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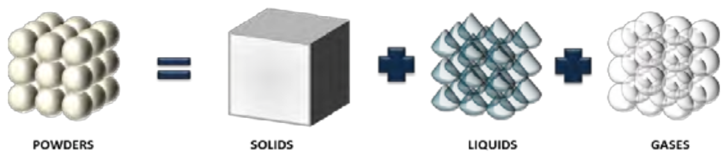

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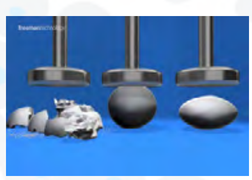

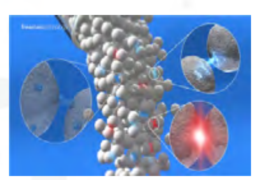

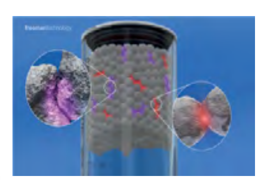

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Hidden Hazard Lurks

Facility finds danger from accumulated dust and effectively addresses it

By Cyrus Fisher, Eli Lilly and Company

COMBUSTIBLE DUST can pose a hidden hazard when accumulation occurs in unseen locations such as in mechanical spaces, above false ceiling, ventilation systems and dust collection systems. Such hazards may be particularly well hidden in certain pharmaceutical manufacturing facilities where use of clean rooms with surrounding mechanical areas are common and the scale of the equipment and facility is relatively modest. Even small quantities of combustible dust may result in a dust cloud flash fire or an explosion capable of significant damage in a plant environment. Although events of this magnitude may not make headline news, the potential impact on an individual present during a flash fire could be life changing.

So, here, I share an example that occurred at Eli Lilly and Company to show how combustible dust may become "hidden" within a dust collection system, and to describe a methodology for safe combustible-dust removal, as well as actions that can prevent future problems.

This example comes from a pharmaceutical blending operation located in a typical clean room. Technicians are preparing to blend 110 kg of dried pharmaceutical powder. All surfaces within the room are dust free and the polished stainless steel blender has just been cleaned. The technicians connect a small 2-in. ventilation trunk between the blender and a port on the clean room wall labeled "to dust collector." The technicians then open the access cover of the blender and press a button to start the dust collector, which is located elsewhere. Seven bags, each containing 16 kg of dried powder, are charged to the blender through the opening. The technicians are wearing personal protective equipment (PPE) to prevent inhalation of the dust but no dust is observed outside the opening. When the product charge is completed, technicians turn off the dust collector and disconnect the 2-in. ventilation trunk. The trunk is visually clean. The self-contained blending operation completes normally. All equipment and the room itself then are cleaned in preparation for the next batch. Lastly, the technicians leave the clean room to check for accumulation of material in a small drum under the dust collector; the drum is empty as always. The technicians know the routine well; they have completed these tasks at least once a week for the last ten years.

By their training, the technicians understand the powder they are handling is a combustible dust. They know the minimum ignition energy (MIE) has been tested at approximately 200 mJ with an average particle size of 27 microns, which means the risk of ignition from an electrostatic discharge from personnel is greatly reduced, and personnel grounding isn't required [1]. The electrical outlets and switches in the clean room look different from others in the area, and signs hang on the doors indicating the room is electrically classified as Class II, Division II for combustible dust. If technicians observe a dust cloud for any reason (e.g., a dropped product bag), they are to immediately leave the area until the cloud settles. In general, technicians believe little if any dusting occurs during loading of product to the blender — a belief supported by the lack of dusting seen during blender loading and emptying the dust collector discharge drum.

The technicians and technical support personnel assumed that because no dust is coming out of the dust collector, no dust is going in. The assumption was

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	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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Figure 1. Inspection revealed that interior of dust collector contained an accumulation ½- to 1-in. thick.

widely believed to be true and even documented in a previously completed formal hazard review. The idea that dust accumulation might be possible simply did not occur to those supporting the blending operation.

A TELLING INSPECTION

In 2012, the facility initiated a hazard review process for all solids handled at the site. This included looking specifically at the dust accumulation risk for each operation. One recommendation stemming from this activity was for engineering to perform an internal inspection of the blending operation dust collector.

Prior to the inspection, the team reviewed available design information for the dust collector and field-verified all ductwork. The system was designed for an airflow of 500 ft^3/min to ensure sufficient capture velocity at the blender opening during loading. The ductwork in the field begins at the clean room wall, where the duct diameter increases from 2 in. to 4 in. and then transitions to a diameter of 6 in. immediately prior to a 15-ft vertical riser. The duct then travels horizontally several hundred feet through multiple mechanical rooms before reaching the dust collector inlet plenum. Portions of this ductwork run above false ceilings. At the inlet plenum, the 6-in. duct expands to a 1-ft × 3-ft rectangle at which point it enters the dust collector. That unit, which is 1 ft in diameter and 3 ft in length, contains four cartridge filters. The dust collector is equipped with a differential-pressure pulsation system to clear the filters under conditions of high pressure drop. At the bottom of the dust collector, a manual slide gate valve leads to the aforementioned drum for dust disposal.

During the engineering inspection, the four cartridge filters were removed and found to be heavily loaded with dust. Internal inspection of the dust collector revealed ½-in.+ layers of dust settled on all horizontal surfaces including the inlet plenum (Figure 1). Samples were taken and submitted for particle-size and MIE testing. The average particle size of the material in the dust collector was 12 microns, half the size of the bulk powder loaded to the blender. That in itself isn't surprising because the dust collector air stream primarily captures fines churned up during blender loading. The MIE for the material in the dust collector was approximately 25 mJ — an order of magnitude less than that of the bulk powder loaded into the blender! With an MIE as low as 25 mJ, the risk of ignition from electrostatic discharges becomes a greater hazard, necessitating enhanced safeguards including personnel grounding [1].

Upon discovery of this fine collected dust, planning commenced for its removal. Engineering personnel led the effort and got assistance from maintenance and operations. The cleaning scope included both the main body of the dust collector and all impacted ductwork. Engineering developed a written cleaning plan. A hazard review team then performed a risk analysis of the proposal. Hazard review teams are routine at this facility due to the significant quantities of solvents utilized. However, site personnel were relatively inexperienced with combustible dust remediation. To ensure a robust review, corporate combustible-dust subject matter experts and the contractors selected to perform the cleaning joined site engineering, operations, maintenance and health/safety personnel to perform a what-if risk analysis of the written cleaning plan.

Using photographs from the field, engineering went over the entire dust collection system with the review team. The MIE data obtained for the dust then were used to list types of ignition sources that would have sufficient energy to ignite a dust cloud if

Figure 2. Ground wire was installed to prevent isolation of metal during cleaning of 4-in. duct.

one formed during the cleaning operation. The hazard review team next focused on two specific areas for risk reduction: 1) identifying safeguards that would prevent/minimize/contain disruption of the dust to prevent formation of a combustible dust cloud during cleaning; and 2) identifying safeguards to minimize all possible ignition sources in the event a combustible dust cloud inadvertently was created.

To minimize the risk of creating a dust cloud, the cleaning plan incorporated multiple recommendations from the hazard review team. First, the order of line breaks and cleaning activities were specified so as to remove dust from easy-to-access areas prior to performing higher-risk line breaks. The goal was to remove as much fuel from the system as possible before performing overhead work with reduced egress options. This included removal of the filter elements and cleaning of the dust collector prior to disassembling overhead ductwork. Second, extra ductwork supports were installed. Adding these supports ensured the ductwork couldn't accidently fall as it was disassembled, disturbing settled dust and potentially forming an ignitable dust cloud. Third, plastic sheeting and glove bags (similar to those used for asbestos remediation) isolated rooms and line breaks. These actions ensured that any dust disturbed wouldn't be able to travel outside the boundaries of the work area, where measures to enhance protection against ignition also were being put in place.

Potential ignition sources were categorized, e.g., charge on metal surfaces (scaffolding, ductwork, etc.), charge on personnel, charge on tools, the vacuum to be used for cleaning, and surrounding electrical equipment. Again, the cleaning plan incorporated multiple recommendations from the hazard review team. Grounding wires were installed in multiple predefined locations including the ductwork (Figure 2), dust collector, scaffolding and any other potentially isolated metal surface. Engineering inspected the contractor air-powered HEPA vacuum equipment. Prior to the cleaning, which took place in August 2013, all operating equipment in the work area was shut down, and an extensive lock-out/tag-out was performed for all electrically powered equipment. Lock out of electrical equipment was accomplished remotely in motor control centers or at electric breaker panels away from the work area. Equipment locked out included motors, heaters, power outlets and control panels. Immediately prior to performing work, engineering met with contractors and maintenance personnel to review the cleaning plan, PPE requirements, and combustible dust hazards. All personnel were instructed to leave the area in the event of a dust cloud. "Danger" tape isolated the entire area; technicians posted at all entrances kept personnel out of the cleaning area.

The planning and coordination for the cleaning activity took several weeks but the cleaning itself required less than six hours. Approximately 10 kg of combustible dust were removed from the system and collected as a wet paste in the bottom of the contractor's vacuum equipment. After cleaning, engineering inspected all ductwork, which was in like-new condition.

PREVENTING FUTURE PROBLEMS

Engineering initiated a root cause investigation into why dust had accumulated and what needed to be implemented to stop accumulation from occurring in the future. The root cause investigation identified two causal factors.

First, designers had inaccurate/incomplete process safety information when the dust collection system was installed over a decade prior to this event. Preliminary design documentation erroneously indicated the product wasn't combustible. As a result, the dust collector system design didn't incorporate standards applicable to combustible dust (isolation/suppression systems, housekeeping program, etc.).

Second, multiple opportunities to identify the risk of accumulating material were missed even after

the material was confirmed to be combustible. One opportunity came after several years of service when an initial combustible-dust hazard assessment was completed on the blending operation/dust collector. At the time, the facility had minimal organizational knowledge regarding combustible dust hazards. Technicians interviewed then stated that little dusting occurred during loading of the blender and no dust ever was discharged from the dust collector. These types of observations prompted the review team to conclude that no dust was being pulled into the dust collector system. The root cause investigation found these observations/conclusions to be inaccurate. The lack of dusting at the blender was due to the successful operation of the dust collector (i.e., dust is pulled away from the operator as intended). The failure to discharge material from the dust collector was traced to a mechanical problem with the internal pulsation system, which likely never had functioned following initial installation. This explained the heavy loading seen on the filters.

Another opportunity to recognize that dust was accumulating arose during completion of routine airflow testing. The investigation found that a 50% drop in airflow was documented in the work history of the dust collector but not flagged as a potential dust-collector operations issue. The reduced airflow rate of 250 ft^3/min sufficed to maintain operator protection from an industrial hygiene perspective, so no actions were taken to restore the airflow to the original design requirement of 500 ft^3/min. The reduced flow and, thus, duct velocity accelerated accumulation. Generally, preventing the settling of materials similar to this product requires a minimum airflow rate of 2,500 ft/min [2]. At 250 ft^3/min, the dust collector system was operating well below this minimum velocity in the 6-in.-diameter line that accounted for the majority of the ductwork in the system. In some cases, nearly 50% of the duct cross-sectional area was found to be plugged, particularly near the bottom of vertical risers where dust settling was prevalent.

Recommendations from the root cause investigation included: upgrading the system design to be suitable for combustible dust service; implementing routine internal inspections; establishing pass/fail criteria for duct velocity measurements; modifying duct sizing to increase airflow velocity; and setting up a program for regular internal cleaning.

The key takeaways from our experience are:

• Accurate material properties are essential for making informed risk-based decisions whenever handling combustible dust. The properties of a specific combustible dust material can vary greatly with changes in particle size. In our case, a 50% reduction in particle size resulted in an order-of-magnitude decrease in MIE and, thus, a far greater risk of a combustible dust flash-fire/explosion. Failure to understand this reduction in MIE might have resulted in less-stringent safeguards during development of the cleaning plan.

• Having all affected parties and subject matter experts take part in performing a thorough hazard analysis is invaluable in confirming that a written plan provides the safest possible path forward for executing a non-routine activity.

• An effective prework safety meeting ensures work is completed in the manner intended by the hazard review team and also provides a final opportunity to address concerns of those performing the work.

In the end, a significant amount of resources went into the uneventful cleaning of a small quantity of accumulated material. The results of the cleaning activity and subsequent investigation were communicated in multiple forums across the organization. Many committed team members actively participated in completing this work. Hopefully, this simple example results in positive outcomes for others vigilantly working to reduce combustible dust risk.

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2. "Industrial Ventilation," 25th ed., American Conf. of Governmental Industrial Hygienists, Cincinnati, OH (2004).

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Benchmark Powder Flowability

New measurement methods now make it easier to evaluate flow

By Robert McGregor, Brookfield Engineering Laboratories, Inc.

PLANT MANAGERS and operations personnel know from experience that certain powders will have flow difficulty. Some can tell by either visually inspecting a powder or feeling it to the touch that processing problems are inevitable. Perhaps the relevant question is: "How great will the challenge be to get the powder to flow through the process?"

Traditional methods for evaluating flow — flow cup, angle of repose measurement, tap test, particle size analysis — have been hit and miss. They actually evaluate physical properties that may contribute to flow behavior, but do not assess flowability in and of itself. Shear cells are becoming the instrument of choice to do this job. Figure 1 shows the basic components for an annular design shear cell. The cell requires a small volume of powder sample, uses a vaned lid to compress the particles together to a defined consolidation stress, and then shears the particles against one another to quantify the frictional resistance for relative movement.

This basic technical approach was scientifically established more than 50 years ago in the minerals industry by the Jenike Shear Cell and led to the creation of ASTM method D6128.

A second test involves use of the wall friction lid (Figure 1c) to measure the frictional resistance of powder sliding down the hopper wall before exiting the bin. The wall friction lid also is useful for running a powder compressibility test which quantifies how density increases from initial "loose-fill" conditions to higher density values as compaction pressure increases.

MODERN TECHNOLOGY

Improvements in instrument design coupled with the PC revolution of the late 20th century have led to

ANNULAR SHEAR CELL

Figure 1. The basic components for an annular design shear cell include the trough with powder sample and filling accessory (a), vane lid (b), and wall friction lid (c).

POWDER FLOW TESTER

Figure 2. A typical shear cell can fit easily onto the work bench in a QC or R&D lab and can process a single sample in as little as 12 minutes.

FLOW FUNCTION GRAPH

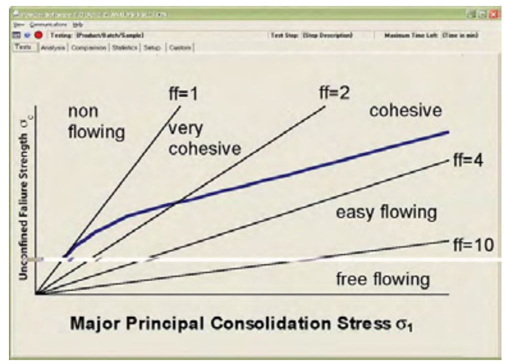

Figure 3. The graph quantifies failure strength (y-axis) versus consolidation stress (x-axis) with defined regions for types of flow behavior.

modern shear cells that are affordable, easy to use, run automatic tests quickly, and provide useful analytical measurements for comparing powders. Figure 2 shows a typical shear cell that fits easily onto the work bench in a quality control (QC) or research and development (R&D) lab and can process a single sample in as little as 12 minutes. Rapid evaluation of flow properties using shear cells is a major revolution in the technology that makes this method practical for everyday use.

FLOW FUNCTION TEST

The flow function is the fundamental test used to evaluate flowability. Standard flow function tests measure the powder sample at five or more consolidation stresses and record the failure strength in each case. (Failure strength is the resistive force or static friction between particles that must be overcome before relative movement can take place.) The data points from the test generate a flow curve similar to the graph shown in Figure 3. Industry has defined regions of flow behavior as indicated, ranging from "non-flowing" to "free flowing". So the flow function offers a convenient tool to quantify the flow behavior of a given powder and variations on that particular formulation.

In order to rapidly characterize a powder, certain pieces of information derived from the flow function may be used for bench-marking purposes:

a) "Flow Index" is the slope of the line drawn from the data point associated with the 5th consolidation stress to the origin.

b) Arching Dimension is the minimum value required for the hopper opening to prevent bridging of the powder in "mass flow" behavior (powder flows downward uniformly toward the hopper opening).

c) Rathole Diameter is the stable annular ring of powder that can form in the bin during "core flow" (also known as "funnel flow") behavior. The size of the ring may vary in diameter as a function of the powder height in the bin.

WALL FRICTION TEST

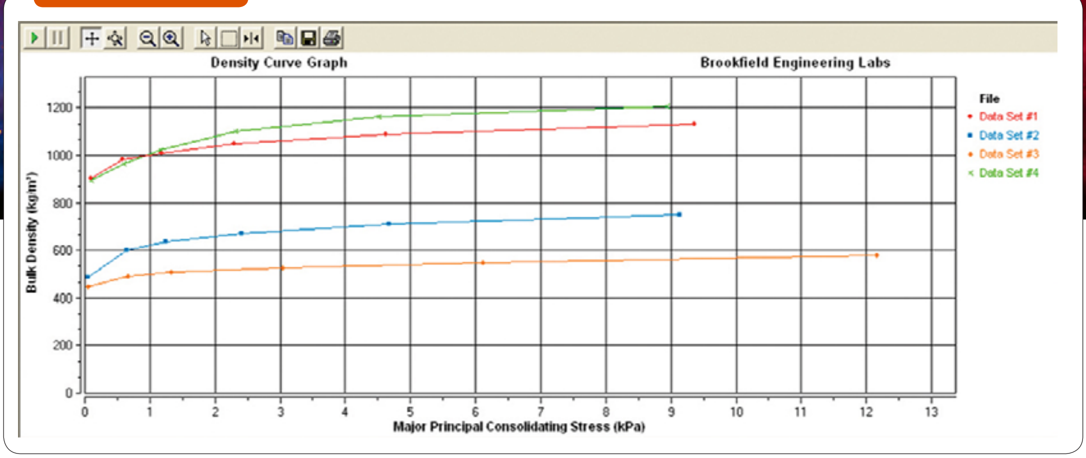

Figure 4. Data reveals the change in powder density with increasing consolidation stress for four different powder samples.

WALL FRICTION TEST

The wall friction test is performed in a similar fashion to the flow function test. Material of construction for the lid is similar to the material in the hopper wall; possible choices may include mild steel with 2B finish, stainless steel, or various plastic materials. Consolidating pressure is applied by the lid to the powder in the trough. The frictional resistance of the powder sliding against the lid at a defined consolidation stress is measured. The process is repeated at increasing consolidating pressures.

Data resulting from the wall friction test gives two important pieces of information.

a) Compressibility ratio is the change in powder density from "loose-fill" condition before start of test to maximum density at highest consolidation stress. Figure 4 shows the graph that provides the raw data to make this calculation.

b) Hopper half angle required to achieve "mass flow" behavior can be calculated when wall friction and flow function data are combined. (For "very cohesive" powders, this angle may be too steep to be practical.)

BETTER FLOW BEHAVIOR

Software used with shear cells will automatically compute all of these values. The person who operates the instrument only requires instruction in proper method for filling the trough with powder. The R&D scientist or engineer can use the test information to evaluate the effects of variations in formulation. Improvements in flow behavior are possible with selection of ingredients that have better flow capability. Use of generic flow aids may also be considered. Operations personnel may decide that modifications to bin design (hopper opening and half angle) are needed to improve flow behavior. The QC department can run quick tests to qualify raw incoming materials used in the formulation. Final product after processing can be tested for compliance with established flow behavior limits for acceptability.

Bench-marking powders for flowability has now become possible with the regular use of measurements from the tests described above. QC may be the important beneficiary when using this new capability because observations and judgment for acceptable product need no longer be subjective. The manufacturer is the ultimate winner because consistent product with predictable flow behavior will now ship to all customers.

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Weigh In Process Safety

A properly designed weigh model can optimize safety and improve efficiency

By Michael Sutton, Mettler Toledo

PROCESS DESIGN and process safety are critical considerations in chemical production and processing. With design and safety paramount at the outset of any new development or equipment retrofit, firms can minimize risk exposure, maximize productivity and position themselves to remain compliant and competitive. Advanced automation technologies continue to drive productivity improvements.

In addition, automating certain industrial tasks reduces people's exposure to workplace hazards. One area that has experienced technological advancements in safety is industrial weighing. Today's weigh modules, which can be used to convert most tanks, hoppers or other vessels into a scale, have been designed to maximize safety without sacrificing accuracy or reliability.

Regulatory scrutiny and employer liability increase the need to evaluate every part of a production facility. The current generation of weigh modules has been designed to improve performance, safety, ease of selection, installation and commissioning. To maximize the value of these safety features and design enhancements, these products were also designed to allow for installation by someone without advanced scale knowledge.

Older or poorly designed weigh modules can compromise the manufacturing process in various ways. Weight data —used in batching, filling, inventory control or other applications — are entirely dependent on the accuracy of the weigh module. A poorly designed weigh module can negatively impact product quality and production yields and diminish organization efficiency.

In addition, an outdated or poorly designed weigh module can cause safety hazards and possible compliance violations. Without extensive experience and expertise, designing a weighing system that takes advantage of today's advanced features can be challenging. Organizations should consider design integrity, load-cell quality, safety requirements and shipping and installation features to make sure they are meeting all requirements.

DESIGN SAFETY

Not all weigh modules are created equal. Some weigh modules have been carefully designed and tested to maximize safety, accuracy and efficiency. Other weigh modules only appear to have these features. It is important to recognize the difference. While the weigh module does weigh the scale, it is also an integral component of the overall support structure.

The weigh module obtains weight as a measurement of vertical force applied by the vessel and contents. This means it is often the only connection between the vessel and the ground. The weigh module

and application must be matched so the vertical force does not exceed the maximum capacity of the load cell. But as the primary connection to the ground, the weigh module must also provide resistance to lateral forces to prevent tipping. So, while the design is important to accuracy, it is critical to safety as well.

Organizations should seek weigh modules that offer anti-lift protection as a standard option. As a field-installed option, anti-lift protection is often overlooked on site. This can be a critical safety feature in windy outdoor conditions or any installation where vehicle traffic can impact the scale. The anti-lift feature protects the scale from tipping. For maximum safety, the anti-lift feature should function without a load cell installed. Seek weigh modules with vertical down-stops to prevent tipping in the event of any suspension hardware failure for added security.

Organizations also should conduct Finite Element Analysis (FEA), a computer-based structural analysis method, in the design of any weigh module. With satisfactory results from computer modeling, the process moves to the production of prototypes.

Prototype testing should first establish that the design can meet the desired specification for both vertical and lateral forces. If the requirements are met, conduct additional testing to identify upper limits of the design capabilities and the failure point. Pursuing design to this level ensures that safety and performance requirements are fully met. The last step is to create drawings, load ratings and installation documentation so the weigh module can be safely and easily integrated into the overall scale design.

In hazardous areas, load cells and weigh modules must also achieve correct certifications to satisfy the requirements of the appropriate governing bodies (FM, ATEX, etc.).

EVALUATING LOAD CELLS

There are several "types" of load cells that are marketed as appropriate load cells for a weigh-module system. But load-cell selection must include a variety of factors. First, make sure the load cells have NTEP, OIML, NEMA and IP ratings to match the application in which they will be used (Figure 2). Beyond basic certifications, there are a variety of other factors that impact the system that should be considered.

LOAD CELL WITH ROCKER-PIN SUSPENSION

Figure 1. A self-aligning rocker-pin suspension design allows some degree of movement to occur without causing damage to the load cell or changes to its accuracy.

Thermal expansion/contraction can create a push/pull force on the vessel supports. In high-traffic production areas, vessels are susceptible to accidental side impacts. Wind forces on exterior tanks or even vibrations from a mixing agitator can reduce load-cell accuracy. Look for a design that takes these and other factors into consideration to achieve the best result. There are load cells available that contain safety or compensation features to deal with those factors.

An example is a self-aligning rocker-pin suspension. This design allows some degree of movement to occur without causing damage to the load cell or changes to its accuracy. In addition, this feature and similar options always return the scale to the ideal weighing position, ensuring repeatability and the highest level of accuracy.

This rocker-pin type suspension in its simplest form allows only limited movement — generally

bidirectional only. A more robust design would typically provide a 360° range of motion to allow expansion/contraction in all directions. This type of design needs to include 360° checking with sufficient strength to stop the scales' movement in all directions to prevent load-cell damage or even tipping.

SHIPPING AND INSTALLATION

In addition to advancements in performance and safety, the modern weigh module design may improve the installation process, resulting in a more robust installation that may be easier to achieve than previous generations. Engineers and product designers have considered every element of the weigh module, including the logistics between production and installation. Look for a weigh module that can be delivered to a site in a shipping/installation "mode." In this mode, the various components are locked in the manufacturer's ideal initial positions in a way that isolates the load cell until installation is complete. In addition to preventing accidental damage to the load cell, this feature also helps to ensure load introduction and equal top-plate travel in all directions upon commissioning (transfer of weight to the cell).

Some products offer alignment and rigidity that is independent of the load cell. In this scenario, the load cell can be installed after rest of the process is complete. This feature has also been designed to allow for quick and easy replacement of a load cell in the event of a failure.

Weigh modules that offer a "shipping/installation" mode also have been designed to provide greater flexibility in the overall design and construction process. With the safeguards that prevent forces to be transferred to the load cell in place, they can fixed to the foundation before the tank is lowered into place or they can be installed to the tank legs at any time before it is lowered to the foundation, even before the tank is shipped to the site.

Following installation, it should be easy to convert the weigh modules from shipping/installation mode to weighing mode in preparation for calibration.

FACTORS TO CONSIDER

Figure 2. When selecting load cells, make sure they have NTEP, OIML, NEMA and IP ratings to match the application in which they will be used.

SUMMARY

Weigh modules are an important component of a weighing system. When selecting one, opt for the modern features discussed here to ensure long equipment life and the best performance. Versatile and feature-rich weigh modules can simplify installation and ensure safety throughout their lifetime.

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Take a Multivariate Approach to Powder Processing

Several methods can help determine which powder properties have the greatest influence on performance

By Jamie Clayton and Brian Armstrong, Freeman Technology

POWDERS ARE widely used in industry. However, they are arguably the most difficult materials to characterize and understand, as evidenced by the many problems encountered in manufacturing processes and final product quality.

This is primarily due to the combination of phases that comprise a powder; solid particles, interstitial gases (typically air) and liquid (e.g. moisture from the atmosphere or a component from upstream processing). The interaction of these phases contributes to the complexity of powder behavior, making the processing of powders equally complex and challenging to predict. In addition, variation in the physical properties of particles contributes to the overall variability of a powder. The range of processes used to manipulate powders subjects them to extremes of stress— from high compaction loads in hoppers, to the dispersed state in fluidized bed dryers and pneumatic conveyors (Figure 1).

The key to successful powders processing is to ensure that a powder's characteristics are suited to the process. This approach demands a thorough understanding of how a powder behaves over a range of conditions, such as in a stationary position, in motion or about to move. Relying on a single, simple parameter is unlikely to provide the required information and result in process interruptions and poor product quality [2].

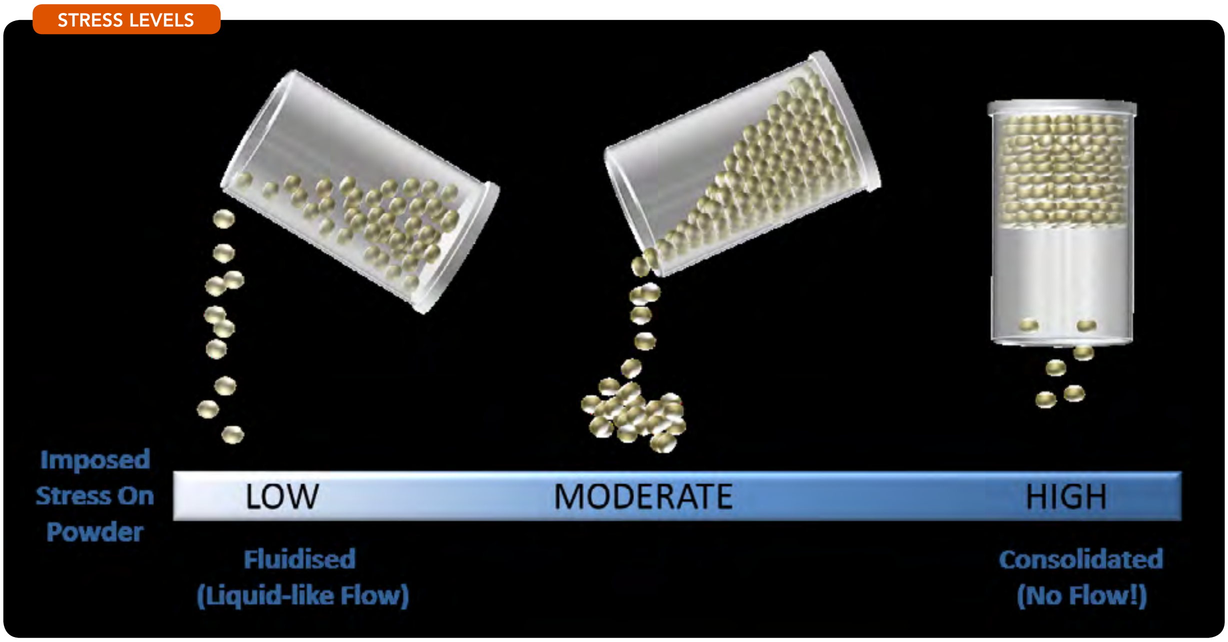

Figure 1. This demonstrates the behavior of powder flow over a range of stress conditions.

WALL FRICTION ANGLE VALUES

DISK NO.	MATERIAL#	SURFACE FINISH	NOTES	R_a VALUE (μM)	WFA (°)
1	SS 316 L	Brushed		0.19	27.5 ± 0.2
2	SS 316 L	Satin finished		0.28	28.8 ± 0.4
3	SS 316 L	Electropolished		0.35	31.5 ± 0.0
4	SS 316 L	Glass pearl treated		0.45	29.0 ± 0.1
5	SS 316 L	Ground, fine		0.61	29.6 ± 0.1
6	SS 316 L	Brushed		1.2	31.0 ± 0.3
7	SS 316 L	Rhenolease MK V	Conductive, PTFE-based	1.85	24.8 ± 0.3
8	SS 316 L	Titanium nitride		N/A	32.1 ± 0.1
9	SS 316 L	CrNi-Coating		N/A	34.8 ± 0.1
10	SS 316 L	NEDOX SF2 coating	Nickel / polymer	0.7	28.1 ± 0.0
11	Aluminium	Tufram coating	Anodized, Polymer	0.91	27.6 ± 0.3
12	Aluminium	Hard slide HSS	Anodized, Polymer	N/A	27.8 ± 0.0
13	PEEK	Milled	Polyetheretherketone	2.39	24.3 ± 0.4
14	POM-C	Milled	Polyoxymethylene copolymer	0.06	13.0 ± 0.1
15	PETP	Milled	Polyethylene terephthalate	2.1	19.5 ± 0.3

Table 1. The table includes descriptions of wall materials and the results of the wall-friction tests.

DETERMINING HOPPER DIMENSIONS

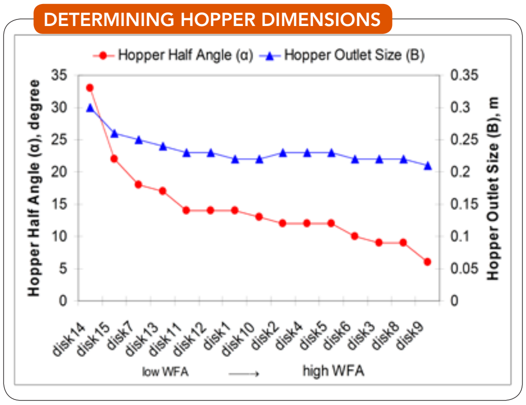

Figure 2. The above graph shows a hopper half angle and outlet size for Respitose with respect to wall friction angle.

Several techniques to measure a powder flow properties have been traditionally used, including Carr's Index, Hausner Ratio, Angle of Repose and Flow through a funnel. These techniques are generally regarded as basic and insensitive because they don't represent the conditions present in manufacture or application [3]. Shear cells have enabled the onset of flow to be quantified for powders under consolidation, which is useful for understanding behavior in hoppers.

However, the introduction of dynamic characterization methods, which simulate process conditions, allow the measurement of a powder's response to various environments. It is therefore possible to directly measure the response to aeration, consolidation

and varying flow rates, as well as quantify bulk properties, such as density, compressibility and permeability. To illustrate how comprehensive powder characterization can assist with process optimization, three examples of common unit operations are examined to determine which powder properties have the greatest influence on performance.

HOPPER SYSTEMS

The most common design method for powders is the specification of hopper dimensions parameters for mass flow, as described by Jenike's [4]. The dimensions are generated using shear, density and wall-friction measurements, and the development of fully automated shear cells has made it possible to generate this information within a few hours. Automated software packages, which rapidly calculate the dimensions, have also made the process simpler and more cost effective. In this example, the effect of the construction material of a conical axisymmetric hopper is examined. The study used a common pharmaceutical excipient, Respitose ML001 and a range of wall materials including finished/coated stainless steel, aluminum and plastic.

Along with the wall friction angle (WFA) values shown in Table 1, shear and density data for Respitose data were gathered enabling hopper dimensions to be determined (Figure 2).

WFA measurements for the 15 materials are highly reproducible and differentiating. They vary from 13° for polyoxymethylene copolymer to 34.8° for CrNi coated 316L stainless steel (SS316L). For both the SS316L and polymers, WFA increases with increasing roughness, (electro-polished SS316L being an exception). But the polymers generate lower WFAs overall despite their higher surface roughness, suggesting that other material properties are important. In addition, polishing the stainless steel does not improve its frictional properties with respect to Respitose.

The influence of WFA on hopper design is

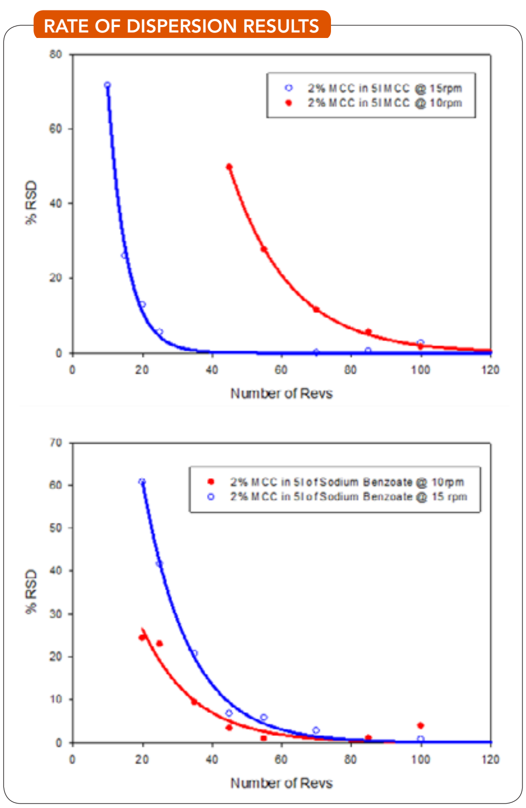

Figure 3. These two graphs compare the rate of dispersion of a radioactive bolus in MCC (top) and Sodium Benzoate (bottom) substrates.

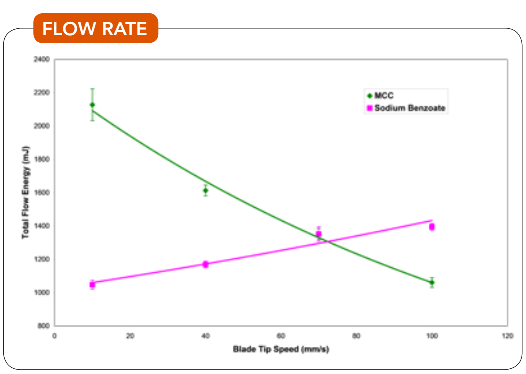

Figure 4. The above graph demonstrates powder response to blade tip speed in a powder rheometer.

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substantial. For the materials in this study, the hopper half angle (angle between the hopper wall and vertical) range from 6° (very steep hoppers) to 33° (very shallow hoppers). Outlet size increases slightly with an increasing hopper half angle to maintain mass flow. This study demonstrates a quick and effective method of evaluating hopper construction materials. The approach could lead to significant savings from the use of less-expensive materials and reduced manufacturing and installation costs. It would also be possible to apply this approach when evaluating coatings or liners for existing hoppers that are required to store different powders.

MIXING

Increasing the rotation rate of a tumble mixer typically increases the rate at which a mixture achieves homogeneity. The graph on the top of Figure 3 shows the rate of dispersion of a radioactive bolus of microcrystalline cellulose (MCC) in an MCC substrate (measured using positron emission tomography [6, 7]) for two different rotational speeds of a 10-liter tumble blender at a 50% fill level. As expected, operating the blender at a higher rotational speed (15 rpm) achieves an acceptable level of mixing earlier than at lower speed (10 rpm).

The graph on the bottom of Figure 3 shows the same experiment using sodium benzoate as the substrate. It can be seen that increasing the rotational speed actually slows the rate of dispersion. There are clear differences between the dispersal mechanisms of the two substrates, which aren't apparent from a visual assessment or from the results of a range of typical powder characterization methods. However, when the materials were studied using dynamic characterization methods, there were clear differences in response to changing the rate of flow. As can be seen in Figure 4, sodium benzoate exhibits an increased resistance to flow (Total Flow Energy) as the flow rate increases while the MCC flows more readily at higher flow rates.

Sodium benzoate particles are mostly platelets compared to the more spherical MCC. Platelets have larger surface contacts and greater mechanical interlocking due to their irregular particle morphology. The ability of the platelets to pass over each other during higher flow rates is clearly reduced and the same phenomenon is observed during the higher speed blending [8]. The platelets are less able to mobilize so interstitial spaces, which would otherwise allow the minor component to disperse within the substrate, are not promoted. In contrast, the spherical MCC particles can move over each other with ease, creating interstitial spaces and allowing the minor component to readily distribute throughout the substrate. No other characterization technique identified this behavior due to the inability to simulate process conditions.

FILLING – POCKET DOSING

The performance of four samples (S1, S2, S3 & S4) in two pocket dosing systems was studied. The results

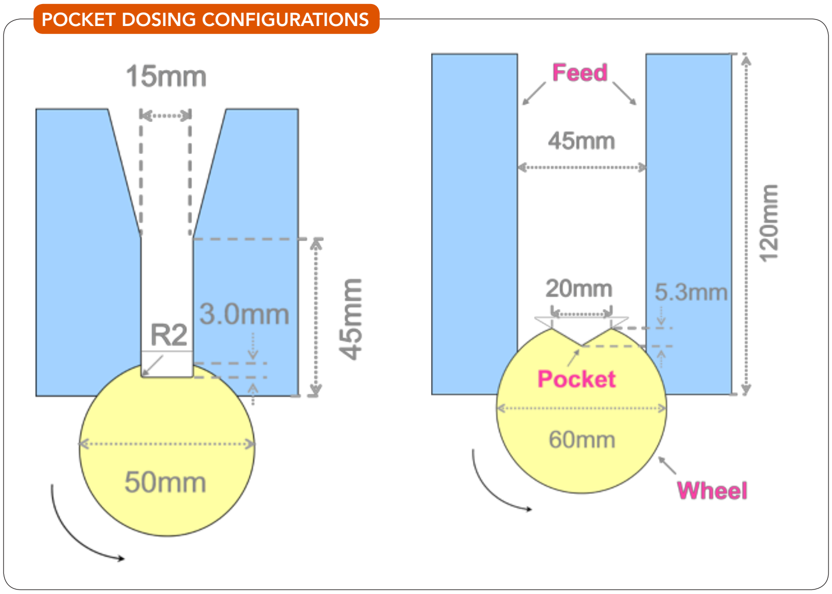
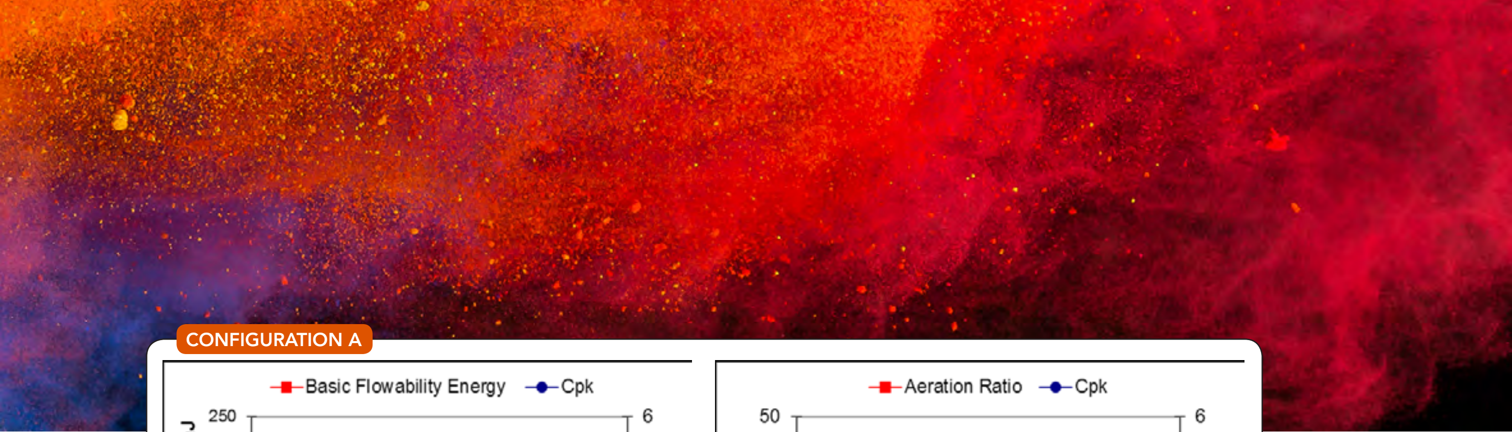


Figure 5. This schematic drawing illustrates the dosing configuration A (left) and B (right).



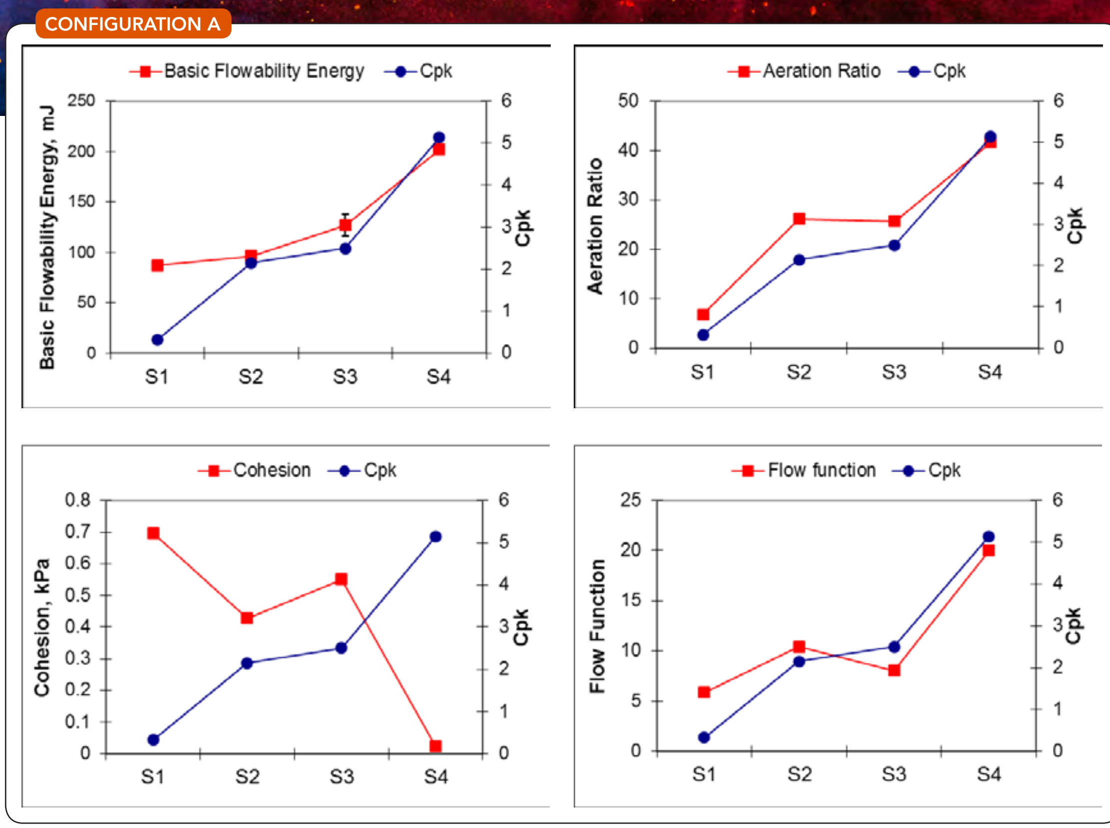


Figure 6. The above graph presents the correlation between dosing performance (Cpk) in configuration A and powder flow characteristics: basic flowability energy, aeration ratio, cohesion and flow function.

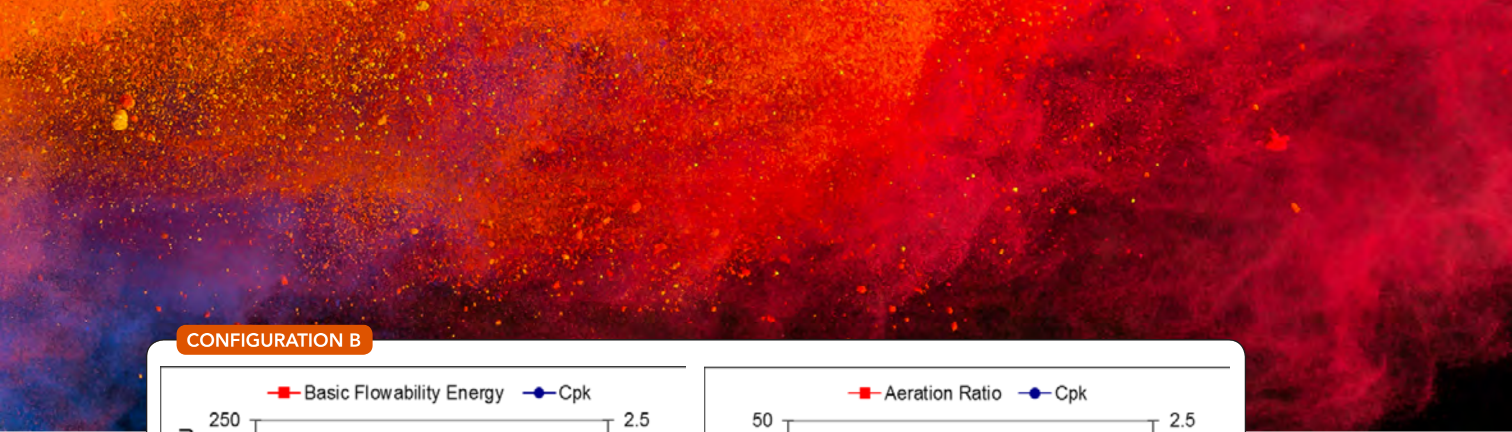
illustrate how different powder properties become influential, depending on the type of pocket, and how formulations must be compatible with process equipment in order to achieve consistent target weight.

Two pocket dosing configurations (A and B) were evaluated using a powder dosing technologies machine. The pocket volumes were similar (~410mm3), but the geometries of the feed hoppers and the pocket shapes were different (Figure 5.) In both cases, powder flows down through a channel to a pocket on a wheel. As the wheel rotates, powder is transferred from the pocket to a waiting receptacle and the wheel completes its rotation back to the filling position.

The process performance of each sample was described by the process capability index, Cpk [9] (Kotz and Johnson, 1993), derived from the weight variance (higher C pk values indicate better weight uniformity). The relationships between four powder flow properties – basic flowability energy (BFE), aeration ratio (AR), cohesion and flow function — and the process performance parameter are shown in Figures 6 and 7. S3 wasn't intended for processing configuration B so no performance data is available.

Powders with high BFE, AR and flow function, along with low cohesion — all indicative of low cohesivity — perform well in configuration A. This indicates that the ability of powders to flow through the feed channel and into the pocket, may dominate filling performance. When the feed diameter is relatively small, powder arching and discontinuous flow, caused by high cohesivity, may dramatically impact weight uniformity.

Opposite correlations were observed in configuration B where cohesive powders performed better. This counter-intuitive result indicates that gravitational flow is less important when the feed diameter is sufficiently large to avoid arching. The interactions between the powder, wheel and feed



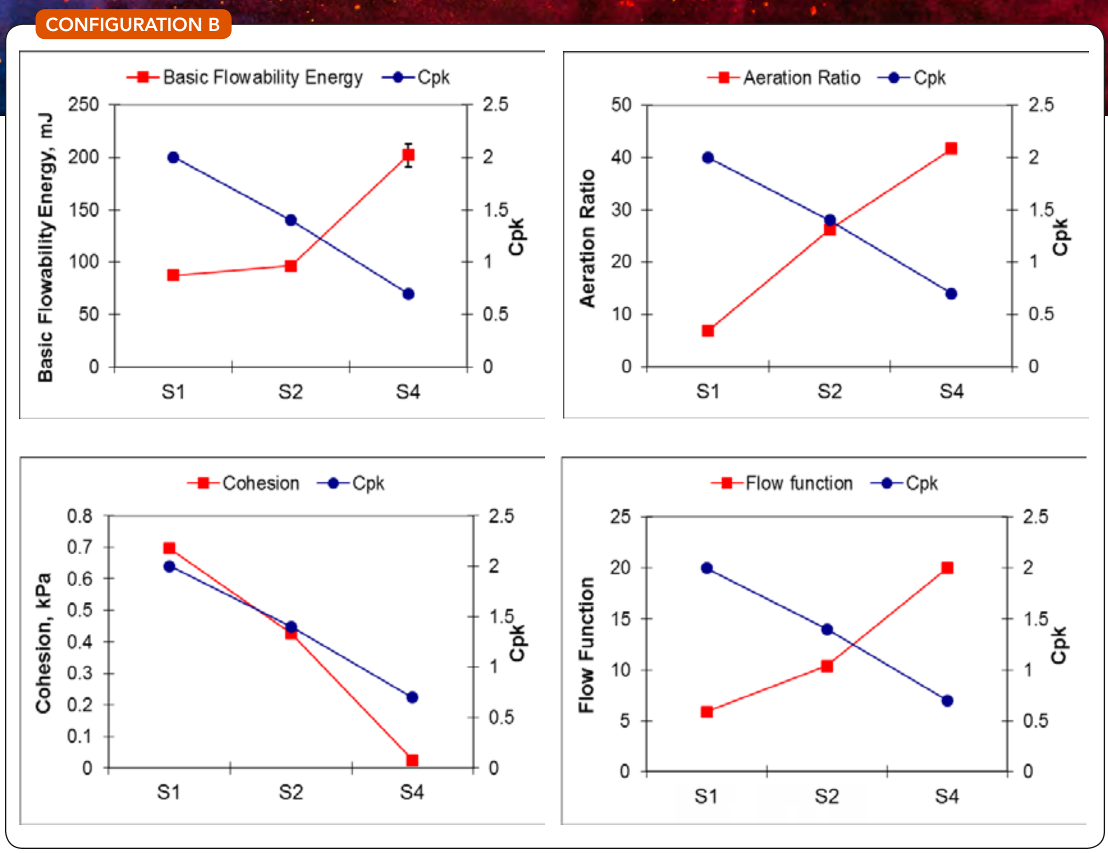


Figure 7. This graph shows the correlation between dosing performance (Cpk) in configuration B and powder flow characteristics: basic flowability energy, aeration ratio, cohesion and flow function.

hopper wall, as the wheel rotates, may dominate filling performance. The response of the powders in the two configurations indicates the importance of understanding the relationship between powder characteristics and process equipment.

POWDER/PROCESS RELATIONSHIPS

Powders are extremely complex and few relationships have been identified that link measurable characteristics to specific process parameters [10]. Although it is convenient to state that powder A is "better" than powder B, the reality is that a single index does not enable a complete understanding of any given powder's performance in all conditions. This article shows that a range of characterization methods are required to ensure a complete understanding of how multiple powder properties can influence process performance.

Comprehensive characterization quickly provides an understanding of why certain powders behave in a specific way within a particular process. Characterizing a powder using a single measurement is unlikely to provide sufficient information to optimize a process.

Equally, knowledge of the conditions experienced by powders in a specific process bottleneck will help identify the tests required to replicate the same response during measurement. This approach allows a database of powder/process relationships to be developed which captures current and historical data to ultimately enable equipment manufacturers and process engineers to improve efficiency and productivity.

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