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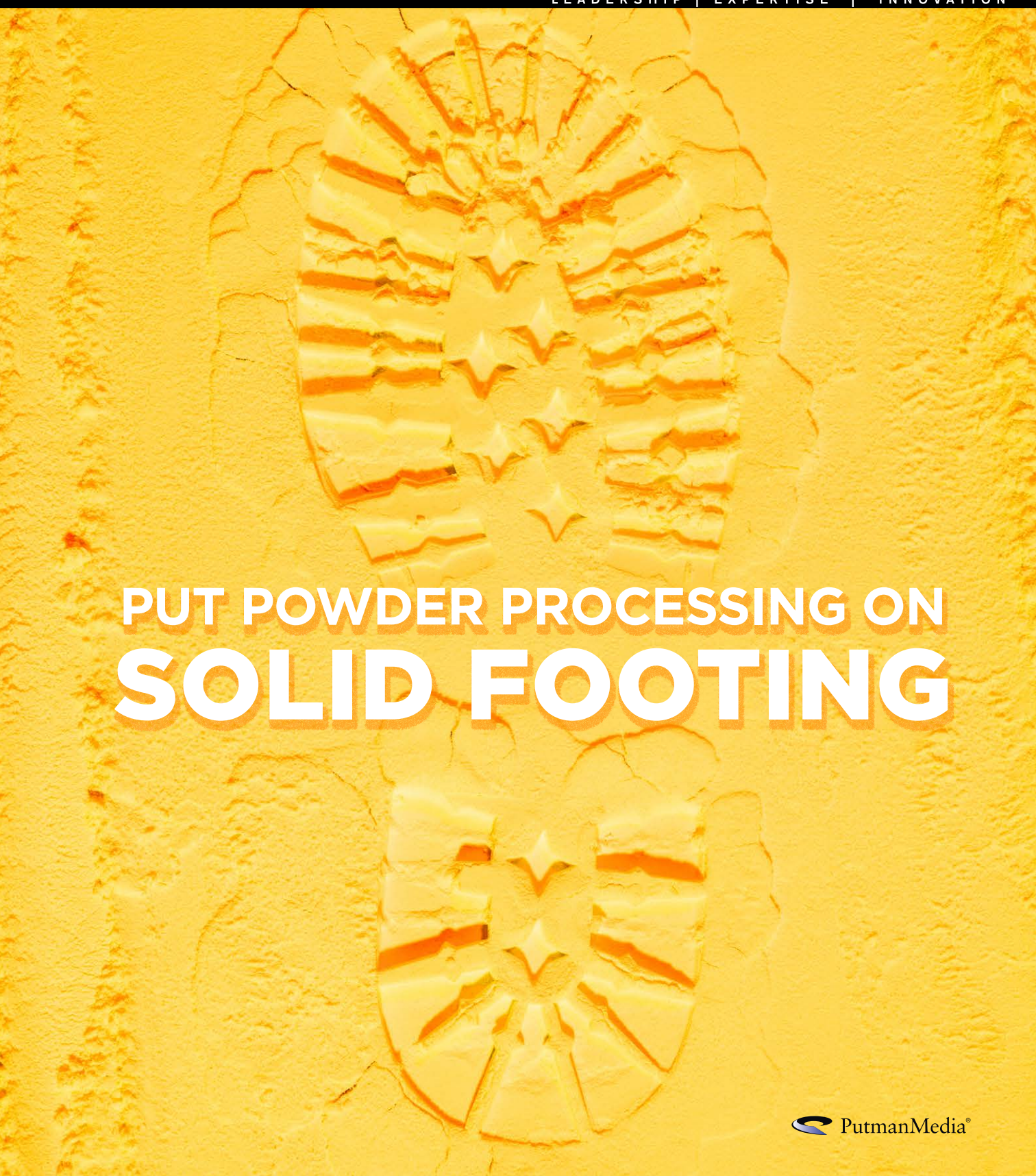

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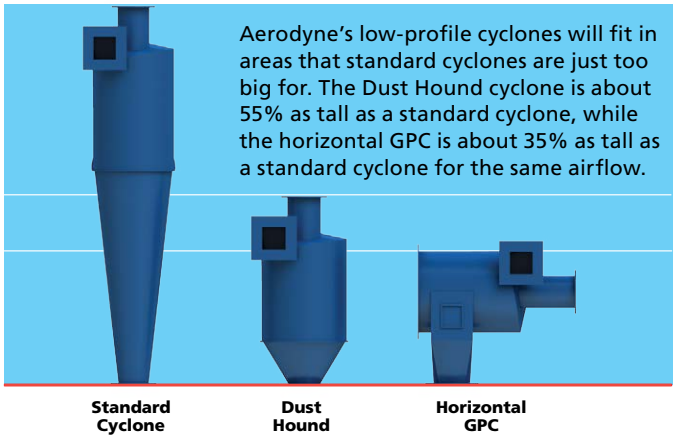

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Is The Homogeneity Of Your Dry Mix Acceptable?

Understand how to correctly validate the results of mixing

By Thomas Lamotte, Nestlé Research & Development Singapore

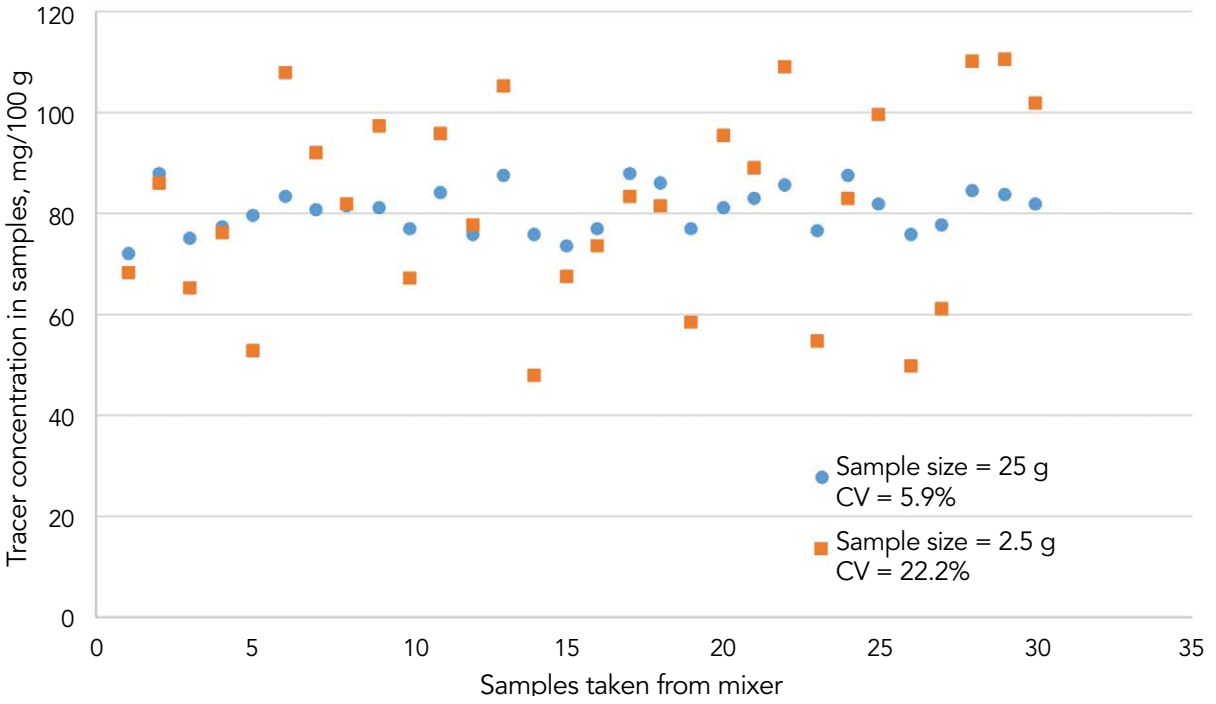
Dry mixing plays an important role in many processes. The operation can take place at different stages of a process, for example at the beginning to mix raw materials or at the end to disperse additives into a product. Regardless, the purpose of the mixer remains the same: creating a mixture homogeneous enough for the intended application.

This article aims to help process designers and plant operators understand what homogeneity in solids processing is, how to characterize it, and how to validate a dry mixing operation by measuring the degree of mixing.

HOMOGENEITY

A perfect degree of homogeneity, or mixture quality, means ingredients appear in the same proportions in any sample taken at any point of a mixture. Of course, this ideal result doesn't occur in the real world. Instead, differences between components, mainly in terms of particle sizes, always will constitute a natural limit to homogenization. (We won't consider coating effects in this article.) Additionally, the sample size matters when checking homogeneity — assessing the same mix using two different sample sizes may lead to different conclusions.

As a consequence, discussing the homogeneity of a mix of solids demands care. The nature of the solids being mixed and the sample size significantly affect the confidence you should place in any measurement of homogeneity. Sample size is a critical parameter (Figure 1). So, always



INFLUENCE OF SAMPLE SIZE

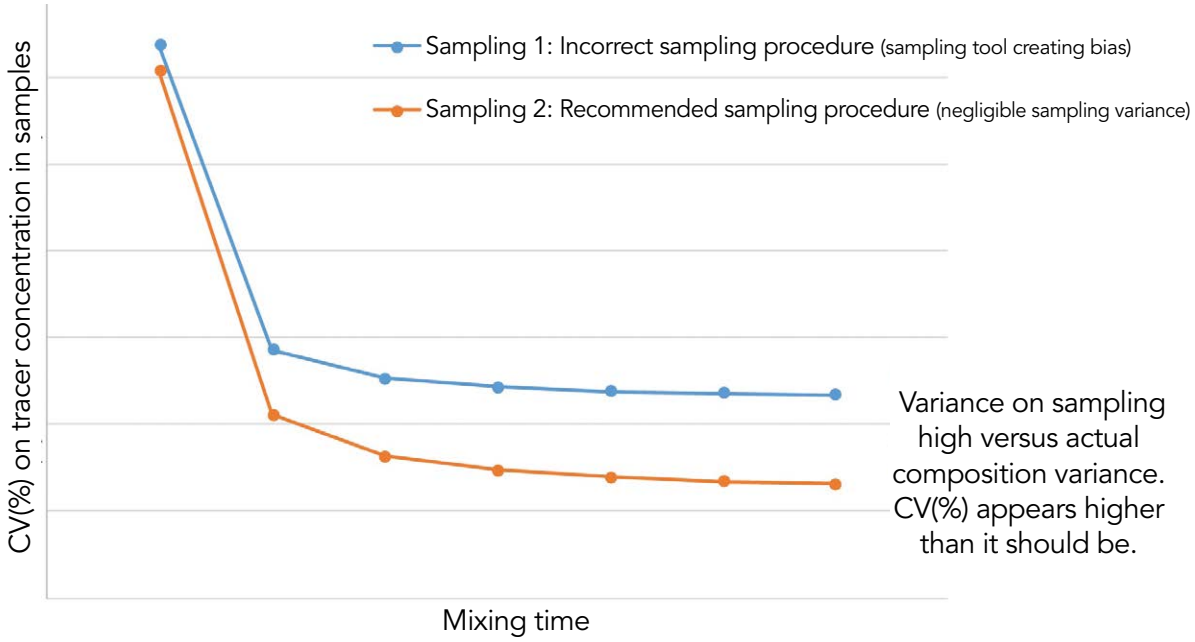
Figure 1. Reducing sample size from the recommended size (25 g) increases the variance of concentration between samples and results in a higher CV.

reflect upon what represents a meaningful sample size for an application; selecting too small a size will make it difficult to determine if the mix is homogeneous, while too large a size almost certainly will make the mix appear homogeneous. For example, if a mixture is intended to be consumed (such as a pharmaceutical or food), the appropriate sample size is the serving size.

The mixing quality is defined in regards to a specific property the processor wants to be equal at any point in the mixture. This sometimes is a physical property like particle size distribution but more often is ingredient composition. Here, we will focus only on composition.

For practical use in processing, the notion of homogeneity must be translated mathematically using statistics. This usually is done by calculating a sample variance (S^2) of the concentration of one component from an analysis of samples taken from the mixer.

The analysis is performed on a tracer, a component of the mixture whose distribution is taken as representative of the state of the mixture. Getting meaningful results, i.e., rather narrow confidence



IMPACT OF SAMPLING METHOD
Figure 2. Results using the same mixer illustrate that incorrect sampling method can lead to wrong conclusions about mixing quality.

intervals, generally requires a minimum of 30 samples from the mix, with a sampling methodology that doesn't leave part of the mix unsampled.

The lower the sample variance is, the higher the degree of mixing or homogeneity and the better the quality of the mix.

CRITICAL FACTORS

Achieving valid insights from samples requires careful attention to three key preliminaries: the desired target value, the choice of tracer and the sampling procedure.

Setting the target value. Measuring mixture quality only makes sense if a processor has set sample variance targets to enable comparing the measurement to an objective. Such targets depend upon the application. Consider two examples:

1. The mixture is used internally for another unit operation on site; the company itself defines the specifications, which may allow quite a large variation to ensure a smooth operation.
2. The mixture is sold directly, e.g., as a pharmaceutical tablet; the specifications must meet legal regulations, which may be very narrow.

This highlights a general point. If the mixture is used in an intermediate process step, the quality of the mix may not be critical and relatively high composition variations

may be acceptable. However, if the mixture is an end product subject to strict regulation (such as a pharmaceutical or food), much more stringent control of the mix quality is required and the validation of the mixer must ensure the mix always will be within specifications.

Let's now look at how to validate the mixture quality and compare the actual state of the mix to a target.

Choosing a tracer. Determining the composition of complex samples (beyond a binary mixture) sometimes is difficult, if not impossible. That's why analysis often is done only on a single component, the tracer.

Selection of the tracer demands care. It shouldn't be a major ingredient, except if the mixture contains only major components. It usually is chosen from small/minor ingredients that often are of interest in the mix (nutrient, active substance, etc.). Because of its low concentration, that component will take longer to homogenize, and thus represents a worst case among all the ingredients.

The tracer should lend itself to easy analysis. It's better if the tracer is part of the mix formula (vitamin, active substance, reactive chemical, etc.). However, sometimes adding another component as the tracer can make sense to ease the mixer validation exercise, for example, adding salt.

Proper sampling procedure. After mixing according to the specified parameters — typically mixing time and speed, batch size and the sequence to fill the mixer — you must take samples to measure the quality of the mix.

The way you take samples is critical for a correct assessment of the degree of homogeneity (Figure 2).

For example, consider a batch mixer containing powder at the end of a mixing cycle. Taking every sample only on the left of the mixer, only in the middle or only on the right makes no sense because the samples must represent the *whole* mix, not only one part.

For this, different methods are possible:

The best approach is to discharge the contents of the mixer and take samples at regular intervals on the *free flowing* powder, from the *very beginning* to the *very end* of the flow. This ensures that no powder area in the mix was left untouched. Sampling must occur in a specific way by quickly "cutting" the flux of powder.

If that approach isn't possible, you must resort to sampling in the mixer. For this, you must use a sampling tool and define a sampling plan that ensures every area of the mixer gets sampled. This method is more difficult because easily reaching every point

of the mixer may prove impossible, and the sampling tool may induce a sampling bias on the results.

ANALYZING THE SAMPLES

After getting the samples, you must analyze each one. In the best case, you can use the whole sample for analysis (e.g., diluted if necessary). However, if the sample is too large, you must divide it. Don't take what's needed for the analysis directly from the sample; this would lead to further sampling bias. Instead, use a sample divider to minimize the risk of mistakes due to re-sampling.

If you're not that familiar or comfortable with the analysis method, it's best to analyze each sample a second time to account for the variability of the analysis. Record the results obtained for each sample.

Calculating the degree of mixing. The mean concentration of the tracer and the variance of the tracer concentration are used to calculate the relative standard deviation (RSD) of the mix, a value reflective of homogeneity:

$$RSD = (S^2)^{½}/\mu \qquad (1)$$

where S^2 is the samples' variance (this isn't the actual variance because samples only can provide an estimation) and μ is the arithmetical average of the samples' concentration calculated from the samples.

Rather than relying on variance or RSD, industry often uses the coefficient of variation (CV), expressed in percentage, to describe the degree of mixing (homogeneity):

$$CV(\%) = [(S^2)^{½}/\mu] \times 100 \qquad (2)$$

Warning: the CV obtained actually has several components; some of these must be calculated to estimate the actual homogeneity variance. The sample variance is calculated via:

$$S^2 = S^2_{mix} + S^2_{analytical} + S^2_{sampling} \qquad (3)$$

The variability due to sampling is very difficult to determine. Thus, in practice, it gets included in the mixture variance. However, for this assumption to give meaningful results, it's critical to sample the mix following the methods explained above — preferably on the free flowing powder — so that the variance due to sampling is negligible compared to the actual mixture variance.

The variability due to analysis might be known if experiments have been done before or can be determined for the particular homogeneity validation by doubling the measurement on the same sample.

You then can calculate S^2_{mix} and followed by $CV_{mix}(\%)$.

Confidence interval. Once you've calculated $CV_{mix}(\%)$, you can't just compare it

to the specification. Indeed, the variance calculated is *not* a true variance but an estimate based on the sampling. If sampling is repeated on the same mix, the value obtained certainly will differ.

You must take this variation into account by calculating a confidence interval, generally at 95%, which corresponds to 2 sigma on each side of the mean, i.e., CV_{mix} is within CV_{lower_limit} and CV_{upper_limit}.

The confidence interval depends on the number of samples; the higher the number of samples, the narrower it is. Good practice usually is to take a minimum of 30 samples per mix.

COMPARISON TO SPECIFICATION

The specification often is given as a maximum acceptable CV on the composition CV_{spec}. It then can be compared to the confidence interval:

$CV_{upper\_limit} < CV_{spec}$ — the mixing is successful because the mix exceeds the specification;

$CV_{lower\_limit} < CV_{spec} < CV_{upper\_limit}$ — the mixing may be successful but it's also possible that the actual variability is higher than the specification; and

$CV_{lower\_limit} > CV_{spec}$ — the mixing homogeneity attained isn't good enough for the application.

For the first case, the processor has achieved the right mixing quality but may wish to test other parameters to optimize the mixing process (e.g., shorten mixing time to increase capacity).

For the two last cases, the plant team must engage in a case-by-case discussion:

- Accept the mix if the application isn't sensitive and CV_{spec} is close to CV_{upper_limit}; or
- Reject the mix and look for root causes (mixer speed, filling rate of mixer, etc.).

ACHIEVE SOLID SUCCESS

Validating mixing performance is crucial for controlling processes that involve dry mixes. You must pay careful attention to key preliminaries, especially defining the sample size, the tracer, the sampling methodology and the analysis technique. Any mistake in sampling or analysis can lead to errors in interpreting mixing quality and, ultimately, to a non-compliant product or a non-optimized process. ●

THOMAS LAMOTTE is a senior process engineer at the Nestlé Research & Development Center in Singapore. Email him at thomas.lamotte@protonmail.com.

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First, Select the Right Blender

Understand the pros and cons of three key kinds of devices

By Tom Blackwood, Contributing Editor

The choice of blender type dramatically can affect end results. Yet, often we put a product in whatever blender is available. That's a mistake. Each blender is unique; they may vary in typical blend time and quality, blender capacity, scaleup, maintenance and attrition. So, let's compare blender types.

The number of options can seem overwhelming. One manufacturer's selection guide I recently looked at showed several hundred blender types. I don't doubt that their differences are real in the eyes of the designer — but I've usually found that the particulate solids characteristics of the material to be blended are more important than some subtle variation in a unit's mechanical design. For simplicity, I suggest concentrating on three overall types of blenders: mechanical, gravity and fluid-assist devices.

MECHANICAL BLENDERS

Most such units are small capacity. Let's look at four options.

- *The double-cone blender* has been the workhorse of the industry, especially for batch operations. Blend times typically run one to two hours. Maintenance is low. Attrition is moderate but particles may segregate on discharge. Internals, which come in many variations (ribbons, baffles, etc.), can improve blend quality and minimize segregation.
- *Paddle or ribbon blenders* provide much lower blend times and often serve in continuous processes. Low-speed units may reach a good blend in one to 15 minutes. In contrast, high-speed devices

The fluidized bed is probably the most effective fluid-assist blender.

may achieve the same blend in five to 30 seconds but the number of fine particles generated may override blend quality. Maintenance is moderate but attrition is high for high-speed devices.

- *Riffle blenders* can match the blend quality of a high-speed blender and create less attrition. However, their maintenance (cleaning) is high and they don't scale up very well.
- *Screw blenders* aren't known for blending applications but can reach a good batch blend in three to five minutes with moderate attrition. Maintenance is high but scaleup is easy.

For continuous operation and the best blend quality, I suggest a loss-in-weight feeder. It scales up easily and creates little to no attrition. The device demands only low to moderate maintenance but often is an expensive alternative.

GRAVITY BLENDERS

Storage of large quantities of bulk material frequently requires some blending to compensate for segregation or process variations. Achieving the best quality blend may call for recirculation via pneumatic conveying. The traditional static flow tube smooths out small variations. However, mass-flow or multi-cone blender designs largely have replaced it because they can reach a high quality blend in a couple of turnovers. Moreover, they are easier to scale up than flow tubes and are lower in maintenance. Other than attrition from the pneumatic conveyor, particle breakage is minimal in these devices.

FLUID-ASSIST BLENDERS

These devices often are costlier than the other two categories but compensate by providing better blend quality. Let's examine four alternatives.

- *The lift-tube blender* could be considered a gravity blender — but recirculation is internal to the device and blending can take place during tank filling, which smooths out production variations over a longer time frame. Scaleup is moderately easy and maintenance is low. Attrition is negligible because the recirculation loop has no feeders or elbows. The design of the internal cone controls blend quality.
- *The fluidized bed* probably is the most effective fluid-assist blender. It works well in batch or continuous operation. Blend

time is on the order of minutes versus hours and scaleup is easy. Most maintenance is external to the device; proper grid design minimizes attrition.

- *The spouted bed*, a cousin to the fluidized bed, is a favorite for coating particles as well as giving a good blend in only one turnover. Attrition is very low, especially with big and light particles, because most attrition occurs as the particles fall back onto the bed. Scaleup is moderately easy; units require little maintenance and cleanup is easy.
- *Blenders using a jet or venturi* work well with fine materials and can give an excellent blend. Scaleup is easy but maintenance can be high. These devices better suit liquid/particle blending.

Throughout this column I haven't talked much about blend quality because this mostly is a function of the particles in the system. Experimental data are a must to define blend quality. ●

TOM BLACKWOOD, Contributing Editor

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Maximize Polyethylene Operations with 80-GHz radar

Accurately detect and measure flake and pellet density variations and anomalies

By Greg Tischler and Jeff Brand, VEGA Americas

Managing polyethylene silos and storage bins in the final stages of production can be a challenging and costly assignment. Polyethylene flakes, or fluff, can be difficult to measure accurately in silos or storage bins. These flakes can have varying densities, tend to collect and build up on any sensors and have low reflective properties. Use of an 80-GHz radar (Figure 1) is a more accurate and less expensive level measurement alternative to other traditional methods.

When polyolefin plants manufacture high-density and linear low-density polyethylene, a chemical reaction creates the product. Following this initial reaction, several steps and processes are taken to separate the product from the excess raw materials. The product, in this case, comes in the form of light, fluffy flakes. These flakes commonly are stored in a fluff silo or storage bin before moving on to the final extruder step, where they're turned into pellets and moved to a different storage silo before shipment. Alternatively, low-density polyethylene goes straight to the extruder and into similar storage silos.

The level inside these vessels is measured and checked in both of these steps — the fluff silo and the storage silo. Traditionally, the level has been tracked using load cell technology. Essentially, a scale is installed beneath the silo, weighing the silo and the material inside. However, these load cells are expensive to install and even more costly to maintain. The load cells cannot tell users whether or not they're maximizing the full vessel, either, because these

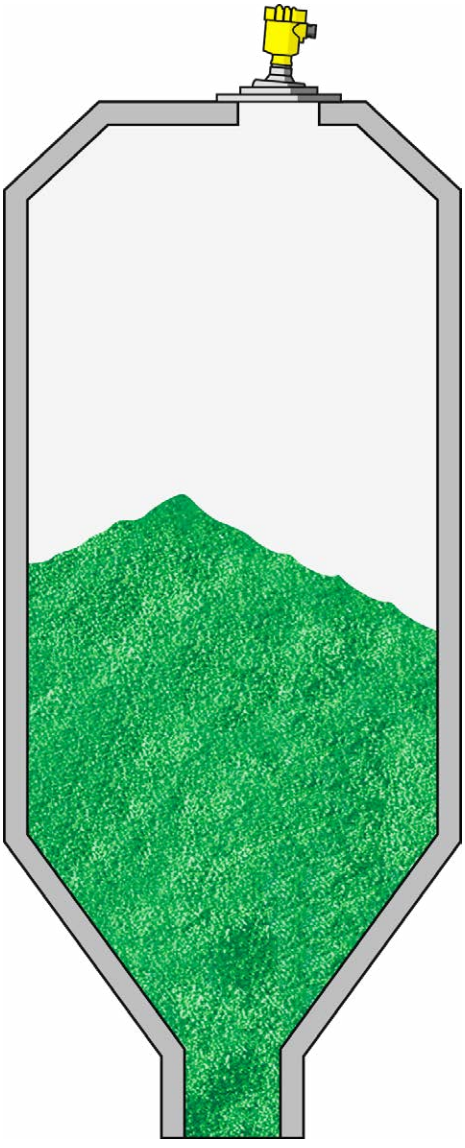
flakes and pellets have varying densities, so different weights will equate to different levels.

Polyethylene comes in many grades, and each grade has a different density. This means the weight of a full silo will be different for two different types of polyethylene. This can lead to an overflowing silo or not fully using a silo when the type of material isn't taken into account.

A DIFFICULT MEDIUM TO MEASURE

Both polyethylene fluff and pellets can be a problematic material for other level measurement sensors. Polyethylene flakes, by their nature, stick to anything and are prone to buildup. This buildup can prevent some sensors, such as ultrasonic devices and lasers, from working properly without constant cleaning and maintenance.

In the past, through-air radars have had trouble measuring polyethylene because of its low dielectric constant and poor reflective qualities. Water has a very high dielectric constant — approximately 80 at room temperature. Polyethylene and plastic pellets, however, have a dielectric constant closer to 1.2. This extremely low number gave previous generations of radars problems obtaining an accurate level measurement when they received a return microwave signal from the product's surface.



80-GHZ RADAR
Figure 1. An 80-GHz radar can more accurately detect and measure polyethylene flakes in silos or storage bins.

These hurdles — buildup and low dielectric constant — have led to the widespread usage of weigh scales (Figure 2) for inventory control in silos at polyolefin plants. However, these mechanical measurements are pricey to install or replace, especially

at older, established plants. Installing or replacing one of these means lifting the entire silo and installing the device underneath — a costly endeavor, especially if it requires shutting down operations. These weigh scales also are expensive to maintain — cleaning, calibrating, and repairing as needed all takes valuable time for maintenance crews. 80 GHz radar provides a less expensive method and also help prevent overflows while maximizing every storage vessel and silo.

RADAR LEVEL MEASUREMENT WITH 80 GHZ

Through-air radar works by emitting radio microwaves from the radar antenna system to the measured product where it is reflected by the product surface and back to the antenna system. The radar sensor uses time of flight to measure level of the product — polyethylene flakes or pellets in this case. The amount of time it takes from emission to reception is proportional to the distance to the product surface. The longer the time of flight, the greater the distance. The distance is inversely proportional to the level in the tank, so the greater the distance, the lower the level.

Real-world benefits of 80-GHz radar can be seen across many applications, including polyethylene silos. The VEGAPULS 69 from VEGA, which is designed for bulk solids, uses enhanced focusing, a dynamic range that can receive more of the microwave signal and software to generate an accurate and reliable echo curve to interpret the level inside the vessel.

SILOS WITH WEIGH SCALES
Figure 2. Weigh scales commonly are used for inventory control in silos at polyolefin plants. However, these mechanical measurements are pricey to install, replace and maintain.

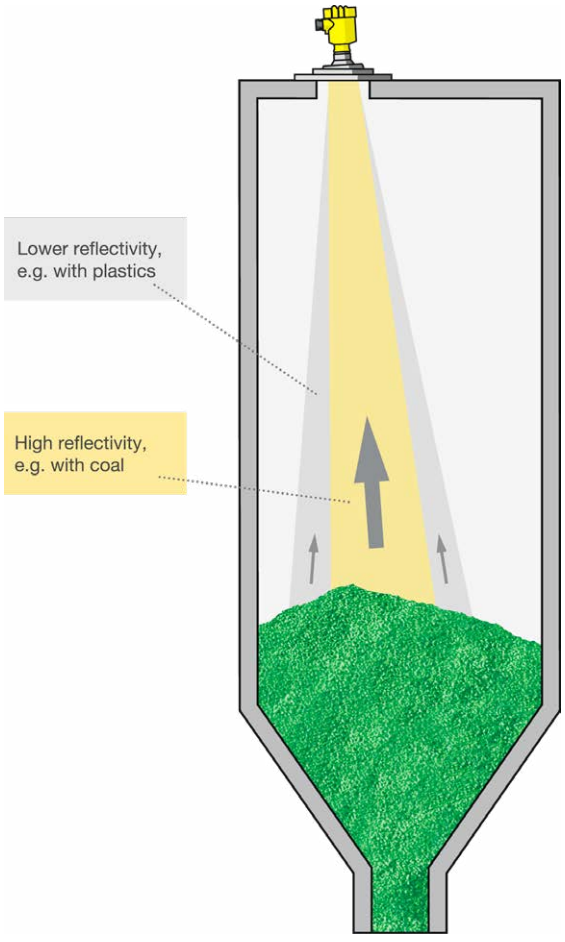
Many factors determine a returned radar signal's strength. Buildup on the antenna

POLYETHYLENE SILOS
Figure 3. The narrow focus of an 80-GHz transmission frequency works well for use in the narrower dimensions of polyethylene silos.

and poorly reflective materials both can influence the return signal, making it difficult to get a precise measurement.

The 80-GHz transmission frequency has a number of benefits, but when it comes to polyethylene silos (Figure 3), which typically are tall, narrow vessels, the narrow focus is especially helpful. The microwave beam's focus depends on a radar transmitter's antenna size and its transmission frequency. As the antenna or the frequency shrinks, the wider and less focused the beam becomes. Conversely, if the antenna or the frequency grows, the beam becomes narrower and more focused. The VEGAPULS 69's beam angle is anywhere between 7° and 14° with process fittings ranging from 3 to 1½ in., giving a range of possibilities for silos of any dimension.

Conductive products reflect almost all microwave energy, but nonconductive products with low dielectric constants, like polyethylene, reflect only a portion of the energy (Figure 4). This means there's a weaker signal from the surface of the product inside the silo. To combat this, some radar sensors use a higher dynamic range. The dynamic range is the measure of which signals a radar sensor can detect, meaning sensors with a large dynamic range are sensitive enough to register weak signals as well as strong ones. This radar sensitivity varies across manufacturers and even among a single manufacturer's instrument line.



REFLECTIVITY
Figure 4. Conductive products reflect almost all microwave energy, but non-conductive products with low dielectric constants, like polyethylene, reflect only a portion of the energy.

This higher sensitivity may lead users and technicians to think a more sensitive radar would be more susceptible to buildup on the sensor itself. Some radar sensors can prevent this by using software to filter out signals the buildup causes. Users can then measure the product as though the sensor is buildup-free.

MORE EFFICIENT OPERATION

Managing polyethylene flake and pellet inventory is now more simplified and more economical with 80-GHz radar. The VEGAPULS 69 can track the level inside the fluff silo accurately before the extruder step and inside the storage silos before shipping. Installation is significantly less expensive than weigh scales, and, in most cases, these sensors can be installed and commissioned without a plant shutdown.

After it's installed, the amount of maintenance and cleaning required for one of these radar sensors is nearly zero, even in the presence of buildup. Recent 80-GHz radar instruments are more focused and more sensitive with more intelligent software than previous versions, allowing users to monitor polyethylene levels effectively and with confidence all throughout the process. ●

GREG TISCHLER is Radar and Guided Wave Radar product manager and **JEFF BRAND** is Pressure and Switching product manager at VEGA Americas. They can be reached at g.tischler@vega.com and je.brand@vega.com.

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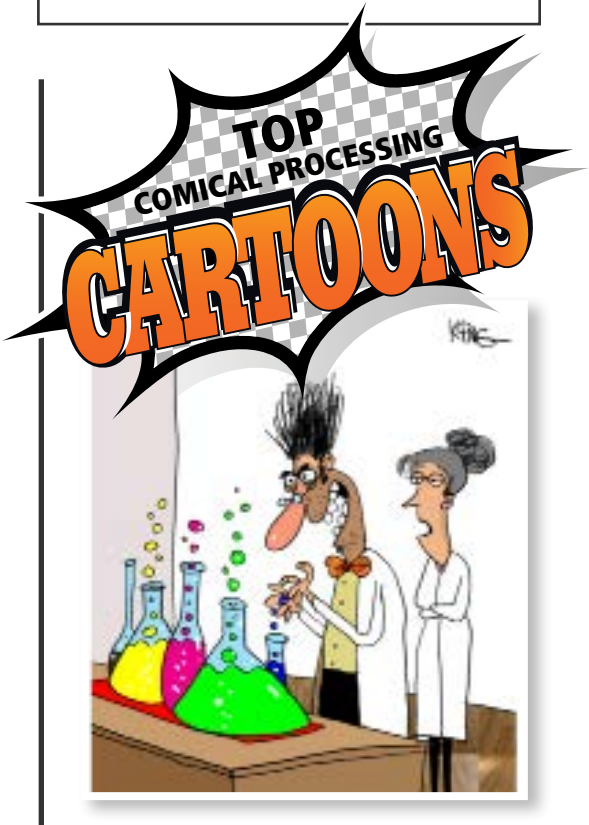

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