increases after yielding, while for the other it increases very rapidly with almost no further increase in the shear stress that is applied.

Having established the yield stress value for each material, it is now possible to run a creep test. Using a constant stress value equal to or slightly higher than the yield stress value, the same materials can be tested for flow behavior in creep mode. Figure 3 shows the type of graph that characterizes this behavior. The y-axis records the amount of spindle rotation while the x-axis is a measure of elapsed time. There is a noticeable difference between the two materials and the one with higher "Deflection Angle" will continue to flow more easily. When the constant stress is removed from the spindle, both materials will "recover" or limit their movement, perhaps even to no movement at all. The graph shows that there is a drop in the "Deflection Angle" value for both.

Quality Control has responsibility for ensuring satisfactory product from batch to batch. R&D recommends the tests that QC performs. The advantage of controlled stress rheometers is that they can easily perform the standard viscosity test, which is a normal part of the pass/fail checks performed on pharmaceutical products. As industry moves to include these additional tests for yield stress and creep, the same instrument can handle the workload. Advancements in instrument design enables controlled stress rheometers to operate in standalone mode on the production floor without need for PC to control and collect data. Test cycle times using these new rheometers are also much quicker than standard bench top viscometers, perhaps by as much as a factor of two. With increasing workloads in labs, finding instruments that can do more automatically is becoming a mantra.

Bottom line is that yield stress and creep are becoming part of the QC list of parameters that must be measured to better qualify production batches. QC has limited time to perform the battery of tests on their plate. Investment in the new generation of controlled stress rheometers offers a solution that deserves investigation. Perhaps it's time to start the process!

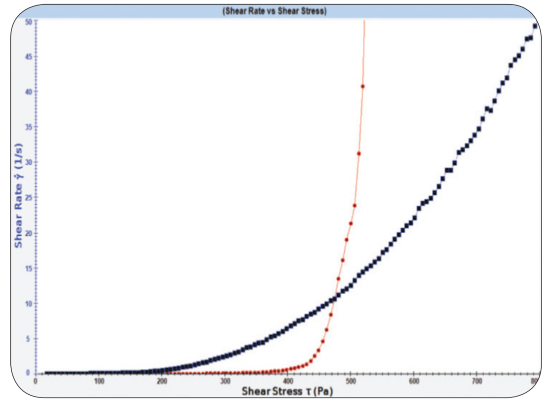

Figure 2: Yield Stress Graph for Two Different Materials

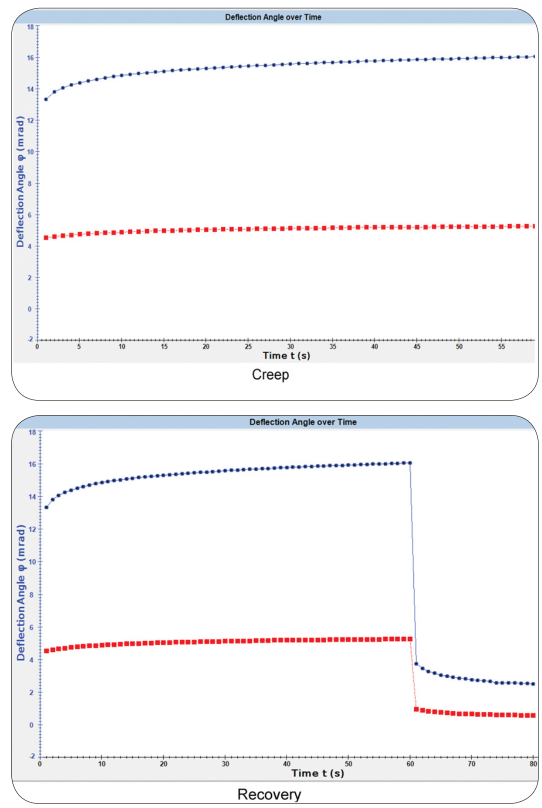

Figure 3: Creep and Recovery Graph for Two Different Materials

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