A quick tutorial on the flow behavior of spray coatings for pharmaceuticals

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Coatings are applied to many of the products we use every day. The function of coatings on cars and appliances is appearance and protection. The coating has no function regarding the performance of the underlying object. On consumable products, the function of the coating can be much more complicated and may include enhancement of flavor, aroma, appearance, and even the longevity of the product. Increasing longevity may be desirable for reasons as simple as long-lasting taste, or as critical as timed-release performance for a medication. A basic requirement, for example, is to keep a medicinal tablet intact until it can be completely swallowed. With such important functions to accomplish, pharmaceutical coatings need to be accurately formulated and precisely applied.

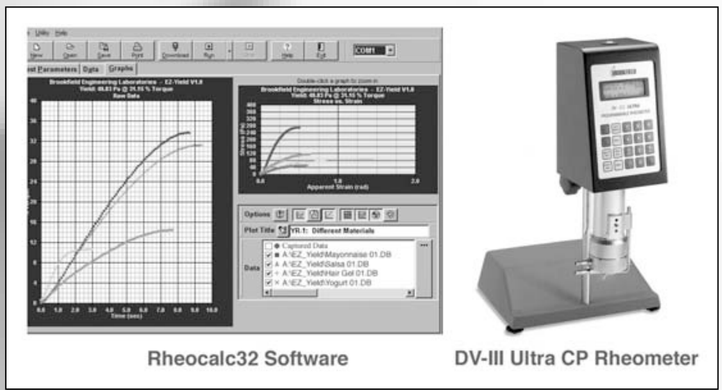

Figure 1 – DV-III C/P Ultra Rheometer with Rheocalc Software used in a QC lab to measure small sample volumes for flow behavior

Figure 2 – AST-100 process viscometer used in a flow line to measure and control viscosity of spray coatings during application

The formulation of a coating is as unique as is its purpose and cannot be covered in a single article. The discussion that follows concerns how coatings are applied and the quality control of that process. The measurement of viscosity is a key parameter that can be used to understand the flow behavior of the coating. The type of equipment used for this purpose is shown in Figure 1 and Figure 2.

Consider what happens to a coating during formulation, processing and application. Formulation mainly involves mixing ingredients. The resulting coatings appear to be liquids, but are more correctly characterized as emulsions laden with solids. Processing as part of the application process may involve pumping the coating through a piping system for delivery to the application area. Application of the coating onto the product often involves spraying while a batch of products are constantly moving in a tumbler, similar to a clothes dryer. Other application processes may dip the product into a pool of coating material on a constantly moving conveyor.

Each of these steps along the way in the manufacturing process subjects the coating material to a variety of conditions. A number of issues can arise from this treatment because emulsions are usually non-

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Newtonian fluids. Non-Newtonian fluids are those whose viscosity changes with rate of shear, or shear rate. One can think of shear rate as flow rate. In typical emulsions the viscosity will fall as flow rate increases. This characteristic is known as pseudoplastic behavior. In practical terms, this means the apparent viscosity of the coating will vary as it moves along through its processing and application path.

Imagine the flow behavior for a newly formulated pharmaceutical coating in the batch tank being slowly stirred while awaiting delivery to the application area. The coating may exhibit a relatively high viscosity at low shear. The impeller blades of the pump then shear the coating, causing a high flow rate as the coating starts on its way to the spray nozzle. At this higher shear rate the coating exhibits a lower viscosity than it did when in the batch tank. The pump motor must be sized correctly to handle the apparent viscosity of the coating both in the high shear area near the impellers as well as through the flow line to the applicator. (Note that shear rate is inversely proportional to the size of the tubing and directly proportional to the velocity of the flow.)

The highest shear rate, and therefore the lowest viscosity, is probably encountered at the spray nozzle due to the relatively small diameter aperture. Why is it important to understand this? It's important because a consistently uniform application of the coating on your products can depend upon the viscosity of the coating emulsion. But which viscosity value is most critical? Another way to think about this question is to consider the apparent viscosity of the coating at various locations in the system; at which location is viscosity most important in controlling and predicting product quality?

Experience suggests that the size of the aerosol droplets and the pattern from the sprayer are the keys to producing a coating of consistent quality on our product. Given this, it is likely that pressure at the spray head and the viscosity of the coating at the shear rate of the spray nozzle are the most important factors to assuring high quality products.

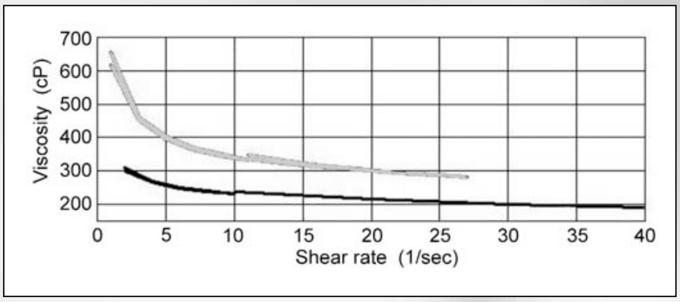

Figure 3 – Flow curves for two pharmaceutical coatings that contain identical ingredients mixed in different proportions

How much change in viscosity are we talking about? Just how "shear thinning" can a coating be, and what range of shear rates might be involved? The degree of pseudoplasticity in any material is dependent upon its components. Shear thinning behavior may appear in any degree from nearly Newtonian to extremely pseudoplastic.
Figure 3 shows the rheology profile of two coatings with identical ingredients. The coatings differ only in the proportion of those ingredients. The coating shown in black does show the characteristic increase in viscosity as shear rate decreases, but the increase is not nearly as dramatic as that for the coating shown in gray. Low shear rates are experienced by the coating in the batch tank while being slowly stirred awaiting application. The coating shown by the gray curve would appear to have much higher viscosity than that shown by the black curve.

As shear rate increases, the viscosity of both coatings falls, but the gray curve falls much more rapidly; it quickly thins at the pump motor as it passes through the flow line on its way to the spray nozzle. The viscosity of both coatings are very similar to each other at the higher shear rates. Although these graphs only show shear rate up to 40 s^{-1}, it appears that almost all of the "shear thinning" behavior has occurred by the time that flow rate is reached. We can assume that, for higher shear rates, there will be little additional decrease in viscosity. Therefore, the shear rates at spray nozzles, which can be very high as shown in Table I, will have minimal impact on a further reduction in viscosity.

Table I – Approximate ranges for shear rates associated with different types of flow behavior

Situation	Typical range of shear rates (s^{-1})	Application
Sedimentation of fine powders in a suspending liquid	$10^{-6} – 10^{-4}$	Medicines, Paints
Leveling due to surface tension	$10^{-2} – 10^{-1}$	Paints, Printing inks
Draining under gravity	$10^{-1} – 10^{1}$	Painting and coating, Toilet bleaches
Extruders	$10^{0} – 10^{2}$	Polymers
Chewing and swallowing	$10^{1} – 10^{2}$	Foods
Dip coating	$10^{1} – 10^{2}$	Paints, Confectionary
Mixing and stirring	$10^{1} – 10^{3}$	Manufacturing liquids
Pipe flow	$10^{0} – 10^{3}$	Pumping, Blood flow
Spraying and brushing	$10^{3} – 10^{5}$	Spray-drying, Painting, Fuel atomization
Rubbing	$10^{1} – 10^{4}$	Application of creams and lotions to the skin
Milling pigments in fluid bases	$10^{3} – 10^{5}$	Paints, Printing inks
High speed coating	$10^{5} – 10^{6}$	Paper
Lubrication	$10^{3} – 10^{7}$	Gasoline engines

Once we've characterized the viscosity of our coating as a function of shear rate using the equipment shown in Figure 1, what do we do next? We know that we have to control the pressure at the spray head because we've already determined that this is important for consistent quality of our coated product. Good pressure regulation or an in-line pressure sensor, or both, would be a good idea.

To scale up for high volume manufacturing, we need to decide what viscosity measurement instrumentation to use. A quick review of the flow curve graph in Figure 3 shows that it probably is not necessary to duplicate the high shear rate at the spray nozzle when measuring viscosity. Most of the viscosity loss due to shear thinning has already occurred at a much lower shear rate. However, we'd probably be wise to stay above 10 s^{-1} when using the coating material shown in grey, because below that shear rate, the viscosity is very shear sensitive. We should be able to establish a meaningful correlation between a good product coating result and the viscosity of the coating material at any point above 10 s^{-1}.

A single point viscosity test may give the desired information. The DV-III Ultra Cone/Plate system with Rheocalc software shown in Figure 1 is ideal for R&D; the instrument can also be used in standalone mode for the QC function. There are also less expensive alternatives available if multiple units need to be purchased. The possibility at measuring viscosity on-line is also a consideration and can be accomplished with the AST-100 In-Line Process Viscometer shown in Figure 2. The advantage to your process is that no time is lost in making adjustments to your spraying process. The output signal from the AST-100 can be used as an input to your process controller to enable automatic adjustments of viscosity.

Once you have investigated the parameters as explained above, your QC requirements should be easy to specify. This will give you confidence that the coatings being applied to your valuable pharmaceuticals meet your requirements.

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