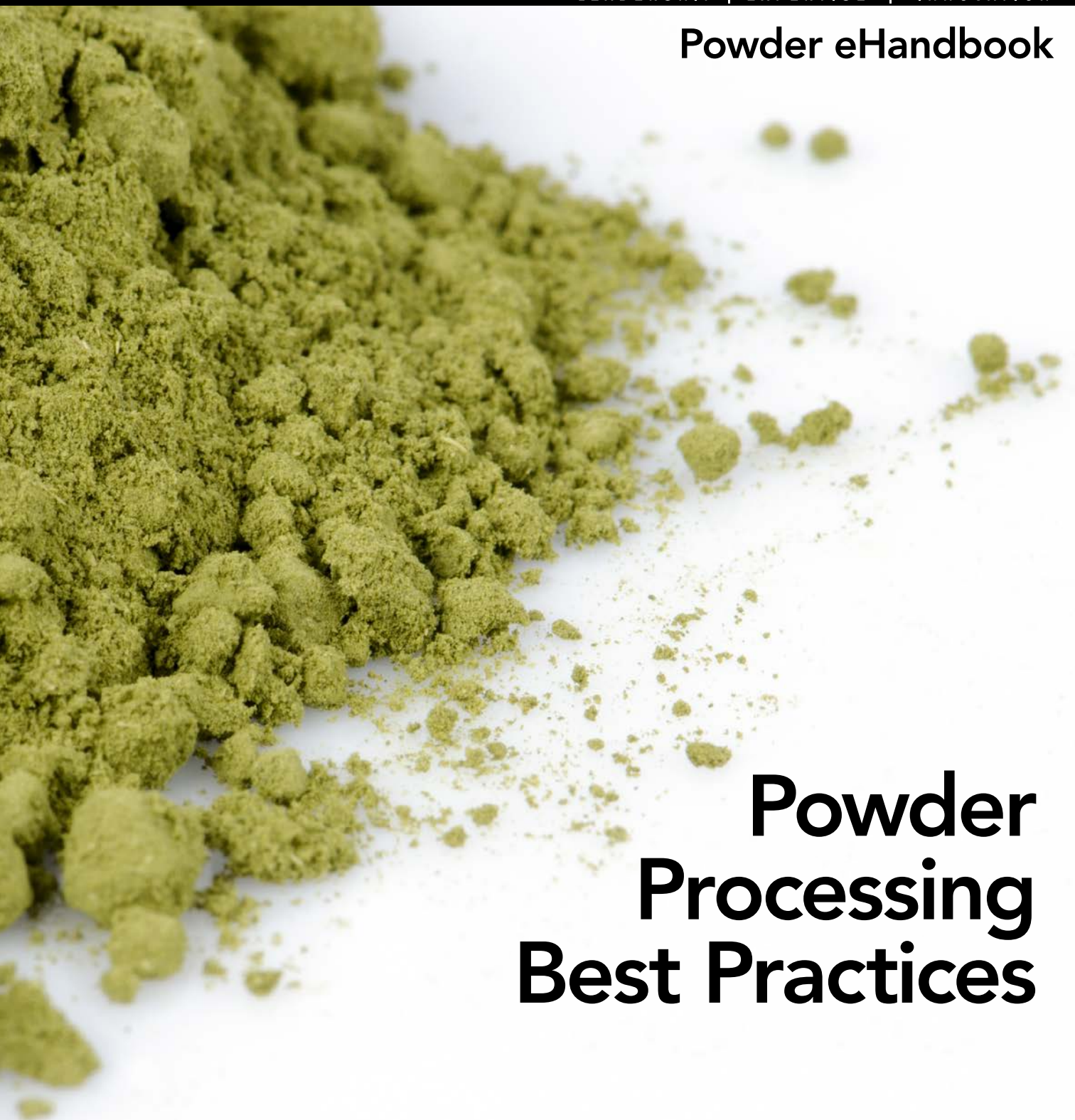


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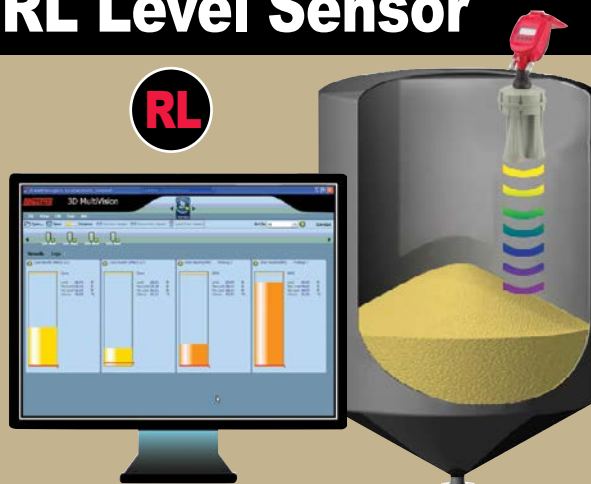
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## Food-Grade Hoses Handle High-Static Applications

Hose design helps dissipate static charges to ground.

**KURIYAMA OF** America's new line of Tigerflex Voltbuster food-grade material-handling hoses have been designed for high-static applications such as the transfer of powders, pellets and other granular materials.

The hose's design helps dissipate static charges to ground, helping prevent static build-up and reducing the potential for dangerous electrostatic discharges. They have been constructed with static dissipative plastic materials, allowing for the free flow of static to the hose's embedded grounding wire. The light-weight design of the hoses can help reduce injuries related to heavier metal hoses.

The "Volt Series" hose-tube construction includes abrasion-resistant food-grade polyurethane to ensure the purity of transferred materials. In addition, the grounding wire has been encapsulated in a rigid PVC helix on the exterior of the hose, eliminating the risk of contaminating the transferred materials. The VLT-SD Series is constructed the same, but has an FDA polyester fabric reinforcement to handle both suction and higher pressure discharge applications. New 2- and 8-in. ID sizes have been recently added to this product line.







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The product line is now approved for use with dusts, gases, hybrid mixtures and metal dusts. The Q-Rohr-3-6T and Q-Rohr-3-6T-AL are ideal for applications found in pharmaceutical, coatings, steel, iron and other industries. Click here for more info.

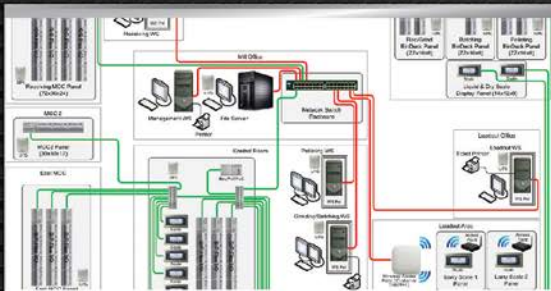
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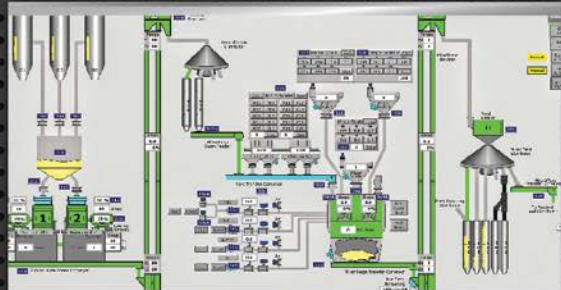
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# Particle Mysteries Take a Powder

Insights promise to enhance processing

By Seán Ottewell, Editor at Large

**MOST MANUFACTURERS** handling powders certainly would agree that achieving a better understanding of the materials' characteristics and how they change during processing could enhance the efficiency and safety of their operations. Fortunately, vendors are making solid progress in demystifying some key factors affecting powders.

For instance, Freeman Technology, Tewkesbury, U.K., and Medford, N.J., is looking beyond the physical properties of the particles and treating powder as a bulk entity. Using its FT4 powder rheometer, the company can evaluate and understand all the parameters that influence process performance.

"Our approach is to simulate the stresses, flow regimes and conditions that a powder may be subjected to in a process, for example, aeration, moisture absorption and electrostatic charging, and directly quantify its response," says Jamie Clayton, operations manager.

One project involved dynamic testing to meaningfully quantify differences in flow behavior between two different samples of sodium nitrite. The aim was to get the same properties in the second sample as in the first, commercially available, one.

A combination of dynamic powder testing and automated imaging showed the process for making the second sample needed modification to produce coarser particles with a smoother regular shape (Figure 1).

"The results here are not specific for sodium nitrite and in general could be true for a wide range of different powders. However, our experience tells us that when working with powders there isn't a one-size-fits-all solution; there is no such thing as a 'good' or 'bad' powder. The real question is, 'What is powder flow?'

because the answer will depend on the material in question, the process concerned and what the formulation scientist or process engineer needs to achieve," notes Clayton.

For example, the properties of powder necessary for efficiency in a bulk bag filling process may significantly differ from those required when manufacturing a pharmaceutical tablet. A common preconception in both cases is that the most-free-flowing powder is needed. However, in reality, a free-flowing powder can introduce its own problems such as dusting, segregation and flooding. So, the key is to identify powders that best suit a particular process and accurately quantify their properties.

"In some unit operations, such as tableting or pneumatic conveying for example, a more cohesive powder may actually be conducive to better results. In all circumstances though, it's important to recognize that any application will consist of a series of stages and, in order to achieve efficient processing and a high-

## PARTICLE SIZE ANALYSIS

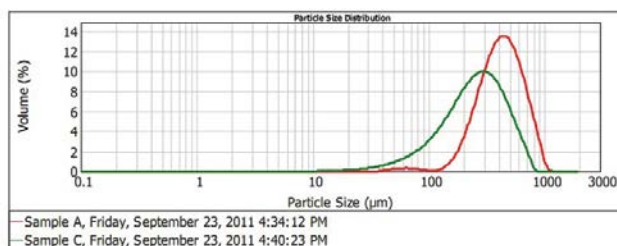


Figure 1. Dynamic powder testing and automated imaging identified the need to modify a process to produce coarser particles with a smoother regular shape. Source: Freeman Technology.



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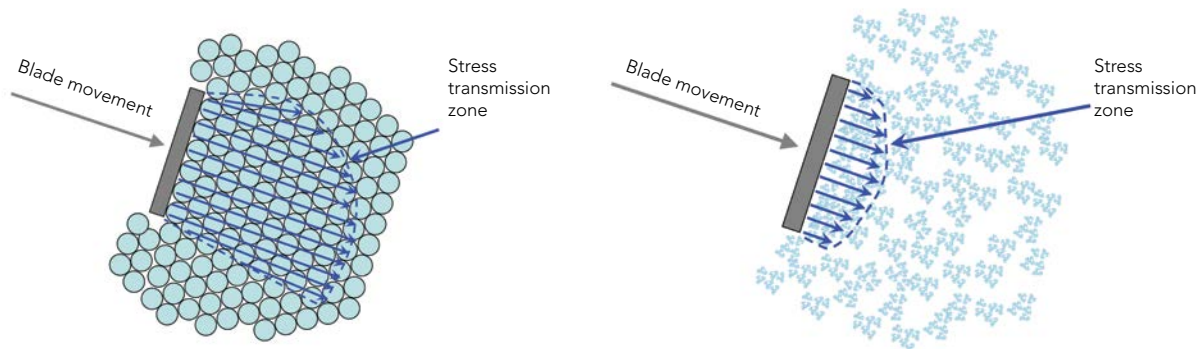


Figure 2. Efficiently packed large particles transmit blade movement through a much larger stress transmission zone than more cohesive powders. Source: Freeman Technology.

quality final product, a given powder must be compatible with all stages within the process,” he adds.

### MOISTURE’S ROLE

Another recent project focused specifically on controlling the impact of humidity on the powder flow properties of lactose and microcrystalline cellulose (MCC). “The main driver for this work was not only to gain a greater understanding of how humidity affects powder flow properties, but also to examine the general assumption that moisture is always detrimental to powder flow. The results illustrated how small quantities of adsorbed moisture can actually lead to an improvement in certain conditions,” Clayton explains.

The two materials were selected for study because they represent powders commonly used in industry and also exhibit varying degrees of hygroscopicity. The results demonstrated that even the small quantities of moisture adsorbed by powders considered to be hydrophobic could significantly impact flow properties.

“I think we always knew that assuming moisture adsorption is always detrimental and that so-called hydrophobic powders aren’t affected by humidity was over-simplistic, but this study allowed us to prove this and demonstrate how influential even minor changes in moisture content can be. It was particularly relevant that some of the greatest changes were seen across a relative humidity range that would be typical of most processing environments,” he notes.

The effect of humidity on powders can be very complex; the test results show how properties such as cohesion can prevail in certain circumstances while mechanical friction or interlocking can dominate

when the powder is subjected to different stress levels (Figure 2). This underscores why a multivariate testing approach is required to simulate the various stages within a specific process and understand how the powder responds.

A further study has looked at factors that influence blending kinetics, a subject still very poorly understood. Based on work carried out as part of a Ph.D. research project at the University of Birmingham, Birmingham, U.K., the study correlated positron emission tomography (PET) data with flow energy measurements. The results suggest that dynamic data may be a reliable predictor of blending behavior and, consequently, useful for developing and optimizing blending processes.

While PET primarily is used in academia and isn’t an established industrial tool, Clayton believes it provides a novel comprehensive method for evaluating mixing efficiency to determine optimum parameters such as speed and mixing time to achieve uniform dispersion.

“In this study we see how two different mixtures require different process equipment settings to achieve a predefined level of acceptance. These different requirements are likely to be due to the different physical properties of the constituent powders but after applying a range of powder characterization techniques, only the FT4 was able to identify and quantify these differences,” he explains.

The company says its reports and white papers — particularly those related to subjects such as caking or humidity — are generating a lot of interest from process engineers and formulation scientists across a wide range of industries.



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## DETERMINATION OF VOLATILE CONTENT OF COATINGS BY RAPID LOSS-ON-DRYING INSTRUMENTATION

	MAX® 4000XL (% Solids)		Convection Oven
Coating 1	74.644	Mean	74.404
	0.111	Standard Deviation	0.400
	14:17	Test time	2 hours

Table 1. Statistical Analysis of Clear Acrylic Resin

	MAX® 4000XL (% Solids)		Convection Oven
Coating 2	48.429	Mean	48.198
	0.261	Standard Deviation	0.377
	10:01	Test time	2 hours

Table 2. Statistical Analysis of Brown Acrylic Resin

	MAX® 4000XL (% Solids)		Convection Oven
Top Coat	60.317	Mean	60.028
	0.111	Standard Deviation	0.035
	7:20	Test time	2 hours

Table 3. Statistical Analysis of the Finish Coat with Additives

For testing paints and coatings, rapid loss-on-drying methods prove to be more desirable than traditional testing methods. Both testing methods were able to provide similar results, but the MAX® 4000XL was able to reduce testing times when compared to the convection oven, and gives a complete profile of the materials as they are being analyzed. This technology can be used to reduce manufacturing throughput times, and provide quality improvement with more information should formulation problems arise.

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“As a company we have experienced great success in the pharmaceutical industry where our technology lends itself well to recent initiatives such as process analytical technology and quality by design. However, the FT4 is used across a diverse range of industries including chemical, food, agriculture, powder coatings and toners. One of our existing users has recently demonstrated how minor variations in the raw ingredients and upstream process can have a major impact on the properties of household detergent tablets,” notes Clayton.

#### OTHER KEY PARAMETERS

Analyses of particle size and shape present their own challenges when it comes to understanding how different operating conditions affect various materials. The main push from its customers is to gain insights for improving product quality and consistency, notes Malvern Instruments, Malvern, U.K. Automation can play a key role in achieving these aims, it stresses.

In one project, the company carried out successful trials of an online particle size analyzer on an active pharmaceutical ingredient (API) milling operation at a commercial site of a major manufacturer.

Like many other API makers, the company used an iterative process that required numerous offline analyses of the milled powder to verify it met the correct product specification. Time consuming and wasteful, this procedure only can show that the properties of the batch are acceptable but reveal no information about manufacturing consistency.

Following the trials, the plant installed an Insitac online particle analyzer (Figure 3). This is linked to the mill's programmable logic controller by an Insitac PC to provide a fully integrated system; Malvern software



Figure 3. Device is fully integrated with mill, enabling automated control which has boosted quality and throughput and cut waste. Source: Malvern Instruments.

handles data exchange. The operator interacts with the PC via the mill's human machine interface and can input set points for the control loop, remotely start and stop the analyzer and mill, perform background tests, and receive particle size results.

The benefits of automating control include improved product quality, increased throughput and less waste — especially in the form of dust. Furthermore, automation opens up a route to semicontinuous operation and real-time release — important long-term goals in reaching operational excellence.

“This system has been used in many milling campaigns, some in excess of 20 tonnes, with consistent quality throughout. Together with streamlining operations and ensuring excellent quality, the embedded process analytical technology (PAT) also has enabled this company to reduce offline sampling — lab work — by

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an amount equivalent to 30% of its initial cost per campaign," says Alon Vaisman, Westborough, Mass.-based application development manager for Malvern.

As the technology moves from early adopters to more mainstream users, Vaisman notes that such PAT solutions are becoming very widespread in the chemical and other industries: "In my view this has become more of a requirement in the western market where competition is very aggressive and improving quality is a big driver. Whether you are talking about total quality manufacturing or Six Sigma, it's all about quality control."

In an effort to further improve its online automation, Malvern has launched specialized software to control the analyzer and peripherals, plus enable integration of the analytical data into the user's control system.

"In the future, I think that more of our work will focus on developing specialized software such as this in order to make integration easier," he adds.

Looking five years ahead, Vaisman believes the needs of the biopharmaceutical and nano sectors will spur new online analytical devices based on technologies now limited to the laboratory, and there will be a wider variety of online tools available than is currently the case. He also foresees traditional manufacturers responding to low-cost competition by opting

for instrumentation that now might be considered too expensive or not designed for such processes, for example as used in the food industry, to boost efficiency.

"There will also be further improvements in the ease of process integration. PAT projects are expensive today in terms of equipment and validation. I hope that an increase in standardization will lead to easier integration and faster validation," he concludes.

Of course, laboratory-based analytical techniques will continue to play an important role at many sites far into the future. So, a major study about how the reliability of analytical instruments and the reproducibility of their results changes over time by Wyatt Technology, Santa Barbara, Calif., a maker of light-scattering instrumentation, should interest many plants. The company focused on results generated by two of its size exclusion chromatography-multi angle light scattering (SEC-MALS) instruments, which are widely used when investigating how particle size and distribution can vary from batch-to-batch in different processes. The company used data collected from an independent analytical laboratory over a five-year period, an unprecedented procedure for such instruments, it believes. "The findings... found extraordinary reproducibility from day-to-day, month-to-month, and year-to-year," says Wyatt. ●

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## Scanners take Volume to a New Level

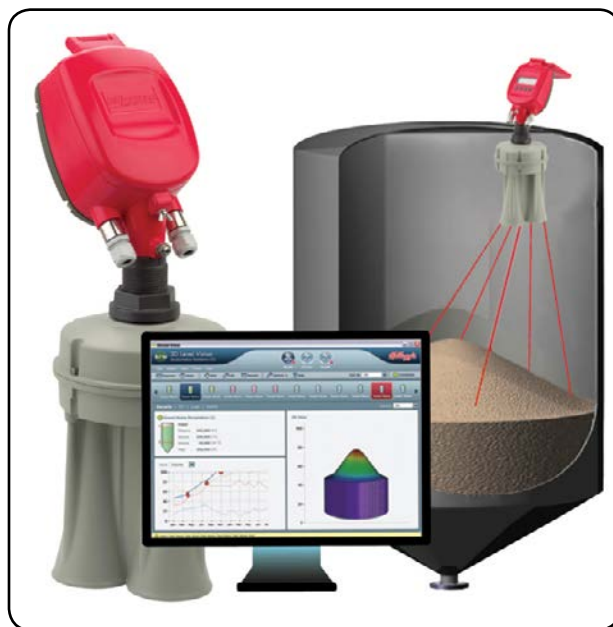
Acoustics-based sensors can help accurately measure the volume of powders in vessels

By Jenny Christensen, BinMaster Level Controls

**MANY DEVICES** are available on the market today for detecting the level of materials in bins, tanks and silos. However, for calculating what's in inventory sometimes level data just isn't enough. To estimate the amount of material on hand and the dollar value of that inventory, a single measurement point might not contain enough data to provide the accuracy needed. Due to their tendency to form irregular topography in the silo, this can be especially true in powders because most don't tend to flow freely. If inventory volume is based upon a single measurement from a single point in the silo, the volume estimate has the potential of being very inaccurate.

### MULTIPLE POINTS REALLY MATTER

Acoustics-based technology used in sensors referred to as 3DLevelScanners, or generically called scanners, is very different from other types of sensors. Like the name implies, these devices scan the material surface to take multiple measurements, taking into account the high and low spots in the silo. Scanning the surface also detects conditions such as cone up or cone down as well as buildup that may be present along the sidewall of the vessel. The data from multiple measuring points



is processed using advanced firmware and algorithms, and when combined with the silo's parameters loaded into the software, a highly accurate volume estimate can be provided. Additional data, such as the highest, lowest and average level of the material also is supplied.

#### SINGLE MEASUREMENT POINT

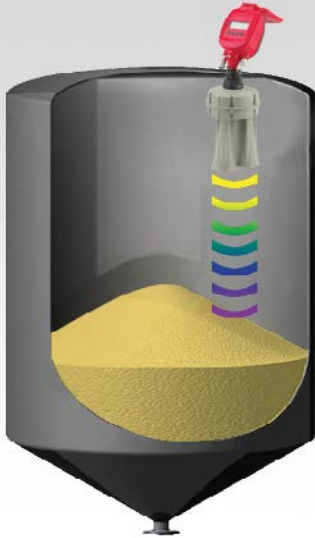


Figure 1. The RL model detects level in a narrow beam and is ideal for bins of all size where a single measurement point is adequate.

#### MULTIPLE MEASUREMENT POINTS



Figure 2. The S model measures multiple points within a 30° beam angle and is ideal for smaller silos.

#### PERFORMS RELIABLY IN HEAVY DUST

One of the greatest challenges powders present is the presence of excessive amounts of dust, which can render some types of sensors inaccurate or unreliable until the dust settles. By operating at very low frequencies, a scanner isn't bothered by dust and can perform consistently and reliably regardless of the conditions in the vessel. This technology has been proven in many different challenging materials such as alumina powder, carbon black, detergents, polyethylene powder, silica granules, fly ash and talc powder.

#### SELF-CLEANING SENSOR MINIMIZES MAINTENANCE

The unique design and materials used to manufacture scanners ensure that the surface resists the buildup of powders that are suspended in the air at the top of the vessel. Plus, the acoustic pulses make a "chirping" sound that resonates and creates an almost imperceptible vibration that helps keep the inside of the scanner clean. This way, the sensor stays clean and operational without the need for running an air purge to the top of the vessel, which can be costly.

#### STANDS UP TO CORROSIVE MATERIALS

When a silo is filled with highly corrosive materials, it's important to outfit the silo with a sensor that can stand up in the toughest industrial environments. For this type of specialized application a scanner that has a HALAR coating on the mechanical parts, VITON for the o-rings and stainless steel for any parts exposed to the material will work reliably over a long period of time. This option is recommended when the sensor is being used to measure corrosive materials such as quick lime, sodium chlorite, potassium hydroxide or other materials on the U.S. DOT Class 8 material list.

#### NON-CONTACT SENSOR PREVENTS CONTAMINATION

With an acoustic sensor, there's nothing that comes into contact with the material, making it very safe to use in powders, granules or other solids of all types. There's no risk of equipment being stuck in deep material or becoming



#### VOLUME MEASUREMENT

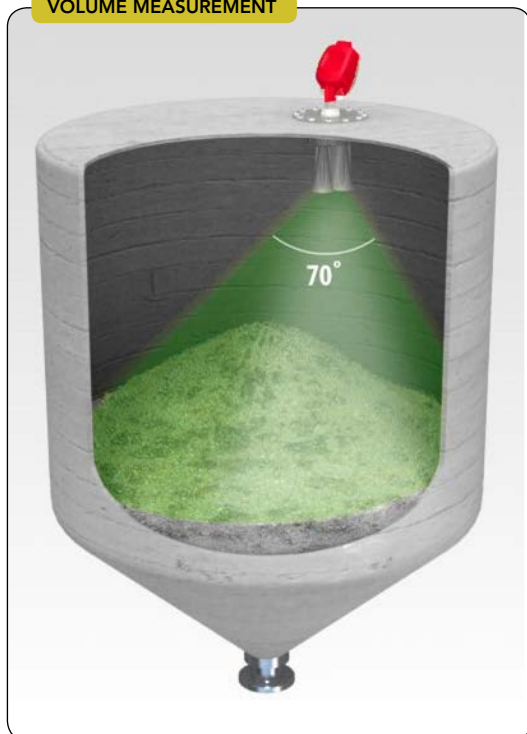


Figure 3. For larger silos, the M model measures multiple points within a 70° beam angle to accurately calculate volume.

detached and potentially damaging structure or equipment in the bottom of the silo. There's no need to replace probes or cables that can wear out over time. Eliminating contact with material helps ensure long life with minimal preventive maintenance or cost.

#### SAFETY INCENTIVES

When inventory monitoring is completely automated, there's no need to climb silos to take measurements. This not only saves time, it eliminates the risk of falls and the resulting injuries and the hassles of insurance and paperwork that comes with accidents. Plants today deal with rigorous OSHA requirements pertaining to climbing and entering silos and risk hefty fines when found in violation. Installing an automated inventory monitoring system allows for material management to be performed from a personal computer in the safety of an office.

#### LEVEL AND VOLUME MEASUREMENT

3DLevelScanners, like most other measurement devices, come in a variety of models and offer a wide range of options to tailor the device to the application. To select the right model for an application, it's extremely important to communicate your expectations of the technology and how you plan to use the data that you get from the device. Many plants are focused upon having a very high level of inventory accuracy. Accuracy of the scanner is driven by a number of variables, starting with information about the size of the vessel, the presence of structure inside it, and the material that's being measured. The installation location also is important as scanner technology measures multiple points, so the device needs to have a clear view of the material surface.

The most basic model of a scanner is referred to as an RL, which is short for reliable level. This model measures material in a narrow beam directly below the device, penetrating high levels of dust and performing where other types of level sensors become unreliable or inaccurate. This model is most often used when highly reliable level measurement data is needed on a continual basis. This model is frequently applied in materials that are less prone to excessive buildup and in narrow or smaller silos.

When more than simple level data is needed, the S model is designed to determine volume based upon an average level in the bin from multiple measurements taken within a 30° beam angle. This model often is used in narrow silos with diameters up to 16 ft and heights up to 200 ft. This model can also be used in wider silos, but with diminished accuracy as the 30° beam angle may not cover the entire material surface.

The M model takes measurements from a broader 70° beam angle making it appropriate for larger diameter silos and silos with uneven material topography. It also generates additional data including the lowest, highest and average distances based upon multiple measurements. Due to its ability to scan the material surface and take into account irregularities, it can provide a very high level of volume accuracy from between one and three percent.

At the top of the model spectrum is the model MV, which does everything the M does, plus adds a unique visualization feature. Using complex algorithms and a lot of processing power, this model generates a 3D image that indicates where the high and low spots are in the silo, shows if the cone is up or down, and detects sidewall buildup. This additional feature can be used to help manage filling and emptying points as well as detect if maintenance is needed to clean out buildup. This model is often used in large silos and in flour-like powders and other types of materials that tend to pile up unevenly.

#### OTHER MEASURES TO IMPROVE ACCURACY

If a bin is very wide or large, two or more scanners can be combined into a multiple-scanner system. By adding a controller that synchronizes the measurements from all of the scanners on a silo, the MVL model is able to provide very high volume accuracy in some of the largest silos. The number of scanners needed on the silo is determined by how large the silo is and the desired level of accuracy. The MVL also can generate a 3D visualization of the material surface from the measurements taken from all scanners. If visualization is not a requirement, an ML model supplies the same level of accuracy without the visual, making it more economical choice when budgets are tight.

Silos can be challenging structures to measure and often they come with some surprises that need a little “work around.” For example, sometimes there is structure in the top of the silo, so a measurement device might try to measure the structure instead of the material. Neck extensions have been designed for scanners that allow them to clear structure and see beyond it to get an accurate measurement of the material. Angled mounting flanges are designed to keep the device level, while angled mounting adaptors can be used when it’s necessary to aim the device in a problematic silo.

Although they have only been in the North American market for about five years, acoustic sensors have already revolutionized inventory management by adding the ability to accurately estimate the volume in a silo without ever leaving the safety of an office. The data derived from these devices improves operations in so many ways by allowing for timely replenishment and purchasing, reducing safety stock, and making inventory valuation far more accurate. For plant personnel, the scanner has been a game changer, keeping personnel safe from climbing silos and making them more efficient. Scanners truly have taken volume to a new level. ●

**JENNY CHRISTENSEN, MBA**, is vice president of marketing, for BinMaster. She can be reached at [jchristensen@garnerindustries.com](mailto:jchristensen@garnerindustries.com).

#### SELF-CLEANING SENSORS



Figure 5. The scanner on a powder silo is covered with dust (a). Inside of the silo, the outside of the unit also is coated with dust (b). However, the self-cleaning sensors inside the unit are clean and fully functional (c).



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## Determine a Coating's Volatile Content

Advancements in rapid loss-on-drying techniques significantly reduce testing time

By James A. Moore, Arizona Instrument LLC

**FOR 40,000** years people have been using paints and coatings for decoration, protection and camouflage in daily life. Early paints contained naturally occurring dyes and used egg yolks, linseed oil, waxes or other natural binders to help it adhere to surfaces. Changes have been made since early formulation, but the paints and coatings industry was revolutionized following World War II when toxic components, such as lead and mercury, began to be removed.

Today synthetic polymers, resins and solvents are used for paint production. These materials outperform their historic predecessors, showing an increased resiliency for weathering effects such as acid exposure, extreme heating and cooling, and water exposure from rain or snow.

Paint is a three component material made of a pigment, a vehicle and carrier. High quality paints are made from high quality materials, and manufacturers need to test materials to ensure that consumers will be happy with the products they purchase. To ensure they have the right blend of components, a manufacturer will run loss-on-drying assays, which examine the amount of material that will volatilize once it is heated.

### TRADITIONAL TESTING METHODS

ASTM International has dedicated method D2369 for determining volatile content of coatings using an oven method test. First approved in 1965, this method effectively determines the volatile content, but is not able to take advantage of new technology that reduces testing and throughput times, which would allow manufacturers to improve the efficiency of their metrology process and allow them to reduce product manufacturing times. Additionally, this method isn't able to provide real-time results for analysis, which would be helpful to immediately diagnose possible manufacturing problems.

### RAPID LOSS-ON-DRYING

Because of technological advancements in traditional loss-on-drying techniques, ASTM International adopted method D7232 in 2006. This method is designed to produce the same result obtained from testing using method D2369, but reduces testing time from 2+ hours to minutes. In addition, the Computrac® MAX® 4000XL analyzer provides in-situ measurements, giving metrologists the ability to diagnose manufacturing problems immediately.

## TESTING AND RESULTS

For this experiment three different materials (Table 1) were tested using ASTM method D2369 and D7232. For method D2369 a convection oven was used, and for D7232 a Computrac® MAX® 4000XL instrument was used. Between sample tests using the MAX® 4000XL, the coatings and pigment were shaken in their containers. A flat pan with paper was used for each test and the materials were placed onto the pan using a 5mL plastic syringe.

The data from the table shows that the two methods provide similar results. However, the MAX® 4000XL showed a significantly tighter testing result range for two of the three sets of testing.

Figure 1 shows the resin begins to volatilize around 100 seconds, suggesting that the idle temperature is cool enough to prevent the material from evaporating prior to data collection. Also, the tail of the rate graph shows that the material has lost all volatile content and the test is allowed to end. This is a typical graph for all three materials that were examined.

## CONCLUSION

For testing paints and coatings, rapid loss-on-drying methods prove to be more desirable than traditional testing methods. Both testing methods were able to provide similar results, but the MAX® 4000XL was able to reduce testing times when compared to the convection oven, and gives a complete profile of the materials as they are being analyzed. This technology can be used to reduce manufacturing throughput times, and provide quality improvement with more information should formulation problems arise. ●

**JAMES A. MOORE** is research chemist for Arizona Instrument LLC. He can be reached at [sales@azic.com](mailto:sales@azic.com).

## TEST METHOD RESULTS

### CLEAR ACRYLIC RESIN

	MAX® 4000XL (% Solids)		Convection Oven
Coating 1	74.644	Mean	74.404
	0.111	Standard Deviation	0.400
	14:17	Test time	2 hours

### BROWN ACRYLIC RESIN

	MAX® 4000XL (% Solids)		Convection Oven
Coating 2	48.429	Mean	48.198
	0.261	Standard Deviation	0.377
	10:01	Test time	2 hours

### FINISH COAT WITH ADDITIVES

	MAX® 4000XL (% Solids)		Convection Oven
Top Coat	60.317	Mean	60.028
	0.111	Standard Deviation	0.035
	7:20	Test time	2 hours

Table 1. Statistical analysis of three different materials show newer test methods, such as the MAX® 4000XL, can reduce testing times when compared to a convection oven.

## DETERMINATION OF SOLIDS FOR BROWN ACRYLIC RESIN

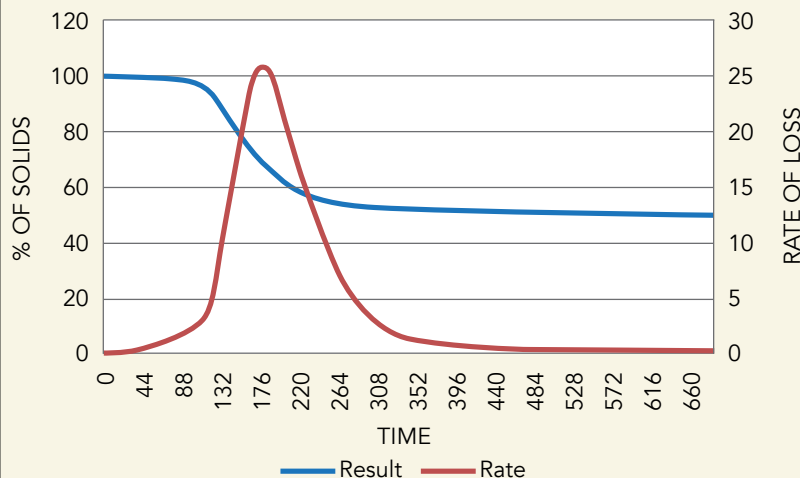


Figure 1. Real time measurements of volatile content and the rate of loss over time.



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## Test Powder Flow with Ease

Advances in test methods help better predict flow behavior at elevated temperature and humidity levels

By Dr. Erik Miller and Michael Olson, Solutia/Eastman Chemical Co., and Vinnie Hebert, Brookfield Engineering Laboratories

**ONE OF** the more difficult characterization tests for bulk solids involves testing at higher temperature and humidity levels. Many materials are susceptible to an increase in temperature and humidity levels, which could cause their flow properties to change. A material that was flowing easily at lower ambient temperature and humidity levels could now jam the feeder system when these levels rise. Too often, the way to address this flow problem involves a hit or miss proposition by merely adding in some flow aid compound, increasing the amount until flow improves, and hopefully before too much is added to affect the base material. Another solution is to fully alter process conditions, which can be a very costly modification.

This unscientific method is fairly common practice in industry. Part of the reason is the absence of an established characterization test for evaluating powders at elevated temperature and humidity levels. However, a test method does exist using a shear cell instrument modified to run at higher temperatures and humidity levels.

### SHEAR CELL BASICS

The shear cell has been utilized for defining powder flow properties accurately for many years. Its concept is well-defined in ASTM D6128 and accepted in the bulk solids industry. The initial design of the cylindrical Jenike shear cell has evolved to the annular Walker shear cell, which generates powder flow data much more rapidly.

With an annular shear cell (Figure 1), powder is sheared against itself at defined consolidation stresses to simulate the conditions when powder particles move downward in a bin

under influence of gravity. This annular configuration ensures the shearing action is uniform across the sample of powder. Information from this test is then utilized to create data for defining standard flow behavior properties such as flow function and bulk density. Mathematical calculations provide additional information on arching dimension and rat-hole diameter.

The flow function test provides a quantitative measure for flowability, far superior in usefulness compared to something like a subjective “cup test” or angle of repose measurement, which are, surprisingly, accepted and still used within many industries.

### TEMPERATURE AND HUMIDITY: THE PROBLEM

Many bulk solids are sensitive to heat and humidity. As these levels rise, either in the feeder system or via outside conditions, flow problems crop up. As the powder gains strength, jams and poor flow conditions begin to appear. Costly downtime and a loss of product are incurred while a search is made for a solution to the flow problems. Certainly, it would be much more cost effective to have the product defined at different temperature and humidity levels.

Solutia Inc., a subsidiary of Eastman Chemical Company, had just such a problem. Solutia’s product line includes a range of high performance PVB interlayers for automotive and architectural glazing applications. With manufacturing operations located around the world, the same PVB resin can encounter a varying process environment caused by geography and seasonal weather. Even within the same plant, the large scale of the operation can see the material moving from a cool climate-controlled pre-processing area, through a nearly tropical, hot operating area

#### ANNUAL SHEAR CELL



Figure 1: Components of an annual shear cell include the powder trough (a) vane lid (b) configured for uniform shearing action. A filling accessory (c) can help to fill the trough with the powder sample.

## POWDER FLOW TESTER



Figure 2: The Brookfield powder flow tester utilizes shear cell methodology and intuitive software to display flow data.

just upstream of the extruder. The matter is further complicated by the need to run a variety of resin types for different products. Clearly, the ability to quantitatively characterize all resin products across the known range of operating conditions was not only beneficial for Solutia, but a critical step in the continuous optimization of processing conditions and equipment.

## TEMPERATURE AND HUMIDITY: THE SOLUTION

After looking at various instruments for testing powders, Solutia settled on Brookfield Engineering's powder flow tester (PFT) (Figure 2), which utilizes shear cell methodology and intuitive software to display flow data. However, the PFT is rated for environmental conditions of 0°C–40°C at 20%–80% relative humidity (RH). While the temperature range was adequate, Solutia needed to ramp humidity levels to 95% RH. They approached Brookfield with the issue.

Solutia planned to place the entire tester inside of a steady state environmental chamber (Blue M CEO-916) to allow for testing at defined temperature and humidity levels necessary to characterize their product. The problem is that at levels of humidity past 80%, moisture would begin to form inside of the PFT instrument and cause the electronics to fail.

Working in concert with Brookfield, the solution was to fully seal the exterior housing of the instrument and add two purge fittings: inlet and outlet. These purge fittings would be used to pump cool air through the instrument while running a test at elevated temperature and humidity levels before processing begins. Brookfield initially tested the modified instrument and then turned it over to Solutia for more robust testing in their environmental chamber on their products.

## DATA RESULTS

One example of an interesting and somewhat unexpected finding provided by the ability to test at defined environmental

## PVB RESIN RESULTS

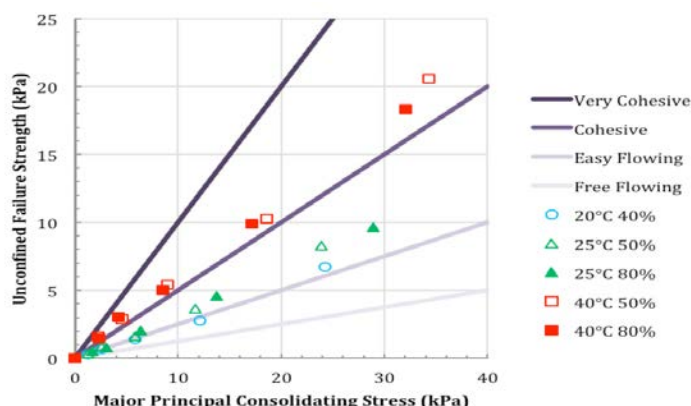


Figure 3: Flow function curves generated on the modified Brookfield PFT shear cell. The data are all for one type of PVB resin, at various temperature (°C) and humidity (%RH) conditions.

conditions is summarized by the flow function curves in Figure 3.

PVB resin, the main ingredient in a very strong yet flexible sheet product, isn't a free flowing material, and becomes less so with increasing temperature. For one particular resin, the flow function changed from easy flowing to very cohesive, spanning three flow regimes purely as a function of a 20°C temperature change. An unexpected result of this test was the relative independence of this resin's flow function to increasing humidity level. It was hypothesized that elevated humidity levels would compound the effects of higher temperature. However, results showed that this was not the case; temperature was the driving factor for flow function behavior. This one example shows the value that the modified PFT was able to provide. Knowing that humidity wasn't a strong influence on performance allowed R&D to concentrate on controlling temperature alone.

## CONCLUSION

When testing powder flow behavior at higher temperature and humidity levels, a solution does exist. The described method utilizing a modified Brookfield PFT has been running successfully at Solutia for almost two years. The data gathered has been valuable to ongoing process development.

Thanks to close cooperation between Solutia and Brookfield to address temperature and humidity level problems, a powder test method and solution for enhanced equipment design were developed jointly. This version of the PFT is now available to the general powder processing industry where similar temperature and humidity issues may require attention. ●

**DR. ERIK MILLER** is advanced mechanical engineer at Solutia/Eastman Chemical Co. He can be reached at [emill1@eastman.com](mailto:emill1@eastman.com). **VINNIE HERBERT** is senior sales engineer at Brookfield Engineering Laboratories, Inc. Contact him at [v\\_herbert@brookfieldengineering.com](mailto:v_herbert@brookfieldengineering.com).

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