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Vacuum Shelf Dryer Provides More-Uniform Cakes

Different approach to endpoint determination leads to greater consistency

By Steve Webb, Eli Lilly and Company

SHELF DRYING is a common unit operation for reducing the liquid solvent content of solid cakes prior to material storage or downstream processing. Where the use of high temperatures could result in product stability issues, shelf dryers may employ vacuum to evaporate volatiles at low temperatures. Pharmaceutical manufacturing, for one, often requires efficient drying at less than ambient temperatures. The combination of shelf drying and sub-atmospheric pressure allows for efficient volatile material evaporation while maintaining the solid cake at relatively low temperatures. Determining the drying endpoint using in-process conditions that accurately predict the solid-cake moisture content minimizes the potential for a need to stop and restart the operation. We have found in a case study that the absolute pressure during a vacuum shelf drying operation correlates closely with the volatiles' content of solid cake and, thus, can serve to regulate drying time.

In this case, we must dry a crystalline solid product to a specified solvent content prior to storage and shipping for downstream processing. Technicians load a batch of wet solids that contain 20–40-wt.% solvent into stainless steel pans. The pans then are placed on jacketed shelves within the dryer and thermocouples are inserted into thermowells installed on the pans to measure cake temperature. The dryer is connected to a vacuum pump separated by a block valve that controls the start and end of drying. The vacuum pump runs at capacity throughout drying, with a flow rate of 40–82 acfm depending upon the operating pressure. The dryer shelf contains recirculated water that is temperature controlled to a specified set point to regulate heat transfer to the pans. Figure 1 shows the dryer and Figure 2 depicts the equipment layout.

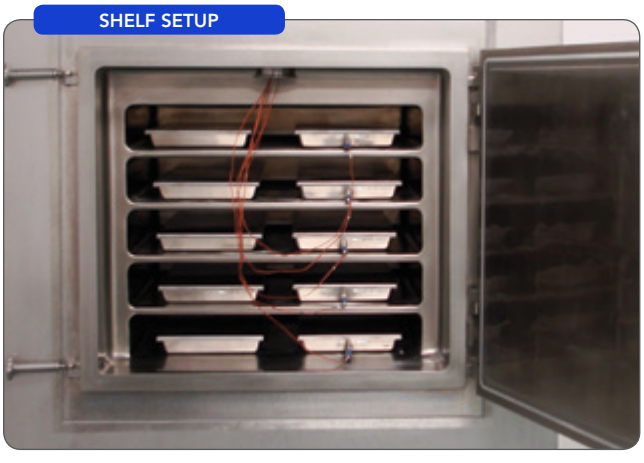

Figure 1. In typical operation, ten pans containing product are placed on jacketed shelves with thermal couples inserted into wells on five pans.

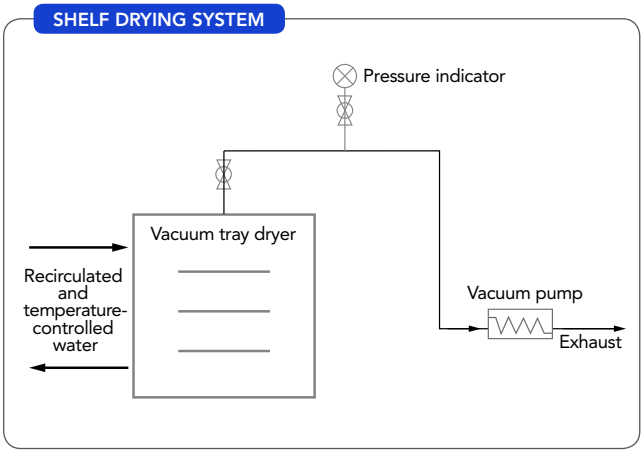

Figure 2. The vacuum pump runs at capacity throughout the drying operation.

Drying starts once the dryer door is sealed and the vacuum supply valve is opened. The shelf transfers conductive heat to the cake. Pressure is measured on piping connecting the dryer to the vacuum pump. The pump runs at full capacity, both removing solvent vapors and continually decreasing the pressure within the dryer. Initial pressure is as high as 7 mm Hg abs., but typically falls to 0.1–0.5 mm Hg abs. by the end of the drying operation. The thermocouples installed into the trays continuously monitor the solid cake temperature. It drops quickly at the start of drying from ambient temperature to between -10°C and -25°C as a result of evaporative cooling. The rate of evaporative cooling is greatest at the start of the drying operation when the cake has its highest volatiles' content. Temperature soon levels off and then slowly increases for the majority of the process due to the ever-decreasing rate of heat transfer from the solids to the vapor as the drying rate falls. By the end of drying, the cake temperature typically is 5–11°C when controlling the jacket to 12°C

CONVENTIONAL STRATEGIES

Control of the drying operation endpoint historically has been based on either a fixed time or a final cake temperature within a fixed time range. Both strategies heavily rely upon a defined time or time range to estimate when drying ends. A time-based approach poses inherent issues because factors that impact the drying rate vary from batch to batch and are difficult to control. The most influential factors are batch size or cake thickness, and initial moisture content.

Additionally, the temperature endpoint strategy has proven ineffective for several reasons. The level of leak rate into the dryer can impact the efficiency of solvent removal but is undetectable by the cake temperature alone. The placement of the temperature sensor within the cake also adds variability because the cake temperature changes as a function of its distance from the shelf. In addition, the cake isn't uniform in initial moisture content, so a single-point temperature measurement within the cake isn't always representative of the average of the entire cake due to lower or higher rates of localized evaporative cooling. These factors result in unacceptably high variability of endpoint moisture content.

In our case, prior to the vacuum dryer operation, the cake undergoes a forced nitrogen convection process that removes excess liquid solvent. As a result, the solvent contents observed in the process always start and end within the falling rate period of drying; therefore, the drying rate always is a function of the moisture content in the cake as mass transfer laws dictate. For this operation, batches enter the dryer with variable initial solid weights and liquid solvent contents. Batch size and the initial solvent content contribute to the volume of volatiles to be removed; larger or wetter batches generally require longer times to dry in the falling rate period of drying. Bigger batch sizes and, thus, thicker cakes also increase resistance to flow because diffusing vapor must travel a more torturous path to the surface. In addition, higher initial solvent content increases drying times because a greater ratio of initial solvent content to solid mass results both in a larger volume of solvent to remove and more-extensive evaporative cooling heat transfer occurring from the solids to the vapor. A higher extent of evaporative cooling removes heat from the solids and generates a lower temperature profile throughout the drying process, slowing the drying rate and extending the drying time.

A DIFFERENT APPROACH

We decided to try to determine drying endpoint based on product solvent content in the cake by indirectly gauging the vapor mass flow rate via pressure measurement. Assuming a low leak rate into the dryer, the pressure at any given time during drying is proportional to the volumetric flow rate as determined by the vacuum pump pressure/flow curve. In this case study, the vacuum pump capacity far exceeds the drying rate; therefore, the operating conditions all are on a sloped portion of the vacuum pump curve where volumetric flow rate continually decreases as pressure falls. Because the vacuum chamber is well sealed with a minimal leak rate and contains a vapor-producing stream from the cake in the falling rate period of drying, the pressure generated at any time reflects the mass flow rate of solvent vapor from the cake.

To check the assumption that the leak rate is minimal, we measured the pressure impact of the leak rate by running the vacuum dryer process without loading product. The pressure generated by the system at equilibrium primarily is a result of the system leaks, including from the vacuum pump. The leak rate pressure was found to be approximately 0.01 mm Hg abs. ±0.005 mm Hg. Because the pressure associated with leaking accounts for only 1%–15% of the total pressure at the endpoint condition, we felt comfort-

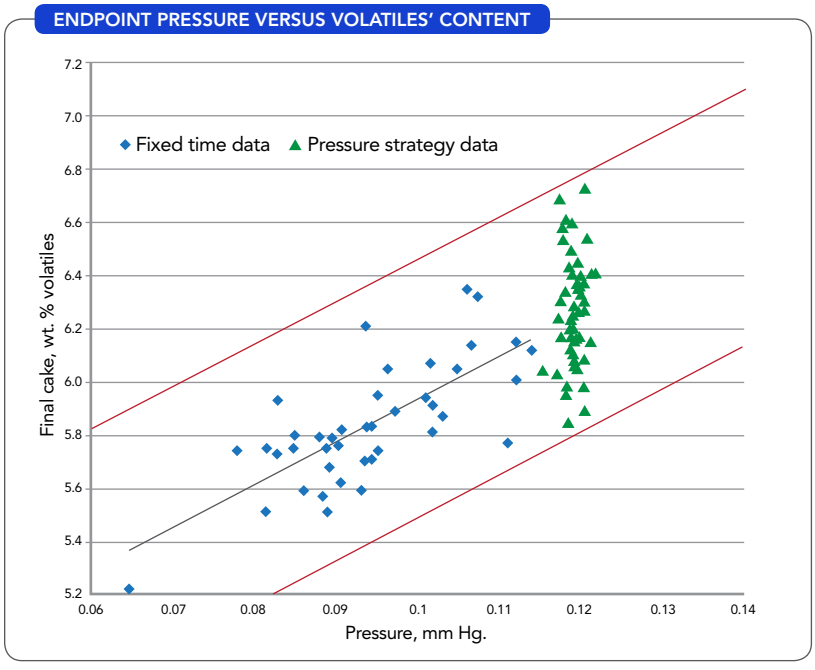

Figure 3. Pressure endpoint strategy enables accurate prediction of average volatiles' content.

able treating the contribution of leak rate as minimal and relatively constant. These data support the conclusion that the large majority of flow through the system when drying stems from solvent vapor evaporative mass transfer from the wet cake, also identified as the drying rate.

The Ideal Gas Law enables calculating the drying mass flow rate from the measured pressure during drying, the volumetric flow rate as determined by the vacuum pump performance curve, and the average cake temperature. The Ideal Gas Law is a good approximation of the actual drying rate as a result of low operating pressures. The drying rate is required to correlate measured pressure and the cake solvent content. However, in this case study, calculating the specific drying rate wasn't necessary because our desire was to predict the final volatiles' content in the cake.

The correlation of drying mass flow rate to the actual cake solvent content in the falling rate period of drying is complex and depends upon several factors including the specific cake's resistance to drying. The unknown geometry of interstitial spaces both between and within the solid particles making up the cake results in complex cake resistances. Therefore, empirical data are required to equate drying rate to a final volatiles' content and complete the correlation to endpoint pressure.

We experimentally determined the relationship between endpoint pressure and cake moisture content by measuring the final solvent content of the cake for 40 batches using a fixed-time drying endpoint strategy. We plotted the endpoint pressure and volatiles' content (wt.%) of each batch on an x-y chart and found a linear correlation for the given data range (Figure 3). We used the best-fit linear relationship to approximate an average volatiles' content that correlates to each endpoint pressure. We fit confidence limits of 99% (shown in red) to the data to show with high confidence the expected range of volatiles' contents for each endpoint pressure condition. In this case, we selected a specific endpoint pressure to achieve an average solvent content of 6.25% while limiting the upper range to 6.8%.

Upon implementing this pressure endpoint drying strategy, we produced a set of 56 batches by controlling the endpoint pressure to 0.12 mm Hg absolute. The result was an average final solvent content of 6.26 wt.%, with a range of 5.85–6.73 wt.%, which closely resembles the targeted average and upper limit. Figure 3 shows that the average volatiles' content with the pressure endpoint drying strategy was predictable by using a linear fit to the fixed time data. Additionally, the range can be predicted because the pressure endpoint drying data remained within the 99% confidence limits set for the fixed time drying data.

We now have implemented the pressure endpoint strategy; drying time (i.e., the time needed to reach the endpoint pressure) varies from -35% to +20% of the previous fixed time. Smaller and initially dryer batches require less time in the dryer while larger and wetter batches need more time. As a result of this strategy, the variability in the final solvent content in the cake has decreased by approximately 50%. These results support the conclusion that using a fixed pressure endpoint can compensate for batch-to-batch variability such as batch size (cake thickness) and initial moisture content.

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Stop What You're Doing

Challenging existing practices may spur significant improvements

By Tom Blackwood, Contributing Editor

AN OLD adage says, "To get what you want, stop doing what isn't working." In industry, we often fall into a routine set by operational procedures aimed at ensuring a safe workplace and cost effectiveness. We rarely question the current practice and review alternatives. In solids processing, many myths and superstitions get perpetuated because we don't fully understand the way nature works or the person who developed the procedure failed to explain it completely.

For instance, in planning an expansion, the choice of dryer came up for review. The existing operation used rotary dryers that often over-dried the product or even melted it on the lifters, requiring a significant amount of downtime. The product was very cohesive even when dry and went through a tacky phase during drying. To make matters worse, inlet-moisture control wasn't very good. An alternative technology — a fluid bed dryer— was suggested. The plant wouldn't even consider the option until the cost estimates came in and then, reluctantly, agreed to a test. Although the rotary dryers posed major operational problems, the plant argued it knew how to address the difficulties while it would have to start the learning curve from scratch with a fluid bed. Never mind how much the rotary drying was costing the site! Fortunately, the fluid bed dryer eventually was selected.

In another situation, the product from a flash dryer would come out burnt or decomposed. The dryer had a very effective control system. However, the plant continually was tuning it to respond to inlet-moisture changes because the upstream centrifuge produced erratic moisture content. The plant recycled some dry product from a baghouse. While the overall moisture was acceptable, fine particles were much dryer and were slow to absorb moisture. These particles never adequately mixed with the other particles and decomposed. Not only should a cyclone have been ahead of the baghouse so material wetter than the fines could be returned to the inlet but also centrifuge control needed improvement. The plant was tuning the wrong part of the process.

Many years ago, a plant installed a fluid bed dryer that from day one was plagued by sporadic fires. The manufacturer suggested raising the gas flow and maintaining that fixed value, which solved the problem; that high velocity was enshrined in the safety manual. However, losses from the dryer and attrition were very high. This soon became a cost issue. An evaluation of the fluidization grid showed the tuyeres were spaced too far apart and the gas jets were impinging on each other, which caused the high attrition. The fires stemmed from not having enough pressure drop over the grid, not low velocity in the bed. A new grid with the correct pressure drop operating at a lower overall gas flow rate eliminated the fires, attrition and excessive product losses due to entrainment. It took a while to get the green light, though, because changing a safety policy is tough and not for the faint of heart.

In another case, a centrifuge produced a wet cake of about 20–25% moisture, which required over a day to dry the solids. To meet high product demand, we considered buying another dryer or a compression filter to squeeze out more water. One day when the centrifuge drain had plugged up, we observed that just before the product was cut, water stopped flowing at the drain. However, as the peeler cut the solids, water gushed out. Nobody had considered slowing down the cut because it always had been done that way. After a few hours of playing with the peeler settings, we were getting moistures of 6–8%. Drying time was 4–5 hours and the product was fluffier. Problem solved!

My company participated in an industry study on project management when I was a young engineer. I wasn't surprised to learn that while the majority of projects in the chemical industry were completed on-time and near budget, those that involved solids seldom were on-time or anywhere near budget. A couple never started up.

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Consider Vacuum Drying

A vacuum drying pump offers several advantages for removing vapor over other methods

By Phil Vibert, Tuthill Vacuum & Blower Systems

DRYING IS a mass transport process normally involving removing or reducing water or other liquid content from a solid or solid-like material by evaporation. Heat is required for evaporation and as a method to remove the vapor formed by the evaporation process. Methods for removing vapor for drying include air drying and vacuum drying.

AIR DRYING PROCESS

With air drying, a gas stream like heated air blows over the product removing the vapor as humidity at atmospheric pressure. Heat transfer takes place through natural or forced convention, and in some cases, microwave radiation.

$$Wv = Wa \times MWv/MWa \times Pv/Pa$$

Where:

- Wv = Weight of Vapor Removed
- Wa = Weight of Air or N.C. Gas Blowing Over Product
- MWv = Molecular Weight of Vapor
- Mwa = 29 = Molecular Weight of Air or of N.C. Gas
- Pv = Vapor Pressure
- Pa = (Atmos – Pv) = Partial Pressure of Air

VACUUM DRYING PROCESS

The vacuum drying process removes vapor via a vacuum system. Heat transfer is primarily through conduction.

$$Q = U\ A\ \Delta T$$

- Q is the total heat
- U is the overall heat transfer coefficient
- A is the effective heat transfer surface area
- ΔT = Tw - Tv is the difference between the dryer wall temperature (Tw) and vaporization temperature (boiling point under vacuum) (Tv) of the liquid to be removed
- ΔT is increased by vacuum drying because Tv is lowered while under vacuum
- $dW/dt = dQ/dt = d(m_V\ Hv)/dt$
- dW/dt is the drying rate
- m_V is the weight of moisture evaporated and Hv is latent heat of evaporation
- Drying time $t = (Hv\ \Delta\ m_V)/(UA\ \Delta T)$

Figure 1 shows the vacuum drying rate process. A-B is the heat up where product temperature increases. B-C shows the evaporation rate increases as surface temperature approaches

PEAK DRYING RATE

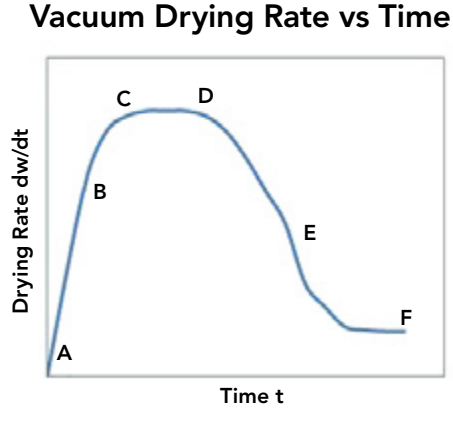

Figure 1. A vacuum pump is always sized based upon peak drying rate, C-D.

heating medium. C-D represents peak drying as temperature of product is at a maximum for evaporation and free moisture is removed. In D-E, heat transfer is reduced via fouling by product and moisture concentration is reduced to lower levels. E-F shows diffusion limited moisture removal at very low concentration.

ADVANTAGES OF VACUUM DRYING

There are many advantages with the vacuum drying process. Vacuum drying removes moisture at a lower temperature which is helpful for temperature-sensitive material. Moisture is removed without exposure to air to reduce oxidation or flammable conditions.

The vacuum drying method allows for recovery of solvents. Lower levels of moisture are removed by controlling both pressure and temperature. Heat recovery is also more controlled. Because the boiling points of the liquids to be removed are lowered under vacuum, the sensible energy consumption is less and Δt increases which reduces drying time. A closed-loop design allows for better recovery of material and air pollution control. And finally, overall the vacuum drying process is safer for drying hazardous materials.

DISADVANTAGES OF VACUUM DRYING

Some of the disadvantages of vacuum drying include the following:

- The upper surface temperature of vacuum dryers is normally limited to 600°F due to indirect heating and elastomer limitations. Direct heat atmospheric dryer temperatures can be higher.
- The surface area for heat transfer through conduction limits the rate of temperature rise in vacuum dryers.
- Atmospheric dryers are limited by volume of hot air (convection) blown over the product.
- Lastly, vacuum drying is normally a batch process rather than continuous since it needs to be an enclosed, sealed process.

VACUUM DRYER TYPES

Three types of vacuum dryers are available: shelf or tray dryer, rotary dryer, and conical or tumble dryer.

Within a shelf or tray dryer, product is placed in trays on shelves that are heated by circulating steam, hot water or hot oil. Hollow heated shelves conduct heat to the product. This process provides gentle handling and heating of product as it is designed for drying materials that cannot be agitated, or for small batches. It works in applications involving the drying of pharmaceuticals, fine chemicals, plastics, ceramics, electronic components and food products.

It is best to use a rotary dryer for drying paste and slurries; materials that may be sticky and need agitation. Its internal rotary agitator reduces sticking and clumping of product. Heat is supplied through a jacketed shell and shaft and internal rotating shaft with paddles or ribbons. The agitated material then exposes surface area for more efficient drying. A rotary dryer normally has the highest heat transfer coefficient. It works in applications involving the drying of pharmaceuticals and chemical along with filter cakes, pastes and food products. Volumes range from 2–300 CF with standard operating pressures of ≥ 10 torr.

A conical or tumble dryer generates a gentle folding action which insures more complete drying. Its overall dryer performance ranks in between the shelf and rotary dryer types. It is designed for free-flowing material. Heat is supplied through a jacketed shell equipped with baffling. The dryer rotates end-over-end to provide a folding action for the material, exposing surface area for efficient drying. A conical dryer is used for drying pharmaceuticals, powders, fibers, plastics, and food products. Volumes range from 1–350 CF with standard operating pressures of ≥ 1 torr.

DESIRABLE CONDITIONS

Vacuum drying's desirable conditions include:

- Pressure should be less than the vapor pressure of the liquid/solvent being removed:

VACUUM PUMP

Figure 2. Liquid ring vacuum pumps can pump the permanent gases, such as air leakage, along with the saturated vapors at the outlet temperature of the condenser.

$P \leq P_V$

- Temperature and pressure can be adjusted to attain this.
- Heat should be a sufficient level to supply the latent heat of vaporization required during drying:
- Heat for drying = (lb/hr Liquid Evaporated) x (Latent Heat)
- Surface Area: large surface area exposed to vacuum and heat.

VACUUM PUMP SELECTION

Dryer applications require the removal of vapors. Vapor load normally dominates vapor/gas load. The most common vacuum pump selected is a precondenser/vacuum pump where the precondenser pumps vapor and the vacuum pump handles gas. The precondenser is typically the most efficient methods of pumping large vapor loads.

The addition of heat is required to evaporate liquids in the dryer. Heat removal is required to pump out vapors via condensers. The heat of condensation is efficiently removed within the condenser. Surface condensers are normally used as opposed to direct contact (spray) condensers. The condensate can either be collected in a condensate tank or passed on to the vacuum pump (if liquid ring is used).

The dryer operating pressure and temperature can determine if a precondenser can be used.

- Determine cooling water temperature or coolant temperature available is Tc.
- Assume a drying pressure Pd ≥ 2Pv + 10 torr where Pv is the vapor pressure of solvent removed @ Tc in torr.

The vacuum pump is used to pump the permanent gases, such as air leakage, along with the saturated vapors at the outlet temperature of the condenser. The most common vacuum pump used would be the liquid ring vacuum pump (Figure 2) because of its attributes:

P ≥ 50 torr	Liquid Ring Vacuum Pump
100 ≥ P ≥ 10 torr	Booster/Liquid Ring System
10 ≥ P ≥ 1 torr	Booster/AE/Liquid Ring System

Other vacuum pumps used in this process include:

Dry Pumps	High exhaust temperature keeps solvent in vapor phase. Condense after discharging. Vapor AIT >> Disch Temp
Oil Sealed Piston or Vane Pumps	P ≤ 10 torr. using gas ballasting

PHIL VIBERT is an application engineer for Tuthill Vacuum & Blower Systems. He can be reached at pvibert@tuthill.com.

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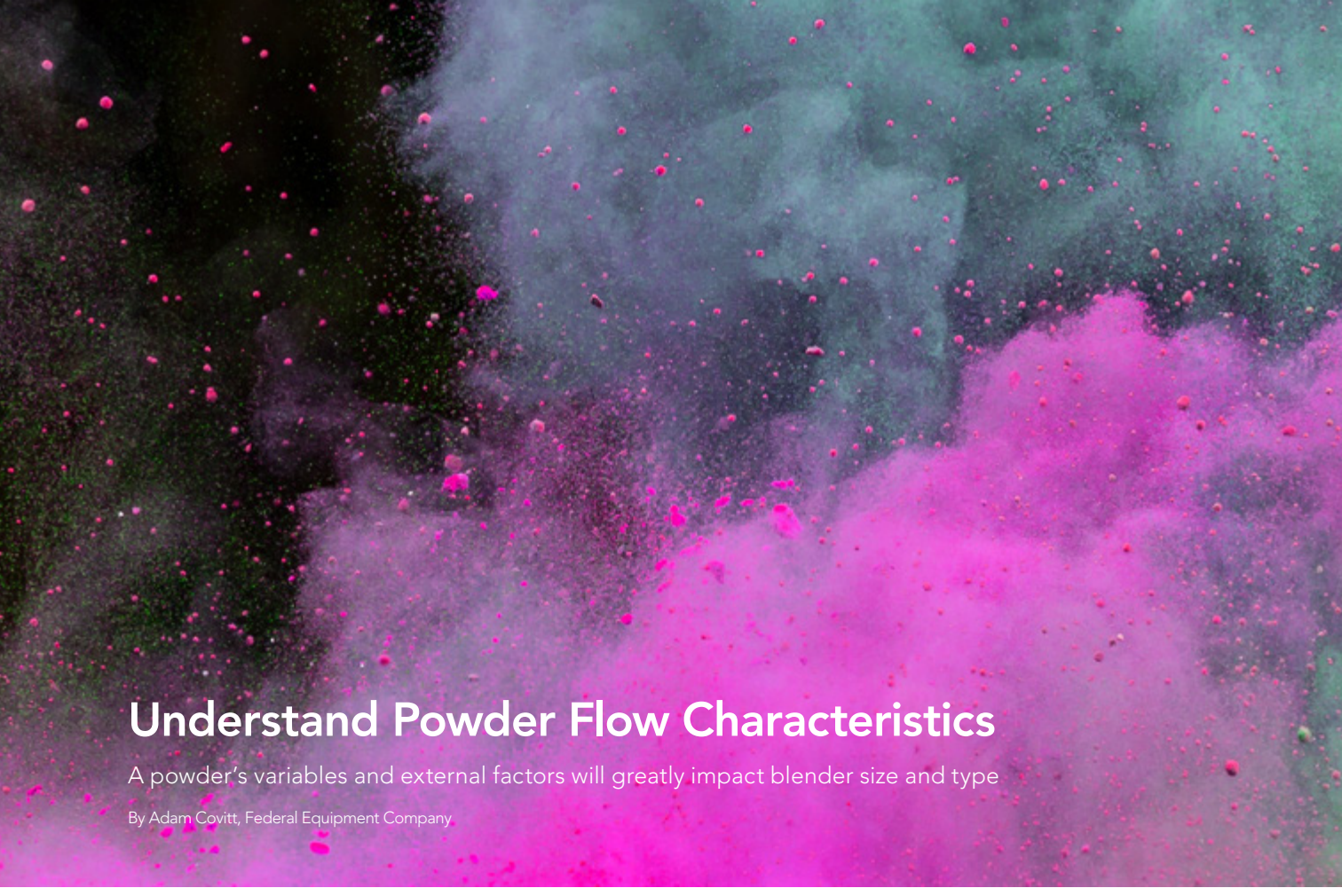

Understand Powder Flow Characteristics

A powder's variables and external factors will greatly impact blender size and type

By Adam Covitt, Federal Equipment Company

THE GOAL of powder blending is a homogeneous, or uniform, consistent mix of materials. The key to selecting a blender that will blend powders together into uniform mixes, also called "bulk solids," is the material's flow characteristics. Free-flowing powders are more likely to blend well together, but not all powders are free-flowing. Understanding the powder's flow characteristics will help determine the best type of blender for the product.

POWDER FLOW CHARACTERISTICS

Powders are the least predictable of all materials in terms of their ability to flow. Two sets of factors determine a powder's flow characteristics: the powder's variables and external factors. Powder variables include the product's bulk density, particle size, size distribution, shape, surface texture, cohesiveness, surface coating, and electro-static charge, among others. External factors include vibration, temperature, humidity, spurious electrical charges, aeration, container surface effects (or wall friction) and storage time. Addressing only one set of variables or partially addressing both sets of variables will lead to flow and, eventually, blend uniformity problems on the production floor. The flow characteristics will help identify the proper type of blender and the powder's weight and density will help determine the size of the blender.

BLENDER SIZE

Blenders are volumetric, which means their sizes are usually measured in terms of their volume capacity such as cubic feet. Powders will not blend well (flow) if the blender is too full or too empty. A safe range of effectiveness is 35–65% of the overall capacity. The product's flow characteristics will be the best indicator for the capacity range of each product mixed in the blender. As a rule of thumb, working capacity of a blender is usually determined as 50% of the total volume of the blender. For example, if a blender has an actual volume of 2 ft^3, then the working capacity will be 1 ft^3.

While blenders are generally sized according to volume, powders are usually measured according to density. Such measurements are either grams per cubic centimeter (gr/cc) or pounds per cubic foot (lbs/ ft^3). Additionally, the request for blended materials usually comes in terms of a specific weight of material such as 15 kilograms (kg) without reference to volume or density. Powders can be very light ("fluffy") or very dense which leads to different volumes of product at the same weight (think of a pound of feathers versus a pound of lead).

The proper blender size for the product is one that will have a working capacity within the effective range to achieve a uniform blend.

The simple way to calculate the proper size versus the product density is to weigh a quart or liter of powder. 16 quarts or 15 liters of a product is equal to 1 ft^3 at a density of 35 lbs/ ft^3. Blenders sizes based on 35lbs/ ft^3 at working capacity: 1 ft^3 = 15kg; 2 ft^3 = 30kg; 3 ft^3 = 45kg; 5 ft^3 = 75kg; 10 ft^3 = 150kg; 20 ft^3 = 300kg; 30 ft^3 = 450kg; and 50 ft^3 = 750kg. Size is not the only factor to consider, the type of blender is important as well.*

BLENDER TYPES

Twin-shell (or "V" blenders), double cone blenders and bin (or tote) blenders are all considered "random" style blenders. These types of blenders also are referred to as "open shell blenders." They randomly mix powders that are already free flowing through the blender's tumbling action. If the products are dense, an intensifier bar can be added which will force the powders to move inside the shell of the blender. Liquids may be added to the bulk solids mixture with a liquids bar. Intensifier bars can be overused which can result in particle break down for dry and friable powders or it can pack powders that are wet or cohesive (sticky).

Ribbon and paddle blenders are excellent ways to mix powders slowly and gently. In a ribbon blender, a double-helix agitator will move materials towards the center of a trough with the outer blade while the inner blades move the materials towards the outside of the trough. Paddles are an alternate design that can be used for small batches relative to the working capacity of the blender and with friable materials.

High shear mixers are generally used for products which are considered immiscible, where the products to not generally form a homogeneous blend. The mixer operates by moving one phase into a continuous phase. The phases, or ingredients, can be solids, liquids or gases. The ingredients are moved with a rotor, or impeller, across other rotors or stators to produce a mechanical force called shear which forces the products to mix. Plow-style mixers are common high shear mixers that can be used for particle size reduction and to produce granules.

These are only a few, common-types of blenders available. Many additional types of mixers can be used for powder blending including planetary mixers, Nauta-style mixers and single and double-arm mixers — choices among many others which can be evaluated applying the same analysis of weight, capacity and flow characteristics. Powder flow characteristics and the weight of the product can be used to determine the proper type and size of the blender needed for the product. Analyzing the weight of the product can be used to estimate the working capacity required which determines the overall size of the blender needed. The flow characteristics will also determine whether the product can be blended in a random type blender, a ribbon or paddle blender, high shear mixer or some other type of blender. ●

**Special thanks to Mike Tousey at Techceuticals, LLC for providing the technical information described in this article.*

ADAM COVITT is vice president for Federal Equipment Company. He can be reached at adam@fedequip.com.

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Check out our vast library of past eHandbooks that offer a wealth of information on a single topic, aimed at providing best practices, key trends, developments and successful applications to help make your facilities as efficient, safe, environmentally friendly and economically competitive as possible.

Upcoming and On Demand Webinars

Tap into expert knowledge. *Chemical Processing* editors and industry experts delve into hot topics challenging the chemical processing industry today while providing insights and practical guidance. Each of these free webinars feature a live Q&A session and lasts 60 minutes.

White Papers

Check out our library of white papers covering myriad topics and offering valuable insight into products and solutions important to chemical processing professionals. From automation to fluid handling, separations technologies and utilities, this white paper library has it all.

Minute Clinic

Chemical Processing's Minute Clinic podcast series is designed to tackle one critical issue at a time — giving you hard-hitting information in just minutes.

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Have a question on a technical issue that needs to be addressed? Visit our Ask the Experts forum. Covering topics from combustion to steam systems, our roster of leading subject matter experts, as well as other forum members, can help you tackle plant issues.

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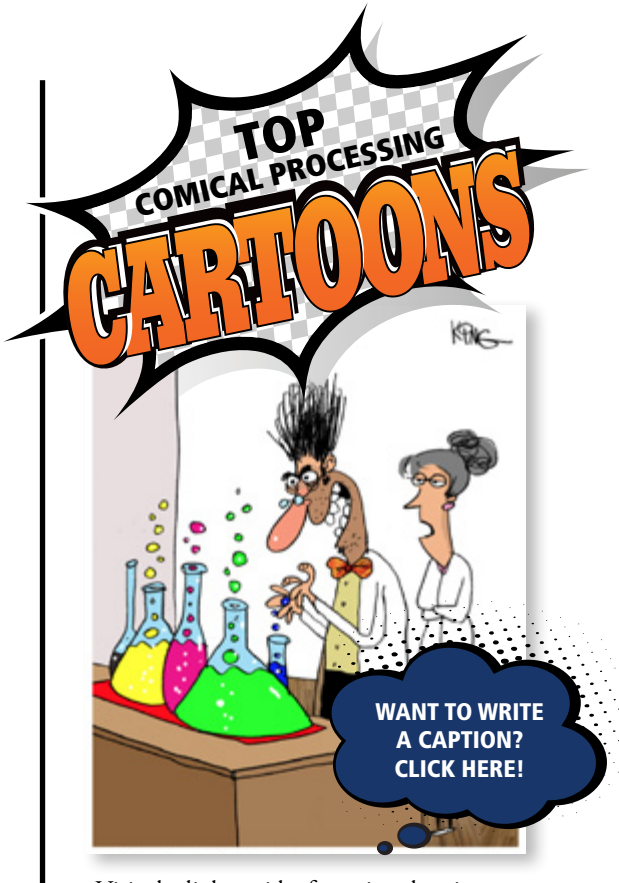

Visit the lighter side, featuring drawings by award-winning cartoonist Jerry King. Click on an image and you will arrive at a page with the winning caption and all submissions for that particular cartoon.