HOW CAN MANUFACTURING GET MORE OUT OF RHEOLOGY?

by Robert G. McGregor

Rheology is the science that investigates the flow behavior of materials. Whether the product is a liquid, cream, paste, or powder, manufacturing transfers raw ingredients through multiple processing stages to create the finished product. The ability to pump, mix, transfer, store and fill the product into shipping containers successfully requires proper rheological analysis by R&D to ensure both flowability and stability at every step.

Figure 1: Brookfield RST Controlled Stress Cone/Plate Rheometer

In the case of liquid and semi-solid products, rheometers are the scientific tool used to evaluate new formulations by R&D. On the production floor and in the QC Lab, viscometers make confirming measurements that the product meets spec. What's the difference between rheometers and viscometers? Can the latter do the job properly so that manufacturing doesn't experience flow behavior problems or instability in the final product? Viscometers can very likely do the job without question and perhaps have capability to do much more if needed.

An example of a Cone/Plate Rheometer is shown in Figure 1. It has the capability to shear the material over a continuously broad range of shear stresses and/or shear rates. Because it can handle many types of materials, ranging from thin liquids (eye solutions) to semi-solids (rubbing ointments), it is a more expensive investment. However, a rheometer can perform a much more comprehensive battery of tests, starting with visco-elastic deformation and yield stress measurement when the material is at rest, to a full viscosity flow curve with thixotropy calculation (time sensitivity to being sheared) when the material is in motion, to recovery and creep (low force flow behavior), and finally temperature profiling (how the viscosity property changes with temperature). Consequently, the versatility of this instrument makes it a must have for R&D.

Fortunately, these rheometers have become less expensive in recent years and the ease of use in standalone mode or under PC control make them potential candidates for both QC and manufacturing. The critical improvement is that they are durable in busy environments and can actually survive on the production floor with multiple operators using the same instrument. Several major companies have moved in this direction because they see value in following the same path as R&D. Does this rule out standard bench top viscometers? No, it does not by any stretch of the imagination.

Figure 2: Brookfield DV2T Controlled Rate Viscometer with Small Sample Adapter

A popular and readily affordable viscometer shown in Figure 2 runs only in controlled rate mode (you define the rotational speed of the spindle and the instrument measures the material viscosity). This type of measurement is called a single point viscosity test. It really gives relatively little information about the overall flow behavior of the sample in test, but R&D has probably done sufficient characterization work to know that this one data point will detect most problem batches of material. So a lot is left on the table in terms of additional information that this type of instrument could provide, such as a viscosity flow curve (how viscosity changes with different shear rates), time sensitivity to shearing (thixotropy), or temperature profiling (how the viscosity is affected in different climates).

Whose decision is it to settle in on the instrument of choice? R&D knows that Manufacturing and QC want simple to use, quick to run the test, rapid clean up and affordable. The truth is that either today's rheometer or viscometer can do the job. So it's really a matter of preference and budget. One consideration of growing importance is durability. The more solidly constructed instrument requires less service, less frequent calibration checks, and delivers longer up time.

The type of material in manufacture can play a role in deciding between rheometer and viscometer. Viscous materials, such as thick ointments, are good candidates for testing by a controlled stress rheometer. The controlled stress mode can make a direct measurement of yield stress, the force required to start the material moving.

(See Figure 3) This measurement can be equated with the startup torque in a motor that drives the mixing pump on the production floor or the squeezing force applied by the end user to get the product out of the tube. So for more viscous products, the rheometer may offer an advantage in terms of additional measurements that could prove useful.

Controlled rate measurements generate "Viscosity Flow Curve" information which characterizes the resistance of the material to flow. (See Figure 4) For most liquids and semi-solids, viscosity decreases as shear rate increases. The objective is to determine how significant this resistance is in terms of the "rate of change" in flow. ("Pseudoplastic" is a term used to describe this type of flow behavior.) If this is the property of greatest interest, then the viscometer can do the job. However, the mechanics of instrument operation lead to the discovery that the rheometer can complete the test in much faster time.

As mentioned earlier in this article, one value on the Viscosity Flow Curve is typically selected as the control point for use by QC in making measurements on production batches of material. Viscometers are used for this single purpose and may therefore earn the incorrect perception that they cannot do more. In general, viscometers are multi-speed instruments which can easily measure many points on the Flow Curve in Figure 4. So the issue is whether to take advantage of this capability and select at least one additional reference point for good measure. For pharmaceutical products that are highly shear thinning, like medical ointments, it might be worth considering. With proper forethought in selecting data points at two separate shear rates, this approach will confirm that the flow behavior is acceptable to both manufacturing as well as to the end user.

There is a well established method for investigating shear thinning behavior of materials using a viscometer. Select two speeds that are a decade apart, perhaps 1 and 10 rpm or 10 and 100 rpm, depending on material thickness. Compare the viscosity values recorded at each speed by computing the ratio of the former to the latter. Since the viscosity measured at the lower speed will almost always be a higher value than the second viscosity, this ratio generates a number greater than 1.0. This ratio is called the "Thix Index" or "TI".

For off-the-shelf medicinal products, like cough syrups, that exhibit a slight change in viscosity behavior, the TI will probably be close to 1. For highly shear thinning materials, like rubbing ointments, the TI could be at least 2, and perhaps as high as 3 or 4. So the TI becomes a very useful tool for improved qualification and certification of pharmaceutical products.

Manufacturing has good reason to consider the added value that comprehensive viscosity data can bring to operations. There are a number of different shear rates that take place during processing in manufacturing. Knowing that the viscosity data fits within established windows for acceptable flow behavior guarantees that process operations will proceed without issue. What may have seemed like an esoteric subject – "Rheology" – has everything to do with making Manufacturing successful.

One final consideration is the use of online process viscometers in manufacturing. Once viscosity data is available for the changes in material behavior throughout the manufacturing process, the possibility of selecting a control point in the process to monitor and control viscosity becomes possible. Although process viscometers have been available for many years, manufacturing operations are not generally aware of the potential they offer. Consistency of product is the objective. Minimizing waste or product that needs to be reworked is the corollary. This is where the viscometer manufacturer can provide assistance in identifying the appropriate location for the installation of the online instrument.

In summary, manufacturing has something to gain with a deeper involvement in the use of Rheology to improve operations. R&D and QC can be helpful partners in making this happen. The bottom line is to produce consistent product while improving processability and meeting customer expectations at the same time.

Figure 3: Controlled Stress Test Provides
Yield Stress Value for Start of Flow

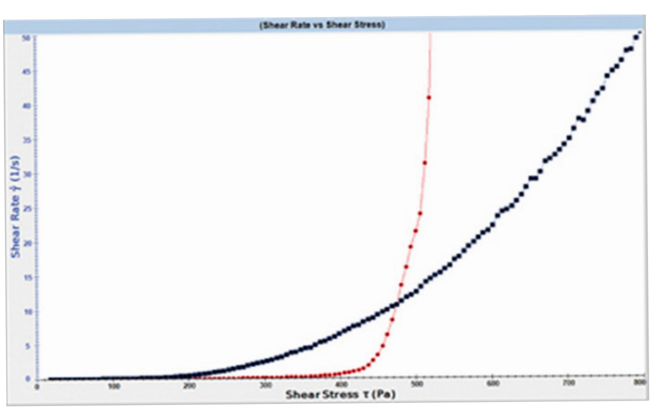

Figure 4: Flow Curve Shows Viscosity vs.
Shear Rate Behavior