Address Challenges Posed by Powders
You have a process material handling challenge...

We have the answer

Hapman will use our broad range of material handling equipment and our 60 years of industry knowledge to provide the right equipment to meet your bulk material handling process challenges.
SUMMARY

DRYING IS a potentially hazardous operation that requires special consideration. To make this operation safer, it is critical to identify the hazards associated with decomposition, fire, and explosion including worst-case scenarios resulting from operational and control failures at elevated temperature. Choosing the right tests will recognize and more importantly quantify hazards, conducting a process hazard analysis identify/evaluate operational problems, and using a risk assessment technique quantifies the hazards in engineering terms. This approach allows for the right level of safeguards because there is a fundamental understanding of the magnitude and probability of an adverse event. This approach meets compliance regulations with Occupational Safety & Health Administration (OSHA) Process Safety Management (PSM), OSHA Combustible Dust National Emphasis Program (Reissued), OSHA Grain handling facilities standard [29 CFR 1910.272], and good engineering practices in general.

BACKGROUND

Statistically, drying can be a hazardous operation. In some cases, drying has contributed to 37% of all plant accidents. Drying is an essential unit operation in the explosive, agricultural, food, pharmaceutical, chemical industries at any stage in production process because it delivers an isolated product for subsequent operations. A fire and/or explosion in this unit operation can be devastating from a safety perspective because it can result in death or injury. In addition, dryer outages can result in significant delay to a research project or a serious business interruption at the commercial level. There needs to be a properly designed plan to identify fire and explosion hazards before scale-up for the desired and upset conditions. For legacy commercial projects, there must be periodic review of the process hazard analysis, and there is a requirement to update dust testing results to current standards. The outcome of the strategy is to allow for quantification of the risks of decomposition, fire, explosion, and plant malfunction and to implement adequate safeguards.

It all starts with identifying wet/cake powders that can undergo exothermic decomposition because they are thermally unstable, shock and/or friction sensitive, prone to self-accelerating decomposition, propagate explosively, or detonatable. Interestingly enough, endothermic decomposition at elevated temperature can result in a dryer explosion due to excessive pressure and the rate of pressure rise as fixed gases are generated very rapidly. Decarboxylation (loss of CO₂) decompositions are a good example of an endothermic decomposition that has been associated with dryer explosions.

STRATEGY

The best strategy to avoid any unwanted events when a wet cake is heated in a dryer is to conduct screening tests to identify potential explosion hazards. The sequence of events involves:

- Examine the molecular structure to determine if there are any chemical functional groups that are considered to be unstable, explosive, or can generate gas upon heating.

- Use the ASTM Computer Program CHETAH to predict the potential for deflagration or detonation of a pure chemical as a part of their hazard evaluation process for the synthesis of new/existing chemicals.

- Conduct small scale heating tests to identify exothermic and endothermic events over a broad temperature range, e.g., 25-400°C using a Differential Scanning Calorimeter (DSC) with high-pressure crucibles.

- Conduct a small scale test to characterize a time/pressure profile for these parameters, as well as the rate of temperature/pressure, e.g., Advanced Reactive Screening Tool (AR SST), Accelerating Rate Calorimeter (ARC), etc.

- If the development chemist observed any color changes to the wet cake as it was drying in a lab oven, this should be interpreted as potential for an air oxidation reaction.

- Conduct a literature search to determine if this material or analogous materials have been involved in an incident. Be aware that if the search fails to find an incident, this is not a basis of safety.

- If the information from the above mentioned items indicates there is a problem, it is would be appropriate to consider the following additional tests:

  - Shock sensitivity test using the BAM Fallhammer technique and a friction test using the BAM Friction tester. If these tests are positive, it is best to talk with one of our consultants to decide on how to proceed.

  - If the material appears to be prone to oxidation it is important to consider some form of aeration test, e.g.,
At a minimum, it is important to determine if the material is a combustible dust or not through a "Go/NoGo" test. If it is combustible, then it would be appropriate to consider an explosion severity test (KST). Again, it is important to speak with one of our consultants about how this data should be interpreted and determine if any other dust explosion tests are needed to characterize this powder.

Additional dust tests that may be needed are:
- Minimum ignition energy (MIE);
- Minimum ignition temperature (MIT);
- Minimum oxygen concentration (MOC).
These tests are needed to help us make decisions regarding the need for an inert atmosphere, electrical classification of the operating area, etc.

**REFERENCES**

RISK MANAGEMENT SERVICES

COMBUSTIBLE DUST EXPLOSION AND FIRE HAZARDS
Onsite dust hazard assessments, OSHA Combustible Dust NEP compliance and other services related to characterizing, preventing and mitigating combustible dust explosion and fire hazards

OSHA PROCESS SAFETY MANAGEMENT (PSM)
Services for all 14 elements of OSHA PSM including PHA as well as human factors and facility siting

SAFE PROCESS SCALE-UP
Process safety to support chemical development programs for facilities including kilo lab, pilot plant and commercial scale plants

PHA/LAYERS OF PROTECTION ANALYSIS (LOPA)
PHA for all phases from design through production. Often paired with PHA, LOPA is a simplified form of risk assessment and assists in PSM and RMP compliance. LOPA helps direct limited resources to the most critical safeguards

INCIDENT INVESTIGATION
Expertise in discovering possible conditions leading to a recent explosion, fire, incident or near miss, performing root cause analysis and testing to include dust explosion severity, flammability and chemical reactivity as well on-site investigation services

PROCESS SAFETY DUE DILIGENCE FOR MERGERS AND ACQUISITIONS
Compliance reviews per regulations and guidelines, gap analysis and prioritized recommendations for future effort

RELIEF SYSTEM DESIGN REVIEW
One of the principal scientists involved in the Design Institute of Emergency Relief Systems (DIERS), Dr. Hans K. Fauske continues to contribute to developments in relief system design technology
WHILE NO one would deny the challenges involved in processing liquids and gases, powders are in a league of their own. Those charged with the design and operation of fluid processing plants have access to extensive physical property databases and well-established tools for property estimation and process simulation. In contrast, there’s a wide range of possible powder characterization techniques — from simple angle of repose to comprehensive analysis with a universal powder tester — but little published data. The process relevance of what information exists often is far from clear.

Powders are complex systems consisting of solids or particulates, a continuous gaseous phase (usually air) and, almost always, a liquid component. Their properties are a function of composition and an array of particle parameters that includes size and distribution, surface roughness and hardness. Vibration, compaction, attrition, segregation and many other factors influence behavior.

This makes measuring powders in a reproducible and meaningful way difficult; predicting flow properties from basic parameters, such as particle size, is well beyond current capabilities. So, powder processors have learned to rely on experience rather than fundamental knowledge. They tend to solve problems using a trial-and-error approach — and often can’t pin down the underlying cause or even why a solution works.

Such a trial-and-error approach can succeed but is time consuming and expensive. Furthermore, the experience gained, unless it extends underlying knowledge, has limited applicability.

DRIVERS FOR PROCESS IMPROVEMENT
As margins tighten any processing inefficiencies become less and less tolerable — many plants already have solved the easiest problems. Further progress requires manufacturers to develop in-depth understanding to push units to their efficiency limits and maximize return on investment. Typical goals may be:

• raising throughput by decreasing downtime or increasing flow rates;
• reducing waste by consistently maintaining product specification and cutting rework;
• switching to lower cost feeds; and
• automating process control.

To achieve these targets the manufacturing team must know what changes to make — and understand the impact that any change will have on the process and product. The starting point has to be the existing experience base but this, in its raw state, only can be used to improve operation within a well-mapped window.

If aspects of processing experience can be correlated with specific powder properties then a better understanding can be developed. Knowing which variables determine how a powder behaves in a given situation is the first step toward more-effective control. Using such knowledge to extend operation outside the established working envelope is the way to meet more-exacting performance standards.

CHARACTERIZING POWDERS
A powder rheometer (Figure 1) can serve as an excellent starting point to investigate process behavior. It determines the energy required to make a sample
flow by measuring the force/torque acting on a blade rotating through the material [1]. Conditioning, which is a gentle agitation step that produces a homogeneous, loosely packed bed prior to measurement, ensures excellent reproducibility. This makes powder rheometry highly differentiating. It can measure powders in a compacted, conditioned, aerated and even fluidized state, to more accurately simulate process conditions.

Modern rheometers not only provide dynamic measurement but also shear and bulk property testing capabilities. It’s therefore possible, using just a single instrument, to gather a comprehensive set of data in a reasonably short time. This encourages the development of databases of powder properties that can include:

- flowability parameters such as Basic Flow Energy (BFE) and Specific Energy (SE) [1];
- shear properties, e.g., yield locus, unconfined yield stress (compression strength), cohesion and internal angle of friction; and
- bulk properties like bulk density, compressibility and permeability.

In addition, investigating specific aspects of powder behavior such as de-aeration, segregation, caking and the effect of moisture and attrition can improve understanding.

Using this database of properties, it’s possible to relate process behavior to the characteristics of the powder. It also becomes feasible to identify those variables that critically impact performance, as the following case study illustrates.

**CHANGING FEEDSTOCK**

A manufacturer wants to consider other suppliers for a powder feed. The operational team strongly resists, though, because feedstock has been switched before and productivity was severely compromised. The powder flow through the plant, particularly from the feed hoppers, now is easily maintained; it was erratic with the alternative material and blockages were common.

In this case, the manufacturer is paying for material with superior handling performance — but there’s limited understanding of the properties that confer this superiority. Better understanding could have predicted failure of the alternative feed. So, the starting point for assuring a new feed is acceptable is to develop a specification that defines the existing material’s success. Unless this can be done, the manufacturer is stuck with the current supplier.

Analysis of the current feed provides data that can be used to understand why it behaves well in the process. For example, the powder may have relatively low cohesion and a low compaction index (CI). A low CI means that flow energy is relatively unaffected by the application of a consolidating pressure. In a hopper, exiting material is compressed by the weight of the powder above it. Under such conditions, this powder won’t form a bridge blocking the hopper outlet and will tend to flow relatively easily. The measured data, therefore, offer insights on why this material may be so easy to handle.

Comparing this material’s properties with those of the alternative feed that proved problematic, of course, would be even more illuminating. Such an analysis might confirm that the feed should have low cohesivity and low CI — and also may indicate that it mustn’t be prone to segregation.

Specifying acceptable values for these parameters and testing prospective materials against these criteria would give some assurance of success.

**USING POWDER PROPERTIES**

The previous example illustrates two ways in which powder characterization data can be used. One approach is to comprehensively measure the powder and then look at all the properties to see how well they behave because of aeration. A small amount of air can dramatically improve flow properties of some powders.
the material fits with the demands of the process.

For example, consider die filling, where powder is used to fill a specific volume of defined shape. Ideally the powder should flow freely into the die, settle and then flow into the required shape as filling proceeds. In this case, logic would suggest that flow behavior under compacting and unconfined conditions will be important. The way in which the powder releases air also has a bearing. A powder with low permeability may trap air, compromising filling or the effectiveness of any subsequent compression step. So, a powder that releases air easily is preferable from this point of view — however, such powders can quickly become immobile because air lubricates powder flow. Achieving optimal performance requires balancing these effects.

Figure 2 presents the results of a study of the effect of air on two powders. The data show how flow energy falls as the velocity of air travelling up through the sample increases. The free-flowing powder’s flow energy drops almost to 0, indicating that it has fluidized; the minimum fluidization velocity occurs at around 2 mm/s. This small amount of air radically improves the material’s flow properties. In contrast, the cohesive material forms channels through which the air escapes.

Shear properties also can affect processability. Figure 3 shows data for the stress required to shear a previously consolidated powder sample as a function of applied normal stress. The two materials exhibit different behavior — one presents a much greater flow resistance. This powder is quite cohesive (C1 value) and thus more likely to form stable bridges that can compromise hopper discharge and die filling than the other material. Powders with low shear strength will tend to flow freely even when compacted. C2 is the cohesion value or shear strength of the powder when not consolidated.

The alternative approach is to compare the properties of materials that process well in the operation with those that perform poorly, as in the following study.

**OPTIMIZING EQUIPMENT USE**

A titanium dioxide producer makes different grades for applications in the paper, plastics, cosmetics and food industries. It uses surface treatment, milling and drying processes to produce the required properties. With some grades, operation is both easy and productive while others demand very close control. The manufacturer instigates a review with the aim of improving throughput and productivity.

The producer needs to understand how the properties of different TiO2 grades affect how they interact with the processing equipment. Existing process experience allows materials to be classified using “Processability Rankings” (PR). For example, a PR of 2 may describe a trouble-free powder that processes consistently at a fast rate. A PR of 9 may indicate a powder that can’t be processed to any degree, while an intermediate value of 6 may signify a powder that performs acceptably if the process is closely monitored or control is fine-tuned. This is a useful way of starting to quantify experience. Factors to take into account when ranking processability include:

- symptoms, circumstances and frequency of any unplanned shutdowns;
- final product quality;
- overall plant reliability — the percent of available time the plant operates; and
- controllability/productivity — the percent of operational time the plant produces material that meets the defined specification.

It also can be beneficial to identify periods when the plant performs poorly; for example, is performance worse on a Monday morning after a weekend shutdown?
Such details can help determine the reasons for poor performance.

Correlating processability rankings with powder properties will pinpoint those parameters that dominate behavior and will increase understanding of how to effectively modify equipment to improve operation. Furthermore, measuring the values of a new material prior to processing will allow prediction of how well it will perform. If done at the development stage, this may enable tailoring properties to achieve better processing behavior.

Combining experience with well-defined reproducibly measured powder properties provides a route toward more effective powder management. Using this approach it becomes possible to:

• define the characteristics of materials that will process well on a given line;
• assess in a process-relevant way differences in materials from various suppliers;
• better understand the impact of hardware modifications;
• more effectively match powder with processing equipment;
• establish effective quality-control criteria for both feed and product;
• understand and address causes of batch-to-batch variability; and
• reduce risk associated with introducing new formulations.

Understanding and knowledge gained from quantifying experience lead to better decisions and actions and, therefore, to a greater likelihood of success.

THE WAY FORWARD

Defining powder flow properties in terms of the array of variables that influence them — particle size and shape, hardness, moisture and air content, for example — is beyond our current capabilities. In addition, correlations between flow properties and processing behavior aren’t yet well established, although progress is being made. However, that doesn’t mean a manufacturer must resort to a trial-and-error approach to process and product development, with its largely subjective and highly specific results. Instead, it’s possible to extract more generally applicable information via powder testers to obtain the understanding required for optimization.

State-of-the-art powder testers that offer shear, bulk and dynamic property measurement give the most-comprehensive insight into powder characteristics. Samples can be analyzed in a consolidated, conditioned, aerated or even fluidized state, and important phenomena such as segregation and attrition also can be thoroughly investigated.

These instruments foster the relating of operating experience to variables that can be reproducibly measured and sensitively differentiate between samples in a process-relevant way. This makes it easier to define properties of a “good” powder for a specific unit operation and to identify characteristics that will cause poor performance. The key to effective processing lies in matching equipment and powder properties so both exhibit optimal performance. The data that universal powder testers provide give insight required to optimize design, operation and troubleshooting of powder processes in this way.

FIVE KEY STEPS TO RANK PROCESSABILITY

Correlating processing experience with powder flow properties is a powerful way to determine which variables critically impact performance. A plant that handles a variety of powders should consider developing Processability Rankings (PR) to support this approach.

To rank the processability of different powders:

1. Carefully define the focus of study — whether the whole process or just a single piece of equipment such as a hopper, granulator, storage bin or conveyor. Different unit operations place different demands on the powder, so it may be beneficial to individually consider them.

2. Identify which powders process well and which are problematic.

3. Describe in detail the issues — causes and effects — associated with poor performance. These may include:

• consolidation in a vibratory environment;
• moisture absorption (hygroscopic materials may absorb moisture and become difficult to handle);
• segregation;
• bridging in hoppers;
• attrition (which can remove a surface coating, change particle shape or generate fines);
• caking in storage;
4. Define and assign processability rankings. For example:
PR1 means “trouble-free” processing;
PR5 means occasional stoppages or quality non-conformance; and
PR9 means frequent stoppages and significant wastage or scrap product.

5. Combine the information in a form that clearly relates operating experience with powder properties (such as Table 1).

### Processability Issues on Tableting Line

<table>
<thead>
<tr>
<th>Material</th>
<th>PR rating</th>
<th>Problems</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formulation 1</td>
<td>2</td>
<td>Potential segregation during transfer</td>
</tr>
<tr>
<td>Formulation 2</td>
<td>5</td>
<td>Bridging and adhesion to machinery</td>
</tr>
<tr>
<td>Formulation 3</td>
<td>8</td>
<td>Weight variability and high wastage</td>
</tr>
</tbody>
</table>

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### Troubleshooting Tips to Diagnose Problems

Many manufacturers dedicate equipment to processing a single powder and can cope well with routine operation. However, change of any type, whether inadvertent or planned, can pose challenges. Even minor modifications to plant or procedures can have a big impact. Use the following pointers to diagnose problems and deal with change more effectively.

- **Startup/shutdown**: length of time of shutdown, effectiveness of clean out and vessel filling method all can impact plant reliability. Define procedures in detail based on an understanding of powder properties and make sure they are adhered to.
- **Operator-to-operator variability**: manual procedures and operation provide significant scope for variation in approach and technique. Identify critical steps, share best practice and enhance underlying knowledge to minimize differences in approach.
- **New source or batch of feed**: avoid experiments on line. Measure the characteristics of new materials first and compare with those of known feeds to confirm similar flow properties.
- **Dealing with processing problems**: Resolving problems with quality or processing is important but so is learning lessons on the cause and future prevention. Measure powder properties to determine which have changed and why.
- **Plant or process change**: new or modified plant or a revised procedure can directly lead to problems or can cause issues downstream. A new mill, for example, can change particle size and shape, resulting in very different powder behavior. Powder characterization will highlight any inadvertent changes to critical properties.
- **External environment**: temperature and especially humidity can affect powder properties. Storage, transportation and vibration in particular can result in serious consolidation and compromise processability. Avoid surprises by determining susceptibility to these factors.

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**Reference**

Dangers can lurk in the most unsuspecting places, causing the most damage to you and your employees. Make sure your facility has the right training to minimize the possibility of an accident or incident occurring. These programs from Summit help you manage your work environment and equipment to avoid any faulty reactions in processes that could lead to an explosion:

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PHASE CHANGES in the downstream processing or handling of solids cause many process problems. These changes can alter the form of the final product or mandate rework of a material. Too often, though, phase changes do not get adequate attention.

Consider these classic problems:
- The particle size of a batch product suddenly changes.
- The filtration time of a slurry alters over time or the cloth blinds unexpectedly.
- A dried product clumps even though the processing conditions in the dryer didn’t vary.
- An evaporator begins to foul unexpectedly.

Many factors can cause these situations, but phase change usually is not the first thing that comes to mind. However, if you are processing an organic, phase change should be one of the first considerations even when your research people say there is only one phase for the material. Polymorphs and solvates in organic and inorganic chemicals can exist briefly enough to dramatically change the outcome of a crystallization process.

My personal experience indicates that about one-third of all crystallization problems stem from polymorphs or solvates. These often are overlooked in the development of a product, which is not surprising given the extreme pressure to get to market. Also, operating conditions can change for an existing product as plants strive to become more efficient. For example, switching to a different, maybe larger, reactor without considering the difference in mixing or reactant addition can play havoc with the process. Even the most sophisticated process-modeling programs don’t take some of the micro-fluidic and reaction effects into account, especially reactor size changes and piping layout. Phase changes frequently are ignored if they don’t show up in the reactor and solid/liquid separator as a design issue.

One very common complaint is about erratic filtration rates, especially in an existing process. After all, in most cases the purpose of the crystallizer is to make solid/liquid separation efficient, not just to give a larger-sized product. The problem can be a drop-off in rate of filtration, pluggage of the filter or variation in the amount of time needed for a batch.

The two examples that follow highlight on how phase change can affect a process.

MORE THAN A SHIPPING CHANGE
A plant had been crystallizing a material and sending the batch to centrifuges to make a wet cake. The cake then went to another location for further processing. As the scale of operations increased, the plant decided to ship the wet cake by railcar. To accommodate this, the slurry from the crystallizer was pumped to a feed tank for the centrifuges, which were located next to the railcar loading station. As you can imagine, the line had to be fairly long and the velocity high to prevent sedimentation. Almost every batch had a decline in the filtration rate. In some cases only half of the contents of the feed tank could be processed. The remainder was either disposed of or reworked. The cloth and the centrifuge manufacturer both were blamed and a wide variety of measures were taken to try to solve the problem.

The plant discovered that by pumping only half of the contents and then refilling the tank from a crystallizer almost all of the slurry could be processed — but at too slow a rate. Average crystal size was not changing but the particle size distribution (PSD) was wider than before the change to the railcar loading system. In this case three factors contributed to the problem:
1. Although the finer crystals were distributed uniformly in the crystallizer, more of the larger crystals were accumulating near the bottom of the vessel, especially just before pump-out of the crystallizer.
2. The pump was located closer to the railcar loading station, which put a lower net positive suction head (NPSH) on the inlet when the flow rate was increased. Also, due to the long distance, the pressure rise across the pump was heating the slurry.
3. The feed tank used a dip leg to avoid splashing in the tank.

While each of these issues normally would not cause a problem, the three together created a poor filtration rate.

Pump-out of the crystallizer allowed the larger crystals to get to the feed tank early in the process. During the last portion of the pump-out, the pump was handling slurry with very fine crystals and a lower
solids’ content. Localized heating resulted in both dissolution of some of the fines and a supersaturated solution in the tank. Larger crystals grew slightly because the dip leg put the solution into the bottom of the tank but some secondary nucleation was occurring. Even though the tank’s impeller provided some recirculation, the larger crystals still collected in the lower portion of the tank.

By removing only half of the tank’s contents before refilling from a crystallizer, the operators discovered that coarser particles could be replenished to provide better filtration rates. However, in the long run the finer crystals would accumulate and eventually blind the centrifuge.

The operators thought that running the impeller when the centrifuge was not being fed would provide adequate recirculation within the tank. However, the impeller could only keep the solids off the bottom of the tank and most of the mixing was caused by the recycle of slurry.

To verify this scenario, PSDs were examined before and after the pump and during the crystallizer pump-out. As expected, the PSD did change as the crystallizer was emptied, with a higher slurry density and larger average particle size at the beginning of the cycle. In addition, finer crystals were “disappearing” after the pump throughout the entire transfer. A close look at the impeller of the pump showed slight signs of cavitation, which meant some of the solvent may have been evaporating.

Two equipment changes and one process change were made based on the PSD studies. The transfer pump was relocated closer to the crystallizer and the impeller in the feed tank was switched to an axial flow design. The feed pump was adjusted so that there is always flow to the feed tank, even during filtration cycles.

**IMPACT OF POLYMORPHS**

A very similar crystallization situation occurred at another plant. There, a large variation in filtration rate occurred, but it was never repeatable. Occasionally, the plant would get a “super” batch that filtered in half the normal time. This was a very desirable, but seemingly random outcome. That batch had a slightly larger and narrower PSD.

Examination of the particles under a microscope revealed that the habit was different. No polymorphs or solvates were known for this material — however, an evaluation of the chemical structure suggested at
least four polymorphs and two solvates. This led to a closer look at process conditions. The standard operating procedure called for cooling of the under-saturated solution until a heat flux was observed in the jacket of the vessel. The difference between the inlet temperature and the outlet temperature was used to mark this heat flux. The temperature was held constant for a fixed period of time and then the batch cooled to the final temperature. In the super batch case, this step-change in heat flux was not observed; cooling occurred below the normal holding temperature at which the heat flux usually was seen. There was no holding period and cooling continued to the final temperature.

What was really happening in the process got lost in translating the research work to the process development and operation. This material exhibited classic enantiotropic behavior: a transition temperature between two polymorphs (alpha and gamma) as shown in Figure 1. The gamma form is unstable and has lower solubility above the transition temperature while the alpha form has the lower solubility below the transition temperature.

The under-saturated solution at the starting temperature has a solute concentration below the solubility of gamma and alpha. As the temperature decreases, the gamma form comes out of solution and crosses the transition temperature. More crystallization occurs until the concentration is outside of the meta-stable limit for the alpha form. Rapid nucleation (the heat flux) of alpha begins and the gamma form begins to dissolve. At that point the rate of cooling is slowed but the damage has been done. The excessive amount of nuclei hinders growth and results in a wider and smaller PSD.

The simple solution to this problem is to dilute the solution to below the concentration that corresponds to the transition temperature. If the volume of solvent that results is too large for the solid/liquid separation, consider other methods for controlling the initial nucleation. Possible remedies are: seeding, cooling below the transition temperature and then heating slightly or installing a “nucleation detector.” In this case, seeding was chosen because it produced a faster overall crystallization time. A cooling crystallizer was not the best choice for this process but it would not be cost effective to replace it.

DON’T BE FAZED
In both of these cases, a key indicator of a phase change issue was the dramatic shift in the ease of filtration. It is important to identify polymorphs and solvates for a product and to determine any critical temperature early in process development and design (see sidebar). Also, watch out for minor changes in the processing layout. Understand what you are measuring and why. In these examples finding the potential cause of the problem was a fairly short and easy process once the fundamentals were examined. Verifying it was much more difficult. However, in most production environments we don’t need a definitive study but can alter the processing conditions to test the causes. Then a more cost-effective solution often can be developed and implemented. ☐
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